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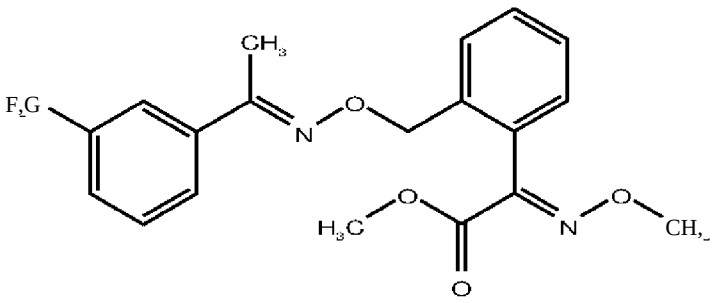
(54) Title: A NOVEL PROCESS FOR THE PREPARATION OF TRIFLOXYSTROBIN



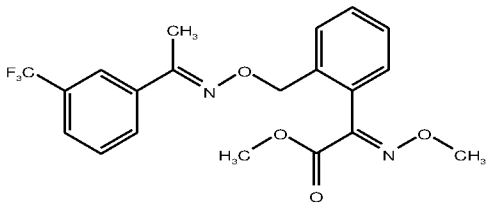
Formula (I)

(57) Abstract: The present invention relates to a novel process for preparation of methyl (αE)-(α- (methoxyimino)-2-[[[(E)-[1-[3-(trifluoromethyl) phenyl] ethylidene] amino] oxy] methyl] benzeneaceate (Trifloxystrobin) compound of formula (I) in free form or in agro chemically acceptable salt form useful as a pest control agent.The present invention also relates to a novel process for preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate of formula (X) intermediate of Trifloxystrobin. [Formula should be inserted here].

Title of the invention - **A NOVEL PROCESS FOR THE PREPARATION OF TRIFLOXYSTROBIN**

FIELD OF THE INVENTION

The present invention relates to a novel process for preparation of methyl (αE)-(α-(methoxyimino)-2-[[[(E)-[l-[3- (trifluoromethyl) phenyl] ethylidene] amino] oxy]methyl] benzeneaceate (Trifloxystrobin) of formula (I) in free form or in agro chemically acceptable salt form useful as a pest control agent.

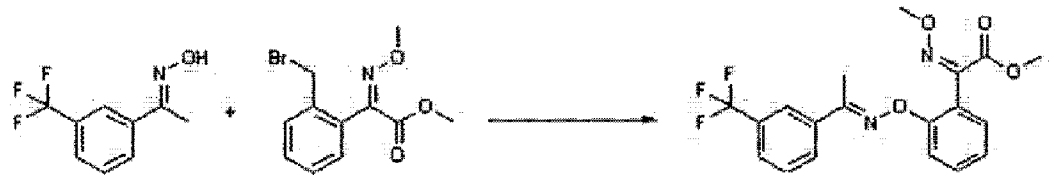


Formula (I)

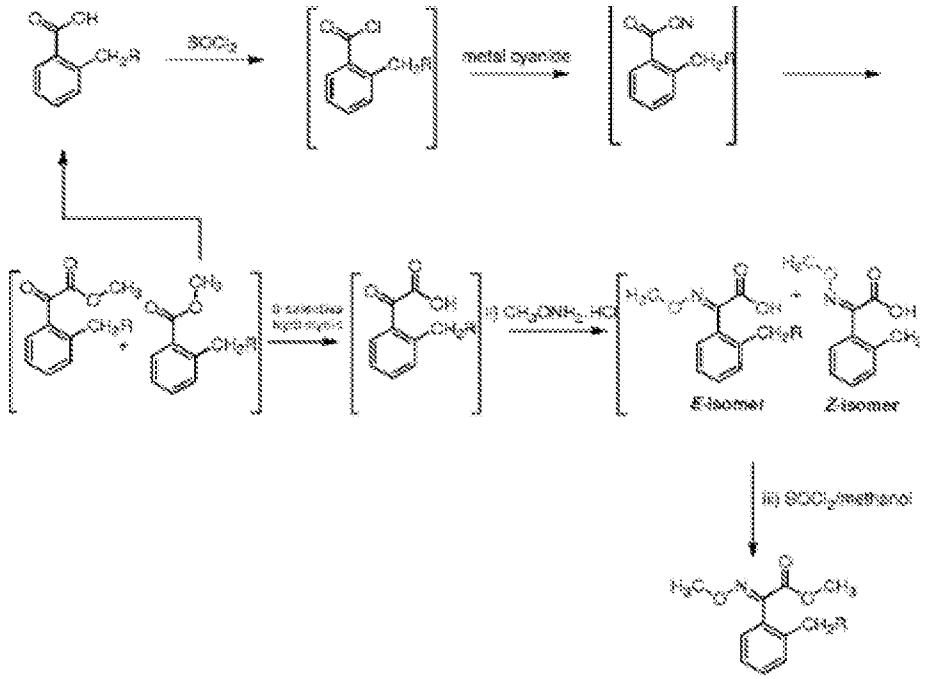
BACKGROUND OF THE INVENTION

Methyl (aE)-(a-(methoxyimino)-2-[[[(E)-[l-[3- (trifluoromethyl) phenyl] ethylidene] amino]oxy] methyl] benzeneaceatebelongs to the strobilurin class of fungicides. Trifloxystrobin and its process for preparation were first disclosed in US5238956.It is most commonly used on bananas, cereals, citrus, coffee, corn, cotton, field beans, grapes, hops, nuts, ornamentals, peanuts, pome fruits, potatoes, rice, small fruits, soybeans, stone fruits, sugar beets, sunflowers, tea, tropical fruits, turf, vegetables and various other crops.

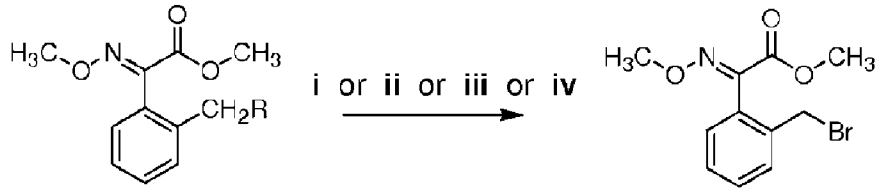
CN103787916Ateaches process for the preparation of Trifloxystrobin. A reaction process of the synthesis method includes water diversion treatment. By virtue of a water carrying process, the influence of water formed in the reaction on the reaction is effectively avoided, thereby reducing side reaction, and improving product yield and product quality. According to the synthesis method, a reaction liquid solvent is directly used for crystallization after the reaction so as to avoid change of crystallization solvent and to avoid cross infection of the solvents, thus further enhancing product quality.



WO2013144924teaches process for the synthesis of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate. It further relates to the conversion of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate, subsequently to substantially pure Trifloxystrobin, compound of formula (I). Synthesis for preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoateas shown in scheme as follows:



WO2013144924 also teaches preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate wherein R is H can be represented as shown in scheme as follows:



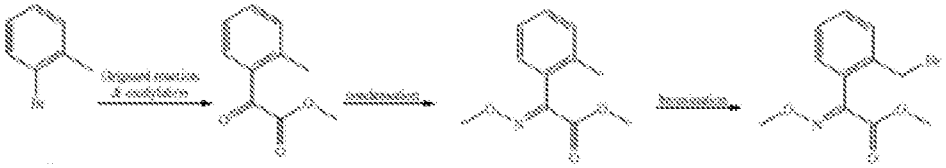
i = NBS / AIBN or benzoyl peroxide

ii = NaBrOs / NaHSOs, UV or ordinary light

iii= Br / H2O2, UV or ordinary light

iv = alkali metal bromide / H2SO4 / H2O2, UV or ordinary light

Rambaud, M. et al., *Synthesis*, 564(1988); and Korean Patent No. 88,673 PCT/KR 95/00020), teaches process for the synthesis ofmethyl (2E)-[2-(bromo methyl) phenyl] (methoxyimino) ethanoateas shown in reaction scheme:



The prior art processes for the preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate suffer from one or other drawback like low yield, high cycle time, by-product formation and formation of a mixture of Z and E isomers. Additionally, recovery of compound of formula (I) is not possible. This leads to an increase in the effluent treatment load, feasibility and high overall cost.

The processes for the preparation of methyl (2E)-[2-(bromo methyl) phenyl] (methoxyimino) ethanoate disclosed in the prior art do not yield E-isomer in good yield and high purity.In a subsequent step, methyl (2E)-[2-(bro momethyl) phenyl] (methoxyimino) ethanoate is converted to Trifloxystrobin.

The prior art processes have some difficulties that may affect overall process economics. Thus WO20 13 144924 process incorporates hydrolysis of acyl cyanide that results a formation of 2-methyl benzoic acid formed due to decarboxylation & resulting formation of about 40 to 50% of acyl cyanide byproduct,2-Methyl Benzoic Acid .

In the light of above disadvantages there is a need to develop an improved process for the preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate in good yield and high purity. Methyl (2E)-[2-(bromo methyl) phenyl] (methoxyimino) ethanoateis further used in the preparation of substantially pure Trifloxystrobin in good yield. There is a need to develop a commercially viable process which has the capacity to produce products in good yield, high purity, and which also has the provision to recover useful starting material and reagents from the by-product formed during the course of the reaction.

