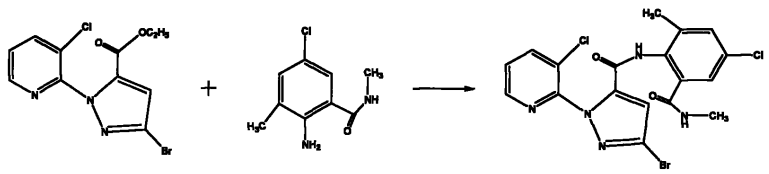
Patents

Synthesis method of chlorantraniliprole pesticide

Abstract

translated from Chinese

The invention discloses a method for synthesizing the pesticide chlorantraniliprole, which belongs to the field of agricultural insecticides, and in particular relates to a method for synthesizing the pesticide chlorantraniliprole. Compound II and III are reacted in an organic solvent at a temperature of 100°C-180°C for 6-10 hours to obtain the target compound I. The advantages of the present invention are: the preparation method is simple and practical, the efficiency is high, and the yield of each step product is above 85%; the reagents used in the synthesis process have low toxicity, the solvent can be recycled and there are few side reactions, so the corrosion to the equipment is small and the The environmental pollution is small; due to the low price of raw materials, the production cost is reduced and the economy is good, so it has a good application prospect.



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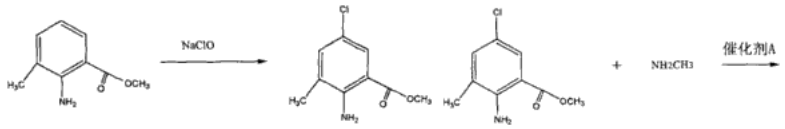
Images (2)



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Landscapes

Organic Low-Molecular-Weight Compounds And Preparation Thereof

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CN101717395A

China

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Other languages: Chinese

Inventor: , , , , ,

Current Assignee : Nankai University

Worldwide applications

2009 CN

Application CN200910228966A events

2009-12-04	Application filed by Nankai University
2009-12-04	Priority to CN200910228966A
2010-06-02	Publication of CN101717395A
Status	Pending

Info: Patent citations (4), Cited by (3), Legal events, Similar documents, Priority and Related Applications

External links: Espacenet, Global Dossier, Discuss

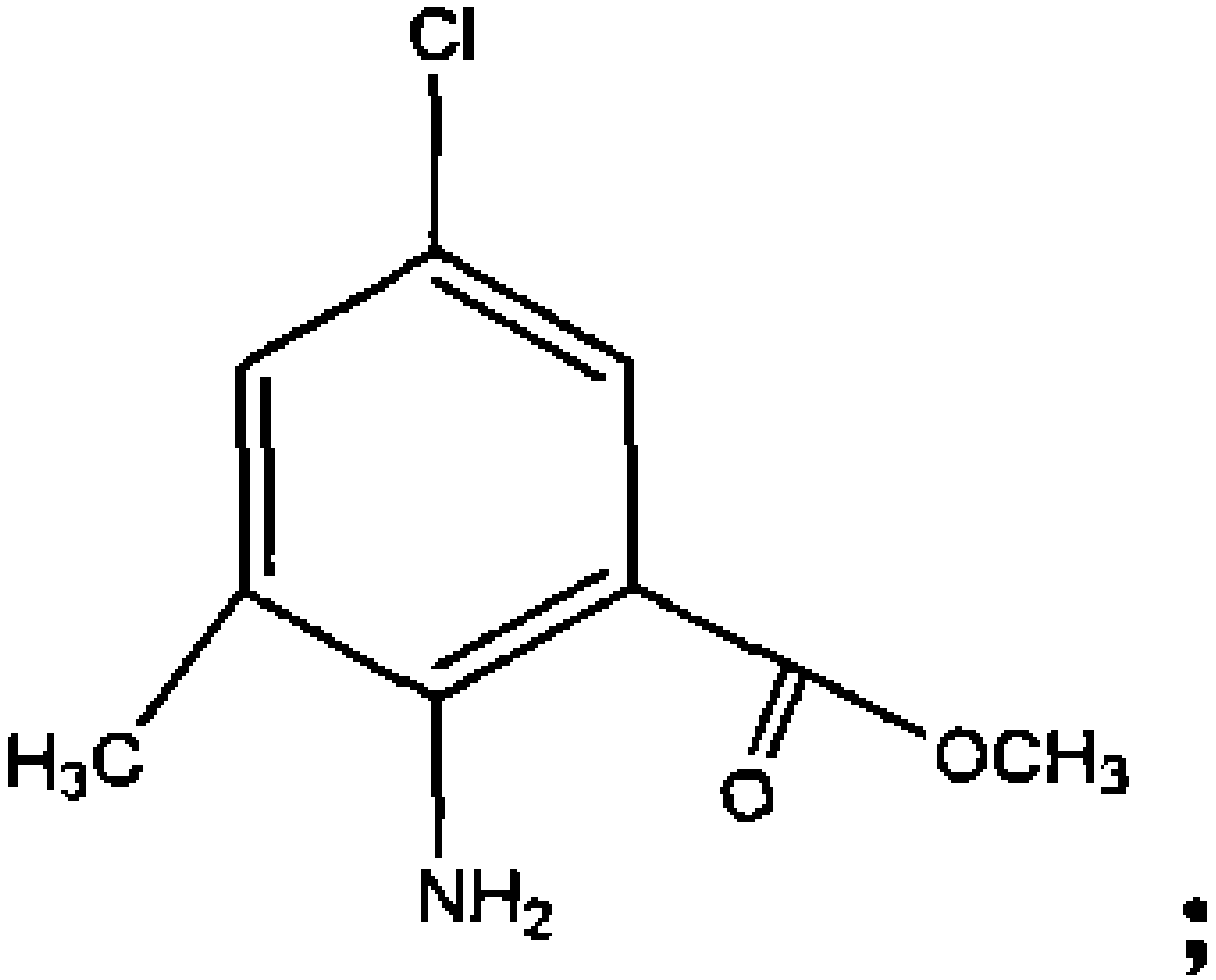
Claims (8)

