

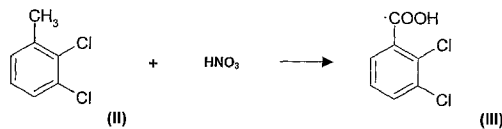
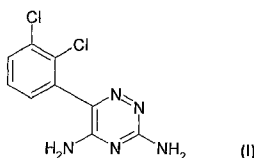
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657/MUM/2006 26 April 2006 (26.04.2006) IN(71) **Applicant (for all designated States except US):** RATNA-MANI BIO-CHEMICALS & PHARMACEUTICALS PVT.LTD. [IN/IN]; Plot No. 6112 & 6113, Opp. Heubach Colour, GIDC, Ankleshwar 393 002 (IN).(72) **Inventors; and**(75) **Inventors/Applicants (for US only):** PATEL, Rameshkumar, Bhagwandas [IN/IN]; Plot No. 6112 & 6113, Opp. Heubach Colour, GIDC, Ankleshwar 393 002 (IN). PATEL, Prasannavadan, Rasiklal [IN/IN]; Plot No. 6112 & 6113, Opp. Heubach Colour, GIDC, Ankleshwar 393 002 (IN).(74) **Agents:** MAJUMDAR, Subhatosh et al.; S. MAJUMDAR & CO., 5, Harish Mukherjee Road, Kolkata 700 025 (IN).(81) **Designated States (unless otherwise indicated, for every kind of national protection available):** AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW(84) **Designated States (unless otherwise indicated, for every kind of regional protection available):** ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).**Published:**

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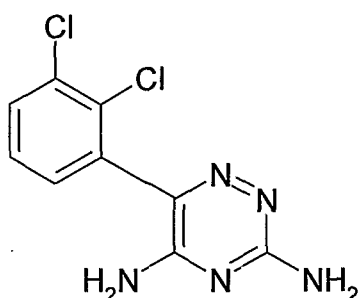
(54) **Title:** AN IMPROVED PROCESS FOR PREPARATION OF LAMOTRIGINE INTERMEDIATES

(57) **Abstract:** An improved process for the preparation of Lamotrigine of Formula (I) and their intermediates wherein an intermediate, 2,3-dichlorobenzoic acid of Formula (III) is prepared in single step. The process for the preparation of Lamotrigine comprises cyano-de-halogenation of 2,3-dichlorobenzoyl chloride of Formula (IV) with a metal cyanide and a phase transfer catalyst. Formula (I) Formula (III) Formula (IV).

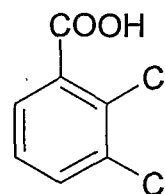
**AN IMPROVED PROCESS FOR PREPARATION OF
LAMOTRIGINE INTERMEDIATES**

FIELD OF INVENTION

The present invention relates to an improved process for the preparation of 3,5-diamino-6-(2,3-dichlorophenyl)-1,2,4-triazine commonly known as Lamotrigine of Formula I and their intermediates in which an intermediate i.e. 2,3-dichloro benzoic acid of Formula (III) is prepared in single step.

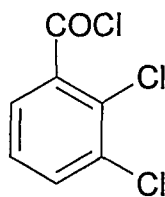


Formula I



Formula III

The present invention further relates to a improved process for the preparation of 3, 5-diamino-6-(2, 3-dichlorophenyl)-1, 2, 4- triazine in which cyano-de-halogenation of 2,3-dichloro benzoyl chloride of Formula (IV) is carried out with a metal cyanide and a phase transfer catalyst in catalytic amount which in turn gives high yield of Lamotrigine.



Formula IV

BACKGROUND OF INVENTION

The chemical species, 6-(2,3-dichlorophenyl)-1,2,4-triazine-3,5-diamine of Formula (I) is generically known as Lamotrigine.

Lamotrigine is a drug of the phenyltriazine class chemically unrelated to existing antiepileptic drugs (AEDs). Lamotrigine is thought to act at voltage-sensitive sodium channels to stabilize neuronal membranes and inhibit the release of excitatory amino acid neurotransmitters (e.g. glutamate, aspartate) that are thought to play a role in the generation and spread of epileptic seizures.

