CHE261 PROCESS ANALYSIS

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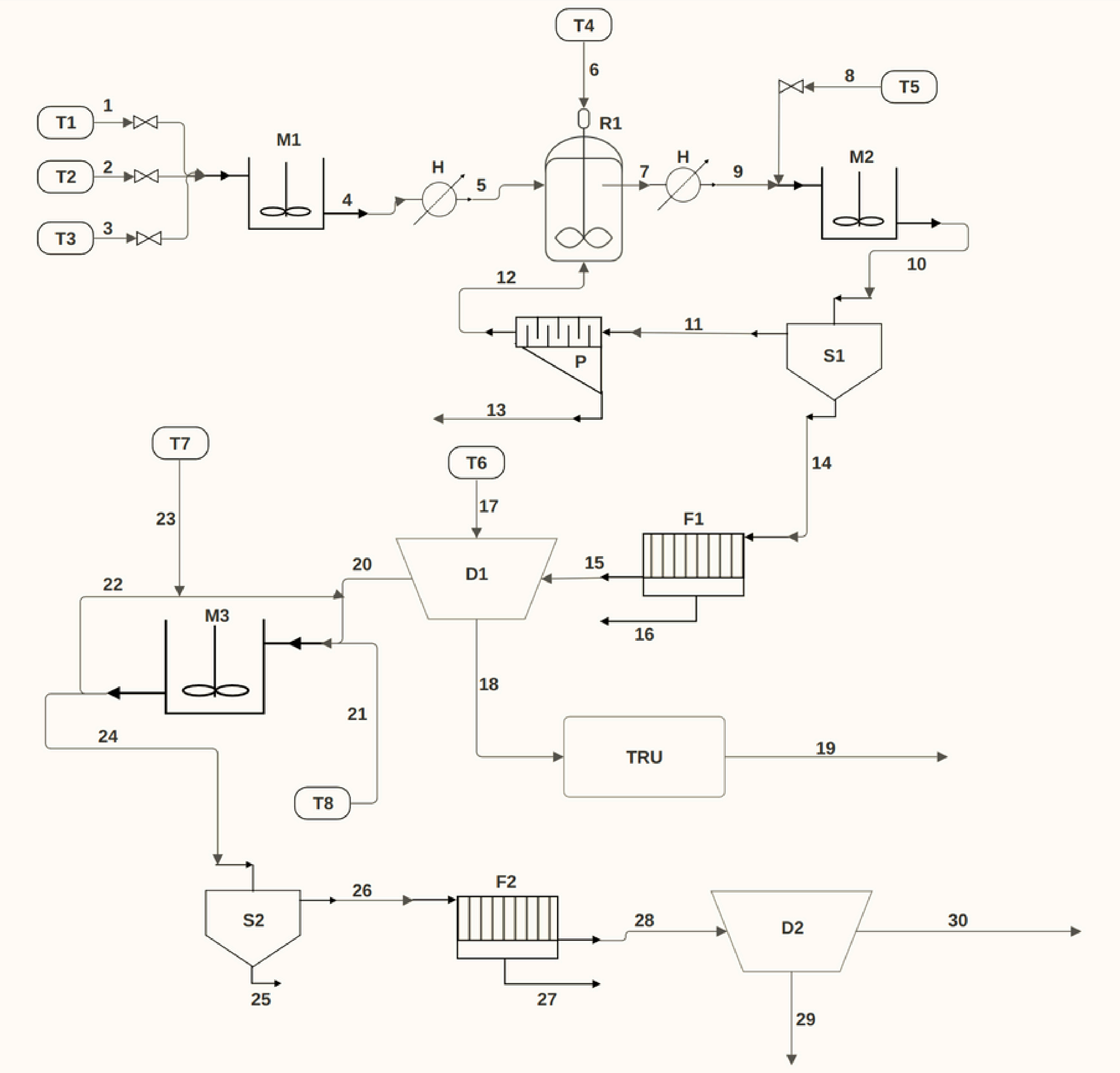
CHEMICAL FORMULA $C_6H_4Cl_2FN_2O_3$

CHEMICAL NAME Fluroxypyr

PROCESS TITLE Synthesis of Fluroxypyr

a) Block Diagram



1. Equipment List & Nomenclature

- T1: 4-Amino-3,5-dichloro-2,6-difluoropyridine
- T2: TBAI (Tetrabutylammonium Iodide) + Potassium Carbonate (K_2CO_3)
- T3: Toluene
- T4: Ethyl Glycolate
- T5: Cold Water
- T6: Na_2SO_4 (Dehydrating agent)
- T7: NaOH Solution
- T8: HCl (Acidification Step)
- M1: Mixer
- R1: Batch Reactor (Reacting Vessel with Stirrer & Heater)
- M2: Jacketed Cooling Mixer
- S1: Two-Layer Liquid Separator
- P: Precipitate Separator (Ouput feed Treatment Unit)
- F1: Nutsche Filter
- D1: Vaccum Dryer
- M3: Jacketed Mixing Vessel
- S2: Two-Layer Liquid Seperator
- F2: Nutsche Filter
- D2: Vaccum Dryer
- TRU: Treatment and Recovery Unit
- H: Heat Exchanger (Cooling)