The present invention is directed to the above drawbacks, the purpose is to provide novel process for the preparation of Trifloxystrobin of formula (I) which is a mild reaction conditions, simple operation, less expensive cost effective process.

OBJECTIVES OF THE INVENTION

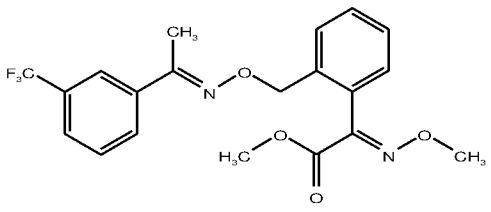
One of the objectives of the present invention is to provide a novel process for the preparation of methyl(aE)-(a-(methoxyimino)-2-[[[(E)-[l-[3(trifluoromethyl) phenyl] ethylidene] amino] oxy] methyl] benzeneacetateof formula (I) in free form or in agro chemically acceptable salt form starting from readily accessible and cheap intermediates which are easy to handle, which process makes it possible to prepare Trifloxystrobin of formula (I) in simple manner and in good yield.

Still another objective of the present invention is to provide novel process for the preparation of an intermediate, (2-Bromomethyl-phenyl)-methoxyimino-acetic acid methyl esterof formula (X), which is further used in the synthesis of final methyl(aE)-(a-(methoxyimino)-2-[[[(E)-[l-[3(trifluoromethyl) phenyl] ethylidene] amino] oxy] methyl] benzeneaceate (Trifloxystrobin) of formula (I) in free form or in agro chemically acceptable salt.

Still another objective of the present invention is to provide novel process for the preparation of an intermediate, oxy-o-tolyl-acetic acid of formula (VI)

DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a novel process for the preparation of methyl(aE)-(a-(methoxyimino)-2-[[[(E)-[l-[3(trifluoromethyl) phenyl] ethylidene] amino] oxy]methyl] benzeneaceate (Trifloxystrobin) of formula (I).

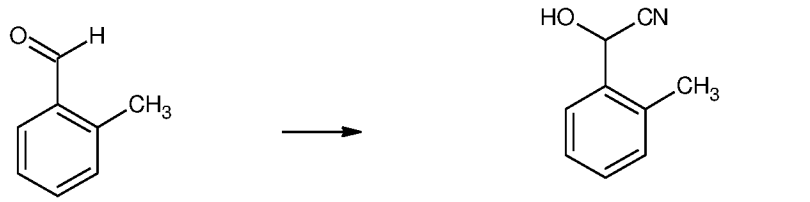


Formula (I)

Thus, the invention provides a simple, economically viable and efficient process for preparing methyl (aE)-(a-(methoxyimino)-2-[[[(E)-[l-[3(trifluoromethyl) phenyl] ethylidene] amino] oxy] methyl] benzeneaceate(Trifloxystrobin) of formula (I)

The invention will now be described in detail in connection with certain preferred and optional embodiments, so that various aspects thereof may be more fully understood and appreciated and briefly described as follows.

In one embodiment the present invention provides a novel process for the preparation of hydroxy-(2-methylphenyl)-acetonitrileof formula (III) by reacting 2-methylbenzaldehyde of formula (II) with a cyanation agent that is selected from but not limited to Sodium Cyanide or Potassium Cyanide in presence of acid or Sodium metabisulfite and solvent.



2-methylbenzaldehyde

Formula (II)

hydroxy(2-methylphenyl)acetonitrile

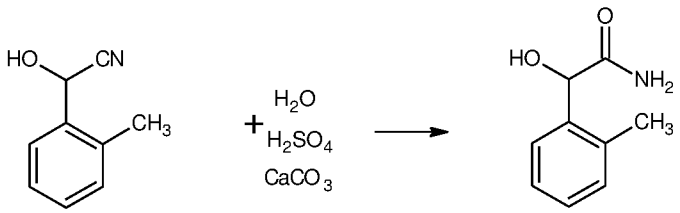
Formula (III)

The process for the preparation of hydroxy (2-methylphenyl) acetonitrileof formula (III) by cyanation was carried with Hydrochloride salt of 2-methylbenzaldehyde of formula (II) orbisulfite adduct of 2-methylbenzaldehyde of formula (II) with Sodium metabisulfite Or bisulfite adduct of 2-methylbenzaldehyde of formula (II) with sulphuric acid.

Suitable solvent is selected from but not limited to water, alcohols, ketones, diols, triols, esters, amides, ethers, hydrocarbons, polar aprotic solvents, polar solvents, chloro solvents, nitriles or mixtures thereof. Polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; alcohols such as methanol, ethanol, propanol, butanol;chloro solvents like methylene chloride,ethylene chloride, chloroform, monochlorobenzene, ; hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane, more preferably toluene.

In another embodiment the present invention provides a novel process for the preparation of 2-hydroxy-2-(2-methylphenyl) acetamide of formula (IV) by hydrolyzing hydroxy (2-methylphenyl) acetonitrile of formula (III) in presence of acids. The aqueous solution of sulphuric acid was added drop wise and with stirring at the temperature of about 50°-25°C for hydrolyzing hydroxy (2-methylphenyl) acetonitrile of formula (III). The resultant crude amide of formula (II) was neutralized by the addition of sodium carbonate with stirring.

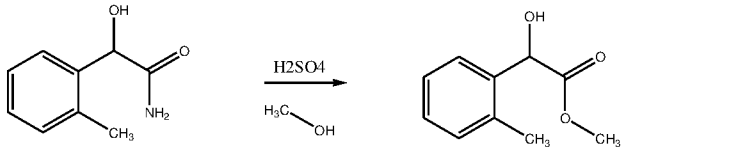
The sodium carbonate is reacted with sulfuric acid in the filtrate to obtain sodium sulphate.



hydroxy(2-methylphenyl)acetonitrile
Formula (III)

2-hydroxy-2-(2-methylphenyl)acetamide
Formula (IV)

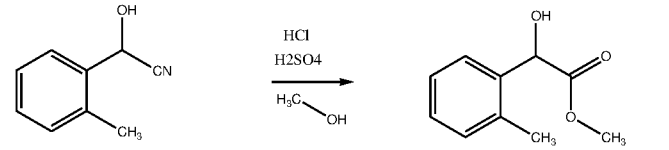
In another embodiment the present invention provides a novel process for the preparation of Hydroxy-o-tolyl-acetic acid methyl ester of formula (VI) by reacting 2-Hydroxy-2-o-tolyl-acetamide formula (IV) with methanol in presence of acids and solvent.



2-Hydroxy-2-0-tolyl-acetamide
Formula (IV)

Hydroxy-O-tolyl-acetic acid methyl ester
Formula (VI)

In another embodiment the present invention provides a novel process for the preparation of Hydroxy-o-tolyl-acetic acid methyl ester of formula (VI) by reacting hydroxy (2-methylphenyl) acetonitrile of formula (III) with methanol in presence of acid and solvent, wherein the acid is selected form hydrochloride acid and inorganic or organic acids or mixture thereof.



Hydroxy-O-tolyl-acetonitrile
Formula (III)

Hydroxy-O-tolyl-acetic acid methyl ester
Formula (VI)

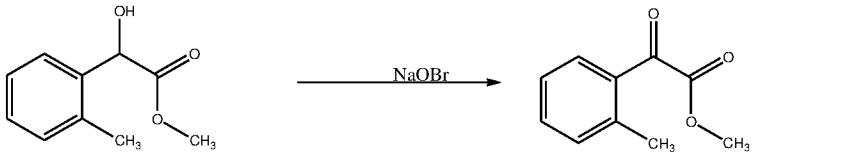
Inorganic or organic acids are added into solution of Hydrochloric acid to generate hydrochloric acid gas. The hydrochloric acid gas passes through sulphuric acid trap into cyanohydrins reaction mass. The purging of hydrochloric gas into reaction mass was carried out at about -15°C to 0° C.Hydrochloric gas and methanol are continually purged in reaction mass at -15 to 10°c for 2 to 10 hours.

Suitable solvent is selected from but not limited to water, alcohols, ketones, diols, triols, esters, amides, ethers, hydrocarbons, polar aprotic solvents, polar solvents, chloro solvents, nitriles or mixtures thereof. Hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane; Polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; Alcohols such as

methanol, ethanol, propanol, butanol; Chloro solvents like methylene chloride, chloroform, monochlorobenzene, and ethylene chloride.

Suitable inorganic or organic acids is selected from but not limited. Inorganic acids are mineral acids such as hydrohalic acids, such as hydrobromic and hydrochloric acids, sulfuric acids, phosphoric acids and nitric acids. Organic acids are aliphatic, alicyclic and aromatic carboxylic acids, dicarboxylic acids, tricarboxylic acids. Examples of such acids are carbonic acid, formic acid, fumaric acid, acetic acid, propionic acid, isopropionic acid, valeric acid, alpha.-hydroxy acids, such as glycolic acid and lactic acid, chloroacetic acid, benzoic acid, methane sulfonic acid, and salicylic acid. Examples of dicarboxylic acids include oxalic acid, malic acid, succinic acid, tataric acid and maleic acid. An example of a tricarboxylic acid is citric acid.

