DESCRIPTION FR2669636

PROCESS FOR PRODUCING 2,5-FURAN dicarboxaldehyde

The invention relates to a process for manufacturing 2,5-furan dicarboxaldehyde (FDC) from sugars, in particular fructose or polyfructans.

The CDF is a base monomer may be used for the synthesis of certain polymers (polyamide, polyurethane) and macrocycles, especially in the pharmaceutical sector.

N1 This compound is currently not commercially produced because of the lack of economically profitable method.

Some publications describe methods for synthesizing FDC from furfural in chlorométhylfurfural, furan and furan dichloromethyl 2.5 2.5 dihydroxymethyl (Ben L. Feringa et al, Synthesis 1988, p. 316-318; Champetier Georges et al, CR Acad.

Sc


Soc., T. 101, 1912, p. 1074-1078; patent DD nO 26542, November 6

1963 K. YU. Novitskii et al, ZH

OBSHCH.KHIM, 1961 V. 31 (2) p.

538-541; Oleink AF et al,

Zh. Org.

Khim, 1970, 6 (12), p. 2632-33), but these methods are implementing long, complex and expensive, and consequently uneconomic.

Other processes use hydroxymethylfurfural (HMF) as a starting compound and oxidizing this compound consist with conventional oxidizing agents such as chromate, barium manganate, manganese dioxide ... (JP 79.09260, January 24, 1979 patent, El Hajj et al, Bull.

Soc.

Ch En 1987, No. 5, p. & 5-860), but these processes lead to significant amounts of effluent, with very low yields and poor selectivity vis-à-vis the FDC.

Furthermore Japanese Patent 55-49368 published nO April 9, 1980 describes a process which consists in performing, in a highly polar aprotic solvent (dimethylsulfoxide: DMSO), a transformation of HMF in the presence of FDC in an electrophile (N2O) .

However, under the conditions of implementation of this Japanese patent, this method has poor performance (57% and 71% in
the examples provided in this patent, the yield is defined as the molar ratio of FDC formed HMF introduced), a poor selectivity vis-à-vis the FDC (obtaining side products such as 2-furan-dicarboxylic acid, 5-formyl -5 carboxylic furan-2), if desired iton a pure product, it is therefore necessary to purifying the obtained FDC and the synthesis process described in this Japanese patent is completed by a step of purification by distillation of DMSO under reduced pressure, followed by recrystallization from the residue.

The low conversion efficiency and the need for this purification step implementation onerous work make the industrial exploitation of this unprofitable process economically.

The present invention relates to an improved method for producing FDC of the type consisting in carrying out a synthesis of HMF in organic medium and oxidizing the obtained HMF in the presence of an electrophilic agent.

The invention aims to eliminate defects of the Japanese method of the same type mentioned above and lead to a method for producing high purity FDC profitable economically.

For this purpose, the method according to the invention consists

(A) a synthesis of HMF (API) by mixing one or more sugars in a strongly polar aprotic solvent family sulfoxides and (A2) by heating the mixture,

then (B) to ensure the conversion of HMF in FDC in the reaction medium in the presence of an electrophilic agent.

The method according to the present invention is characterized in that

(A3) is added to the highly polar aprotic solvent according to the third group solvent diethyl (DEC), methyl isobutyl ketone (MIBK), dichloromethane (DCM) or ethyl acetate,

(C) after processing of HMF, the water was added into the reaction mixture to create an aqueous liquid phase, said first phase, from the highly solubilizing polar aprotic solvent, solubilizing the FDC formed in the liquid phase formed by the third organic solvent, said second phase,

(D) the two liquid phases are separated in order to obtain the second phase containing the FDC,

(E) focuses iton said second phase by evaporation phase FDC third solvent,

(F) is carried out from the concentrated phase obtained crystallization FDC

(G) and the CDF obtained is filtered.

Experiments have shown that the method of the invention resulted in a transformation efficiency and selectivity near 100% FDC and capable of manufacturing a high purity FDC.