2. Process Stream List & Nomenclature

- 1: 4-Amino-3,5-dichloro-2,6-difluoropyridine
- 2: TBAI (Tetrabutylammonium Iodide) + Potassium Carbonate (K_2CO_3)
- 3: Toluene (Solvent Stream)
- 4: Mixed Feed Entering Reaction Vessel (R1)
- 5: Pre-Heated Feed Mixture
- 6: Dropwise Ethyl Glycolate Addition (T4)
- 7: Reaction Mixture: Ester + Byproducts
- 8: Cold Water Stream (T5)
- 9: Cooled Reaction Mixture
- 10: Stream Output from Cooling Mixer M2
- 11: Aqueous Phase component of Stream 10
- 12: Recycled Catalyst Stream(TBAI + K_2CO_3)
- 13: Waste Water Stream
- 14: Organic Phase component of Stream 10
- 15: Filtered Organic Phase Component
- 16: Discarded waste stream
- 17: Na_2SO_4 to remove any residual water
- 18: Evapourated Toluene stream sent for recovery
- 19: Recovered Toulene pumped back to stream 3 as Input
- 20: Dry Fluroxypyr Ethyl Ester
- 21: NaOH (1M) Solution
- 22: Fluroxypyr-Sodium Salt
- 23: Dropwise HCl Addition (Acid Precipitation)
- 24: Fluroxypyr
- 25: Ethanol Removed
- 26: Fluroxypyr in water
- 27: Impurities and unreacted ester removed
- 28: Fluroxypyr + Water
- 29: Waste Water
- 30: Fluroxypyr (>95% Purity)

b) Unit Operations and Process Conditions

1. Raw Material Handling and Feeding

- Storage Tanks (Tanks-1 to Tanks-7)
  - Used for storing reactants, solvents, and catalysts.
- Pumps
  - Transfers raw materials from storage tanks to reactors.
- Flow Control Valves
  - Regulates feed flow into reactors.

2. Reaction Stages

- Reaction 1: Esterification (Ester Intermediate Synthesis)

$$C_5HCl_2F_2N_2 + C_4H_8O_3 \rightarrow C_9H_8Cl_2F_2N_2O_3 + H_2O$$

  - Batch Reactor (Jacketed & Agitated)
    - Reaction Type: Nucleophilic Substitution (Esterification)
    - Temperature: 80–100°C
    - Pressure: Atmospheric
    - Residence Time: 10–12 hours
    - Reactants:
      - 4-Amino-3,5-dichloro-2,6-difluoropyridine
      - Ethyl Glycolate
    - Solvent: Toluene
    - Catalysts:
      - Tetrabutylammonium iodide (Phase Transfer Catalyst)
      - Potassium Carbonate (K_2CO_3) (Acid-Scavenger/Base)
    - Byproducts: Water

- Reaction 2: Hydrolysis (Fluroxypyr Sodium Salt Formation)

$$C_9H_8Cl_2F_2N_2O_3 + NaOH \rightarrow C_7H_6Cl_2F_2N_2O_3Na + C_2H_5OH$$

  - Jacketed Mixing Vessel (Agitated)
    - Reaction Type: Hydrolysis
    - Temperature: 90°C
    - Pressure: Atmospheric
    - Residence Time: 2 hours
    - Reactants: Fluroxypyr Ethyl Ester + NaOH (1M Solution)
    - Solvent: Water
    - Byproducts: Ethanol

- Reaction 3: Acidification (Fluroxypyr Formation)

$$C_7H_6Cl_2F_2N_2O_3Na + HCl/H_2SO_4 \xrightarrow{\text{pH } 1\text{–}2} C_7H_6Cl_2F_2N_2O_3 + NaCl/Na_2SO_4$$

  - Jacketed Mixing Vessel (Agitated)
    - Reaction Type: Acid-Base Neutralization
    - Temperature: ~25–30°C (Ambient)
    - Pressure: Atmospheric
    - Residence Time: ~30 minutes
    - Reactants: Fluroxypyr Sodium Salt + Dilute HCl / H_2SO_4
    - Solvent: Water
    - Byproducts: NaCl / Na_2SO_4 (depending on acid used)

3. **Post-Reaction Processing**

- Cooling & Phase Separation
  - Jacketed Cooling Mixer (Mixer + Heat Exchanger)
    - Function: Cools down the reaction mixture to facilitate phase separation.
    - Cooling Medium: Chilled water or glycol.
    - Exit Temperature: ~25–30°C.

  - Two-Layer Separator
    - Function: Separates organic and aqueous phases.
    - Organic Phase: Contains Fluroxypyr Ethyl Ester.
    - Aqueous Phase: Removed to eliminate impurities and catalyst residues.