In another embodiment the present invention provides a novel process for the preparation of Hydroxy-o-tolyl-acetic acid methyl ester of formula (VII) by oxidation of Oxo-o-tolyl-acetic acid methyl ester of formula (VI) in presence of oxidizing agent and a catalytic amount of a weak base such as 2,2,6,6-tetramethylpiperidine 1-oxyl, hereinafter abbreviated as TEMPO.



Hydroxy-O-tolyl-acetic acid methyl ester
Formula (VI)

Oxo-O-tolyl-acetic acid methyl ester
Formula (VII)

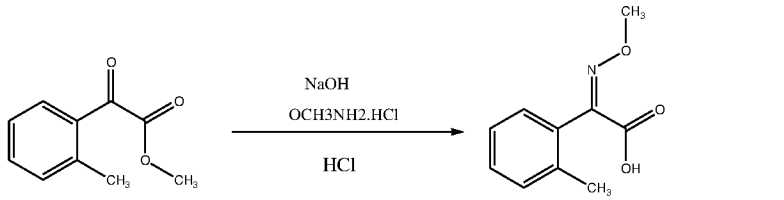
The reaction of Oxo-o-tolyl-acetic acid methyl ester of formula (VI) expressed by the above reaction with oxidizing agent to obtain Hydroxy-o-tolyl-acetic acid methyl ester of formula (VII) is performed in the range of -10°C to 10°C, preferably in the range of -5°C to 5°C. Oxidizing agent is used in the range of 1.0 to 2.0 molar equivalents to Oxo-o-tolyl-acetic acid methyl ester of formula (VI). It is preferred to use halogenated hydrocarbon as a solvent such as dichloromethane, chloroform, carbon tetrachloride and 1, 2-dichloroethane.

Oxidizing agent is selected from Sodium hypobromite (NaOBr), Potasium Hypobromite (KOBr), N-bromosuccinimide, bromine isocyanuric acid, peracids, 2KHSOs, KHSO4, K2SO4, H2O2, and mixtures thereof.

Sodium hypobromite and potassium hypobromite can be generated easily by reacting 2 equivalents of sodium hydroxide or potassium hydroxide with 1 equivalent of bromine in aqueous solution in the range of -5 ~ 0°C *[Org. Syn. Coll., 5, 8 (1973)]*.

After oxidation, unreacted oxidizing agent such as sodium hypobromite can be decomposed easily by treatment with salts of thiosulfate (sodium thiosulfate 100ml of water and 124g of Na2S203- 5H20). If necessary, high purity of the desired product can be obtained by conventional methods.

In another embodiment the present invention provides a novel process for the preparation of Methoxyimino-o-tolyl-acetic acid of formula (VIII) by reacting Oxo-o-tolyl-acetic acid methyl ester of formula (VII) with Methoxyamine hydrochloride in presence of a base in an solvent optionally in the presence of phase transfer catalyst such as, TBAB, TEBAC, and base used is selected from but not limited to sodium hydroxide, potassium carbonate, sodium carbonate, triethylamine, diethylamine or mixtures thereof.

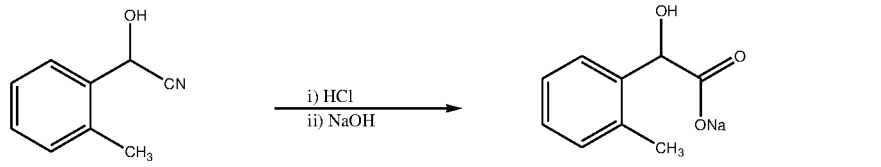


Oxo-O-tolyl-acetic acid methyl ester
Formula (VII)

Methoxyimino-O-tolyl-acetic acid
Formula(VIII)

Suitable solvent is selected from but not limited to water, alcohols, ketones, diols, triols, esters, amides, ethers, hydrocarbons, polar aprotic solvents, polar solvents, chloro solvents, nitriles or mixtures thereof. Polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; alcohols such as methanol, ethanol, propanol, butanol; chloro solvents like methylene chloride, chloroform, monochlorobenzene, , and and ethylene chloride; hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane, more preferably toluene.

In another embodiment the present invention provides a novel process for the preparation of Hydroxy-O-tolyl-acetic acid sodium salt of formula (Va) by reacting hydroxy (2-methylphenyl) acetonitrile of formula (III) in presence of inorganic or organic acids.The cone .Hydrochloric acid was added drop wise and with stirring at the temperature of about 25°C-50°C followed by carried out hydrolysis in presence of base.



Hydroxy-o-tolyl-acetonitrile

Formula-(III)

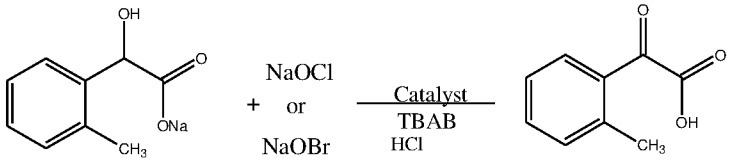
Hydroxy-o-tolyl-acetic acid sodium salt

Formula(Va)

Suitable solvent is selected from but not limited to water and chloro solvents like Dichloromethane, Dichloroethane, chloroform, monochlorobenzene, hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane, more preferably toluene.

In another embodiment the present invention provides a novel process for the preparation of Hydroxy-o-tolyl-acetic acid of formula (V)by oxidation of Hydroxy-O-tolyl-acetic acid sodium salt of the formula (Va) using sodium hypochlorite or Sodium Hypobromite in presence of metal oxide catalysts such as Platinum, Ruthenium, Rhodium, Molybdenum incatalytic amount and/or a weak base such as 2,2,6,6-tetramethylpiperidine 1- oxyl, hereinafter abbreviated as TEMPO or its derivatives such as 4-acetoxy-2,2,6,6-tetramethylpiperidinyl-l-oxyetcin presence of phase transfer catalysts selected from the group consisting of quaternary ammonium cations, quaternary phosphoniumcations, and cyclic polyethers such as tricaprylylmethylammonium chloride, methyl tributyl ammonium chloride, methyl tributyl ammonium fluoride, tetrabutyl ammonium bromide, tetrabutyl ammonium fluoride, tetrabutyl ammonium hydrogen sulfate, triethyl benzyl ammonium chloride, tetrabutylphosphonium bromide, tetrabutylphosnhonium chloride, tetraoctylphosphonium bromide, and mixtures thereof.

Obtained resultant sodium salt of oxo-toyl-acetic acid followed by treated with the acids such as Hydrochloric Acid or Sulphuric Acid in presence of solvents such as water and chloro solvents like Methylenedichloride, Ethylenedichloride, chloroform, monochlorobenzene, hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane, more preferably toluene to give the oxo-o-toyl-acetic acid.



Hydroxy-o-tolyl-acetic acid sodium salt

Formula(Va)

Oxo-o-tolyl-acetic acid

Formula(V)

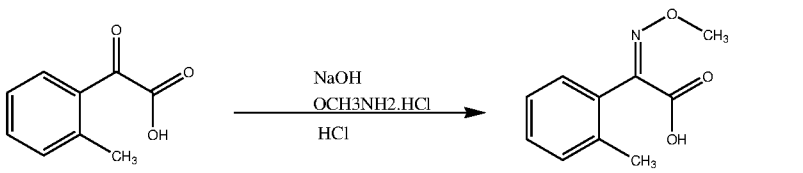
The reaction of Oxo-o-tolyl-acetic acid of formula (V)expressed by the above reaction prepared by with oxidizing agentis performed in the range of -10°Cto50°C, preferably in the range of -5°Cto5°C. Oxidizing agent is used in the range of 2 mole %tol0 mole % toHydroxy-o-tolyl-acetic acid sodium salt of formula (Va). It is preferred to use halogenated hydrocarbon as a solvent such as dichloro methane, chloroform, carbon tetrachloride and 1, 2-dichloroethane and more preferably dichloroethane.

Oxidizing agent is selected from Sodium hypobromite (NaOBr), Potasium Hypobromite (KOBr), N-bromosuccinimide (NBS), bromineisocyanuric acid, peracids, Benzoyl peroxide, free radical initiator AIBN, $2KHS0_5$, $KHS0_4$, K2SO4, H2O2, and mixtures thereof.

Sodium hypobromite and potassium hypobromite can be generated easily by reacting 2 equivalents of sodium hydroxide or potassium hydroxide with 1 equivalent of bromine in aqueous solution in the range of -5 ~ 0°C *[Org. Syn. Coll., 5, 8 (1973)]*.

After oxidation, unreacted oxidizing agent such as sodium hypobromite can be decomposed easily by treatment with salts of thiosulfate (sodium thiosulfate 100ml of water and 124g of Na2S203- 5H20). If necessary, high purity of the desired product can be obtained by conventional methods.