Selectivity is defined by the number of moles of DCF formed, based on the number of moles of consumed HMF.

The conversion yield is defined as the number of moles of DCF formed to moles of HMF synthesized (as in the above-referred Japanese patent) in the invention the starting material is a sugar which is a commercial product available directly and the overall yield (number of moles of DCF obtained relative to the number of moles of introduced sugars) is of the order of 75 to 80.

By high purity, it means a content by weight greater than 98% FDC.

These remarkable performance can be explained by the original functions that fill the third solvent and water in the process of the invention, the third solvent has dual function, firstly, to make the conversion reaction totally selective visà FDC-screw, on the other hand, to enable the extraction of the product FDC which dissolves in said third solvent to form an organic phase, the water has a function of solubilizing the aprotic solvent and thus avoid a passage thereof in the third solvent.

The FDC solubilized in entirety in the third solvent to the exclusion of any other compound can be easily separated by evaporation and crystallization to yield a compound of high purity.

It is therefore rejected all the flaws of the Japanese method.

According to a preferred embodiment of the method used, is selected as the highly polar aprotic solvent, dimethylsulfoxide (DMSO), which leads to more efficient synthesis of HMF, due to its specific properties (excellent solvent intramolecular dehydration selective, high affinity for water, power stabilization against HMF).

Furthermore, the method of the invention is advantageously implemented using as starting sugars fructose or polyfructans leading to more efficient synthesis of HMF while being cheap and common sugars.

The synthesis of HMF is preferably operated under the following conditions, which lead to higher yields of synthesis
(A1) or the mixture of the starting sugar in the highly polar aprotic solvent to obtain a solution having a weight content of between 200 g and 1000 g of sugar per kg of aprotic solvent,

(A2) heating the sugar solution and strongly polar aprotic solvent of between 1000 °C and 1700 °C for a time between 2 and 8 hours.

Furthermore, according to a mode of implementation of the preferred process (A3) is selected as the third solvent

December, which leads to better performance processing

HMF in FDC because of its chemical structure, leading to a total selectivity and its specific properties (high affinity for the FDC, low boiling point, good indication of evaporation).

The addition of this third solvent is advantageously performed in a proportion between 5 and 20 g of solvent per gram of sugar introduced.

This proportion can solubilize all FDC product and obtain the above-mentioned selectivity, while maintaining high concentrations of avoiding handling of too large volumes of solvent.

After addition of the third solvent can be, where appropriate, by a method like the step (A4) for removing at least a fraction of the water formed in the reaction medium during the synthesis of HMF. This removal is in particular carried out when the sugar is in the form of an aqueous syrup or when the synthesis of HMF is conducted under conditions of high initial sugar concentrations, as it is then led to a high proportion of water in environment with the risk of lowering the processing performance of FDC in HMF.

The electrophilic agent selected to effect the conversion of HMF is in particular a compound from the following group: acetic anhydride mixed with orthophosphoric acid dicyclohexyl-carbodiimide, or phosphoric anhydride.

The first compound is preferably selected because of its low cost and its property of initiating the conversion reaction in the absence of a foreign catalyst.

The molar proportion thereof relative to the starting sugar is preferably selected between 1 and 5, a proportion of this agent to allow the DMSO to activate the stoichiometry of the oxidation reaction of HMF.

According to another preferred feature of the process, (C) after processing of HMF, iton added water in the reaction medium so that it contains a weight proportion of water of between 0.23 and 0.9 relative to strongly polar aprotic solvent contained in the medium.

This amount of water can completely solubilize the DMSO to be separated without causing the FDC formed.

The method of the invention can in particular be used in the following conditions, which seem the most interesting economic

(A3) the third solvent is added to the end of the synthesis reaction of HMF after a period of between 4 and 6 hours after completion of warm-

(B) the processing is carried out in HMF

FDC cooling the medium to a temperature between 250 °C and 800 °C and adding the electrophile,

(C) is added to the end one 1eau of the transformation reaction after a period of between 4 and 6 hours after the addition of electrophile,

(D) separating the two liquid phases is carried out by decantation while maintaining the temperature between -5 °C and 300 °C,

(E) is concentrated by evaporation until the second liquid phase saturation of the third solvent FDC

(F) the crystallization of the FDC is prepared by adding a crystallization solvent to the concentrated phase and cooling the solution to a temperature between 50 °C and 200 °C.