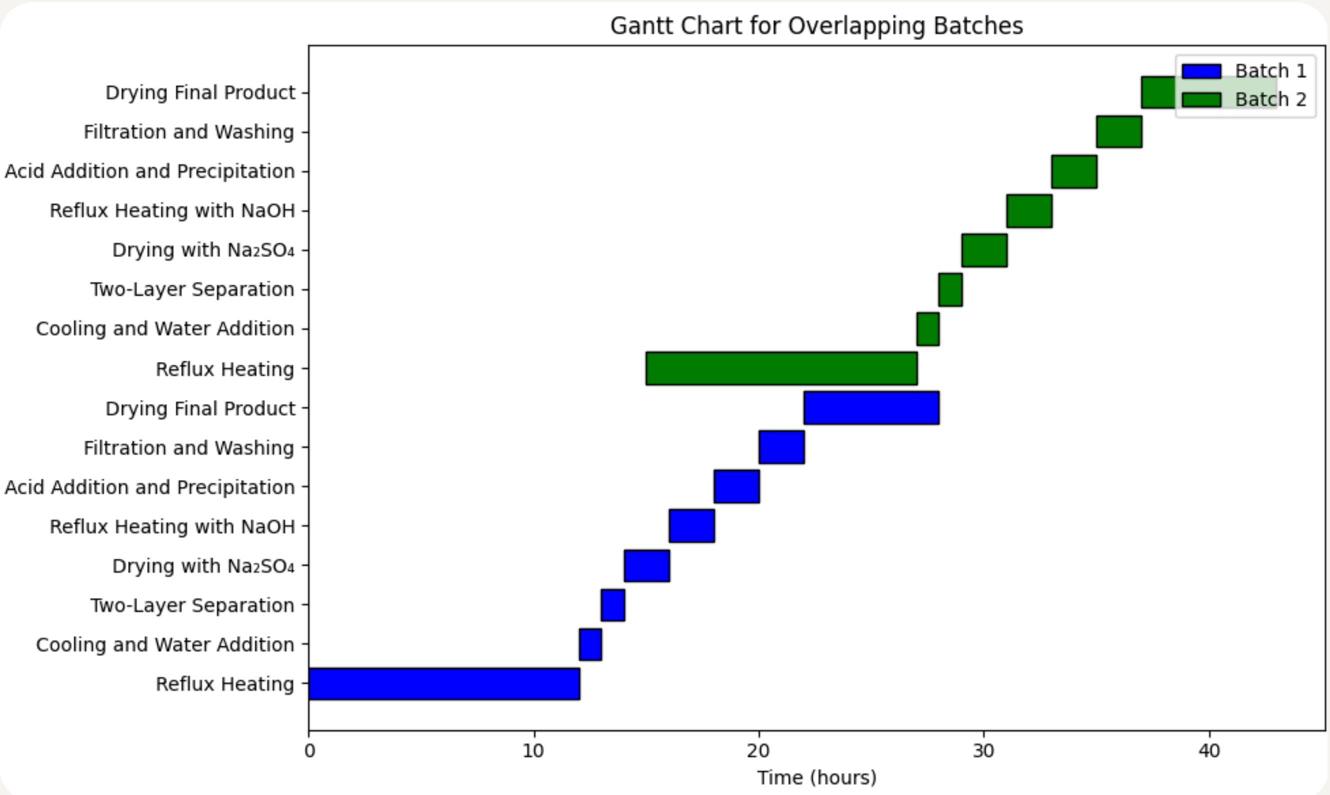
- Purification & Drying
  - Nutsche Filter
    - Type: Solid-Liquid Separation.
    - Purpose: Isolates the Fluroxypyr Ethyl Ester (after phase separation) and Fluroxypyr final product (after hydrolysis & acidification).
  - Vacuum Tray Dryer
    - Type: Vacuum Tray or Rotary Dryer.
    - Vacuum Level: 50–100 mmHg.
    - Purpose: Removes residual solvents (toluene, ethanol, water) from the isolated product.

Optimization of Filtration & Drying

Initially, the plan was to reuse the same Nutsche filter and vacuum dryer for both Fluroxypyr Ethyl Ester and the final Fluroxypyr product. However, after creating a Gantt chart, we realized this caused a major bottleneck in the process.