Lamotrigine is disclosed in US Patent 4602017. The process for the preparation of lamotrigine disclosed in US Patent 4602017 comprises steps as follows:

1. **Preparation of 2,3-Dichlorobenzoic Acid**

Step (a) A solution of 2,3-dichloriodobenzene in sodium dried ether was added dropwise to magnesium turnings and a crystal of iodine with warming so as to form a Grignard reagent.

Step (b) The mixture of Step (a) was stirred and refluxed then cooled and transferred dropwise, under nitrogen, into a stirred mixture of sodium dried ether containing solid carbon dioxide.

Step (c) The mixture of Step (b) was stirred for 2 hours, left overnight to warm to room temperature, then treated with ice and aqueous hydrochloric acid, and the product extracted with ether. The combined ether extracts were washed with water then repeatedly extracted with an aqueous sodium hydroxide. These basic solutions were combined, stirred with activated charcoal for 10 minutes, filtered and the cooled filtrate was acidified with concentrated hydrochloric acid at 10.degree. C.

Step (d) The resultant solid was filtered off, washed with water (2.times.20 mis) and dried in vacuo. Yield 77.6%,

2. **Preparation of 2,3-Dichlorobenzoyl Chloride**

A mixture of 2,3-dichlorobenzoic acid and thionyl chloride was heated to reflux. The cooled solution was evaporated down in vacuo and distilled under nitrogen.

Yield 35.5 g (85%), b.p. 146.degree.-148.degree. C. at 31 mm of mercury pressure.

3. Preparation of 2,3-Dichlorobenzoyl Cyanide

- 5 Step (a) A mixture of cuprous cyanide, potassium iodide and xylene was refluxed in an atmosphere of nitrogen under a Dean and Stark trap so as to remove all trace of water.
- Step (b) A solution of 2,3-dichlorobenzoyl chloride in sodium dried xylene was added drop wise to the above mixture of dry cuprous cyanide and xylene. The resulting mixture was stirred and heated to reflux for a further 72 hours. The cooled mixture was filtered and the solid washed well with sodium dried xylene.
- 10 Step (c) The filtrate and washings were combined and evaporated down in vacuo to give oil. Yield 32 g (94%).

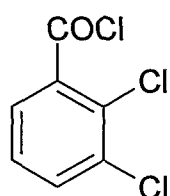
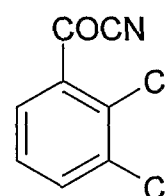
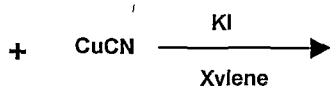
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4. Preparation of 3,5-Diamino-6-(2,3-dichlorophenyl)-1,2,4-triazine

- Step (a) A solution of 2,3-dichlorobenzoyl cyanide in dimethylsulphoxide was added dropwise to a stirred suspension of aminoguanidine bicarbonate which has been treated with an aqueous nitric acid at a temperature of ca 25.degree. C.
- 20 Step (b) The mixture was stirred for 3 hours, then left to stand at room temperature for 7 days. The cooled mixture was stirred and basified with aqueous ammonia at 20.degree.C then stirred with ice cooling, filtered and the resulting solid washed thoroughly with water and finally dried in vacuo.
- 25

It is noted that the process for preparation of 2,3-dichlorobenzoic acid of Formula (III) disclosed in US Patent 4602017 gives low yield and it is not industrially viable. Also time required for the preparation of 2, 3-Dichlorobenzoyl Cyanide is 72 hrs.

US patent 6639072 discloses the process for the preparation of lamotrigine and intermediates thereof. The process for preparation of 2,3-dichlorobenzoic acid of Formula (III) disclosed in US Patent 6639072 comprises series of reactions starting with catalytic reduction of 2,3 dichloro nitro benzene and converting the resulting amino compound to 2,3 dichloro benzonitrile by diazotization followed by cyanation using metal cyanide. The hydrolysis of 2,3 dichloro benzonitrile in acidic or alkaline condition produces 2,3 dichloro benzoic acid. Further compound of formula **III** is converted to acid chloride with the help of chlorinating agents such as Thionyl Chloride or Phosphorous trichloride or Phosphorous pentachloride. The acid chloride of formula IV is further reacted with metal cyanide and potassium iodide in Monochlorobenzene to obtain 2,3 Dichlorobenzoylnitrile of Formula V.