The use of a crystallization solvent allows to obtain FDC very high purity (greater than 99.5%) and very good (no staining or smell parasite).

For economy, the crystallization may optionally be effected by simple cooling from the concentrated solution.

The crystallization solvents which are preferably used are the following group of solvents: 1-trifluoro, 1,2 1,2,2 trichloro ethane, petroleum ether, cyclohexane, volumetric proportion of between 2 and 5 1 1 by solvent of concentrated solution.

The method of the invention lends itself to recycling of solvents.

After separation of the two liquid phases (D), the highly polar aprotic solvent can be recovered from the aqueous phase by
distillation under reduced reuse for new synthesis pressure.

The third solvent, in turn, is recovered in two stages: firstly, directly after evaporation (E), on the other hand, after crystallization (F), by operating a distillation of the liquid phase, and the fractions recovered are recycled for a new implementation of the process.

The following description with reference to the single figure of the attached drawing shows schematically a plant for implementing the method of the invention and illustrates examples of implementation.

The installation shown for example in the single figure includes a synthesis reactor 1 (capacity: 5 liters) which is supplied with raw materials from a sweet tank 2 and DMSO from a reservoir 3.

The reactor 1 is equipped with a water extraction system 4.

The reactor is temperature-controlled and equipped with a mechanical stirrer.

The synthesis medium is collected at the outlet of the reactor 1 in a tank 5 and is sent to a reactor 6 FDC transformation. This reactor is supplied with a third solvent in the reservoir 7.

This reactor is also fed with electrophilic agent contained in a reservoir 8.

The shift reactor 6 has a water extraction system 9 d1.

It is temperature regulated and equipped with a mechanical stirrer.

After transformation, the reaction medium from the reactor 6 is collected in a tank 10 and is sent to a liquid / liquid extractor 11.

Water contained in a reservoir 12 is introduced at the top of the extractor 11.

The residue in the bottom of extraction (water / DMSO) is collected in a storage bin 13.

The sample flows into a storage tank 14 and is sent to an evaporator 15.

The vapors are condensed in December in a condenser 16 to be recycled to the tank 7.

The crude saturated FDC is sent to a crystallizer 17 which is supplied with crystallization solvent (cyclohexane) from a reservoir 18.

The FDC precipitate is separated from the solution in a filter system 19, is recovered and stored in a hopper 20 for drying.

After filtration, the liquid phase is collected in a tank 21 and is sent to a distillation unit 22.

The third solvent vapors and cyclohexane are separately condensed in a condenser 23 to be recycled to the respective tanks 18 and 7.

General Protocol Implementation examples

The raw material is sugar Dfructose; a1 amount is dissolved in 1000 g of anhydrous DMSO and the solution is introduced into the synthesis reactor 1.

Sugar mixture / DMSO is kept under mechanical stirring and heated to a temperature a2.

When the temperature rise is complete, the reaction mixture is colored in black and quickly, after a time a3, a4 a quantity of solution is collected in the tank 5, the analysis shows that it contains an amount HMF a5, a6 of H20 per 1000 g of DMSO (analysis by thin layer chromatography and gas chromatography).

The yield of the synthesis is equal to HMF a7 for llégard HMF selectivity equal to a8 (HMF moles formed relative to the number of moles of sugar consumed).

After cooling the mixture to room temperature, the solution contained in the tank 5 is introduced into the shift reactor 6.

It is diluted in a quantity b1 third solvent.

6 The reactor is brought to a temperature for a period b2 of 30 minutes, so as to extract a fraction of the water present in the solution (9 extractor).

A quantity b3 electrophilic agent is introduced into the shift reactor 6.