Since drying takes 6 hours, by the time the first batch finished drying, the next batch was already ready for filtration. This meant the second batch had to wait, delaying the overall workflow.
To solve this, we added a second Nutsche filter and vacuum dryer, allowing:

- Parallel processing of two batches at a time
- No idle time between filtration and drying
- Increased throughput without delaying subsequent batches



c) Material Balance

Mass Balance for the Reaction Process

Step 1: Iteration for Toluene Volume Selection

The reaction requires 4-Amino to be dissolved in toluene. The solubility of 4-Amino in toluene is given as

$$0.1\,\frac{\text{kg}}{\text{L}}.$$

Since we have a maximum reactor volume constraint of 10,500 L, we iteratively estimated the appropriate toluene volume:

- **Chosen Toluene Volume:** 9,500 L
- **Maximum Dissolved 4-Amino:**

$$9,500\,\text{L} \times 0.1\,\frac{\text{kg}}{\text{L}} = 950\,\text{kg}$$

Moles of 4-Amino: To determine the amount in moles, we use the molar mass of 4-Amino (198.99 g/mol):

$$\frac{950\,\text{kg} \times 1000\,\frac{\text{g}}{\text{kg}}}{198.99\,\frac{\text{g}}{\text{mol}}} \approx 4,774.16\,\text{mol}.$$

Thus, the number of moles of 4-Amino in the reaction is approximately 4,774.16 mol.

Step 2: Determining Other Reactants Using Stoichiometric Ratios

Ethyl Glycolate (1.1 Equivalents of 4-Amino):
The reaction requires 1.1 equivalents of Ethyl Glycolate relative to 4-Amino:

$$4,774.16 \times 1.1 \approx 5,251.58\,\text{mol}.$$

Mass of Ethyl Glycolate: (Molar Mass = 104.06 g/mol)

$$\frac{5,251.58 \times 104.06}{1000} \approx 546.5\,\text{kg}.$$

Volume of Ethyl Glycolate: (Density = 1.04 kg/L)

$$\frac{546.5}{1.04} \approx 525.2\,\text{L}.$$

Potassium Carbonate (K_2CO_3) (1.2 Equivalents of 4-Amino):

$$4,774.16 \times 1.2 \approx 5,729.00\,\text{mol}.$$

Mass of K_2CO_3: (Molar Mass = 138.21 g/mol)

$$\frac{5,729.00 \times 138.21}{1000} \approx 791.3\,\text{kg}.$$

Volume of K_2CO_3: (Density = 1.89 kg/L)

$$\frac{791.3}{1.89} \approx 419.7\,\text{L}.$$

Tetrabutylammonium Iodide (TBAI) (5 mol% Catalyst):

$$4,774.16 \times 0.05 \approx 238.71\,\text{mol}.$$

Mass of TBAI: (Molar Mass = 369.37 g/mol)

$$\frac{238.71 \times 369.37}{1000} \approx 88.2\,\text{kg}.$$

c) Material Balance

Volume of TBAI: (Density = 1.52 kg/L)

$$\frac{88.2}{1.52} \approx 58.0\,\text{L}.$$

Total Volume Before Reaction:

$$9,500\,(\text{Toluene}) + 525.2\,(\text{Ethyl Glycolate}) + 419.7\,(\text{K}_2\text{CO}_3) + 58.0\,(\text{TBAI}) \approx 10,502.9\,\text{L}.$$

Since this is within the 10,500 L limit, the reaction setup is valid.

Step 3: Reaction Progress and Yield Calculation

The reaction proceeds with a 92% yield, meaning that 92% of 4-Amino reacts completely, while 8% remains unreacted.

Reacted 4-Amino (92%):

$$4,774.16 \times 0.92 \approx 4,392.23\,\text{mol}.$$

Unreacted 4-Amino (8%):

$$4,774.16 \times 0.08 \approx 381.93\,\text{mol}.$$

Mass of Unreacted 4-Amino:

$$\frac{381.93 \times 198.99}{1000} \approx 75.9\,\text{kg}.$$

Ethyl Glycolate Consumption:

Since Ethyl Glycolate is 1.1 equivalents, the amount that reacts is:

$$5,251.58 \times 0.92 \approx 4,832.65\,\text{mol}.$$

Thus, the unreacted Ethyl Glycolate is:

$$5,251.58 - 4,832.65 \approx 418.93\,\text{mol}.$$

Mass of Unreacted Ethyl Glycolate:

$$\frac{418.93 \times 104.06}{1000} \approx 43.7\,\text{kg}.$$

Volume of Unreacted Ethyl Glycolate:

$$\frac{43.7}{1.04} \approx 42.0\,\text{L}.$$

Fluroxypyr Ethyl Ester Formed (92%):

Since 4-Amino fully converts to product:

$$\text{Product Formed} \approx 4,392.23\,\text{mol}.$$

Mass of Fluroxypyr Ethyl Ester: (Molar Mass = 314.66 g/mol)

$$\frac{4,392.23 \times 314.66}{1000} \approx 1,382.0\,\text{kg}.$$

Volume of Fluroxypyr Ethyl Ester: (Density = 1.14 kg/L)

$$\frac{1,382.0}{1.14} \approx 1,210.4\,\text{L}.$$

Water Produced:

Water formed equals the moles of reacted 4-Amino:

$$\text{Water Formed} \approx 4,392.23\,\text{mol}.$$

c) Material Balance

Mass of Water: (Molar Mass = 18.02 g/mol)

$$\frac{4,392.23 \times 18.02}{1000} \approx 79.1\,\text{kg}.$$

Volume of Water: (Density = 1.00 kg/L)

$$\frac{79.1}{1.00} \approx 79.1\,\text{L}.$$

Updated Calculation for Excess K_2CO_3:

After correcting the calculation, the excess moles of K_2CO_3 is 1,336.77 mol instead of 1,000 mol.