**Formula IV****Formula V**

US Patent 6639072 employs the use of hazardous chemicals like sodium cyanide and hydrogen for the preparation of 2, 3 Dichloro nitrobenzene. Also the process is three stage preparation starting with 2, 3 Dichloro nitrobenzene. The percentage yield by this method is only about 60 %. All these factors makes the process industrially non viable.

European Patent No. 0 963 980 discloses process for the preparation of lamotrigine and intermediates thereof. In European Patent No. 0 963 980 2,3-dichlorobenzoic acid of Formula **III** is prepared by oxidation of 2,3 dichloro benzaldehyde with hydrogen peroxide and sodium hydroxide in presence of t-butyl alcohol. The percentage yield obtained in this patent is 75% only. Also the manufacturing of 2, 3-Dichloro benzaldehyde requires 2, 3 - Dichloro toluene of Formula **II** as a starting material.

US Patent 6111101 discloses an improved process for the preparation of lamotrigine which process comprises the step of reacting 2,3-dichlorobenzoylchloride with cuprous cyanide in presence of acetonitrile and a cosolvent to produce dichlorobenzoyl cyanide of Formula (V),
5 said dichlorobenzoyl cyanide is reacted with aminoguanidine bicarbonate to produce an intermediate product, which is cyclized in presence of aqueous potassium hydroxide to produce 3,5-diamino-6-(2,3-dichlorophenyl)-1,2,4-triazine (lamotrigine). US Patent 6111101 replaces potassium iodide with acetonitrile and a co-solvent to obtain product of formula V. The reported reaction time is 16 hrs. But potassium iodide and acetonitrile are costly and
10 hazardous thereby making it industrially non viable.

Thus there is a long felt need in the prior art for the process of preparation of lamotrigine which yields 2,3 dichloro benzoic acid of Formula (III) in single step and gives high yield. Further the reaction time required for 2, 3 dichlorobenzoyl cyanide of Formula (V) is
15 reduced.

OBJECTS OF INVENTION

Thus it is an object of the present invention to provide an improved process for the preparation of 3, 5-diamino-6- (2, 3-dichlorophenyl)-1, 2, 4- triazine (lamotrigine) that
20 overcomes problems associated with the prior art.

It is further object of the present invention to provide a process for the preparation of an intermediate 2, 3 dichloro benzoic acid of Formula (III) in single step.

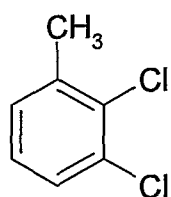
25 It is further object of the present invention to give 2, 3 dichloro benzoic acid in high yield.

Yet another object of the present invention to provide a process for the preparation of 2,3-Dichlorobenzoyl Cyanide of Formula (V) which employs use of a phase transfer catalyst that reduces the time required for the reaction.

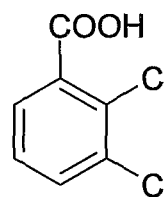
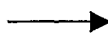
SUMMARY OF INVENTION

According to an aspect of the present invention there is provided An improved process for the preparation for 6-(2,3- dichlorophenyl)-1,2,4 - triazine- 3,5 - diamine of the formula I which comprises step of preparation of 2,3 dichloro benzoic acid of Formula (III) by reacting

5 2,3 dichloro toluene of formula II with dilute nitric acid of 20 - 25% at a temperature from about 110 °C to about 160 °C underpressure of about 5 kg/cm² to about 15 kg/cm².

**Formula II**

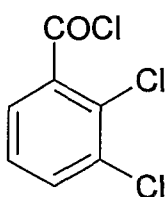
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HNO₃**Formula III**

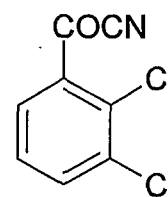
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According to further aspect of the present invention there is provided An improved process for the preparation for 6-(2, 3- dichlorophenyl)-1,2,4 - triazine- 3,5 - diamine of the formula I which comprises step of preparation of 2,3-Dichlorobenzoyl Cyanide of Formula (V) by reacting 2,3 - dichloro benzoyl chloride of Formula (IV) with metal cyanide in presence of

15 phase transfer catalyst and in solvent.

