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Drugs intermediates 2-formylcarbonylbenzoic acid synthesis method

FIELD OF THE INVENTION

The present invention relates to drugs intermediates 2-formylcarbonylbenzoic acid synthesis method.

GENERAL BACKGROUND

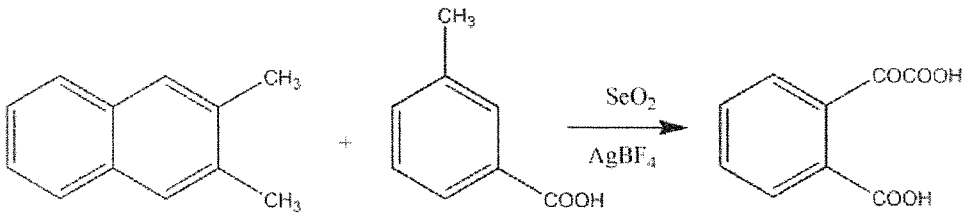
2-formylcarbonyl benzoic acid is mainly used for chemical reagents, fine chemicals, pharmaceutical intermediates, materials intermediates, however, most of the existing synthetic methods are using naphthalene as reactants, it is complicated and the final yield is not very high. Therefore, it is necessary to propose a new synthetic method for further improving the quality and yield of the product and reducing the byproduct content, it has important economic significance.

SUMMARY

The purpose of the present invention is to provide drugs intermediates 2-formylcarbonylbenzoic acid synthesis method, comprises the following steps:

(i) the reaction vessel was added 3 mole 2,3-dimethylnaphthalene, 1.5 L ethanol solution, and 4-5 mol 3-carboxymethyl, controlling the stirring speed at 110-130 rpm, maintaining the temperature to 40-46℃, keeping for 90-110 min, continuing to add 20g selenium dioxide powder, silver boron fluoride powder 30-50g, raising the solution temperature to 50-58℃, keeping for 130-150 min, filter, washed with potassium bromide solution, washed with isobutyric acid solution, and with methyl chloroacetate solution, dehydrated with the dehydrating agent to obtain 2-formylcarbonylbenzoic acid; wherein, the mass fraction of the ethanol solution in step (i) is 20-27%, the mass fraction of the potassium bromide described in step (i) is 10-16%, the mass fraction of the isobutyric acid solution of step (i) is 50-55%, the mass fraction of the methyl chloroacetate solution in step (i) is 70-78%, the dehydrating agent in step (i) is any one of anhydrous magnesium sulfate and activated alumina.

Throughout the reaction process can be the following reaction formula:



Advantage of the present invention is that: reducing intermediate links reaction, decreasing the reaction time and improving the reaction yield.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

The following examples with reference to specific embodiments of the present invention are further illustrated:

drugs intermediates 2-formylcarbonylbenzoic acid synthesis method.

Embodiment 1

The reaction vessel was added 3 mol 2,3-dimethylnaphthalene, 1.5 L ethanol solution with a mass fraction of 20%, 4 mol 3-carboxymethyl, controlled stirring speed at 110 rpm, kept at 40 °C for 90 min , continue to add 20 g selenium dioxide powder, 30g silver borofluoride powder, raising the temperature of the solution to 50℃ for 130 min, filter, washed with potassium bromide solution with a mass fraction of 10%, washed with isobutyric acid solution with a mass fraction of 50%, washed with methyl chloroacetate solution with a mass fraction of 70%, and dehydrated with anhydrous magnesium sulfate dehydrating agent to obtain 506.34g 2-formylcarbonylbenzoic acid, yield 87%.

Embodiment 2

The reaction vessel was added 3 mol 2,3-dimethylnaphthalene, 1.5 L ethanol solution with a mass fraction of 23%, 4.5 mol 3-carboxymethyl, controlled stirring speed at 120 rpm, kept at 43 °C for 100 min, continue to add 20 g selenium dioxide powder, 40g silver borofluoride powder, raising the temperature of the solution to 53℃ for 140 min, filter, washed with potassium bromide solution with a mass fraction of

13%, washed with isobutyric acid solution with a mass fraction of 52%, washed with methyl chloroacetate solution with a mass fraction of 73%, and dehydrated with activated alumina dehydration agent, 2-formylcarbonyl benzoic acid 529.62g, yield of 91%.

Embodiment 3

The reaction vessel was added 3 mol 2,3-dimethylnaphthalene, 1.5 L ethanol solution with a mass fraction of 27%, 5 mol 3-carboxymethyl, controlled stirring speed at 130 rpm, kept at 46 °C for 110 min , continue to add 20 g selenium dioxide powder, 50g silver borofluoride powder, raising the temperature of the solution to 58℃ for 150 min, filter, washed with potassium bromide solution with a mass fraction of 16%, washed with isobutyric acid solution with a mass fraction of 55%, washed with methyl chloroacetate solution with a mass fraction of 78%, and dehydrated with anhydrous magnesium sulfate dehydrating agent to obtain 2-formylcarbonyl benzoic acid 541.26g, the yield of 93%.

Claims

1. Drugs intermediates 2-formylcarbonylbenzoic acid synthesis method, comprises the following steps:

(i) the reaction vessel was added 3 mole 2,3-dimethylnaphthalene, 1.5 L ethanol solution, and 4-5 mol 3-carboxymethyl, controlling the stirring speed at 110-130 rpm, maintaining the temperature to 40-46℃, keeping for 90-110 min, continuing to add 20g selenium dioxide powder, silver boron fluoride powder 30-50g, raising the solution temperature to 50-58℃, keeping for 130-150 min, filter, washed with potassium bromide solution, washed with isobutyric acid solution, and with methyl chloroacetate solution, dehydrated with the dehydrating agent to obtain 2-formylcarbonylbenzoic acid; wherein, the mass fraction of the ethanol solution in step (i) is 20-27%, the mass fraction of the potassium bromide described in step (i) is 10-16%, the mass fraction of the isobutyric acid solution of step (i) is 50-55%.

2. Drugs intermediates 2-formylcarbonylbenzoic acid synthesis method according to claim 1 wherein the mass fraction of the methyl chloroacetate solution in step (i) is 70-78%.

3. Drugs intermediates 2-formylcarbonylbenzoic acid synthesis method according to claim 1 wherein the dehydrating agent in step (i) is any one of anhydrous magnesium sulfate and activated alumina.