- **Mass of Excess K_2CO_3:**

$$\frac{1,336.77 \times 138.21}{1000} \approx 184.75\,\text{kg}.$$

- **Volume of Excess K_2CO_3:**

$$\frac{184.75}{1.89} \approx 97.75\,\text{L}.$$

Step 4: Updated Mass Balance Tables

Before Reaction

Component	Moles (mol)	Mass (kg)	Volume (L)
4-Amino	4,774.16	950.0	–
Ethyl Glycolate (1.1 eq)	5,251.58	546.5	525.2
K_2CO_3 (1.2 eq)	5,729.00	791.3	419.7
TBAI	238.71	88.2	58.0
Toluene	–	–	9,500.0
Total	–	2,376.0	10,502.9

Table 1: Mass Balance Before Reaction

After Reaction (Updated)

Component	Moles (mol)	Mass (kg)	Volume (L)
Unreacted 4-Amino	381.93	75.9	–
Unreacted Ethyl Glycolate	418.93	43.7	42.0
Fluroxypyr Ethyl Ester	4,392.23	1,382.0	1,210.4
Water Produced	4,392.23	79.1	79.1
Excess K_2CO_3 (Updated)	1,336.77	184.75	97.75
TBAI	238.71	88.2	58.0
Toluene	–	–	9,500.0
Total	–	1,853.65	10,493.7

Table 2: Mass Balance After Reaction (Updated)

c) Material Balance

Mixer

Step 5: Final Mass Balance Before and After Mixing

Before Mixing (Mass Before Adding Water)

Component	Mass (kg)	Volume (L)	Moles (mol)
Unreacted 4-Amino	75.9	–	381.93
Unreacted Ethyl Glycolate	43.7	42.0	418.93
Fluroxypyr Ethyl Ester	1,382.0	1,210.4	4,392.23
Water (Produced in Reaction)	79.1	79.1	4,392.23
Excess K_2CO_3	184.75	97.75	1,336.77
TBAI (Catalyst)	88.2	58.0	238.71
Toluene (Solvent) (Corrected)	8,227.0	9,500.0	–
Total Before Mixing	10,080.65	10,487.25	–

Table 1: Before Mixing (Mass Before Adding Water)

Added Water for Separation

Component	Mass (kg)	Volume (L)	Moles (mol)
Water Added	4,750.0	4,750.0	263,888.9

Table 2: Added Water for Separation

Updated Total Mass & Volume After Mixing:

$$\text{Total Mass After Mixing} = 10,080.65 + 4,750 = 14,830.65\,\text{kg},$$
$$\text{Total Volume After Mixing} = 10,487.25 + 4,750 = 15,237.25\,\text{L}.$$

After Mixing: Partition into Water Phase & Organic Phase

Water Phase (Aqueous Layer) — Contains Water-Soluble Components

Component	Mass (kg)	Volume (L)	Moles (mol)
Water (Added + Produced)	$4,750.0 + 79.1 = 4,829.1$	4,829.1	268,281.13
Excess K_2CO_3	184.75	97.75	1,336.77
TBAI	88.2	58.0	238.71
Unreacted Ethyl Glycolate	43.7	42.0	418.93
Total Water Phase	5,145.75	5,026.85	–

Table 3: Water Phase (Aqueous Layer)

c) Material Balance

Component	Mass (kg)	Volume (L)	Moles (mol)
Toluene (Solvent) (Corrected)	8,227.0	9,500.0	–
Fluroxypyr Ethyl Ester (Product)	1,382.0	1,210.4	4,392.23
Unreacted 4-Amino	75.9	–	381.93
Total Organic Phase	9,684.9	10,710.4	–

Table 4: Organic Phase (Toluene Layer)

Organic Phase (Toluene Layer) — Contains Non-Water-Soluble Components

Final Mass Balance Check:

$$5,145.75\,\text{kg (Water Phase)} + 9,684.9\,\text{kg (Organic Phase)} = 14,830.65\,\text{kg}$$

Mass is correctly balanced!

Final Volume Check:

- Water Phase: 5,026.85 L
- Organic Phase: 10,710.4 L
- **Total After Mixing:** 15,237.25 L

Final Validation:

- Corrected total mass after adding water: 14,830.65 kg
- Ethyl Glycolate now correctly assigned to the water phase
- Clear phase separation into aqueous and organic layers
- Volume remains within operational limits

c) Material Balance

Two-Layer Separator

Adjusting for Partial Recovery

Since the recovery of K_2CO_3 and TBAI is not 100%, we need to factor in losses during the recovery process.

Assumptions for Partial Recovery

- K_2CO_3 Recovery Efficiency: 80% recovered, 20% lost.
- TBAI Recovery Efficiency: 85% recovered, 15% lost.