**Formula IV**

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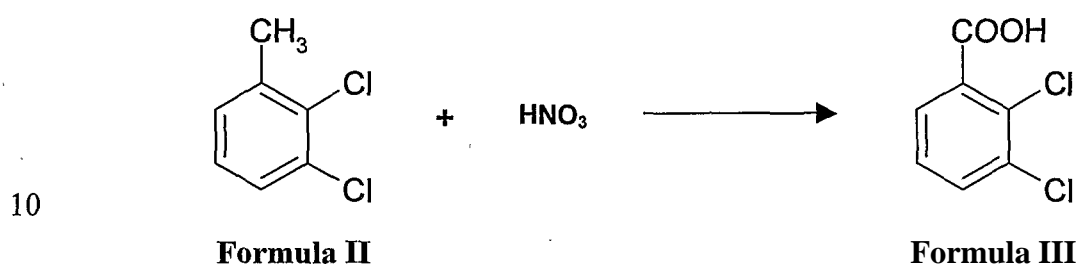
CuCN**PTC****Solvent****Formula V**

20

DETAILED DESCRIPTION

The inventors of the present invention have addressed the need of an improved process for the preparation of 3, 5-diamino-6-(2, 3-dichlorophenyl)-1, 2, 4- triazine (lamotrigine) of Formula (I).

- 5 Accordingly inventors have found out that when 2, 3 - dichloro toluene of Formula (II) is reacted with dilute nitric acid in the presence of temperature and pressure ,2,3 dichloro benzoic acid of Formula (III), an important intermediate for the preparation of 3, 5-diamino-6-(2, 3-dichlorophenyl)-1,2,4- triazine is formed in a single step and high yield.



According to present invention preferred strength of dilute nitric acid is about 15 % to about 35%. The most preferred strength of dilute nitric acid is 20 - 25 % .

15

According to present invention preferred temperature employed in the preparation of Formula (III) is about 80°C to about 160°C. The most preferred temperature employed in the preparation of Formula (III) is about 130 - 145 °C.

- 20 According to present invention preferred pressure applied in the preparation of Formula (III) is about 5 kg/cm² to about 15 kg/cm². The most preferred pressure applied in the preparation of Formula (III) is 10 - 15 kg/cm².

According to present invention 2, 3-dichloro benzoic acid of Formula (III) is obtained in yield of 90- 98 % .

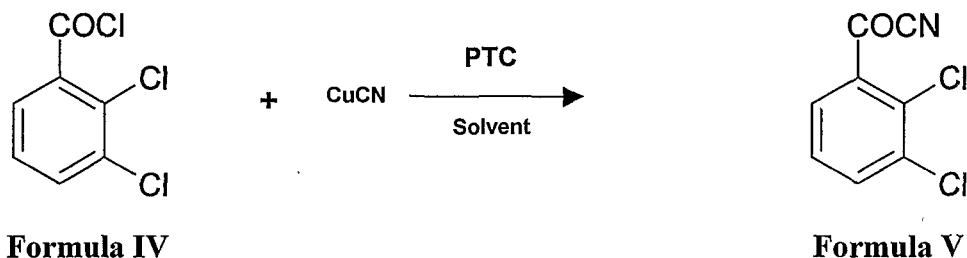
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The present invention has following advantages:

- 2, 3-dichloro benzoic acid of Formula (III) is obtained in most economical and safe way.
- It eliminates use of hazardous and toxic raw materials.
- The multi stage processing is also eliminated.
- 2, 3-dichloro benzoic acid of Formula (III) is obtained in high yield which in turn gives high yield of lamotrigine.

2, 3 - dichloro benzoyl chloride of Formula IV is obtained by reaction of 2, 3 - Dichloro benzoic acid of formula III with thionyl chloride at 80°C for 4 hrs. Removal of excess thionyl chloride by distillation gives the product of formula IV.

Further present inventors have found with sustained effort that the reaction time required for the preparation of 2,3-Dichlorobenzoyl Cyanide of Formula (V), an intermediate for the preparation of lamotrigine, is reduced when 2,3 - dichloro benzoyl chloride of Formula (IV) is reacted with metal cyanide in presence of Phase transfer catalyst in solvent.



According to present invention Alkali metal cyanides/ transition metal cyanides can be employed. The preferred metal cyanide is selected from Cuprous cyanide, potassium cyanide, sodium cyanide or mixture of cuprous cyanide & sodium cyanide. The most preferred metal cyanide according to present invention is cuprous cyanide.

According to present invention the most preferred range of metal cyanide employed in the reaction is 1.2 - 1.7 mole.

