Nature of Invention: Chemical molecule and synthesis route

Applicant: Chimique Inc.

Inventors: Sujal Jain

Lokesh Yadav

Anisurya Jana

Chemical Formula: $C_9H_{10}ClN_5O_2$

Chemical Name: Imidacloprid

Chemical synthesis routes:

1. Synthesis of Imidacloprid at lab scale :-

Raw materials and Chemicals:-

- 2-nitroimino imidazolidine
- 2-chloro-5-chloromethyl pyridine
- potassium carbonate
- acetonitrile
- DMF

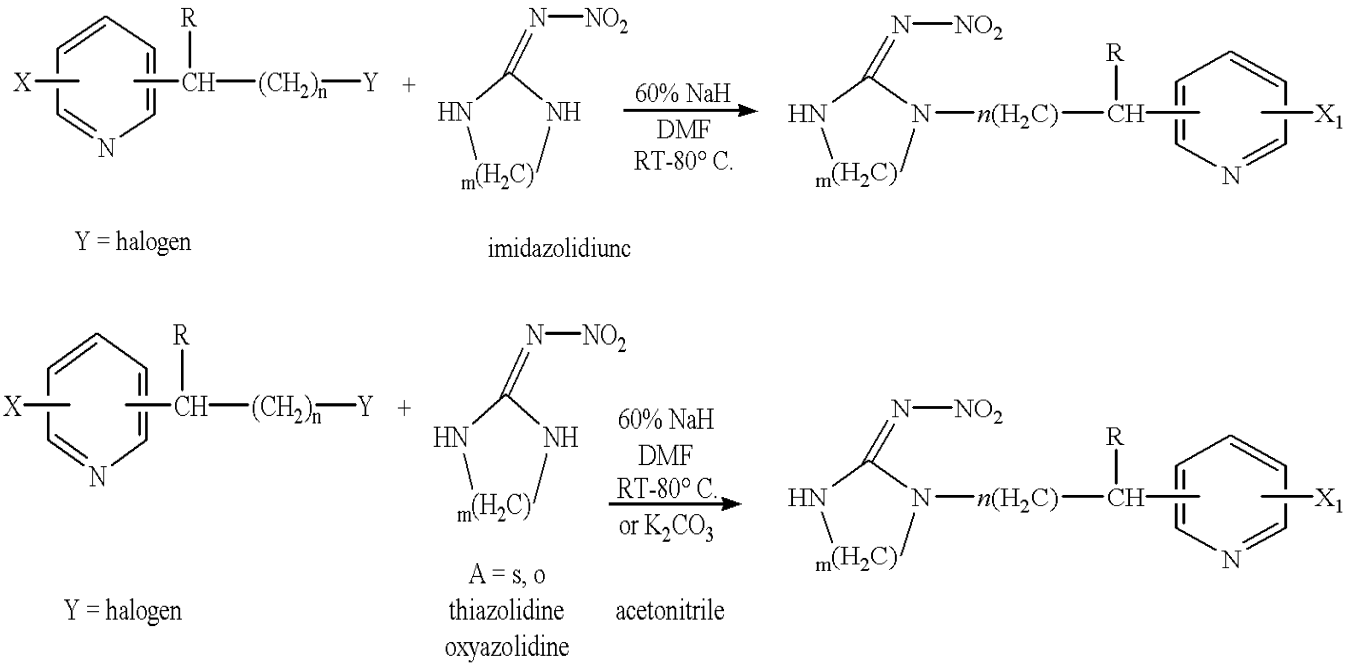
Synthesis process:-

The process for preparing imidacloprid involves reacting 2-nitroimino imidazolidine with 2-chloro-5-chloromethyl pyridine in the presence of an alkali carbonate in an organic solvent under reflux conditions. The 2-chloro-5-chloromethyl pyridine is added dropwise and continuously to the mixture at a rate of less than 0.03 equivalent per minute.Below the stepwise process described for the experiment conducted in the laboratory:

- Dissolve 7.8 g (60 mmol) of 2-nitroimino imidazolidine and 12.1 g (87.5 mmol) of potassium carbonate in 60 ml of acetonitrile in a reflux flask.
- Heat the mixture to achieve reflux operating conditions around 80 degree celsius.
- Dissolve 8.1 g (50.0 mmol) of 2-chloro-5-chloromethyl pyridine in 40 ml of acetonitrile.