Updated Mass Balance After Partial Recovery

Recovered Components from Water Phase

Component	Initial Mass (kg)	Recovery Efficiency (%)	Recovered Mass (kg)	Lost to Waste (kg)
Excess K_2CO_3	184.75	80	147.8	36.95
TBAI	88.2	85	75.0	13.2
Total Recovered	272.95	–	222.8	50.15

Table 1: Recovered Components from Water Phase

Updated Water Phase (Sent to Waste)

Component	Mass Before Recovery (kg)	Mass Lost to Waste (kg)
Water (Added + Produced)	4,829.1	4,829.1
K_2CO_3 (20% lost)	184.75	36.95
TBAI (15% lost)	88.2	13.2
Unreacted Ethyl Glycolate	43.7	43.7
Total Waste Water	5,145.75	4,922.95

Table 2: Updated Water Phase Sent to Waste

Updated Organic Phase (Retained for Further Processing)

Component	Mass (kg)	Volume (L)	Moles (mol)
Toluene (Solvent) (Corrected)	8,227.0	9,500.0	–
Fluroxypyr Ethyl Ester (Product)	1,382.0	1,210.4	4,392.23
Unreacted 4-Amino	75.9	–	381.93
Total Organic Phase (Retained for Purification)	9,684.9	10,710.4	–

Table 3: Updated Organic Phase Retained for Further Processing

c) Material Balance

Phase	Mass Before Recovery (kg)	Recovered (kg)	Waste (kg)
Water Phase	5,145.75	222.8	4,922.95
Organic Phase	9,684.9	–	–
Total	14,830.65	–	–

Table 4: Final Mass Balance After Partial Recovery

Final Mass Balance After Partial Recovery

Notes:
Organic phase mass remains unchanged. Recovered catalysts (222.8 kg) can be reused in future reactions. 50.15 kg of catalyst loss must be accounted for in waste disposal.

Next Step: Final Purification of Organic Phase
Now that we've accounted for partial catalyst recovery, the organic phase (Toluene + Product + Unreacted 4-Amino) will be purified to isolate Fluroxypyr Ethyl Ester.

Filter Dryer

Overview

This mass balance details the separation, drying, and recovery process to obtain pure Fluroxypyr Ethyl Ester, while ensuring proper handling of byproducts and waste.

Step 1: Initial Organic Phase Before Drying

After water phase separation, the organic phase contains non-water-soluble components:

Component	Mass (kg)	Volume (L)	Moles (mol)	Notes
Toluene (Solvent)	8,227.0	9,500.0	–	To be recovered
Fluroxypyr Ethyl Ester (Product)	1,382.0	1,210.4	4,392.23	Target product
Unreacted 4-Amino	75.9	–	381.93	Small residue
Residual Water	79.1	79.1	4,392.23	Needs removal
Total Organic Phase	9,684.9	10,789.5	–	Before drying

Table 5: Initial Organic Phase Before Drying

Step 2: Sodium Sulfate (Na_2SO_4) Addition for Water Removal

To remove 79.1 kg of residual water, we add Na_2SO_4 (50% w/w absorption capacity).

Component	Mass (kg)	
Na_2SO_4 Added	39.55	
Hydrated $Na_2SO_4 \cdot 10H_2O$	118.65	(Forms solid waste)
Residual Water Removed	79.1	(Fully absorbed)

Table 6: Sodium Sulfate Addition for Water Removal

Mass After Na_2SO_4 Addition:

c) Material Balance

Component	Mass (kg)	Total Mass (kg)
Organic Phase (Before)	9,684.9	–
Added Na_2SO_4	39.55	–
Total Before Filtration	9,724.45	

Table 7: Mass After Na_2SO_4 Addition

Step 3: Filtration – Removing Hydrated Na_2SO_4

Filtration removes the solid hydrated Na_2SO_4 and the absorbed 79.1 kg water.

Component Removed	Mass (kg)	
Hydrated $Na_2SO_4 \cdot 10H_2O$	118.65	(Filtered out as solid waste)
Residual Water	79.1	(Completely removed)
Total Waste Removed	118.65	

Table 8: Filtration Process

Mass After Filtration:

Component	Mass (kg)	Total Mass (kg)
Before Filtration	9,724.45	–
After Filtration	9,605.8	–

Table 9: Mass After Filtration

Step 4: Vacuum Drying – Removing Toluene

Objective: Recover toluene by evaporation, leaving behind the final dry product.