In another embodiment the present invention provides a novel process for the preparation of Methoxyimino-o-tolyl-acetic acid of formula (VIII) by reactingOxo-o-tolyl- acetic acid of formula (V) with Methoxyamine hydrochloride in presence of a base in an solvent optionally in the presence of phase transfer catalyst selected from the group consisting of quaternary ammonium cations, quaternary phosphoniumcations, and cyclic polyethers such as tricaprylylmethylammonium chloride, methyl tributyl ammonium chloride, methyl tributyl ammonium fluoride, tetrabutyl ammonium bromide, tetrabutyl ammonium fluoride, tetrabutyl ammonium hydrogen sulfate, triethyl benzyl ammonium chloride, tetrabutylphosphonium bromide, tetrabutylphosnhonium chloride, tetraoctylphosphonium bromide, and mixtures thereof. Base used is selected from but not limited to sodium hydroxide, potassium carbonate, sodium carbonate, triethylamine, diethylamine or mixtures thereof.

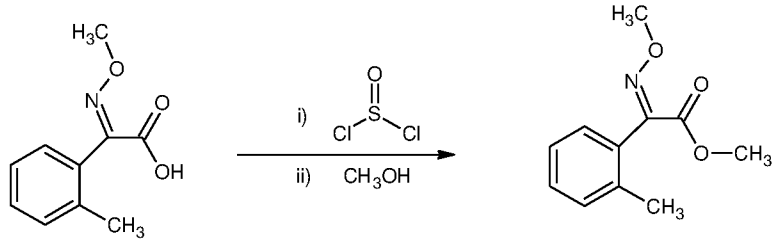


Oχo -σ-tolyl-acetic acid
Formula (V)

Methoxyimino-o-tolyl-acetic acid
Formula(VIII)

Suitable solvent is selected from but not limited to water, alcohols, ketones, diols, triols, esters, amides, ethers, hydrocarbons, polar aprotic solvents, polar solvents, chloro solvents, nitriles or mixtures thereof. Polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; alcohols such as methanol, ethanol, propanol, butanol;chloro solvents like methylene chloride, chloroform, monochlorobenzene, and ethylene chloride; hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane, more preferably toluene.

In another embodiment the present invention provides a novel process for the preparation of methyl (2E)-(methoxyimino) (2-methylphenyl) ethanoate of formula(IX) by methylation of (2E/Z)-(methoxyimino) (2-methylphenyl) Ethanoicacid of formula (VIII) in presence of thionyl chloride or triphosgene using methanol solvent.

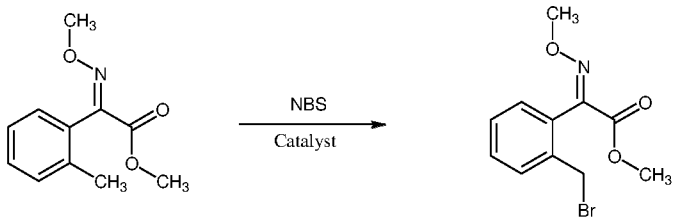


(2EZ)-(methoxyimino)(2 -msthylphenyl)ethanoic acid
Formula **(VIII)**

methyl(2E)-(methoxyimino)(2 -methylphenyl)ethanoate
Formula **(IX)**

The solvents used in step of the methylation are chloro solvents like methylene chloride, chloroform, monochlorobenzene, and ethylene chloride; hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane; polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; alcohols such as methanol, ethanol, propanol, butanol; or mixtures thereof.

In another embodiment of the present invention provides a novel process for the preparation of methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate of formula (X) by brominatingmethyl (2E)-(methoxyimino) (2-methylphenyl)Ethanoate of formula (IX) in presence of brominating agent and an organic solvent optionally in the presence of a phase transfer catalyst.



Methoxyimino-o-tolyl-acetic acid methyl ester (2-Bromomethyl-phenyl)-methoxyimino-acetic acid methyl ester

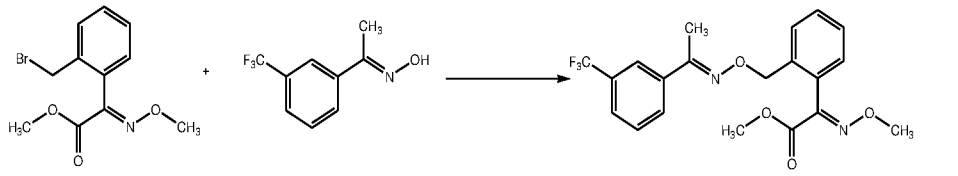
Formula (IX) Formula(X)

The solvents used in step of the bromination are polar aprotic solvents such as acetone, DMF, acetonitrile, DMSO, sulfolane; alcohols such as methanol, ethanol, propanol, butanol; chloro solvents like methylene chloride, chloroform, monochlorobenzene, and ethylene chloride; hydrocarbon solvents like toluene, xylene, heptane, cyclohexane and hexane or mixtures thereof. These solvents are liquid at room temperature and preferably have a boiling point of 35°C to 130°C, more preferably 50°C to 100°C and in particular of 75°C to 85°C.

Brominating agents are selected from but not limited to phenyltrimethyl ammoniumtribromide; dibromodimethylhydantoin (DBDMH); potassium bromide; Sodium bromide; Br_2; hydrobromic acid; N-bromophthalimide; N-bromoacetamide, N-bromosuccinimide (NBS) or mixtures thereof.

In the process of the present invention the bromination agent is added to a solution of a compound of Formula (IX) in a solvent. During the addition of the bromination agent the mixture is preferably kept at a temperature within the range of from room temperature to reflux temperature. After the addition of the bromination agent is completed the reaction mixture is preferably stirred for an additional time period of few minutes to 5 to 50 hours maintaining temperature between 20°C to 60°C. The crude product methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate of formula (X) is purified by recrystallizationin presences of ether class of organic solvent such as diethylether, di-isopropylether, methyl tert-butyl ether or mixture thereof.

In a preferred embodiment, the invention provides a novel process for the synthesis of Methoxyimino-[2-[l-(3-trifluoromethyl-phenyl)-ethylideneaminooxymethyl]-phenyl]-acetic acid methyl ester of formula (I), which comprises reacting (2E)-[2-(bromo methyl) phenyl] (methoxyimino) ethanoate of formula (X) with l-(3-Trifluoromethyl-phenyl)-ethanoneoxime of formula (XI) in presence solvent and optionally in presence of a base or phase transfer catalyst.



(2-Bromomethyl-phenyl)-methoxyimino -acetic acid methyl ester

Formula (X)

1-(3-Trifluoromethyl-phenyl) -ethanone oxime

Formula (XI)

(E-isomer)

Methoxyimino-{2-[l-(3-trifluoromethyl-phenyl)-ethylideneaminooxymethyl]-phenyl)-acetic acid methyl ester

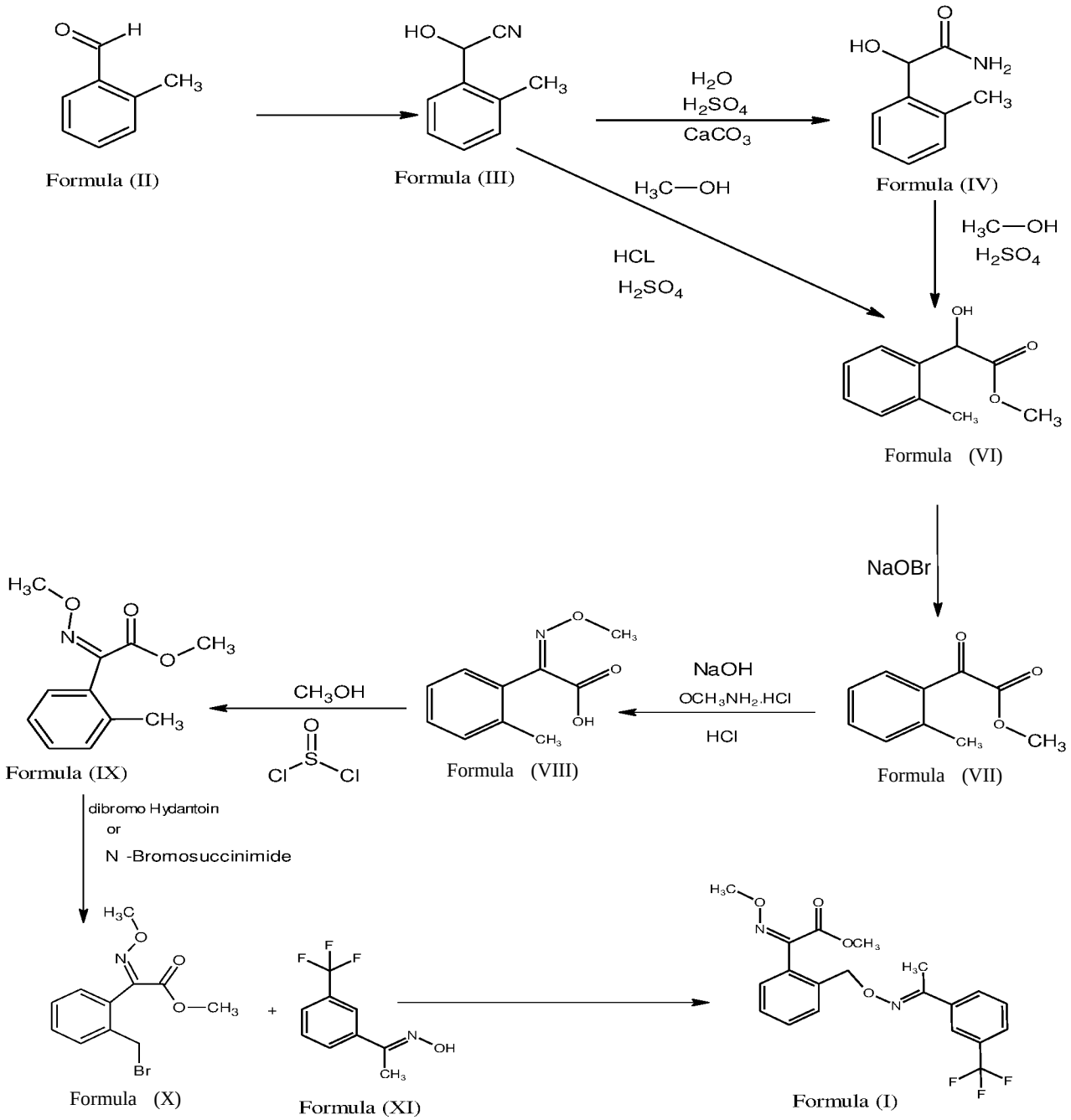
Formula (I)