According to present invention all Tetra alkyl ammonium halides can be employed. The preferred phase transfer catalyst is selected from tetra butyl ammonium iodide or tetra butyl ammonium bromide or quaternary phosphonium salt, or crown ethers, or alkylated polyethylene glycole or tetra butyl ammonium hydrogen sulphate. The most preferred PTC
5 according to present invention is tetra butyl ammonium bromide or tetra butyl ammonium iodide..

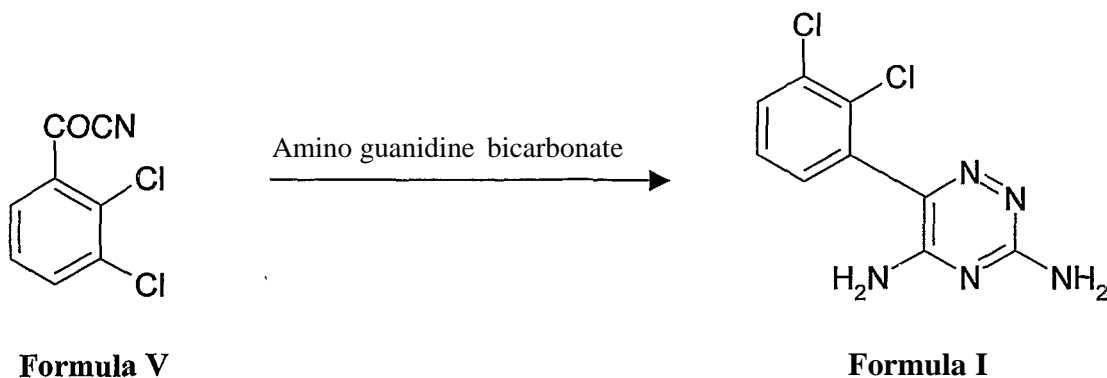
According to present invention the preferred range of phase transfer catalyst is 0.002 to 1.0 mole. The most preferred range of PTC according to present invention is ...0.005 to 0.1
10 mole.

According to present invention the preferred solvent is selected from Xylene , mono chloro benzene , toluene , 1,2 Dichlorobenzene or aromatic hydro carbone or halogenated aromatic hydro carbon. The most preferred solvent according to present invention is xylene , mono
15 chloro benzene.

In a mixture of cuprous cyanide in 300 ml xylene in presence of tetra butyl ammonium iodide or tetra butyl ammonium bromide, a solution formula IV in 200 ml xylene or mono chloro benzene is added under nitrogen atmosphere. The resultant reaction mixture is heated to
20 reflux for 8 hrs., cooled and filtered the solid. Xylene or mono chloro benzene is distilled off under reduced pressure to obtain the product of formula V. The overall reaction volume of solvent with this invention is reduced to 2.4 lit from 2.9 lit per kg of formula IV. The entire reaction is conducted under nitrogen atmosphere.

The dichlorobenzoyl cyanide of formula V is further reacted with aminoguanidine
25 bicarbonate and cyclising the reaction product to produce Lamotrigine of formula I

10

**Formula V****Formula I**

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Advantages of using PTC:

1. Reaction time is reduced
2. Use of KI a costly reagent has been eliminated
3. PTC is used in catalytic amount that is 0.06 mole.

10

The details of the invention, its objects and advantages are explained hereunder in greater details in relation to non-limiting exemplary illustrations. The examples are merely illustrative and do not limit the teaching of this invention and it would be obvious that various modifications or changes in the procedural steps by those skilled in the art without departing from the scope of the invention and shall be consequently encompassed within the ambit and spirit of this approach and scope thereof.

15

EXAMPLES:

20 **Example 1:**

200 gm (1.24 mole) 2, 3 dichloro toluene of formula II was charged in a autoclave fitted with reflux condenser. 710 gm (2.48 mole) of 22% nitric acid was added and autoclave was closed. Slowly it was heated to 140°C temp. The pressure obtained at this temperature was 10 Kg/ cm². The reaction at this temperature was continued for further 4 hrs. The reaction was cooled down and the vent was opened to release the NO_x gases.

25

The reaction mass was dumped in 1 lit water and filtered. The wet cake was washed several times to make acid free. The quantity obtained was 215 gms of purity 99% having melting point 166 - 170 °C.