Component Removed	Mass (kg)	
Toluene (Recovered)	8,227.0	(Recycled for next batch)

Table 10: Vacuum Drying Process

Mass After Drying:

Component	Mass (kg)	Total Mass (kg)
After Filtration	9,605.8	–
After Drying (Final Product)	1,378.8	–

Table 11: Mass After Drying (Final Product)

Step 5: Final Output and Recovery

Final Mass Balance Validation

Notes:

c) Material Balance

Component	Mass (kg)	Recovery (%)
Fluroxypyr Ethyl Ester (Final Product)	1,378.8	99.7
Toluene (Recovered for Reuse)	8,227.0	100
Hydrated Na_2SO_4 + Water Waste	118.65	–

Table 12: Final Output and Recovery

Initial Mass (Organic Phase + Na_2SO_4)	9,724.45 kg
Final Mass (Product + Recovered + Waste)	9,724.45 kg

Table 13: Final Mass Balance Validation

- Water fully removed using Na_2SO_4.
- Toluene completely recovered for reuse.
- High-purity Fluroxypyr Ethyl Ester obtained (99.7% yield).
- Minimal process losses (only hydrated Na_2SO_4 waste).

Hydrolysis, Acidification of Fluroxypyr Ethyl Ester and Final Separation of Fluroxypyr

1. Process Overview

Reaction:

Fluroxypyr ethyl ester reacts with NaOH to form Fluroxypyr sodium salt and ethanol. The sodium salt is then acidified to form Fluroxypyr.

Step 1: Hydrolysis

$$\text{Fluroxypyr-Ester} + \text{NaOH} \rightarrow \text{Fluroxypyr-Na (Product)} + \text{Ethanol (Byproduct)}$$

Step 2: Acidification

$$\text{Fluroxypyr-Na} + \text{HCl} \rightarrow \text{Fluroxypyr} + \text{NaCl}$$

Step 3: Separation

The final product, Fluroxypyr, is separated from the reaction mixture using a Nutsche filter followed by a vacuum dryer.

- NaOH is taken in excess to ensure complete conversion.
- Hydrolysis reaction yield = 98% to achieve an overall process yield of 90%.
- No mass loss occurs, only conversion.

2. Input Mass Calculation

3. Yield and Output Calculation

Step 1: Hydrolysis Output Calculation

Step 2: Acidification Output Calculation

c) Material Balance

Component	Mass (kg)	Moles (mol)	Notes
Fluroxypyr ethyl ester	1,382.0	4,392.23	Limiting reagent
NaOH (Excess)	189.99	4,750.0	Base for reaction
Water in NaOH solution	4,559.76	–	Water component
HCl (for acidification)	149.3	4,093.2	Required for conversion
Total Input	6,280.06	–	

Table 14: Input Mass Calculation

Component	Mass (kg)	Calculation	Notes
Fluroxypyr sodium salt	1,250.0	$4,392.23 \times 0.98 \times 290.58/1000$	Product (98% yield)
Ethanol	198.3	$4,392.23 \times 0.98 \times 46.07/1000$	Byproduct
Unreacted Ester	27.6	$1,382.0 \times 0.02$	2% Residual
Excess NaOH	17.8	$(4,750.0 - 4,304.38) \times 40/1000$	Unreacted Base

Table 15: Hydrolysis Output Calculation

Component	Mass (kg)	Calculation	Notes
Fluroxypyr	1,176.4	$1,250.0 \times 290.58/308.58$	Final Product
NaCl	73.6	$1,250.0 \times 58.44/308.58$	Byproduct

Table 16: Acidification Output Calculation

Total Output:

Total Output Mass (kg)	1,721.3	(Sum of all outputs)

Table 17: Total Output Mass from Hydrolysis and Acidification

Step 3: Separation Output Calculation (Post Nutsche Filter and Vacuum Dryer)

After the acidification step, Fluroxypyr is separated from the reaction mixture using a Nutsche filter and then dried in a vacuum dryer to obtain pure Fluroxypyr.

Component	Mass (kg)	Calculation	Notes
Fluroxypyr (Pure)	1,176.4	*Same as from acidification step*	Final Product
Residual NaCl	73.6	*Same as from acidification step*	Byproduct
Total Output After Separation	1,176.4	(Sum of products and byproducts post-separation)	

Table 18: Separation Output Calculation

4. Mass Balance Validation

The mass balance confirms that all input mass is accounted for within the output components.

5. Key Observations

- 98% Hydrolysis Conversion Efficiency: Minimal unreacted ester (2% remains).

c) Material Balance

Total Input	6,280.06 kg
Total Output	6,280.06 kg
Discrepancy	0 kg (No mass loss)

Table 19: Mass Balance Validation

- Acidification Completes the Process: HCl fully reacts with Fluroxypyr sodium salt.
- Separation with Nutsche Filter: Effectively isolates Fluroxypyr from the mixture.
- No Mass Discrepancy: All input materials are converted into products/byproducts.
- Water Included in NaOH Solution: Water content has been accounted for to ensure the mass balance is correct.

6. Final Observations

- Hydrolysis Yield: 98%, consistent with patent CN106187872A.
- Acidification Completeness: HCl fully neutralizes sodium salt (pH 1–2).
- Effective Separation: The Nutsche filter and vacuum dryer allow for the isolation of pure Fluroxypyr.