The reactor is maintained at the temperature for a b5 b4 duration.

The yield of the transformation is to b6 FDC (FDC molar ratio of the HMF formed).
After transformation, a quantity of b7 solution is collected in the tank 10.

The analysis of this solution revealed that it contains an amount of DMSO b8 bg FDC, b10 HMF, and b11 third solvent.

After cooling, the reaction mixture obtained is introduced into the liquid / liquid extractor 11.

The DRSO is extracted from the reaction medium by adding a water quantity c1 to a temperature of 150 °C.

After separation, the analysis of the amount d1 of the samples collected in reservoir 4 reveals that it contains a quantity of DMSO d2, d3 and d4 of FDC third solvent.

This extract is concentrated by passing through the evaporator 15 at a temperature of 250 °C, so as to wait a saturated solution FDC.

The vapors are condensed and third solvent recycled.

The concentrated syrup containing an amount of e1 FDC e2 is diluted by a volume of solvent of crystallization in the crystallizer 17.

The crystallization yield is e3 (FDC crystallized mass relative to the mass of FDC introduced into the crystallizer).

The medium temperature is then quickly lowered to 50 °C and held for 30 minutes.

The crystals formed are filtered vacuum.

The filtrate containing the third solvent and the crystallization solvent is collected in the tank 21 and directed to distillation apparatus 22.

The crystallization solvent and the third solvent is recovered and recycled to the respective tanks 18 and 7.

Example 1

In this example, the reagents and solvents are

- Third solvent: DEC
- Electrophile: acetic anhydride
- Crystallization solvent: cyclohexane.

The values of the various parameters are the following amount of D-fructose 200 g = a1 (for 1000 g of DMSO), synthesis temperature HMF a2 = 1500 °C, duration of the synthesis of HMF a3 = 6 hours, amount of solution collected the end of synthesis a4 = 1200 g, the amount of HMF in a5 solution = 119 g, amount of water in this solution continues a6 = 50.7 g, yield of the synthesis of HMF a7 = 85, the selectivity in respect of the synthesis HMF a8 = 90% quantity DEC b1 = 1745 g, a proportion of 8.73 g per g of sugar introduced. extraction of the water temperature b2 = 83 °C,

. amount of acetic anhydride b3 = 188 g, an
molar ratio to equal to 1.67 sugar
. temperature processing HMF / FDC b4 = 800 °C,
. duration of the transformation b5 = 2 hours
performance processing b6 = 93%. amount of solution collected after processing

b7 = 3079 g. amount of DMSO in the solution b8 = 922 g. amount of FDC in the solution b = 109.4 g. remaining amount of HMF b10 = 2.3 g. December in the amount of b11 solution = 1745 g, amount of water added to the extraction c1 = 638 g, a
weight ratio water / DMSO ratio equal to 0.69,. amount of collected extracts d1 = 1852 g. amount of DMSO extract d2 = 1 g.
amount of FDC extract d3 = 106.4 g amount CED extract d4 = 1723.3 g. FDC amount of concentrated syrup e1 = 106.4 g, an
concentration in the medium equal to 60 g / l (corresponding to
saturation of December). cyclohexane volume added 5320 ml e2 =,. e3 crystallization yield = 99%.

In a general manner, the method in this example was obtained 105.2 g of crystals of FDC purity of about 98% for an amount of 200 g of D-fructose (FDC is a potential of 137 8 g).
The overall yield of the process FDC equals 76.3 i.

Example 2

In this example, the reagents and solvents are - third solvent: MIBK - electrophile: dicyclohexyl-carbodiimide, - crystallization solvent: trifluoro-1,1,2,2 trichloro 1.2 ethane.

The parameter values are as follows a1 = a2 = 450 g 1600 C a3 = a4 = 4:00 = 1450 g = 268 g a5 a6 a7 = 114.5 g = 85 g = 90 g a8 b1 = 7000 g, a proportion of 15.5 g per g of sugar introduced, b2 = b3 = 637.6 C 870 g and has a molar ratio to equal to 1.23 sugar.