In a preferred embodiment, base is selected from the group comprising like alkali or alkaline earth metal hydroxide, alkali or alkaline carbonate, alkali or alkaline bicarbonate, organolithium, organomagnesium compounds, organic base and the like or mixtures thereof. Alkali or alkaline earth metal hydroxide, alkali or alkaline carbonate, alkali or alkaline bicarbonate used herein above are selected from NaOH, KOH, LiOH, NaHC03, KHC03, LiHC03, Na2C03, K2C03, Li2C03, Mg(OH)2, Ca(OH)2, CaC03, MgC03, Ba(OH)2, Be(OH)2, BaC03, SrC03, (CH3)3COK and the like or mixtures thereof. Organolithium used herein above are selected fromn-BuLi, sec-BuLi, t-BuLi, MeLi, PhLi. Organomagnesium used herein above are selected from phenylmagnesiumbromide, ethylmagnesium bromide. Organic base used herein above are selected from nitrogen containing base such as pyridine, piperidine, dimethyl amino pyridine, picolines, diisopropyl ethyl amine, triethyl amine and the like or mixtures thereof.

Suitable solvent is selected from but not limited to water, methyl isobutyl ketone, acetone, DMF, acetonitrile, DMSO, sulfolane; methanol, ethanol, propanol, butanol;methylene chloride, chloroform, monochlorobenzene, and ethylene chloride; toluene, xylene, heptane, cyclohexane and hexane.

In a preferred embodiment, phase transfer catalyst is selected selected from the group consisting of quaternary ammonium cations, quaternary phosphoniumcations, and cyclic polyethers such as tricaprylylmethylammonium chloride, methyl tributyl ammonium chloride, methyl tributyl ammonium fluoride, tetrabutyl ammonium bromide, tetrabutyl ammonium fluoride, tetrabutyl ammonium hydrogen sulfate, triethyl benzyl ammonium chloride, tetrabutylphosphonium bromide, tetrabutylphosnhonium chloride, tetraoctylphosphonium bromide, and mixtures thereof.

The above process- 1 can be represented stepwise as shown below:



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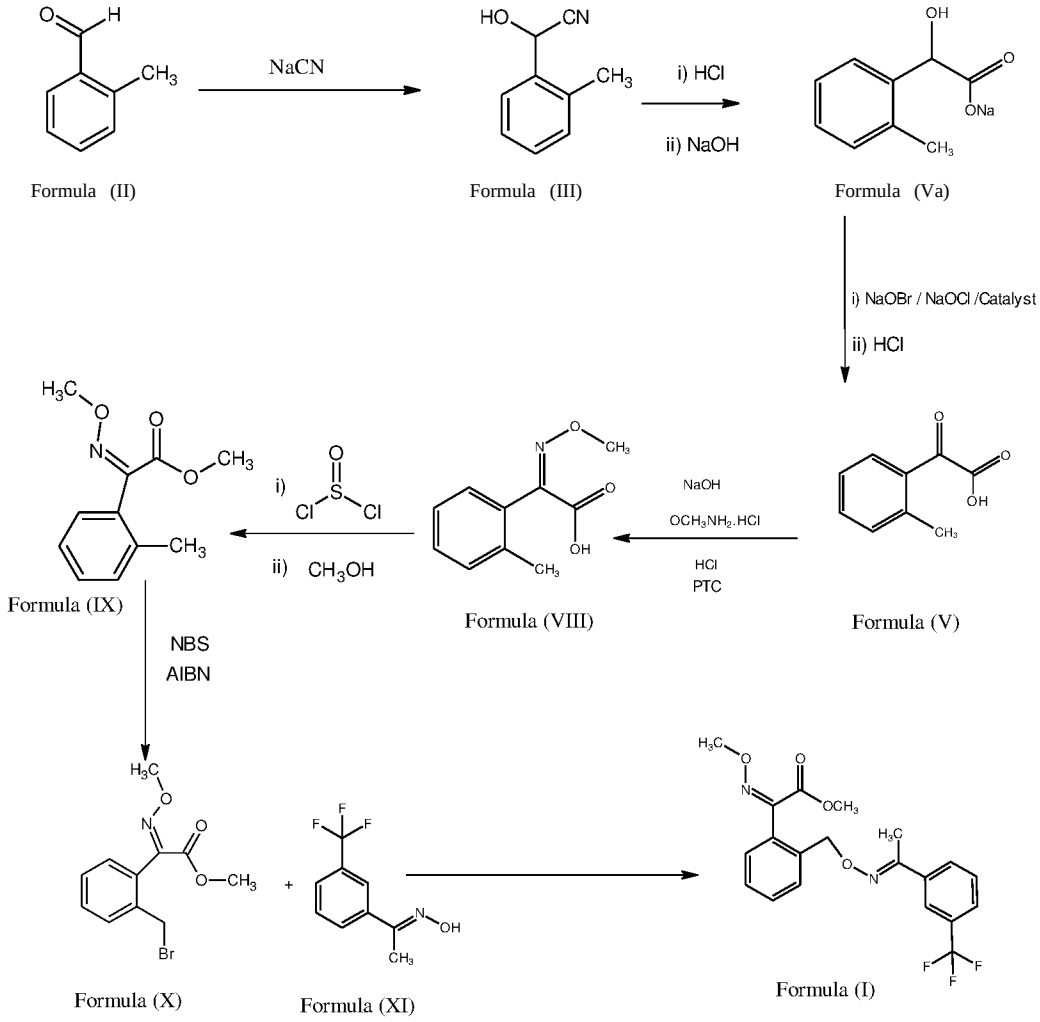
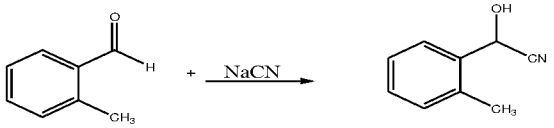
The above process-2 can also be represented stepwise as shown below:



The process of the invention is illustrated with reference to the following Examples and is not intended to limit the scope of the invention. Any permutations and modifications in the process are possible without limiting the scope of the invention.

Example 1; Preparation of Hydroxy-o-tolyl-acetonitrile (Process: A)



2-Methyl benzaldehyde (120.15gm 1.0 mole) was slowly added with starring and within 0.50 hours at temperature 8°C to 10°C in four necked flask contain solution of water (125ml) and sodium cyanide 49.00gm (l.Omole).The solution was prepared by added sodium cyanide 49.00 gm (1.0 mole) drop wise to water (125ml) the mixture was stirred over 30 minutes, during which

the temperature was maintained at -5°C to 5°C and then temperature was raised to 8°C to 10°C. After completion of addition reaction mass was stirred for 15 minutes and then 40 % sulfuric acid [83.0 gm sulphuric acid (0.84 mole) in 125 ml water] was added over a period of three hours, the temperature was maintained between 10°C and 20°Candreaction mixture was stirredfor fifteen minutes. After completion of reaction the product was extracted in dichloroethane and dried over anhydrous sodium sulfate. The dichloroethane layer taken as such for next reaction .In dichloroethane layer contains Hydroxy-o-tolyl-acetonitrile having purity >98.5% and the yield is 98-99 % of the theoretical amount.