Hide Dependent

1. the synthetic method of a chlorantraniliprole pesticide, synthesis step is:

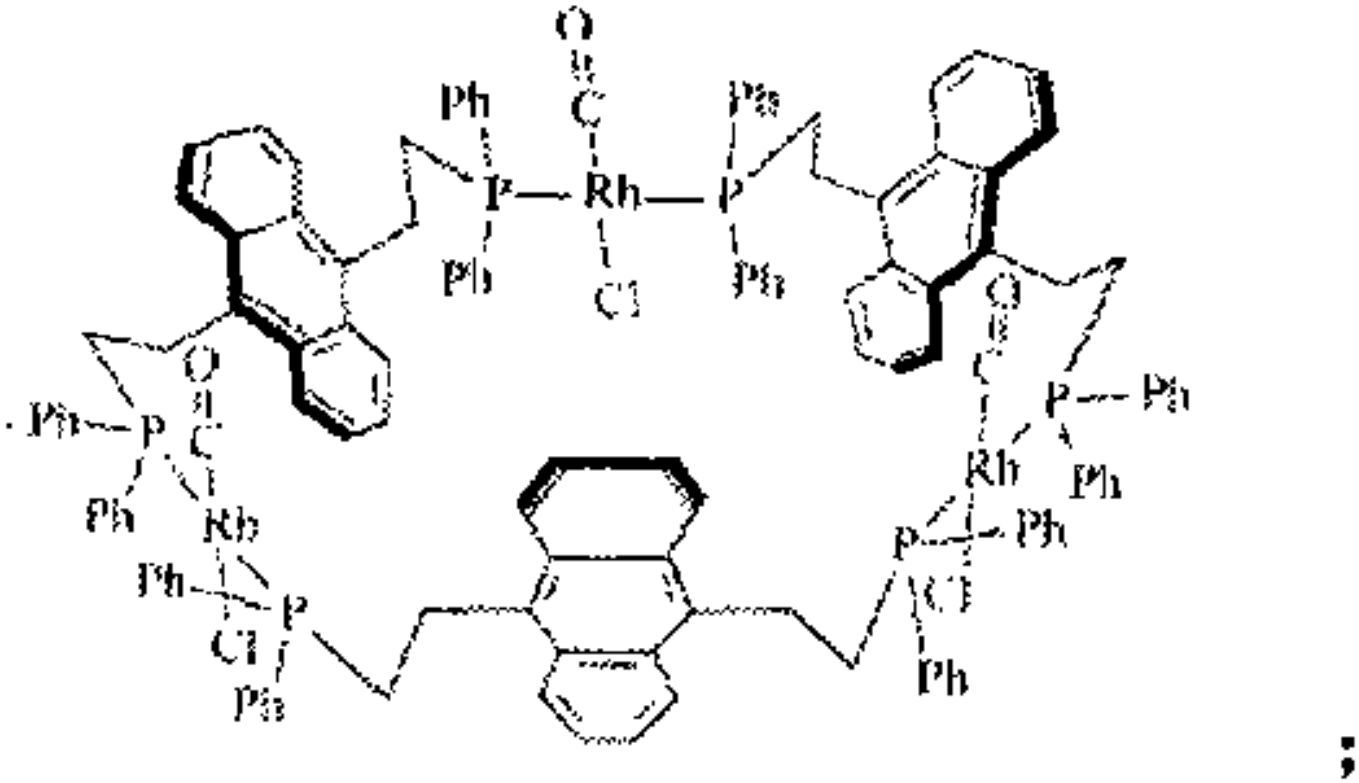
1) 3-methyl-2-amino-5-chloro benzoic ether, i.e. preparation of general formula compound IV:

With 3-methyl-2-Methyl anthranilate is starting raw material, hypochlorous sodium solution and Glacial acetic acid are chlorination reagent, with organic solvent and water jointly as solvent, it is lower than in temperature to mix under-5 °C the condition carries out chlorination, react half an hour, separate organic phase, make 3-methyl-2-amino-5-chloro benzoic ether after the drying, general formula is