5 **Example 2:**

200 gm (1.05 mole) of 2,3 dichloro benzoic acid of formula III is reacted with 249 gm(2.09 mole) of thionyl chloride at 80 °C temp for 4 hrs. Excess thionyl chloride was removed to obtain 2,3 Dichloro benzoyl chloride of formula IV. The quantity obtained was 200 gms of purity 99.2% (by HPLC).

10

Example 3:

In a mixture of 300 ml Xylene, 128 gm cuprous cyanide (1.43 mole) and 3 gm (0.008 mole) tetra butyl ammonium iodide, the solution of 200 gm (0.95 mole) 2, 3 Dichloro benzoyl chloride in 200 ml xylene was added. The resultant reaction mixture was refluxed for 8 hrs.

15 Reaction mixture was filtered and xylene was distilled off under

reduced pressure to obtain 2, 3 Dichloro benzoyl cyanide of formula V. The quantity obtained was 155 gms of purity 99.32% (by GC) having melting point 58-62 °C

Example 4:

20 In a mixture of 300 ml Mono chloro benzene, 128 gm cuprous cyanide (1.43 mole) and 3 gm (0.008 mole) tetra butyl ammonium iodide, the solution of 200 gm (0.95 mole) 2, 3 Dichloro benzoyl chloride in 200 ml MCB was added. The resultant reaction mixture was refluxed for 8 hrs. Reaction mixture was filtered and Mono chloro benzene was distilled off under reduced pressure to obtain 2, 3 Dichloro benzoyl cyanide. The quantity obtained was 152
25 gms of purity 99.15% (by GC) having melting point 59-62 °C.

Example 5:

In a mixture of 300 ml Xylene , 128 gm cuprous cyanide (1.43 mole) and 3 gm (0.009 mole) tetra butyl ammonium bromide, the solution of 200 gm (0.95 mole) 2, 3 Dichloro benzoyl

chloride in 200 ml xylene was added. The resultant reaction mixture was refluxed for 8 hrs. Reaction mixture was filtered and xylene was distilled off under reduced pressure to obtain 2, 3 Dichloro benzoyl cyanide. The quantity obtained was 152.5 gms of purity 99.2% (by GC) having melting point 58-61 °C

5

Example 6:

In a mixture of 300 ml mono chloro benzene , 128 gm cuprous cyanide (1.43 mole) and 3 gm (0.009 mole) tetra butyl ammonium bromide, the solution of 200 gm (0.95 mole) 2, 3 Dichloro benzoyl chloride in 200 ml monochlorobenzene was added. The resultant reaction mixture was refluxed for 8 hrs. Reaction mixture was filtered and monochlorobenzene was distilled off under reduced pressure to obtain 2, 3 Dichloro benzoyl cyanide. The quantity obtained was 154 gms of purity 99.41% (by GC) having melting point 57-63 °C.
(Kindly provide details of monochlorobenzene removal)

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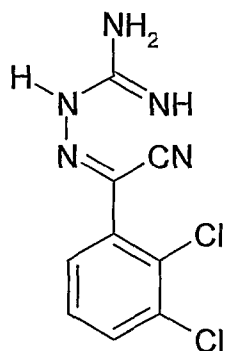
15 **Example 7:**

In 600 ml chilled water, 1100 gm of Sulphuric acid was added followed by addition of 200 (1.5 mole) gm aminoguanidinebicarbonate lot wise. To this mixture 200 gm (0.954 mole) 2, 3 - dichlorobenzoyl cyanide was added. Resultant reaction mixture was stirred at 55 - 60 Deg.C for 6 hrs. Reaction mass was filtered and solid cake was further added in water and pH of this suspension was adjusted to 9 to 10 with the help of aqueous NaOH and stirred for 0.5 hrs. Suspension was filtered and solid cake was washed thoroughly with water. Resultant wet cake was dried at 80 Deg.C to get Schiff base of formula VI.