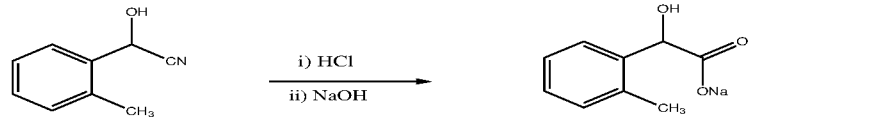
Example 2:Preparation of Hydroxy-o-tolyl-acetonitrile (Process: B)

2-Methylbenzaldehyde (24.03gm, 0.2 moles) was charged to solution of Con. Hydrochloric acid (18 ml, 21.6 g, and 0.2 mole) and water (17ml) in a 200 ml three neck flask at 0°C.The resulting mixture was cooled to -5°C to give slurry of 2-Methylbenzaldehyde hydrochloride. The reaction mixture was charged with solution of sodium cyanide (11.00 g, 0.22 moles) in 40 grams of water, drop wise and stirred mixture over 30 minutes, during which the temperature was maintained at -5°C to -l°C.During the addition a yellow solution was obtained after approximately half the cyanide solution has been added but slurry is again formed towards the end of the addition. Stirring was continued for one hour after the addition was complete. Hydrochloric acid (1 ml) was added to bring the mixture to pH=7. After completion of reaction the product was extracted in ethyl acetate was added and the mixture warmed to 10°C whereupon two clear layers were obtained. Ethyl acetate layer was removed and evaporated to give a 28 gm Hydroxy-o-tolyl-acetonitrile having purity >96% and the yield is 95 % of the theoretical amount

Example 3:Preparation of Hydroxy-o-tolyl-acetonitrile (Process-C)

2-Methylbenzaldehyde (120.15 gm 1.0 mole) was charged to solution of Sodium bisulfite (105 gm 1.0 mole) in water (475 gm) at room temperature and temperature was maintained below 35°C. To the reaction mixture sodium cyanide (49 gm 1.0 mole) was addedin portions to the solution and the temperature was maintained between 25°Cto 35°C. An oil phase separated out as an upper layer and this was separated from the lower aqueous layer. The aqueous layer was then extracted with dichloroethane and the resulting dichloroethane extracts were combined with the separated oil layer. Organic layer was dried over anhydrous sodium sulfate, and subjected to vacuum distillation at a heating bath temperature of 40°C to remove the dichloroethane. The residue obtained of Hydroxy (2-methylphenyl) acetonitrile was 132 grams. The yield of Hydroxy (2-methylphenyl) acetonitrile was 90%.

Example 4: Preparation of Hydroxy-o-tolyl-acetic acid sodium salt (Process-A)

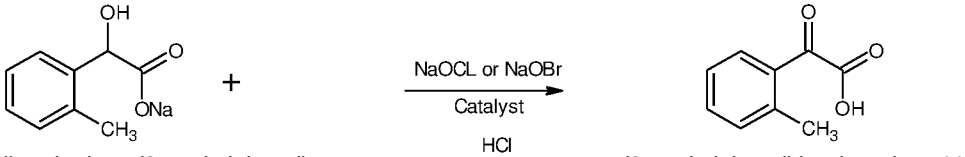


Hydroxy-o-tolyl-acetonitnle
Formula-(III)

Hydroxy-o-tolyl-acetic acid sodium salt
Formula(IVa)

Hydroxy-O-tolyl-acetonitrile (147.17gm 1.0 mole)was added to 300 ml dichloroethane in four necked flask equipped with an overhead stirrer, thermometer and dropping funnel and further added Tetrabutyl ammonium bromide (1.0 mole%). The reaction mass was treated with Hydrochloric acid (125 gm, 1.03 mole, 30% HCl) at 30°C. The reaction mass was maintained for 20 to 22 hours at 30°C. After completion of reaction product was separated in aqueous layer and organic layer. The aqueous layer with product was further hydrolysed in presence of 47% caustic lye (sodium hydroxide 100 gm in 112 gm water, 2.5 moles) at 30°C and the reaction was maintained for 30°C to40°C for 3 to 4 hours in presence of 1.0 gm Tetrabutylammonium bromide. After completion of reaction, reaction mass was washed with solvents such as Toluene, Xylene, EDC, MDC ETC, etc. The product obtained Hydroxy-o-tolyl-acetic acid sodium salt (182 to 183gm) having purity > 97% and the yield is 97 to98 % of the theoretical amount.

Example 5: Preparation of Oxo-o-tolyl-acetic acid (Process-A)



sodium hydroxy(2-methylphenyl)acetate
Formula (Va)

(2-methylphenyl)(oxo)acetic acid
Formula (V)

Sodium hypobromite (NaOBr) was generated by bromine with sodium hydroxide solution (700ml of water and 96g (2.4mol) of sodium hydroxide Cooled the reaction mixture to -5°C),Bromine (62ml 1.2mol) was added into a dropping funnel and dropped slowly to the sodium hydroxide solution for 2 hours, keeping the internal temperature at -5°C to generate sodium hypobromite (NaOBr).

Hydroxy-o-tolyl-acetic acid sodium salt (188.16 gm lmol) in 700ml of dichloromethane was charged to the solution of sodium hypobromite (NaOBr) and 4-acetoxy-2, 2, 6, 6-tetramethylpiperidinyl-l-oxy (5 mole%) and 10 ml of IN sulfuric acidso that the temperature was not exceeding 0°C.The reaction mixtures was stirred for 24 hours at room temperature. After

completion of reaction pH was adjusted to 3.0 to 4.0 with hydrochloric acid.The organic layer and aqueous layer was separated.The aqueous layer was extracted twice with dichloromethane. Removed solvent on a rotary evaporator under reduced pressure to get a target compound. 154.31gm oxo-o-tolyl acetic acid having purity <97% and Yield = 94%.

Example 6: Preparation of Oxo-o-tolyl-acetic acid (Process: B)

Hydroxy-o-tolyl-acetic acid sodium salt (37.63g 200 mmol) dissolved in methylene chloride (100 ml) and charged sodium hypochlorite (148.8 g 240 mmol), and cool at 5°C. To the reaction mixture 4-acetoxy-2, 2, 6, 6-tetramethylpiperidinyl-l-oxy (86mg 0.4 mmol) and sodium hydrogen carbonate (3.02 g 0.18 mmol) was added. The reaction mass was maintained for 24 hours at same temperature.After disappearing the starting material reaction terminated by adding 100 ml of 5% sodium thiosulfate aqueous solution. Organic and aqueous layer where separated andthe organic phase washed with 50 ml of 5% sodium thiosulfate aqueous solution and then with 100 ml of 1% sodium carbonate aqueous solution. Solvent was removed on a rotary evaporator under reduced pressure to obtain Oxy-o-tolyl -acetic acid (30.06gm (92% Yield).

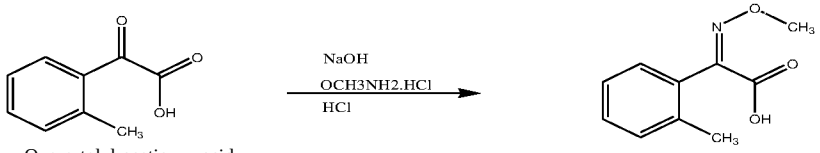
Example 7: Preparation of Oxo-o-tolyl-acetic acid (Process: C)

Hydroxy-o-tolyl-acetic acid sodium salt (18.81g 100 mmol) was dissolved in methylene chloride (50 ml) in 200 ml flask and was charged with of sodium hypochlorite (72.6 g (purity 12.3%, 120 mmol)), and cool at 6°C. To the reaction mixture was added 43 mg (0.2 mmol) of 4-acetoxy-2, 2, 6, 6-tetramethylpiperidinyl-l-oxy and further added 10 ml of IN hydrochloric acid. The reaction mass was maintained for 1 hour. After the completion of reaction, reaction was stopped by adding 10 ml of 5% sodium thiosulfate aqueous solution and separated by organic and aqueous layer. The organic phase was washed with 25 ml of 5% sodium thiosulfate aqueous solution and then 50ml of 1% sodium carbonate aqueous solution. Solvent was removed on a rotary evaporator under reduced pressure to obtain compoundoxy-o-tolyl -acetic acid. Wt. =16.41gm. The yield =90%

Example 8: Preparation of Oxo-o-tolyl-acetic acid (Process: E)

According to the same procedure as described in Example 7, Hydroxy-o-tolyl-acetic acid sodium salt (18.81 g 100 mmol) was dissolved in methylene chloride (50 ml), and tehn treated with 72.6 g (purity 12.3%, 120 mmol) of sodium hypochlorite, 5 mole% (2,2,6,6-Tetramethyl-piperidin-l-yl-)oxyl (TEMPO), 5mole% TBAB and 10 ml of IN sulfuric acid, to obtain compound oxy-o-tolyl -acetic acid (15.03 g). The yield was 92%.

Example 9: Preparation of Methoxyimino-o-tolyl-acetic acid

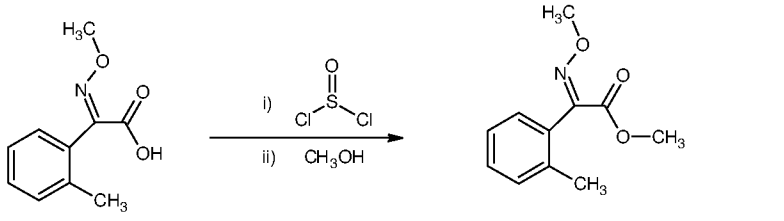


Oxo-o-tolyl-acetic acid
Formula (V)

Methoxyimino-o-tolyl-acetic acid
Formula(VIII)

Oxo-o-tolyl-acetic acid (164.16 gm 1.0 mole) was dissolved in 300 ml Dichloromethane in four necked flask equipped with stirrer, thermometer, addition funnel arrangement. and in four necked flask. The raeaction mass was charged with Tetrabutyl Ammonium Bromide (2.0 gm) and sodium hydroxide solution slowly at 40°C to 45°C within 2 hour.After completion of addition of sodium hydroxide solution reaction mass was maintained at 45°C for 2 to 3 hours. After 0.5 hour settling time organic layer and aqueous layer was separated. In the aqueous layer 300 ml Dichloromethane and 30% solution of Methoxyamine Hydrochloride (125gm, 1.5 mole) was charged at 30°C to 40 °C. the reaction mass was maintained for 1hour at 45°C. The reaction mass was separated by layer separation after 0.5 hour settling time separated the organic layer and aqueous layer.Organic layer contains Methoxyimino-o-tolyl-acetic acid (197gm) and the product was taken as such for water removal azeotropically. The final product obtained wasmethoxyimino-o-tolyl acetic acid (181.60 gm). % Yield = 94% %Purity = 97% (E & Z isomer).