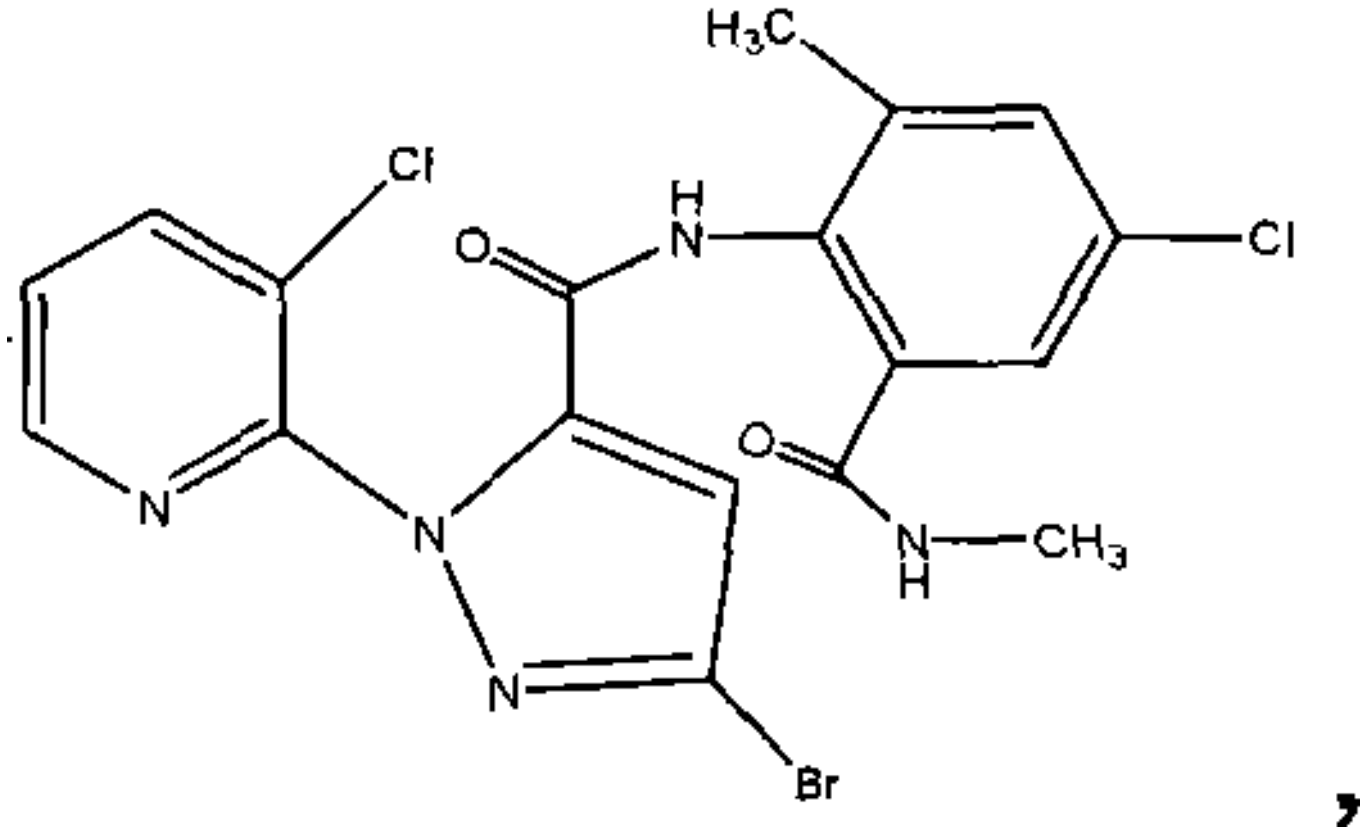
2) preparation of general formula compound II:

The ethanolic soln of 3-methyl-2-amino-5-chloro benzoic ether, methylamine and catalyst A add in the flask, be under 50 100 °C the condition in temperature, reacted 6 hours, separate solvent after reducing to normal temperature and pressure, the crystal that obtains is dissolved with methylene dichloride, add activated carbon then, under 70 80 °C of conditions, stirred 1 hour, can make product compound II after suction filtration is spin-dried for, general formula is The general formula of catalyst A is



3) preparation of Rynaxypyr compound:

II and III are in organic solvent, and temperature is that reaction made the Rynaxypyr Compound I in 6-10 hour under 100 °C of-180 °C of conditions, and general formula is



The general formula of compound III is

2. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1, it is characterized in that, the described chlorine bleach liquor's of step 1) weight percent concentration is 10%, 3-methyl-2-Methyl anthranilate and chlorine bleach liquor's weight ratio is 1: 10 15, and the weight ratio of chlorine bleach liquor and Glacial acetic acid is 10: 1.

3. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that, the amount ratio of the described 3-methyl of step 1)-2-Methyl anthranilate and solvent is 40g 60g/L.

4. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that, the described organic solvent of step 1) is methylene dichloride or trichloromethane.

5. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that, the volumetric ratio of described organic solvent of step 1) and water is 1.5: 1.

6. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that step 2) described methylethylolamine solution weight percentage concentration is 25-30%, the weight ratio of ester group compound IV and methylethylolamine solution is 2-10: 1.

7. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that step 2) described catalyst consumption is the 0.5-1% of 3-methyl-2-amino-5-chloro benzoic ether weight.

8. the synthetic method of chlorantraniliprole pesticide as claimed in claim 1 is characterized in that, the organic solvent of step 3) is selected from toluene, dimethylbenzene, trimethylbenzene, inclined to one side trichlorobenzene, one or more among dioxane or the DMF.

Description

A kind of synthetic method of chlorantraniliprole pesticide

Technical field

The invention belongs to the agricultural insecticide field, be specifically related to a kind of synthetic method of chlorantraniliprole pesticide.

Background technology

Because sterilant, sterilant are in use for some time, insect, germ etc. are biological can to produce resistance to it, therefore, need the constantly novel compound of invention with desinsection, fungicidal activity, and have the new variety of carrying out the mass production possibility industrial, and chlorantraniliprole pesticide has just satisfied above various requirement, and it has higher prevention effect to armyworm under the concentration of 50ppm.And be difficult for and existing sterilant generation cross resistance, lower to fish, honeybee, hydrobiont, natural enemy and mammalian toxicity, very friendly to environment, and to agricultural-food noresidue influence, good with the mixed performance of other agricultural chemicals.

Chlorantraniliprole pesticide is a class broad spectrum pesticide, is mainly used in the lepidoptera pest of preventing and treating various crop, and other insects are also had preventive effect preferably.Its mechanism of action is to activate blue Buddhist nun's alkali acceptor, discharges the calcium of storing in unstriated muscle and the striated muscle cell, causes that the muscle adjusting is weak, the fiber crops illness, until insect death.The mode of action is stomach toxicity and contact toxicity, and stomach toxicity is the main mode of action.Can in stem, leaf surface infiltration plant materials, also can move by the root absorption with at xylem.

