Applicant: VortexChem

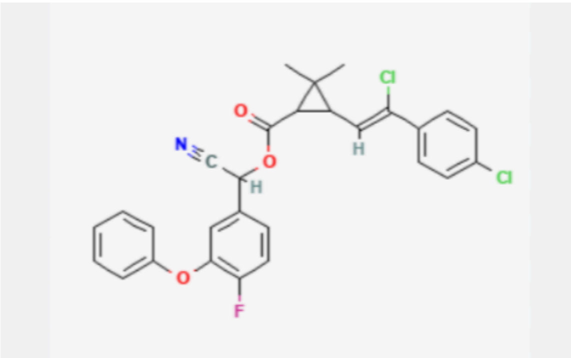
Inventors: Minshul Agrawal, Akshat Swarup

Chemical Formula: $C_{28}H_{22}Cl_2FNO_3$

Chemical Name: **Flumethrin**

Cyano(4-fluoro-3-phenoxyphenyl)methyl
3-[(Z)-2-chloro-2-(4-chlorophenyl)ethenyl]-2,2-dimethylcyclopropane-1-carboxylate

Chemical Structure:



Chemical synthesis routes:

Primary Synthesis Route

The synthesis of **Flumethrin**, a synthetic pyrethroid, involves two major steps:

1. **Preparation of Bayticol P Acid Chloride**
2. **Condensation of Bayticol P Acid Chloride with Sodium Cyanide and 4-Fluoro-3-Phenoxybenzaldehyde**

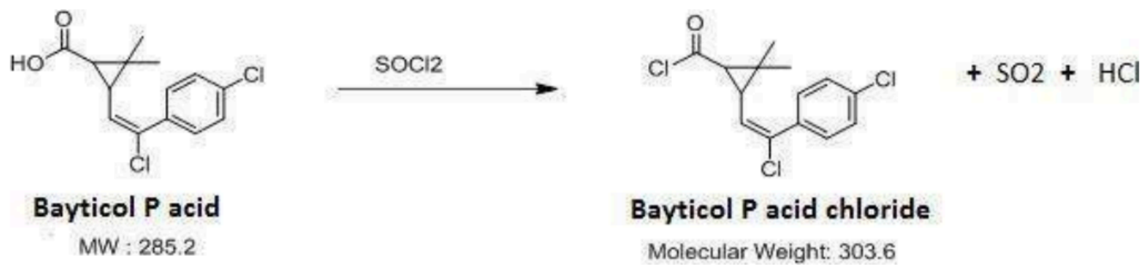
Each step requires controlled reaction conditions, solvent recovery, and purification techniques to ensure high yield and purity. Optimizing reaction parameters such as temperature, catalyst concentration, and reagent addition rate significantly impacts process efficiency and product purity.

Step 1: Preparation of Baytícol P Acid Chloride

Bayticol P Acid undergoes **chlorination with thionyl chloride ($SOCl_2$)** in **toluene**, catalyzed by **dimethylformamide (DMF)**, forming **Bayticol P Acid Chloride**. This reaction liberates **sulfur dioxide (SO_2) and hydrogen chloride (HCl) gases**, which are removed efficiently to prevent side reactions.

Chemical Equation

$$\text{Bayticol P Acid} + \text{SOCl}_2 \rightarrow \text{Bayticol P Acid Chloride} + \text{SO}_2 + \text{HCl}$$



Raw Materials

- **Bayticol P Acid** – Primary reactant
- **Thionyl Chloride ($SOCl_2$)** – Chlorinating agent
- **Toluene** – Solvent
- **Dimethylformamide (DMF)** – Catalytic promoter
- **Water** – Used for neutralization
- **Sodium Hydroxide**– Neutralizes acidic by-products

Reaction Conditions and Lab-Scale Preparation

- **Reaction Setup:** In a **round-bottom flask**, add **toluene** as the reaction medium.
- **Addition of Reagents:** Slowly introduce **Bayticol P Acid and thionyl chloride** while stirring under a nitrogen atmosphere to prevent unwanted oxidation.
- **Catalysis:** Add a small amount of **DMF** to promote chlorination and accelerate the reaction rate.
- **Heating:** Reflux the mixture at **80–100°C** until gas evolution ceases, ensuring complete conversion of the acid to the acid chloride.

- **Solvent Removal:** Distill off excess **$SOCl_2$** and recover **toluene** for reuse, minimizing solvent waste.
- **Product Isolation:** The **Bayticol P Acid Chloride** is obtained via **vacuum distillation** to enhance purity and yield.

Separation & Purification Steps

- **Solvent Recovery: Toluene is distilled and reused** to optimize process economics.
- **Gas Neutralization: SO_2 and HCl are scrubbed using an aqueous NaOH solution**, preventing atmospheric pollution.
- **Purification:** Excess reagents and impurities are removed via **vacuum distillation**, ensuring a highly pure acid chloride with minimal residual solvents.