ExamplelO: Preparation of Methyl(2E)-(methoxyimino)(2-methylphenyl)ethanoate (Process-A)



(2E/Z)-(methoxyimino)(2-methylphenyl)ethanoic acid
Formula (VIII)

methyl(2E)-(methoxyimino)(2-methylphenyl)ethanoate
Formula (IX)

N, N-Dimethylformamide (5gm) and 380 ml EDCwas charged in four necked flask equipped with an overhead stirrer, thermometer and dropping funnel.Methoxyimino-o-tolyl-acetic acid (193.20 gm 1.0 mole) in EDC andthionyl chloride(178.5gm 1.50 mole) was charged in reaction mass at 40 to 45°c & completed it within 2 to 3 hours. The reaction mass was maintain for 10 to 12 hours at 60°C till the z-isomer conversion is less than 1%.After completion of reaction degassing the reaction mass by purging nitrogen gas for 1 hour to remove HCl and SO2 gas.Solvent was recovered by atmospheric distillation and finally under reduced pressure. Then charge 200 ml

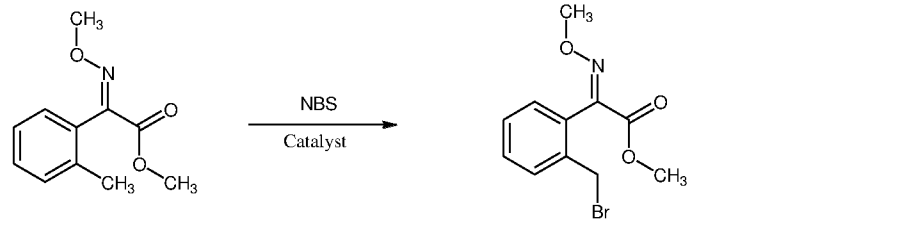
methanol in reaction mass and reflux for 2 hours till to get clear solution.Then gradually the reaction mass was cooled upto 10°C.Filter the reaction mass and treated with chilled methanol wash to cake. Wt. of Methyl (2E)-(methoxyimino) (2-methylphenyl) ethanoate= 186.50 gm
% yield = 90% ; % Purity>97%

Example!!: Preparation of Methyl (2E)-(methoxyimino) (2-methylphenyl) ethanoate

Process: B

N, N-Dimethylformamide (5 gm) in EDC 300 ml was charged in four necked flask equipped with thermometer, stirrer, and additional funnel arrangement. Methoxyimino-o-tolyl-acetic acid (193.20 gm 1.0 mole) in EDCand Triphosgene (119 gm 0.40 mole) was added in reaction mass at 40°C to 45°Cand completed it in within 2 to 3 hours and further reaction was maintained for 10 to 12 hours at 60°C till the z-isomer conversion is less than l%.If z-isomer is greater than 1% then add triphosgene again maintain for another two hours.Degassing the reaction mass by purging nitrogen gas by 1 hour to remove HC1 and SO2 gas. Solvent was recovered by atmospheric distillation and finally under reduced pressure. Then charge 190 ml methanol in reaction mass and reflux for 2 hours till to get clear solution. Then gradually the reaction mass was cooled upto 10°C. Filter the reaction mass and treated with chilled methanol wash to cake. Wt. of Methyl (2E)-(methoxyimino) (2-methylphenyl) ethanoate =195 gmto 196 gm. % yield = 94to 95% ;
% Purity>97%

Example 12: Preparation of methyl(2E)-r2-(bromomethyl)phenyll(methoxyimino) ethanoate



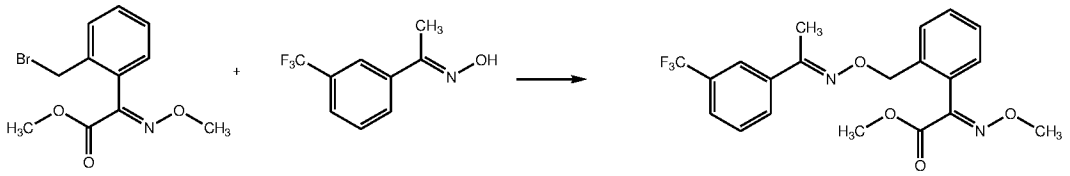
Methoxyimino-o-tolyl-acetic acid methyl ester — Formula (IX)

(2-Bromomethyl-phenyl)-methoxyimino-acetic acid methyl ester — Formula(X)

Methyl (2E)-(methoxyimino)(2-methylphenyl)ethanoate (207.23 gm, 1.0 mole) and N-bromosuccinamide (232 gm 1.30 mole) was charged in 1000 ml Acetonitrile.Reaction mixture was maintained at 58°C to62°C for 2 to 4 hours.After completion of reaction recovered acetonitrile under reduced pressure.The reaction mixture was turned to reddish in color and the process of the reaction monitored byTLC. After completion of reaction recovered acetonitrile under reduced pressure. The reaction mixture was charged with 100 ml water and 300 ml diisopropyl ether. The solutions was washed with 10%aqueous sodium bisulfate solution2*300 ml.Finally

washed with water 2*300 ml.Organic layer dried over sodium sulphate and chill to -5°C and filtered. The solid washed with chill Diisopropyl ether to obtain methyl (2E)-[2-(bromoMethyl) phenyl] (methoxyimino) ethanoate. Wt. of the product= 241 gm % Purity of the product= 98% ; %Yield =84%

Example 13: Preparation of Trifloxystrobin



(2-Bromomethyl-phenyl)-methoxyimino -acetic acid methyl ester

Formula (X)

1-(3-Trifluoromethyl-phenyl) -ethanone oxime

Formula (XI)

Methoxyimino-{2-[l-(3-trifruoromethyl-phenyl)-ethylideneaminooxymethyl]-phenyl}-acetic acid methyl ester

(E-isomer)

Formula (I)

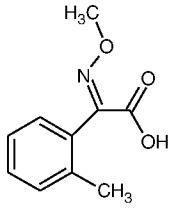
(2-bromomethyl-phenyl)-methoxyiminoacetic acid methyl ester (315 gm,1.10 mole)was dissolved in 1500 ml methyl isobutyl ketone.To this solution was added Intermediate-II (203. 16gm 1.0 mole) at room temperature followed by potassium carbonate (145gm 1.05 mole).The reaction mixture was heated to 115°c for 12 to hours. After completion of reaction mixture was cooled to room temperature and filtered to remove potassium bromide by-product and excess potassium carbonate. The solid obtained after filtration was washed with methyl isobutyl ketone and recycled in next batch for the preparation of Trifloxystrobin. The filtrate was washed with water and dried over anhydrous sodium sulphate and concentrated under vacuum to obtain crude product. The crude product was recrystallised in methanol to give off white colored product of Trifloxystrobin =348 gm. % Yield= 85% ; %Purity>98%

CLAIMS:

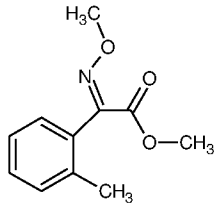
We Claims

[Claim 1], An novel process for the preparation of the compound of formula (X) which comprises the steps of :

a) methylation of (2E/Z)-(methoxyimino) (2-methylphenyl) Ethanoic acid compound of formula (VIII) in presence of thionyl chloride or triphosgene and solvent to obtain methyl (2E)-(methoxyimino) (2-methylphenyl) ethanoate compound of formula (IX);

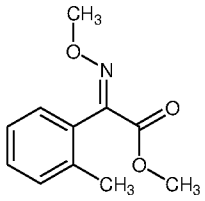


Formula (VIII)

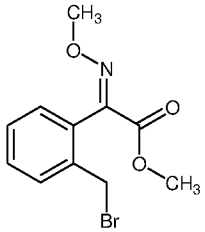


Formula (IX)

b) brominating methyl (2E)-(methoxyimino) (2-methylphenyl) Ethanoate compound of formula (IX) in presence of brominating agent and an organic solvent optionally in the presence of a phase transfer catalyst to obtain methyl (2E)-[2-(bromomethyl) phenyl] (methoxyimino) ethanoate of formula (X)



Formula (IX)



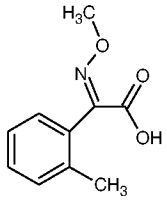
Formula(X)

[Claim 2]. A novel process as claimed in claim 1 wherein, suitable solvent is selected from the group consistingmethylene chloride, chloroform, monochlorobenzene, ethylene chloride, toluene, xylene, heptane, cyclohexane, hexane, acetone, DMF, acetonitrile, DMSO, sulfolane, methanol, ethanol, propanol, butanol, or mixtures thereof.