Based on the advantage of above-mentioned this kind agricultural chemicals, be badly in need of a kind of application and production that economy height, yield is good, side reaction is few and environmental pollution is little synthetic method promote this kind agricultural chemicals that has at present.And present document " WO2006068669, WO2008010897, Bioorganic & amp; Medicinal Chemistry Letter 2007 (17) 6274-6279 " synthetic method of this kind compound of introducing;

characteristics are separately all arranged; but exist difficulty on certain operation and the economy for industrialized big production, and such as: yield is low, pollution is big, aftertreatment is complicated, the high deficiency of cost.

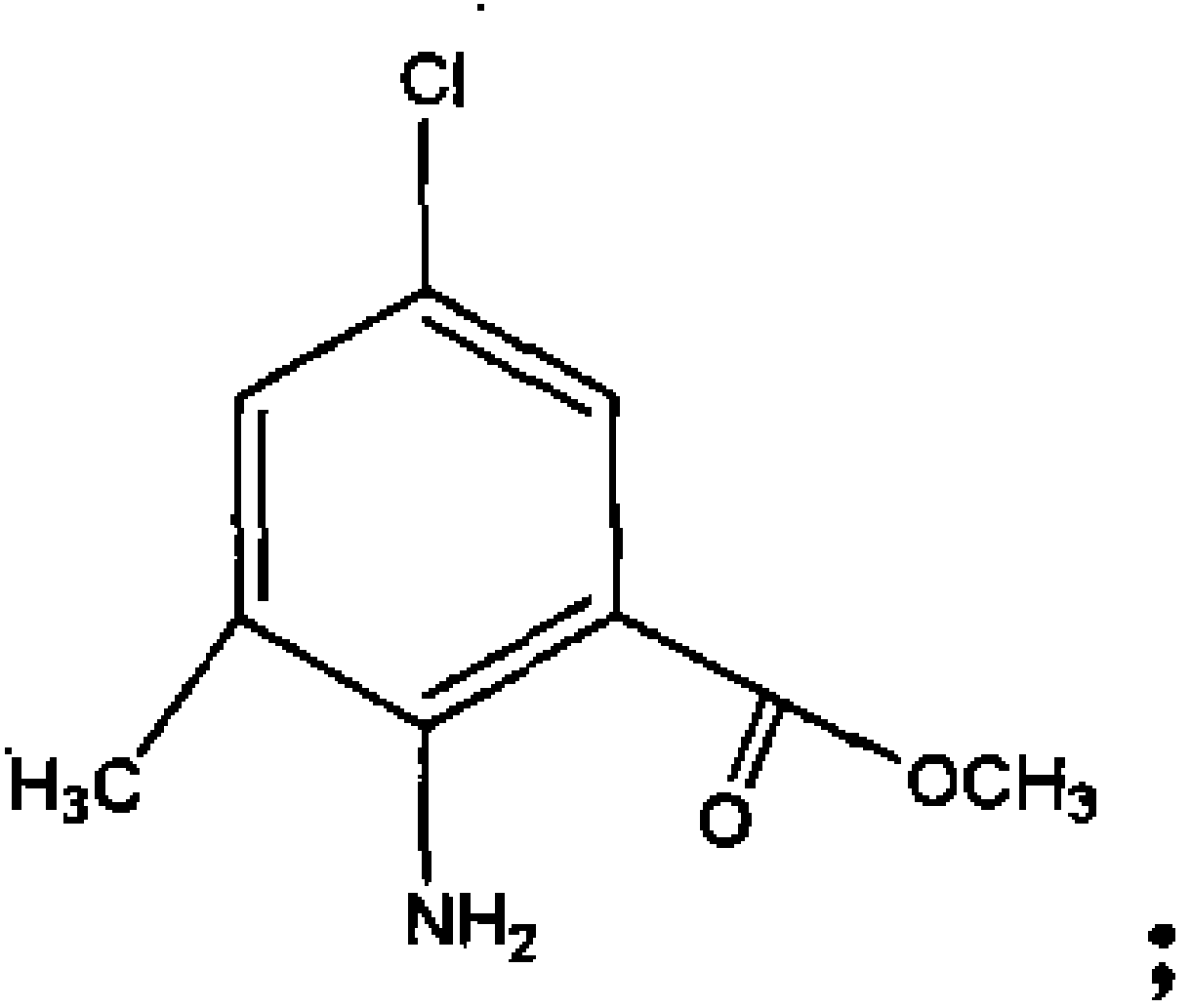
Summary of the invention

The synthetic method that the purpose of this invention is to provide a kind of simple and practical, economy is high, yield is high, side reaction is few and environmental pollution is little chlorantraniliprole pesticide.

The synthetic method of chlorantraniliprole pesticide of the present invention, synthesis step is:

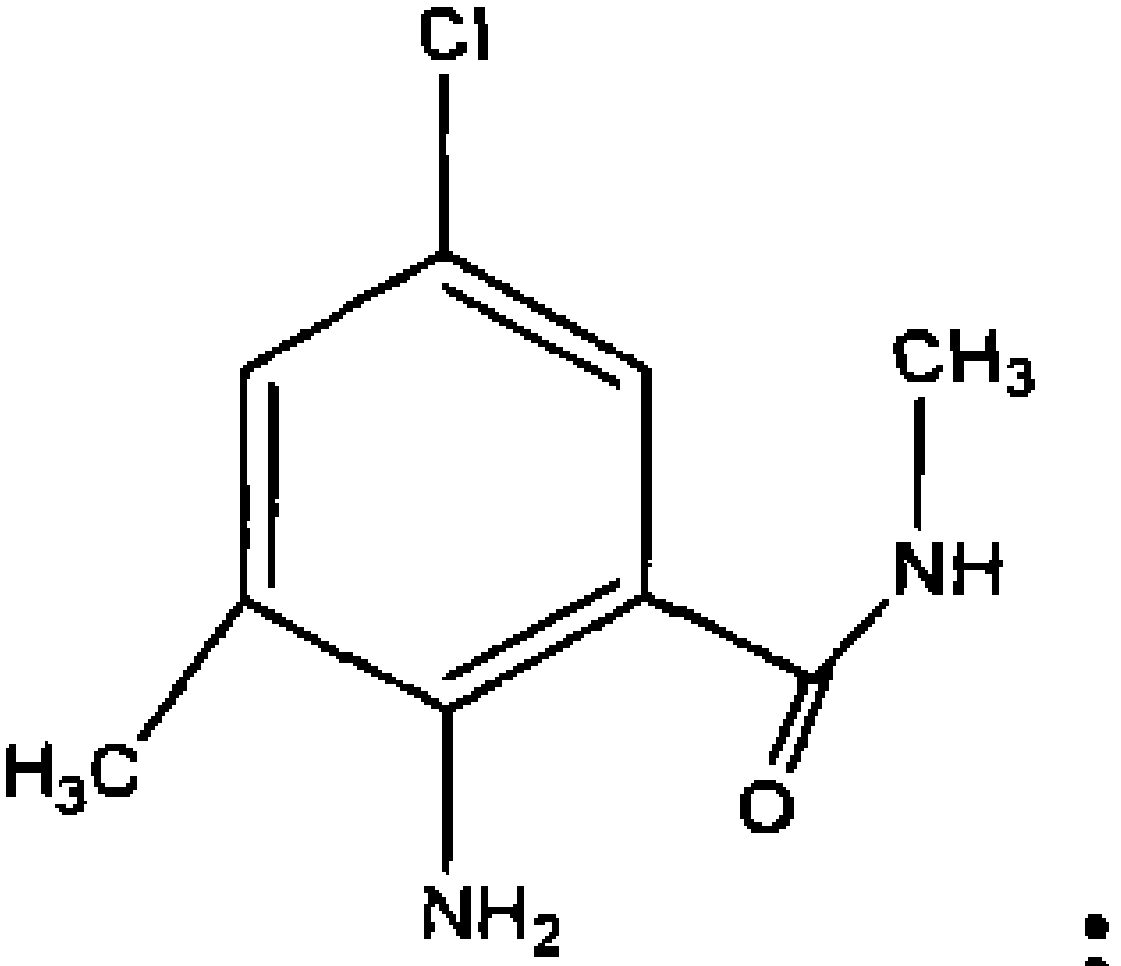
1) 3-methyl-2-amino-5-chloro benzoic ether, i.e. the preparation of general formula compound IV, as Fig. 1:

5 chlorinations of 3-methyl-2-Methyl anthranilate, be starting raw material promptly with 3-methyl-2-Methyl anthranilate, hypochlorous sodium solution and Glacial acetic acid are chlorination reagent, with organic solvent and water jointly as solvent, it is lower than in temperature to mix under-5 °C the condition carries out chlorination, reaction half an hour, separate organic phase, make 3-methyl-2-amino-5-chloro benzoic ether after the drying, general formula is



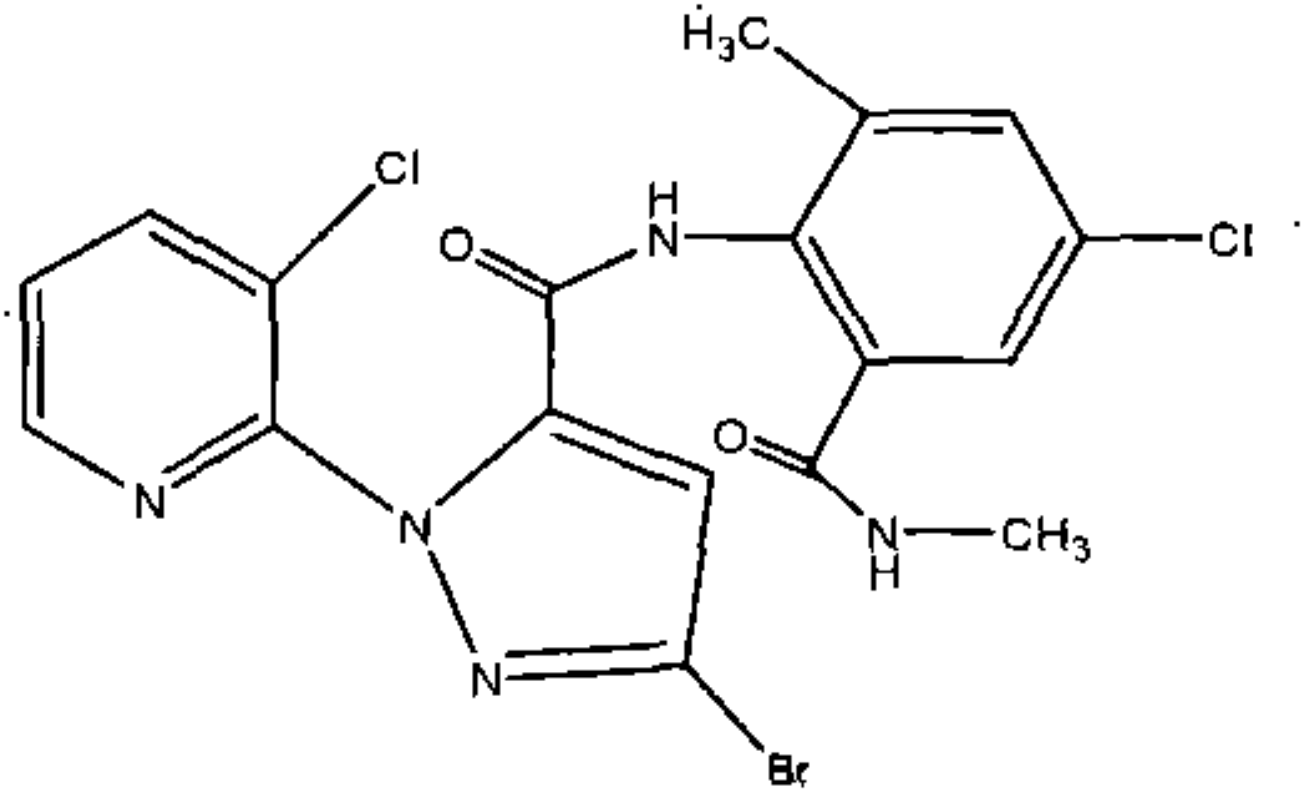
2) preparation of general formula compound II, as Fig. 2:

The amidation of ester group compound IV is to carry out under catalyzer, 3-methyl-2-amino-5-chloro benzoic ether, methylamine solution and catalyst A add in the flask, be under 50 100 °C the condition in temperature, reacted 6 hours, and reduced to and separate solvent and catalyzer behind the normal temperature and pressure, the crystal that obtains is dissolved with methylene dichloride, add activated carbon then, stirred 1 hour under 70 80 °C of conditions, can make product compound II after suction filtration is spin-dried for, general formula is



3) preparation of Rynaxypyr Compound I: the general formula of compound III is:

II and III are in organic solvent, and temperature is that reaction made target compound I in 6-10 hour under 100 °C of-180 °C of conditions, and general formula is



The described chlorine bleach liquor's of step 1) weight percent concentration is 10%, and 3-methyl-2-Methyl anthranilate and chlorine bleach liquor's weight ratio is 1: 10 15, and the weight ratio of chlorine bleach liquor and Glacial acetic acid is 10: 1.

The amount ratio of the described 3-methyl of step 1)-2-Methyl anthranilate and solvent is 40g 60g/L.

The described organic solvent of step 1) is methylene dichloride or trichloromethane.

The volumetric ratio of described organic solvent of step 1) and water is 1.5: 1.

Step 2) described methylethylolamine solution weight percentage concentration is 25-30%, and the weight ratio of ester group compound IV and methylethylolamine solution is 2-10: 1.

The organic solvent of step 3) is selected from toluene, dimethylbenzene, trimethylbenzene, inclined to one side trichlorobenzene, one or more among dioxane or the DMF.

Advantage of the present invention is: the preparation method is simple and practical, the efficient height, and each goes on foot the yield of product more than 85%; The reagent toxicity that adopts in the building-up process is low, solvent can be recycled and side reaction is few, thus little to equipment corrosion, environmental pollution is little; Because cost of material is cheap, production cost is reduced, therefore good economy performance has a good application prospect.

Description of drawings

Fig. 1 is the reaction formula that generates general formula compound IV.

Fig. 2 is the reaction formula that generates general formula compound II.

Fig. 3 is the reaction formula that generates general formula compound I.

Embodiment:

Embodiment 1: the preparation of Compound I

1) 3-methyl-2-amino-5-chloro benzoic ether

In temperature is-15 0 °C container, add methylene dichloride 150mL, 3-methyl-2-Methyl anthranilate 10g, Glacial acetic acid 10g and water 100ml, the amount ratio that is 3-methyl-2-Methyl anthranilate and solvent is 40g/L, the volumetric ratio of organic solvent and water is 1.5: 1, reduce to-10 -8 °C, the weight percent concentration that adds 100g is 10% chlorine bleach liquor, the weight ratio that is methyl o-aminobenzoate and chlorine bleach liquor is 1: 10, the weight ratio of chlorine bleach liquor and Glacial acetic acid is 10: 1, maintain the temperature at-8 °C, react half an hour, separate organic phase, make the white crystals of 2-amino-5-chloro benzoic ether after the drying; Yield is 98%.Through magnetic resonance detection is the purpose product, ^{1}HNMR (300M, CDCl $_3$): δ (ppm) 2.15 (s, 3), 3.80 (s, 3), 5.50 (s, 2H), 7.18 (s, 1H), 7.74 (s, 1H).

2) ammonia of 3-methyl-2-amino-5-chloro benzoic ether is separated

The 3-methyl that makes-2-amino-5-chloro benzoic ether and methylethylolamine solution add in the flask, add 3-methyl-2-amino-5-chloro benzoic ether 10g, weight percent concentration is the methylethylolamine solution 20g of 25-30%, the weight ratio that is 3-methyl-2-amino-5-chloro benzoic ether and methylethylolamine solution is 1: 2, catalyst A (0.05g), be that catalyst consumption is 0.5% of 3-methyl-2-amino-5-chloro benzoic ether weight, be under 50 60 °C the condition in temperature, reacted 12 hours, separate solvent after reducing to normal pressure, the crystal that obtains is dissolved with methylene dichloride, add activated carbon then, stirred 1 hour under 70 80 °C of conditions, can make product general formula compound II behind the suction filtration, productive rate is 95%.Through magnetic resonance detection is the purpose product, ^{1}HNMR (300M, CDCl $_3$): δ (ppm) 2.14 (s, 3), 2.96 (s, 3), 5.53 (s, 2H), 6.03 (s, 1H), 7.09 (s, 1H), 7.16 (s, 1H).