Also in this example, is added a quantity of 20.2 g of orthophosphoric acid to the reaction medium in order to initialize processing HMF / FDC this addition is necessary in the case of using dicyclohexyl carbodiimide.

Furthermore this solution contains an amount of 692.7 g of solid urea, which is separated by filtration dicyclohexyl-carbodiimide.

The overall yield of the process FDC in this example is equal to 76%, of the same order as that of Example 1 (purity: 98 "p), however, the mode of implementation of the example 1 will preferred in the current economic conditions because of the lower cost of the electrophile.

Example 3

In this example, the reagents and solvents are - third solvent: DEC - electrophile: acetic anhydride - crystallization solvent: petroleum ether.

The parameter values are as follows a1 = a2 = 300 g 1600 C a3 a4 = 4:00 = 1300 g = 167.5 g a5 a6 a7 = 71.7 g = 80% = 85% a8 b1 = 2618 g, a proportion of 8.7 g per g of sugar introduced, b2 = 83 °C 265.2 g = b3, and has a molar proportion relative to sugar equal to 800 C = 1.67 b4 b5 b6 = 2:00 b7 = 96% b8 = 4106.5 g = 898.3 g = 157.9 g bg b10 = b11 = 3.3 g 2618 g C1 = 638 g, a weight ratio equal to 0.71 eau/DMS0 d1 = 2772.3 g d2 = 1.1 g d3 = d4 = 238 g 6988.2 g e1 = 238 g, a concentration equal to 36 g / l ( corresponding to saturation), e2 = e3 = 19 821 ml 98%

The overall yield of FDC is equal to 74 s.

The use of petroleum ether as solvent of crystallization leads to a near-quantitative recovery of FDC with a degree of purity higher than 99.5%.

Example 4

In this example, the reagents and solvents are - third solvent: MIBK - electrophile: acetic anhydride - crystallization solvent: petroleum ether.

The parameter values are as follows a1 a2 = 200 g = 1500 C = a3 a4 6:00 = 1200 g = 119 g a5 a6 a7 = 50.7 g = 85 * = 90 g a8 b1 = 2734 g, a proportion of 13.7 g per g of sugar introduced, b2 = b3 = 183.7 C 870 g and has a molar ratio to equal to 1.67 sugar b4 b5 = 700 C = 4:00 b7 b6 = 93% = 4063, 4 g b8 = 929 g = 108.9 g bg b10 = b11 = 4.5 g 2734 g C1 = 638 g, a weight ratio equal to eau/DMS0 a 69 d1 = d2 = 1 g 2840.6 g d3 d4 = 105.6 g = 2722.2 g e1 = 105.6 g, a concentration equal to 36 g / l (corresponding to saturation), e2 = e3 = 8833.5 ml 98.5%

The overall yield of FDC is equal to 4p 76.5 and purity of FDC got 98%.

The use of MIBK as solvent leads to a third processing time HMF / FDC longer if one wishes to obtain a return of the same order.
Process for the manufacture of furan-2,5-dicarboxaldehyde

The invention relates to a process for the manufacture of high purity furan-2,5-dicarboxaldehyde (FDC). This process consists in synthesising hydroxymethylfurfural (HMF) by mixing one or more sugars in a solvent of DMSO type and by heating the mixture and in then converting the HMF obtained to FDC in the reaction mixture in the presence of an electrophilic agent. The process is characterised in that, at the end of the synthesis of the HMF, a third solvent, especially diethyl ketone (DEK), is added to the mixture and, after conversion of the HMF, water is added to the reaction mixture with a view to creating a liquid aqueous phase which will dissolve the DMSO. The FDC formed dissolves in the liquid organic phase and can be easily separated from the latter in order to obtain a stable compound of high purity.
### Patents cited in the search report

#### 1. PROCEDE DE FABRICATION DU 5-HYDROXYMETHYLFURFURAL

<table>
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<tr>
<th>Inventor</th>
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<th>CPC</th>
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#### 2. PRODUCTION OF 2* 55FURANDICARBOXYALDEHYDE

