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Medicine intermediates terephthalic acid synthesis method

FIELD OF THE INVENTION

The present invention relates to medicine intermediates terephthalic acid synthesis method.

GENERAL BACKGROUND

Terephthalic acid is used to produce polyethylene terephthalate. Another important application of terephthalic acid is the production of plasticizers, which includes two categories: one is the dioctyl terephthalate, which is the product of terephthalic acid and 2-ethylhexanol esterification reaction, it is a high flash point, high specific resistance of high-quality plasticizer, especially for heat and insulation requirements of the production of high cable material; the second is a polyester plasticizer, it is a terephthalic acid and polyols, such as diethylene glycol, triethylene glycol, glycerol, propylene glycol, butanediol. Such as the product of the esterification polycondensation reaction, the relative molecular mass is generally between 1000 and 4000, and the relative molecular mass ratio of the polyester as a plasticizer is much smaller than that of the polyester for chemical and plastic packaging. However, most of the existing synthetic methods are using p-phenylethanone and potassium permanganate as reactant, 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.

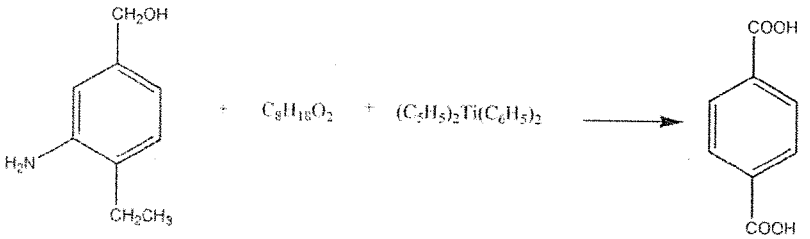
SUMMARY

The purpose of the present invention is to provide medicine intermediates terephthalic acid synthesis method, comprises the following steps:

(i) 2 mol 4-ethyl-5-amino-benzyl alcohol and 4-5 mol 2,5-dimethyl-2,5-hexanediol were added to the reaction vessel, raised the temperature

of the solution to 60-67 ℃, controlled the stirring speed at 170-190 rpm, kept for 130-170 min, then add 3-4 mol dicyldiphenyl titanium in 6 to 9 times, raised the temperature of the solution to 80-86 ℃, kept for 70-90 min, precipitated solid, filtered, the solid washed with potassium bromide solution, washed with dipentyl ether solution, 4-heptanone solution, reduced solution temperature to 3-6 ℃, recrystallized in diisopropyl ether solution, dehydrated with dehydration, got the finished product terephthalic acid; wherein, the 2,5-dimethyl-2,5-hexanediol solution has a mass fraction of 80-85% as described in step (i), and the mass fraction of the potassium solution in step is 40-45%, the mass fraction of the dipentyl ether solution in step (i) is 75-83%, the mass fraction of the 4-heptanone solution in step (i) is 85-90%, the mass fraction of the diisopropyl ether solution is 92-96%.

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:

medicine intermediates terephthalic acid synthesis method.

Embodiment 1

2 mol 4-ethyl-5-amino-benzyl alcohol, 4 mol a 2,5-dimethyl-2,5-hexanediol solution with a mass fraction of 80% were added to the reaction vessel, and the temperature of the solution was raised to 60 °C, controlled the stirring speed at 170 rpm, kept it for 130 min, then added 3 mol dicyldiphenyl titanium in 6 times, raised the

temperature of the solution to 80 ℃ for 70 min, precipitated solid, filter, filter cake washed with potassium bromide solution with mass fraction of 40%, washed with dipentyl ether solution with mass fraction of 75%, and washed with 4-heptanone solution with mass fraction of 85%, the solution temperature was reduced to 3℃, recrystallized in diisopropyl ether solution with mass fraction of 92%, dehydrated with dehydration, got the finished terephthalic acid 288.36g, yield 89%.

Embodiment 2

2 mol 4-ethyl-5-amino-benzyl alcohol, 4.5 mol a 2,5-dimethyl-2,5-hexanediol solution with a mass fraction of 82% were added to the reaction vessel, and the temperature of the solution was raised to 63 °C, controlled the stirring speed at 180 rpm, kept it for 150 min, then added 3.5 mol dicyldiphenyl titanium in 7 steps, raised the temperature of the solution to 83℃ for 80 min, precipitated solid, filter, filter cake washed with potassium bromide solution with mass fraction of 42%, washed with dipentyl ether solution with mass fraction of 80%, and washed with 4-heptanone solution with mass fraction of 87%, the solution temperature was reduced to 4℃, recrystallized in diisopropyl ether solution with mass fraction of 94%, dehydrated with dehydration, got the finished terephthalic acid 294.84g, yield 91%.

Embodiment 3

2 mol 4-ethyl-5-amino-benzyl alcohol, 5 mol a 2,5-dimethyl-2,5-hexanediol solution with a mass fraction of 85% were added to the reaction vessel, and the temperature of the solution was raised to 67 °C, controlled the stirring speed at 190 rpm, kept it for 170 min, then added 4 mol dicyldiphenyl titanium in 9 times, raised the temperature of the solution to 86 ℃ for 90 min, precipitated solid, filter, filter cake washed with potassium bromide solution with mass fraction of 45%, washed with dipentyl ether solution with mass fraction of 83%, and washed with 4-heptanone solution with mass fraction of 90%, the solution temperature was reduced to 6℃, recrystallized in diisopropyl ether solution with mass fraction of 96%, dehydrated with dehydration, got the finished terephthalic acid 301.32g, yield 93%.

Claims

1. Medicine intermediates terephthalic acid synthesis method, comprises the following steps:

(i) 2 mol 4-ethyl-5-amino-benzyl alcohol and 4-5 mol 2,5-dimethyl-2,5-hexanediol were added to the reaction vessel, raised the temperature of the solution to 60-67 ℃, controlled the stirring speed at 170-190 rpm, kept for 130-170 min, then add 3-4 mol dicyldiphenyl titanium in 6 to 9 times, raised the temperature of the solution to 80-86 ℃, kept for 70-90 min, precipitated solid, filtered, the solid washed with potassium bromide solution, washed with dipentyl ether solution, 4-heptanone solution, reduced solution temperature to 3-6 ℃, recrystallized in diisopropyl ether solution, dehydrated with dehydration, got the finished product terephthalic acid; wherein, the 2,5-dimethyl-2,5-hexanediol solution has a mass fraction of 80-85% as described in step (i), and the mass fraction of the potassium solution in step is 40-45%, the mass fraction of the dipentyl ether solution in step (i) is 75-83%.

2. Medicine intermediates terephthalic acid synthesis method according to claim 1 wherein the mass fraction of the 4-heptanone solution in step (i) is 85-90%.

3. Medicine intermediates terephthalic acid synthesis method according to claim 1 wherein the mass fraction of the diisopropyl ether solution is 92-96%.