3) Compound I is synthetic

Assemble dropping funnel on the 250ml there-necked flask, mechanical stirrer and steam sleeve divide fluidization tower (column length 15-20cm), and capital is a still head.Still head is equipped with thermometer and downward-sloping condenser.In flask, add 31.35 (0.11mol) 1-(3-chloropyridine-2-yl)-3-chloro-1H-pyrazoles-5-carboxylic acid, ethyl ester (III) and 50ml anhydrous dimethyl benzene, flask is put be heated to 145-150 °C in the oil bath.Start stirring, in 30min, drip the solution that 18.45g (0.1mol) 3-methyl-2-amino-5-chlorobenzoyl methylamine and 50ml dimethylbenzene are formed, about Dropwise 5 min, head temperature promptly reaches 75-78 °C, and ethanol begins to steam.Have 6-7ml distillate (ethanol) to be collected in the 10ml graduated cylinder in 1h approximately, the temperature of still head reduces, and the expression reaction is finished.

Solution in the flask is inclined to the 250ml beaker, and scrub flask with 10ml dimethylbenzene.Subtract and steam 60ml dimethylbenzene and be placed on room temperature, slowly separate out solid, filter and obtain pale brown look solid, yield is 86%, is purpose product Compound I through magnetic resonance detection, ^{1}HNMR (300M, CDCl $_3$): δ (ppm) 2.15 (s, 3H), 2.93 (s, 3H), 6.24 (s, 1H), 7.07 (s, 1H), 7.18 (d, 2H), 7.36 (dd, 1H), 7.84 (dd, 1H), 8.44 (dd, 1H), 10.09 (s, 1H).

Embodiment 2: the preparation of Compound I

1) 3-methyl-2-amino-5-chloro benzoic ether

In temperature is-15 0 °C container, add trichloromethane 150mL, 3-methyl-2-Methyl anthranilate 10g, Glacial acetic acid 10g and water 100ml, the amount ratio that is 3-methyl-2-Methyl anthranilate and solvent is 40g/L, the volumetric ratio of organic solvent and water is 1.5: 1, reduce to-10 -8 °C, the weight percent concentration that adds 100g is 10% chlorine bleach liquor, the weight ratio that is methyl o-aminobenzoate and chlorine bleach liquor is 1: 10, the weight ratio of chlorine bleach liquor and Glacial acetic acid is 10: 1, maintain the temperature at-8 °C, react half an hour, separate organic phase, make the white crystals of 2-amino-5-chloro benzoic ether after the drying; Yield is 95%.Magnetic resonance detection is the purpose product, ^{1}H NMR (300M, CDCl $_3$): δ (ppm) 2.15 (s, 3), 3.80 (s, 3), 5.50 (s, 2H), 7.18 (s, 1H), 7.74 (s, 1H).

2) ammonia of 3-methyl-2-amino-5-chloro benzoic ether is separated

The 3-methyl that makes-2-amino-5-chloro benzoic ether and aqueous methylamine solution add in the flask, add 3-methyl-2-amino-5-chloro benzoic ether 10g, weight percent concentration is the methylethylolamine solution 100g of 25-30%, the weight ratio that is 3-methyl-2-amino-5-chloro benzoic ether and aqueous methylamine solution is 1: 10, catalyst A (0.1g), be that catalyst consumption is 1% of 3-methyl-2-amino-5-chloro benzoic ether weight, be under 80 100 °C the condition in temperature, reacted 5 hours, separate aqueous solvent after reducing to normal pressure, the crystal that obtains is dissolved with methylene dichloride, add activated carbon then, stirred 1 hour under 70 80 °C of conditions, can make product behind the suction filtration, productive rate is 98%.Through magnetic resonance detection is the purpose product, ^{1}HNMR (300M, CDCl $_3$): δ (ppm) 2.14 (s, 3), 2.96 (s, 3), 5.53 (s, 2H), 6.03 (s, 1H), 7.09 (s, 1H), 7.16 (s, 1H).

3) preparation of Rynaxypyr Compound I:

Assemble dropping funnel on the 250ml there-necked flask, mechanical stirrer and steam sleeve divide fluidization tower (column length 15-20cm), and capital is a still head.Still head is equipped with thermometer and downward-sloping condenser.In flask, add 31.35 (0.11mol) 1-(3-chloropyridine-2-yl)-3-chloro-1H-pyrazoles-5-carboxylic acid, ethyl ester (III) and 50ml anhydrous dimethyl benzene, flask is put be heated to 145-150 °C in the oil bath.Start stirring, in 30min, drip the solution that 18.45g (0.1mol) 3-methyl-2-amino-5-chloro-benzoyl methylamine and 50ml dimethylbenzene are formed, about Dropwise 5 min, head temperature promptly reaches 75-78 °C, and ethanol begins to steam.Have 6-7ml distillate (ethanol) to be collected in the 10ml graduated cylinder in 1h approximately, the temperature of still head reduces, and the expression reaction is finished.

Solution in the flask is inclined to the 250ml beaker, and scrub flask with 10ml dimethylbenzene.Subtract and steam 60ml dimethylbenzene and be placed on room temperature, slowly separate out solid, filter and obtain pale brown look solid, yield is 86%.Through magnetic resonance detection is purpose product Compound I,

^{1}HNMR (300M, CDCl $_3$): δ (ppm) 2.15 (s, 3H), 2.93 (s, 3H), 6.24 (s, 1H), 7.07 (s, 1H), 7.18 (d, 2H), 7.36 (dd, 1H), 7.84 (dd, 1H), 8.44 (dd, 1H), 10.09 (s, 1H).