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<td>MORIKAWA SHIYUNICHI</td>
<td>NOGUCHI KENKYUSHO</td>
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### 1. Hydroxymethyl Furfural Oxidation Methods

**Inventor:** LILGA MICHAEL A [US] HALLEN RICHARD T [US] (+3)

**Applicant:** Battelle Memorial Institute [US]

**CPC:** C07D307/44

**IPC:** C07D307/46

**Publication info:** US2010152470 (A1) 2010-06-17

**US8193382 (B2) 2012-06-05**

**Priority date:** 2006-10-31

### 2. PROCESSES FOR THE PREPARATION AND PURIFICATION OF HYDROXYMETHYL FURALDEHYDE DERIVATIVES

**Inventor:** SANBORN ALEXANDRA J [US]

**Applicant:** Archer Daniels Midland Company

**CPC:** C07C51/00

**IPC:** C07C51/245

**Publication info:** US2009281338 (A1) 2009-11-12

**US8058458 (B2) 2011-11-15**

**Priority date:** 2004-12-10

### 3. PROCESSES FOR THE PREPARATION AND PURIFICATION OF HYDROXYMETHYL FURALDEHYDE AND DERIVATIVES

**Inventor:** SANBORN ALEXANDRA J [US]

**Applicant:** Archer Daniels Midland Co [US] SANBORN ALEXANDRA J [US]

**CPC:** C07D307/42

**IPC:** C07D307/46

**Publication info:** WO2006063220 (A2) 2006-06-15

**WPO2006063220 (A3) 2007-01-11**

**Priority date:** 2004-12-10

### 4. PROCESS FOR PREPARING 2,5-DIFORMYL FURAN FROM CARBOHYDRATES

**Inventor:** GRUSHIN VLADIMIR HERRON NORMAN (+1)

**Applicant:** Du Pont [US]

**CPC:** C07D307/46

**IPC:** C07B61/00

**Publication info:** WO03024947 (A1) 2003-03-27

**Priority date:** 2001-09-17

### 5. METHOD FOR SELECTIVELY PREPARING 2,5-FURANDICARBOXALDEHYDE FROM 5-HYDROXYMETHYL FURAN 2-CARBOXALDEHYDE

**Inventor:** DURAND GERMAIN [FR] FAUGERAS PIERRE [FR] (+6)

**Applicant:** AGRICHIMIE SA [FR] DURAND GERMAIN [FR] (+7)

**CPC:** C07D307/46

**IPC:** C07D307/46

**Publication info:** WO9617836 (A1) 1996-06-13

**Priority date:** 1994-12-07
Process for the manufacture of furan-2,5-dicarboxaldehyde

The EPO does not accept any responsibility for the accuracy of data and information originating from other authorities than the EPO; in particular, the EPO does not guarantee that they are complete, up-to-date or fit for specific purposes.

Legal status of FR2669636 (A1) 1992-05-29; FR2669636 (B1) 1994-05-20:

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Code Expl.: NOTIFICATION OF LAPSE

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### Family list: FR2669636 (A1) — 1992-05-29

1. **Process for the manufacture of furan-2,5-dicarboxaldehyde**


Last updated: 09.10.2013  Worldwide Database  5.8.11.5; 93p
CLAIMS FR2669636

CLAIMS

1 / - A process for manufacturing 2,5 furan dicarboxaldehyde (FDC) of high purity, wherein (A) a synthesis hydroxymethylfurfural (HMF) (Ai) prepared by mixing one or more sugars in a strongly polar aprotic solvent of sulfoxides family and (A2) by heating the mixture, then (B) converting the obtained HMF FDC in the reaction medium in the presence of an electrophilic agent, characterized in that (A3) is added to the highly polar aprotic solvent according to the third group solvent diethyl (DEC), methyl isobutyl ketone (MIBK), dichloromethane (DCM) or ethyl acetate,

(C) after processing of HMF, the water was added into the reaction mixture to create an aqueous liquid phase, said first phase, from the highly solubilizing polar aprotic solvent, solubilizing the FDC formed in the liquid phase formed by the third organic solvent, said second phase,

(D) the two liquid phases are separated in order to obtain the second phase containing the FDC,

(E) said second phase FDC third solvent evaporation phase is concentrated,

(F) is carried out from the concentrated phase obtained crystallization FDC

(G) and the CDF obtained is filtered.