[Claim 3], A novel process as claimed in claim 1 wherein, brominating agent are selected from the group consisting phenyltrimethyl ammoniumtribromide; dibromodimethylhydantoin (DBDMH); potassium bromide; Sodium bromide ; Br_2; hydrobromic acid; N-bromophthalimide; N-bromoacetamide, N-bromosuccinimide (NBS), in Combination with the AIBN or Benzoyl peroxide or mixtures thereof.

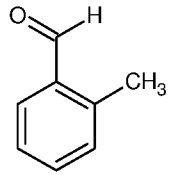
[Claim 4]. A novel process as claimed in claim 1 wherein phase transfer catalyst selected from the group consisting Tetrabuylammonium bromide, Tetrabutyammonium chloride, of quaternary ammonium cations, quaternary phosphoniumcations, and cyclic polyethers such as tricaprylylmethylammonium chloride, methyl tributyl ammonium chloride, methyl tributyl ammonium fluoride, tetrabutyl ammonium bromide, tetrabutyl ammonium fluoride, tetrabutyl ammonium hydrogen sulfate, triethyl benzyl ammonium chloride, tetrabutylphosphonium bromide, tetrabutylphosnhonium chloride, tetraoctylphosphonium bromide, and mixtures thereof.

[Claim 5], A novel process for the preparation of the compound of the formula (VIII) comprises the steps of :

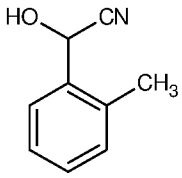


Formula (VIII)

a) Reacting the compound of the formula (II) with Metal Cyanide in presence of acid and water to obtain compound of the formula (III)

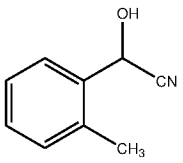


Formula (II)

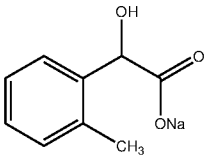


Formula (III)

b) Hydrolysed the compound of the formula (III) by using acid and sodium hydroxide in presence of solvent to obtain compound of the formula (Va).

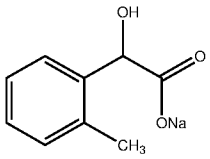


Formula-(III)

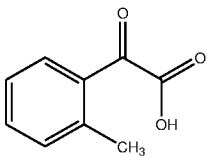


Formula(Va)

c) Oxidation of the compound of the formula (Va) with oxidizing agent to obtain compound of the formula (V)

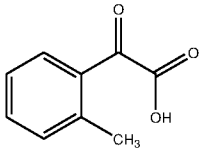


Formula(Va)

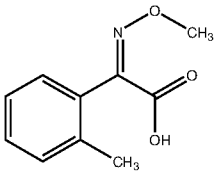


Formula(V)

d) Compound of the formula (V) reacted with Methoxyamine hydrochloride in presence of base and chlorinated solventselected from the group consisting Dichloroethane, Dichloromethane, Toluene,xylene monochlorobenzene to obtain the compound of the formula (VIII)

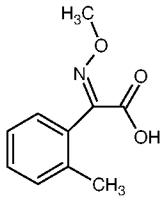


Formula (V)



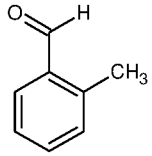
Formula(VIII)

[Claim 6], A novel process for the preparation of the compound of the formula (VIII) comprises the steps of :

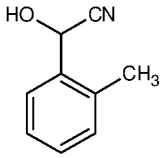


Formula **(VIII)**

a) Reacting the compound of the formula (II) with Metal Cyanide in presence of acid and water to obtain compound of the formula (III)

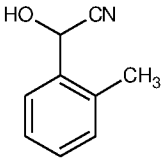


Formula (II)

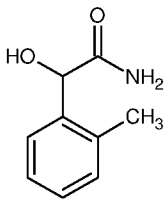


Formula (III)

b) hydrolyzing hydroxy (2-methylphenyl) acetonitrile of formula (III) in presence of acids to obtain compound of compound of formula (IV);

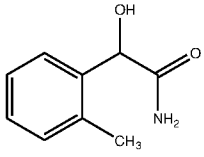


Formula (III)

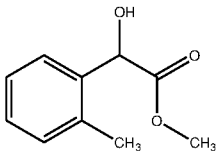


Formula (IV)

c) reacting2-Hydroxy-2-o-tolyl-acetamide compound of formula (IV) with methanol in presence of acids to obtain compound of compound of formula (VI)



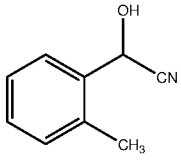
Formula (IV)



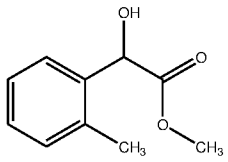
Formula (VI)

OR

reacting hydroxy (2-methylphenyl) acetonitrile of formula (III) with methanol in presence of acid to obtain compound of compound of formula (VI)

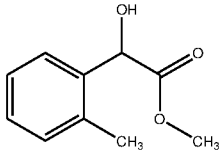


Formula (III)

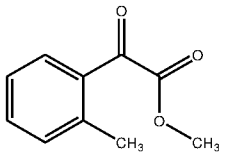


Formula (VI)

d) oxidation of Oxo-o-tolyl-acetic acid methyl ester of formula (VI) in presence of oxidizing agent and a catalytic amount of a weak base 2,2,6,6-tetramethylpiperidine 1- oxyl (TEMPO) to obtain the compound of the formula (VII)

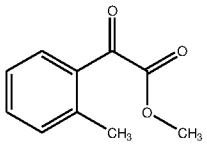


Formula (VI)

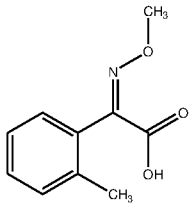


Formula (VII)

e) Reacting Oxo-o-tolyl-acetic acid methyl ester of formula (VII) with Methoxyamine hydrochloride in presence of a base and optionally in the presence of phase transfer catalyst to obtain the compound of the formula (VIII)



Formula (VII)



Formula(Vffl)

[Claim 7], A novel process as claimed in claim 6 & 7 wherein the metal cyanide are Sodium Cyanide, Potassium Cyanide, Cuprous Cyanide.

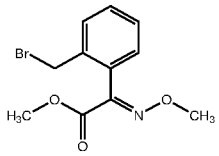
[Claim 8]. A novel process as claimed in claim 6 & 7wherein the acid are Con. hydrochloric acid or Hydrochloride gas.

[Claim 9]. A novel process as claimed in claim 6 & 7wherein oxidizing reagents are Sodium Hypochlorite (NaOCl), Sodium Hypobromite (NaOBr).

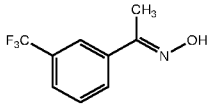
[Claim 10]. A novel process as claimed in claim 6 & 7wherein oxidizing catalysts selected from the group consisting Ruthenium Dioxide,Tempo (2,2,6,6-Tetramethyl-piperidin-l-yl)oxyl), 4-acetoxy-2,2,6,6-tetramethylpiperidinyl-l-oxy,Tetrabutyl ammonium bromide or mixture thereof.

[Claim 11], A novel process as claimed in claim 6 & 7wherein methoxyimination carried out in presence of base selected from the group consisting sodium hydroxide potassium hydroxide, sodium carbonate, potassium carbonate.

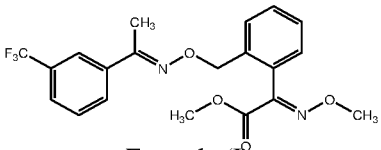
[Claim 12]. An improved process for the preparation of the compound of the formula (I) by reacting compound of the formula (X) with the compound of the formula (XI) in presence of solventselected from Toluene Xylene, Methylisobutyl ketone, acetonitrile, N, N-Dimethylformamideand optionally in presence of a base or phase transfer catalyst.



Formula (X)



Formula (XI)



Formula (I)

[Claim 13]. A novel process as claimed in claim 12 wherein baseis selected from Sodium Hydroxide, Potassium Hydroxide, Sodium Carbonate, Potassium Carbonate, triethylamine.

[Claim 14]. A novel process as claimed in claim 12 wherein phase transfer catalyst selected from the group consisting of quaternary ammonium cations, quaternary phosphoniumcations, and cyclic polyethers such as tricaprylylmethylammonium chloride, methyl tributyl ammonium chloride, methyl tributyl ammonium fluoride, tetrabutyl ammonium bromide, tetrabutyl ammonium fluoride, tetrabutyl ammonium hydrogen sulfate, triethyl benzyl ammonium chloride, tetrabutylphosphonium bromide, tetrabutylphosnhonium chloride, tetraoctylphosphonium bromide, and mixtures thereof.

[Claim 15]. A novel process as claimed in claim 12 wherein the compound of the formula (I) obtained having purity >97%.