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Peroxide second tertbutyl organic intermediates synthesis method

FIELD OF THE INVENTION

The present invention relates to a method for preparing a pharmaceutical intermediate which belongs to the field of organic synthesis, more particularly, relates to peroxide second tertbutyl organic intermediates synthesis method.

GENERAL BACKGROUND

Peroxide second tert butylas cross-linking agentcan be used in silicone rubber, synthetic rubber and natural rubber, polyethylene, EVA and EPT. As polymerization initiator, it can be used in polystyrene and polyethylene. Most of the existing synthesis method adopt tertbutyl as raw material, and under the action of sulfuric acid, the hydrogen peroxide reaction with hydrogen peroxide is produced, and the reaction of tertbutyl hydrogen peroxide can be made with tertbutyl alcohol. But it need to use the synthetic method of sulfuric acid, hydrogen peroxide as raw materials, reaction raw sulfuric acid corrosion resistance is stronger, the production equipment corrosion resistant ability of the demand is higher, health hazards for the operation personnel also is large, it is not conducive to safety production. The hydrogen peroxide of reactive materials is an explosive and powerful oxidizer. In the sealed container of suitable temperature, the gas phase explosion can be generated. Hydrogen peroxide has a strong corrosive effect. Inhalation of steam or fog is strongly irritating to the airway. Direct contact with liquid can cause irreversible damage and even blindness. These increase the risk factor for the synthesis process and are not conducive to safe production. The above analysis shows that there are many deficiencies in this synthesis method, so it is necessary to put forward a new synthetic method.

SUMMARY

Based on the technical problems of the background technology, the purpose of the present invention is to provide peroxide second tert butyl organic intermediates synthesis method, comprises the following steps:

A: 2-methyl-2-propylamine, potassium sulfate solution were added to the reaction vessel, controlled the mixing speed at 110-130 rpm, controlled the reaction temperature

of the solution to 5-10℃, added isopropyl alcohol aluminum, added the second glycol two ethyl ether solution in batches in 30-50min, continued to react for 90-120min;

B:then added nickel fluoride power, controlled the mixing speed at220-250rpm to react for 2-4h, let stand for 30-50min, layered, washed with sodium chloride solution for30-40min, washed with 1-pentanol solution for 20-40min, recrystallized in 2-hexone solution, dehydrated with dehydration, got the finished product peroxide second tert butyl.

Preferably, the potassium sulfate solution has a mass fraction of 10-16%.

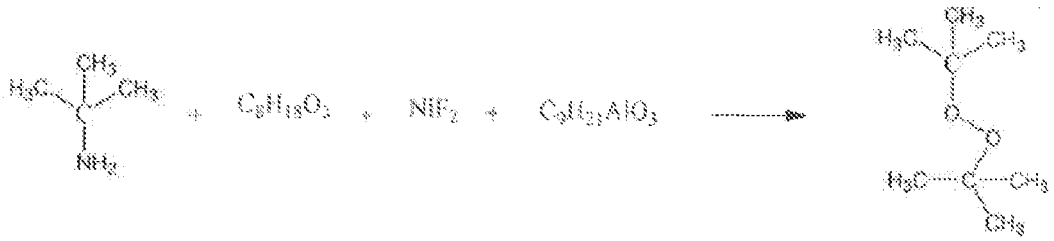
Preferably, the mass fraction of second glycol two ethyl ether solution is 20-25%.

Preferably, the sodium chloride solution has a mass fraction of 15-21%.

Preferably, the 1-pentanol solution has a mass fraction of 40-46%.

Preferably, the mass fraction of 2-hexone solution is 60-65%.

Throughout the reaction process can be the following reaction formula:



Compared with the synthetic method disclosed in the background art, the invention provides peroxide second tert butyl organic intermediates synthesis method, it do not need to use sulfuric acid, hydrogen peroxide as raw materials, to avoid the reaction of raw materials for sulfuric acid corrosion damage to the equipment as well as the harm to the health of the operation personnel. It also avoids the risk of hydrogen gas explosion from strong oxidant hydrogen peroxide and hydrogen peroxide strong corrosive production operators respiratory and eye injury risk. It can reduce a lot of the reaction of the intermediate links, shorten the reaction time, and also improve reaction yield, at the same time, the present invention provides a new synthetic route which has laid a good foundation for further enhancing the yield of the reaction.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

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

Embodiment 1

Peroxide second tert butyl organic intermediates synthesis method, comprises the following steps:

A:2mol2-methyl-2-propylamine, and 900ml potassium sulfate solution with a mass fraction of 10%were added to the reaction vessel, controlled the mixing speed at 110rpm, controlled the reaction temperature of the solution to 5℃, added 4mol isopropyl alcohol aluminum, added 4mol the second glycol two ethyl ether solution with a mass fraction of 20% in two batches in 30min, continued to react for 90min;

B:then added 2mol nickel fluoride power, controlled the mixing speed at220rpm to react for 2h, let stand for 30min, layered, washed with sodium chloride solution with a mass fraction of 15% for30min, washed with 1-pentanol solution with a mass fraction of 40% for 20min, recrystallized in 2-hexone solution with a mass fraction of 60%, dehydrated with anhydrous calcium sulfate dehydration, got the finished product peroxide second tert butyl285.284g，yield of 97.7%.

Embodiment 2

Peroxide second tert butyl organic intermediates synthesis method, comprises the following steps:

A:2mol 2-methyl-2-propylamine, and 900ml potassium sulfate solution with a mass fraction of 13%were added to the reaction vessel, controlled the mixing speed at 120rpm, controlled the reaction temperature of the solution to 7℃, added 5mol isopropyl alcohol aluminum, added 5mol the second glycol two ethyl ether solution with a mass fraction of 23% in three batches in 40min, continued to react for 100min;

B:then added 3mol nickel fluoride power, controlled the mixing speed at230rpm to react for 3h, let stand for 40min, layered, washed with sodium chloride solution with a mass fraction of 18% for35min, washed with 1-pentanol solution with a mass fraction of 43% for 30min, recrystallized in 2-hexone solution with a mass fraction of 63%, dehydrated with anhydrous calcium sulfate dehydration, got the finished product peroxide second tert butyl285.868g，yield of 97.9%.

Embodiment 3

Peroxide second tert butyl organic intermediates synthesis method, comprises the

following steps:

A:2mol 2-methyl-2-propylamine, and 900ml potassium sulfate solution with a mass fraction of 16%were added to the reaction vessel, controlled the mixing speed at 130rpm, controlled the reaction temperature of the solution to 10℃, added 6mol isopropyl alcohol aluminum, added 6mol the second glycol two ethyl ether solution with a mass fraction of 25% in four batches in 50min, continued to react for 120min;

B:then added 4mol nickel fluoride power, controlled the mixing speed at250rpm to react for 4h, let stand for 50min, layered, washed with sodium chloride solution with a mass fraction of 21% for40min, washed with 1-pentanol solution with a mass fraction of 46% for 40min, recrystallized in 2-hexone solution with a mass fraction of 65%, dehydrated with anhydrous calcium sulfate dehydration, got the finished product peroxide second tert butyl286.744g，yield of 98.2%.

The embodiment of the present invention are merely preferred embodiment of the present invention, but the range of the present invention is not limited this, and any person who is familiar with those skilled in the arts, within the technical range of the present invention. It is intended that the technical solution and its inventive concept be replaced or modified equivalently with reference to the range of the invention.

Claims

1. Peroxide second tert butyl organic intermediates synthesis method, comprises the following steps:

A: 2-methyl-2-propylamine and potassium sulfate solution were added to the reaction vessel, controlled the mixing speed at 110-130 rpm, controlled the reaction temperature of the solution to 5-10℃, added isopropyl alcohol aluminum, added the second glycol two ethyl ether solution in batches in 30-50min, continued to react for 90-120min;

B:then added nickel fluoride power, controlled the mixing speed at 220-250rpm to react for 2-4h, let stand for 30-50min, layered, washed with sodium chloride solution for30-40min, washed with 1-pentanol solution for 20-40min, recrystallized in 2-hexone solution, dehydrated with dehydration, got the finished product peroxide second tert butyl.

2. Peroxide second tert butyl organic intermediates synthesis method according to claim 1 wherein the potassium sulfate solution has a mass fraction of 10-16%.

3. Peroxide second tert butyl organic intermediates synthesis method according to claim 1 wherein the mass fraction of second glycol two ethyl ether solution is 20-25%.

4. Peroxide second tert butyl organic intermediates synthesis method according to claim 1 wherein the sodium chloride solution has a mass fraction of 15-21%.

5. Peroxide second tert butyl organic intermediates synthesis method according to claim 1 wherein the 1-pentanol solution has a mass fraction of 40-46%.