2 / - Method of manufacture according to claim 1, characterized in that (A1) is used as the highly polar aprotic solvent, dimethylsulfoxide (DDS0 D) and as starting sugar, fructose or polyfructans.

3 / - Method of manufacture according to one of claims 1 or 2, characterized in that (Ai) or the starting sugars are mixed in the highly polar aprotic solvent to obtain a solution having a weight content of between 200 g and 1000 g of sugar per kg of the aprotic solvent.

4 / - Production method according to one of claims 1 to 3, wherein (A2) is the solution of sugar and strongly polar aprotic solvent between 1000 and 1700 C during a heating period of between 2 and 8 hours .

5 / - Production method according to one of claims 1 to 4, characterized in that (A3) is used as the third solvent December

6 / - Process for manufacturing according to one of claims 1 to 5, characterized in that (A3) of the third solvent is added in a proportion between 5 and 20 g of solvent per gram of sugar introduced.

7 / - Process for manufacturing according to one of claims 1 to 6, characterized in that, after adding the third solvent, (A4) is removed at least a fraction of the water formed in the reaction medium at the synthesis of HMF.

8 / - Process for manufacturing according to one of claims 1 to 7, wherein (B) the processing is carried HMF in the presence of an electrophilic agent of the following group: acetic anhydride, dicyclohexyl carbodiimide mixed acid orthophosphoric, or phosphoric anhydride.

9 / - method according to claim 8, wherein (B) the acetic anhydride is used as electrophilic agent in a molar ratio relative to the
starting sugar of between 1 and 5.

10 / - Production method according to one of claims 1 to 9, characterized in that (C) after processing of HMF, water is added to the reaction medium so that it contains a weight ratio of water between 0.23 and 0.9 relative to the highly polar aprotic solvent contained in the medium.

11 / - Production method according to one of claims 1 to 10, characterized in that

(A3) the third solvent is added to the end of the synthesis reaction of HMF after a period of between 4 and 6 hours after completion of warm-

(B) the processing is carried out in HMF

FDC cooling the medium to a temperature between 250 C and 800 C and adding the electrophile,

(G) the water is added at the end of the transformation reaction after a period of between 4 and 6 hours after the addition of the electrophile.

12 / - Production method according to one of claims 1 to 11, characterized in that (D) separating the two liquid phases is carried out by decantation while maintaining the temperature between -5 C and 300 C.

13 / - Production method according to one of claims 1 to 12, characterized in that (e) was concentrated by evaporating the second liquid phase to the saturation in the third solvent FDC.

14 / - Production method according to one of claims 1 to 13, characterized in that (F) performs lton FDC crystallization by adding a solvent of crystallization in the concentrated phase and cooling the solution to a temperature between 50 C and 200 C.

15 / - method according to claim 14, characterized in that (F) a crystallization solvent of the following group is used: trifluoro1 1.2 1,2,2 trichloro ethane, petroleum ether, cyclohexane, in volume fraction between 2 and 5 1 of solvent per one concentrated solution.

16 / - Production method according to one of claims 1 to 15, characterized in that, after separation of the two liquid phases (D), the highly polar aprotic solvent content is recovered in the aqueous phase by distillation under reduced pressure, for recycling of said aprotic solvent.

17 / - Production method according to one of claims 1 to 16, characterized in that the third solvent is recycled, on the one hand, directly after evaporation (E), on the other hand, after crystallization (F), operating in a distillation of the liquid phase, the crystallization solvent separated in said distillation being recycled itself.