Patent Citations (4)

Publication number	Priority date	Publication date	Assignee	Title
CN101298451A *	2007-04-30	2008-11-05		Benzamide compounds and use thereof
CN101333213A *	2008-07-07	2008-12-31		1-substituted pyridyl-pyrazol acid amide compounds and use thereof
CN101337959A *	2008-08-12	2009-01-07		2-amino-N-oxybenzamidecompounds with insecticidal activity
CN101492387A *	2009-03-09	2009-07-29		Preparation for 2-amino-5-chlorine-N,3-dimethyl benzamide
Family To Family Citations				

* Cited by examiner, † Cited by third party

Cited By (3)

Publication number	Priority date	Publication date	Assignee	Title
JP2014511367A *	2011-01-28	2014-05-15	イー・アイ・デュポン・ドウ・ヌムール・アンド・カンパニー	Method for preparing 2-aminobenzamide derivatives
WO2017219768A1 *	2016-06-21	2017-12-28		Polymorph of chlorantraniliprole and preparation method therefor
CN115181031A *	2021-04-02	2022-10-14		Preparation method of 2-amino-5-chlorobenzoic acid derivative
Family To Family Citations				

* Cited by examiner, † Cited by third party, ‡ Family to family citation

Similar Documents

Publication	Publication Date	Title
CN101830853B	2012-08-29	Nitroimidazoline derivatives and preparation method thereof and application thereof
CN101402622B	2012-09-19	Synthesis and Application of 4,6,4'-Trihydroxy-3',5'-Dimethoxy-Arangone and Its Derivatives
CN110330455A	2019-10-15	Amide derivatives and its preparation method and application
CN101717395A	2010-06-02	Synthesis method of chlorantraniliprole pesticide
CN102351800A	2012-02-15	Method for preparing 5-methylbenzimidazole-2-methyl carbamate
CN101367746B	2011-05-11	Novel method for synthesizing -metolachlor
CN104292166B	2016-04-27	The chloro-2-cyano group of 4--N, N-dimethyl-5-p-methylphenyl imidazoles-1-sulphonamide synthetic method
CN105198801A	2015-12-30	N-(4-chloro-3-picolyl)-2-(3-methylbenzamide) benzamide as well as preparation method and application thereof
Huang et al.	2017	Design, synthesis and biological evaluation of 1H-pyrazole-5-carboxamide derivatives as potential fungicidal and insecticidal agents
CN112920079A	2021-06-08	Preparation method of amide compound
CN109265381B	2020-10-27	Cyano-containing phthalic diamide derivative and preparation and application thereof
CN117327016A	2024-01-02	Preparation method of fenpyrad intermediate
CN109452280A	2019-03-12	A kind of process using carbon dioxide production insecticide
CN105906591A	2016-08-31	Synthesis method of 2-amino-gamma-butyrolactone hydrochloride
CN103319343A	2013-09-25	Pyrethroid compound, and preparation method and applications thereof
CN103819327A	2014-05-28	Method for synthesizing 3,6-dichloro-2-methoxy benzoic acid
CN105646359A	2016-06-08	Arylpyrazol sulfonyl aminated derivative as well as ultrasonic radiation synthesis method and application of arylpyrazol sulfonyl amidated derivative
CN102320998B	2014-08-20	Phthalic diamide compounds containing 2'-hydroxy hexafluoro isopropyl group and application thereof
CN112410808A	2021-02-26	Synthesis method of anthranilate pesticide containing N-pyridylpyrazole
CN104926702A	2015-09-23	Preparation method for 2-methylmercapto-4-thrifluoromethyl benzoate
CN106831488A	2017-06-13	A kind of 5- (3,4- di-substituted-phenyls)-hydroresorcinol class compound and its application

CN103755680A	2014-04-30	Thiocyclam synthesis method
CN102557917A	2012-07-11	Method for preparing 2,4,6-trimethylphenylacetic acid
CN101367721B	2011-07-20	Method for preparing benzene acetic acid with phenylacetonitrile hydrolyzation in ammonia-containing high temperature liquid water medium
CN105001211B	2017-09-26	A kind of sulphone amide derivative and its application in agricultural

Priority And Related Applications

Priority Applications (1)

Application	Priority date	Filing date	Title
CN200910228966A	2009-12-04	2009-12-04	Synthesis method of chlorantraniliprole pesticide

Applications Claiming Priority (1)

Application	Filing date	Title
CN200910228966A	2009-12-04	Synthesis method of chlorantraniliprole pesticide

Legal Events

Date	Code	Title	Description
2010-06-02	C06	Publication	
2010-06-02	PB01	Publication	
2010-07-21	C10	Entry into substantive examination	
2010-07-21	SE01	Entry into force of request for substantive examination	
2014-05-07	C12	Rejection of a patent application after its publication	
2014-05-07	RJ01	Rejection of invention patent application after publication	**Application publication date**: 20100602

Concepts

machine-extracted

Download Filter table

Name	Image	Sections	Count	Query match
pesticide		title,claims,abstract,description	18	0.000
Chlorantraniliprole		title,claims,abstract,description	17	0.000
chlorantraniliprole		title,claims,abstract,description	17	0.000
synthesis method		title	1	0.000
organic solvent		claims,abstract,description	13	0.000
preparation method		claims,abstract,description	11	0.000
solvent		claims,abstract,description	11	0.000
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chemical reaction reagent		claims,abstract,description	4	0.000
biosynthetic process		claims,abstract,description	3	0.000
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method	abstract,description	4	0.000
side reaction	abstract,description	4	0.000
corrosion	abstract,description	2	0.000
corrosion	abstract,description	2	0.000
insecticide	abstract,description	2	0.000
synthesizing effect	abstract	2	0.000
low toxicity	abstract	1	0.000

Show all concepts from the description section