Yield and Purity

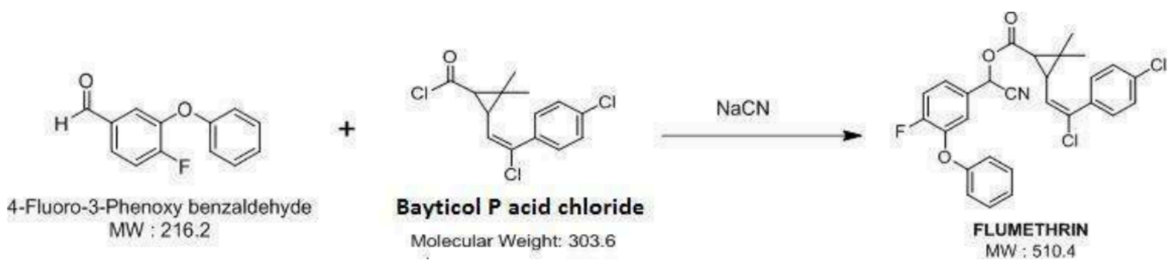
- **Estimated Yield:** ~95%
- **Purity: >98%**, ensuring minimal side product formation and impurity accumulation.

Step 2: Preparation of Flumethrin

Bayticol P Acid Chloride undergoes **nucleophilic substitution** with **sodium cyanide (NaCN) and 4-fluoro-3-phenoxybenzaldehyde** in **toluene**, yielding **Flumethrin**. A **phase-transfer catalyst, tetrabutylammonium bromide (TBAB),** enhances reaction efficiency by improving the solubility of reactants in the organic phase.

Chemical Equation

$$\text{Bayticol P Acid Chloride} + \text{NaCN} + \text{4-Fluoro-3-Phenoxybenzaldehyde} \rightarrow \text{Flumethrin} + \text{By-products}$$



Raw Materials

- **Bayticol P Acid Chloride** – Primary reactant

- **Sodium Cyanide (NaCN)** – Nucleophilic reactant
- **4-Fluoro-3-Phenoxybenzaldehyde** – Key aromatic component
- **Toluene** – Solvent
- **Sodium Bisulfite** – Used for impurity removal
- **Water**– Facilitates separation
- **Tetrabutylammonium Bromide (TBAB)** – Phase-transfer catalyst

Reaction Conditions and Lab-Scale Preparation

- **Reaction Setup:** In a **reaction vessel**, prepare a **toluene** solution under an inert nitrogen atmosphere to prevent unwanted oxidation.
- **Addition of Reagents:** Introduce **Bayticol P Acid Chloride, sodium cyanide, and 4-Fluoro-3-Phenoxybenzaldehyde**, ensuring slow addition to prevent uncontrolled exothermic reactions.
- **Catalyst Addition:** Add **TBAB** to facilitate **phase transfer**, allowing efficient interaction between reactants in different solvent phases.
- **Temperature Control:** Maintain the reaction at **50–70°C**, as excessive heating may cause decomposition or undesired by-product formation.

- **Filtration:** Remove **insoluble impurities and by-products**, including excess sodium cyanide.
- **Solvent Recovery: Toluene is distilled and recycled**, significantly reducing overall process costs.

- **Final Purification:** Perform **aqueous washing and crystallization**, followed by controlled drying to obtain **high-purity Flumethrin**.

Separation & Purification Steps

- **Solvent Recovery: Toluene is distilled and reused**, ensuring minimal waste and reducing solvent costs.
- **Aqueous Extraction:** Unreacted **sodium cyanide** and other polar impurities are removed via a **multi-stage aqueous wash** using sodium bisulfite.
- **Filtration & Crystallization:** Crude **Flumethrin is purified through recrystallization**, ensuring high yield and purity.

Yield and Purity

- **Estimated Yield:** ~90%
- **Purity:** ~90–100%, depending on the efficiency of purification and solvent removal.

We conducted an in-depth literature review for alternative synthesis routes of Flumethrin but did not find any feasible or well-documented method beyond the established process.

References:

https://environmentclearance.nic.in/writereaddata/Online/TOR/10_Feb_2022_18360085055790452AdditionalDocuments.pdf? **Page 47-48 (Flumethrin)**

https://www.benchchem.com/product/b136387

https://biologyinsights.com/flumethrin-structure-action-synthesis-and-application-techniques/

List the contributions of each author:

Minshul Agrawal - Researched the primary synthesis method of Flumethrin, studying reaction mechanisms, key reagents, and factors affecting yield and purity. Analyzed research articles and patents to understand reaction conditions, solvent recovery techniques, and purification steps. Examined the role of catalysts and temperature control in optimizing the synthesis process and ensuring efficient product formation.

Akshat Swarup - Studied research articles and worked on the Lab scale preparations and purification steps.

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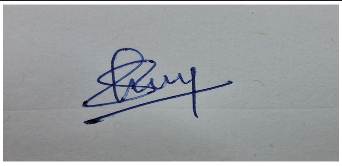
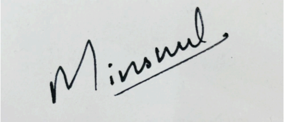
Kumar Shubham	230587	
Minshul Agrawal	230652	
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