- Add the 2-chloro-5-chloromethyl pyridine dropwise and continuously into the reflux flask over 0.5 hours, maintaining a rate of about 1.5 ml/minute.
- Let the mixture cool for a while
- After completion of the reaction, filter the mixture. .
- Concentrate the filtrate by using the filter paper .

The yield is determined by liquid chromatography; for this process it is around 84.30 %.



2. Alternate synthesis process:-

Raw materials and Chemicals:-

- Ethylene Diamine
- Chlorine
- Nitroguanidine
- 3-methylpyridine

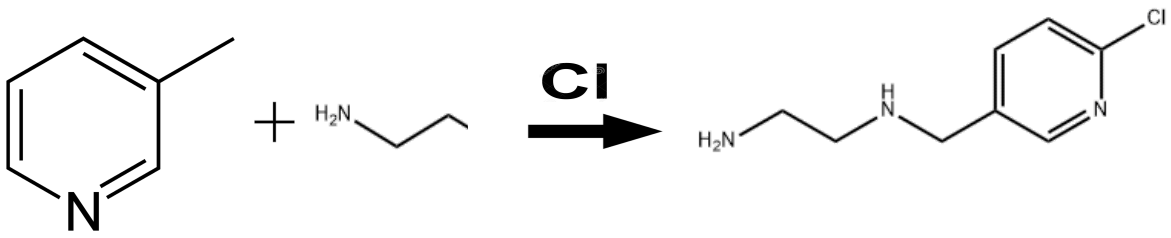
- 2-amino-5-methylpyridine

Synthesis process:-

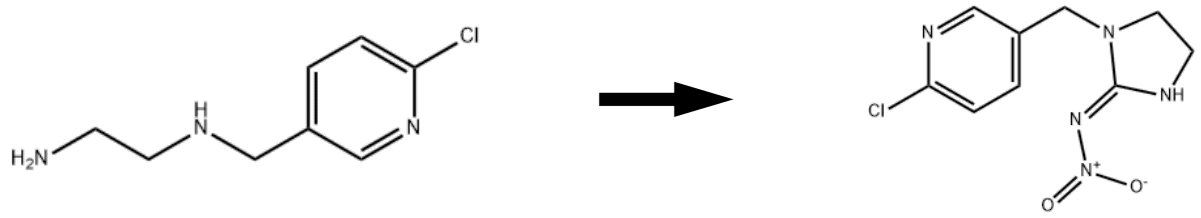
- Perform the Chichibabin reaction by reacting produce 3-methylpyridine to 2-amino-5-methylpyridine .
- Transform 2-amino-5-methylpyridine to the chloride using the Sandmeyer reaction in the presence of hydrogen chloride.
- Conduct chlorination of the methyl group to produce intermediate .
- Without isolating the intermediates, carry out the substitution of the active chloride with ethylene diamine to obtain compound Imidacloprid.
- The final product is formed by ring formation with nitroguanidine.

The multistep process yields the product at a purity of >95%.
Yield is 85%.

Chichibabin reaction:-



3- Methyl-pyridine **ETHYLENEDIAMINE** **N-(2-CHLORO-5-PYRIDYLMETHYL) ETHYLENEDIAMINE**



N-(2-CHLORO-5-PYRIDYLMETHYL) ETHYLENEDIAMINE **Imidacloprid**

References:

https://patents.google.com/patent/US6307053B1/en

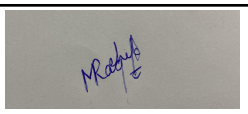
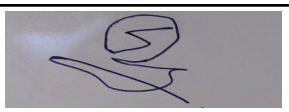
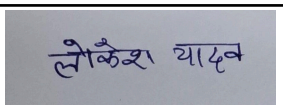
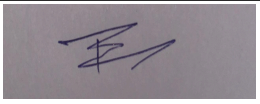
https://www.sciencedirect.com/topics/chemistry/imidacloprid

https://www.chemicalbook.com/synthesis/imidacloprid.html

https://www.chemicalbook.com/synthesis/n-2-chloro-5-pyridylmethyl-ethylenediamine.htm

List the contributions of each author:

- Author 2,4 and 5 carried out the literature search.
- Authors 1,2 and 3 found the reaction steps, product yield, necessary separation steps.

Name	Roll No	Signature
Mehek Agarwal	220646	
Sujal Jain	221100	
Lokesh Yadav	220594	
Anisurya Jana	220152	
Ram Aggarwal	220864	
Vyom Pratap Singh	221211