20

Yield : 180 gm

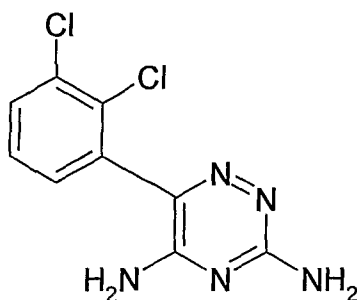
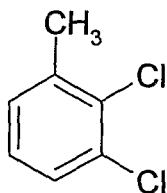
Purity : 98% (By HPLC)

**Formula VI**

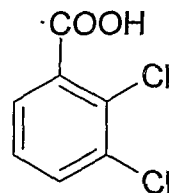
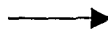
Resultant product of formula VI obtained from 2,3 - dichloro benzoyl cyanide of formula V was refluxed in 750 ml of 1% KOH solution for 1.5 hrs to give white solid. It was filtered
5 and washed with water to give Lamotrigine of formula I

CLAIMS

1. An improved process for the preparation for 6-(2,3- dichlorophenyl)- 1,2,4 - triazine- 3,5 - diamine of the formula I which comprises step of preparation of 2,3 dichloro benzoic acid of Formula (III) by reacting 2,3 dichloro toluene of formula II with
- 5 dilute nitric acid at a temperature from about 110 °C to about 160 °C under pressure of about 5 kg/cm² to about 15 kg/cm².

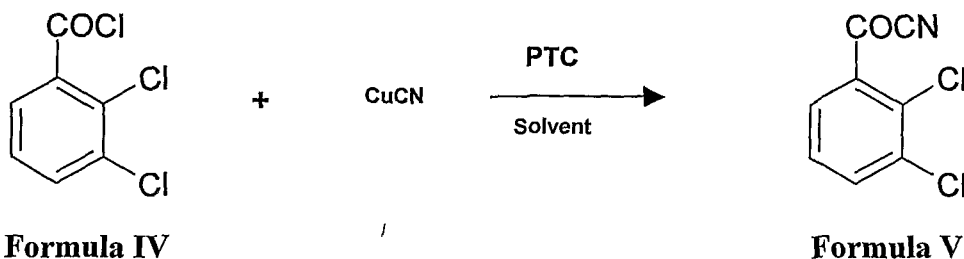
**Formula I****Formula II**

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HNO₃**Formula III**

2. The process as claimed in claim 1 wherein the temperature employed is 130 - 145°C.
3. The process as claimed in claim 1 wherein the pressure employed is 10 - 15 kg/cm².
4. The process as claimed in claim 1 wherein the preferred strength of the dilute nitric acid is about 15 % to about 35 %.
5. The process as claimed in claim 1 wherein the most preferred strength of the dilute nitric acid is 20 - 25 %.
6. An improved process for the preparation for 6-(2,3- dichlorophenyl)- 1,2,4 - triazine- 3,5 - diamine of the formula I which comprises step of preparation of 2,3- Dichlorobenzoyl Cyanide of Formula (V) by reacting 2,3 - dichloro benzoyl chloride

of Formula (IV) with metal cyanide in presence of phase transfer catalyst and in solvent.



7. The process as claimed in claim 6 wherein the said metal cyanide is selected from cuprous cyanide, potassium cyanide, sodium cyanide or mixture of cuprous cyanide & sodium cyanide.

10 8. The process as claimed in claim 6 wherein the said metal cyanide is cuprous cyanide.

9. The process as claimed in claim 6 wherein the preferred range of metal cyanide used in the reaction is 1.2 to 1.7 mole and most preferred range used is 1.5 mole.

10. The process as claimed in claim 6 wherein the said phase transfer catalyst is selected from tetra butyl ammonium iodide, tetra butyl ammonium bromide , or quaternary phosphonium salt, or crown ethers, or alkylated polyethylene glycole or tetra butyl ammonium hydrogen Sulphate.

11. The process as claimed in claim 6 wherein the said phase transfer catalyst is tetra butyl ammonium iodide or tetra butyl ammonium bromide.

12. The process as claimed in claim 6 wherein the preferred range of phase transfer catalyst employed in the reaction is 0.002 to 1 mole and most preferred range of phase transfer catalyst is 0.005 to 0.1 mole.

13. The process as claimed in claim 6 wherein the solvent is selected from xylene, monochlorobenzene toluene, 1, 2 Dichlorobenzene or aromatic hydro carbone or halogenated aromatic hydro carbon.

14. The process as claimed in claim 6 wherein the said solvent is xylene or monochlorobenzene.

15. The improved process for the the preparation for 6-(2,3- dichlorophenyl)- 1,2,4 - triazine- 3,5 - diamine of the Formula I substantially as hereinbefore described with reference to the preferred aspects, embodiments and features of the invention and illustrated with reference to the examples.