Conclusion

The mass balance integrates the separation process with the Nutsche filter and vacuum dryer. All mass is accounted for with no loss in the system.

Reactor Sizing and Cost Estimation

Reactor Capacity Calculation

- The required reactor volumes are calculated using mass balance data and an densities of all the components. The actual reactor design capacity is determined considering only 70% of the total volume can be utilized.

- **Stage 1: Batch Reactor (Input)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine	950	~1.6	Dissolved in Toluene
Toluene	8265	~0.87	9500
Ethyl Glycolate	546	~1.04	525.48
K_2CO_3	791.3	~1.89	418.68
TBAI	88.2	~1.52	58.03
Total Volume			10,502.16

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 10,502.16/0.7
  = 15003.08 L

<u>Reactor Sizing and Cost Estimation</u>

- **Stage 1: Batch Reactor (Output)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	Dissolved in Toluene
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Toluene	8265	~0.87	9500
Ethyl Glycolate (unreacted)	43.7	~1.04	42
K_2CO_3 (unreacted)	184.75	~1.89	97.75
TBAI	88.2	~1.52	58.03
Water	79.1	~1	79.1
Total Volume			10,909.53

Note: TBAI and K_2CO_3 are soluble in the water produced, and Fluroxypyr Ethyl Ester is soluble in toluene. The actual total volume will be lower than the listed sum (~10,500 L) due to dissolution effects.

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 10,493.7/0.7
  = 14991 L

Reactor Sizing and Cost Estimation

- **Stage 2: Water addition and Mixing (Input)**

Volume Estimation

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	Dissolved in Toluene
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Toluene	8265	~0.87	9500
Ethyl Glycolate (unreacted)	43.7	~1.04	42
K_2CO_3 (unreacted)	184.75	~1.89	97.75
TBAI	88.2	~1.52	58.03
Water	79.1	~1	79.1
Total Volume			10,493.7

Note: An additional 4,750 L of water is added during this stage. This will increase the total volume of the reactor contents. The updated total volume, considering the water addition, will be:

$$\text{Total Volume} = 10{,}493.7\ \text{L} + 4{,}750\ \text{L} = 15{,}243.7\ \text{L}$$

Reactor Sizing and Cost Estimation

- **Stage 2: Water Addition and Mixing (Output)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	Dissolved in Toluene
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Toluene	8265	~0.87	9500
Total Volume	**Organic Phase**		~ 10,000

Water (Added + Produced)	79.1 + 4750	~1	4829.1
K_2CO_3 (unreacted)	184.75	~1.89	Dissolved in Water
Ethyl Glycolate (unreacted)	43.7	~1.04	Dissolved in Water
TBAI	88.2	~1.52	Dissolved in Water
Total Volume	**Aqeous Phase**		4829.1 L

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 15243.7/0.7
  = 21,776.7 L

Reactor Sizing and Cost Estimation

- **Stage 3: Two- Layer Seperator (Output)**

Volume Estimation:

Note for Two-Layer Separator Slide:

The two-layer separator efficiently divides the reaction mixture into:

- Aqueous Phase (4,829.1 L): This phase, containing **K_2CO_3**, **ethyl glycolate**, and **TBAI**, is directed to further treatment and catalyst recovery processes. The recovered K_2CO_3 can be recycled back to the process, improving economic efficiency.
- Organic Phase (≈10,000 L): This phase, containing the **Fluroxypyr Ethyl Ester** product and trace amounts of unreacted 4-Amino-3,5-dichloro-2,6-difluoropyridine dissolved in toluene, will be the focus of our subsequent analysis and purification steps.

The separation efficiency significantly impacts both product yield and purity in downstream processing.

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	381.93 (Dissolved in Toluene)
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Toluene	8265	~0.87	9500
Total Volume	**Organic Phase**		~ 10,000

Reactor Sizing and Cost Estimation

- **Stage 4: Nutsche Fliter and Vaccum dryer(Input)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	381.93 (Dissolved in Toluene)
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Toluene	8265	~0.87	9500
Total Volume	**Organic Phase**		~ 10,500

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 10500/0.7
  = 15,000 L

Reactor Sizing and Cost Estimation

- **Stage 4: Nutsche Fliter and Vaccum dryer(Output)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4(Partially dissolved in Toluene)
Total Mass	1382	**Dried Product**	

Component	Mass (kg)	Density (kg/L)	Volume (L)
4-Amino-3,5-dichloro-2,6-difluoropyridine (unreacted)	75.9	~1.67	381.93 (Dissolved in Toluene)
Toluene	8265	~0.87	9500
Trace Impurities	<5		-
Total Volume	**Recovery Stream**		~ 9,500

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 9500/0.7
  = 13,571 L

Reactor Sizing and Cost Estimation

- **Stage 5: Jacketed Mixing Vessel (Input)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
Fluroxypyr Ethyl Ester	1382	~1.22	1132.4
NaOH (1 M)	189.99	1.53	124.24
Water in NaOH	4559.6	1	4559.6
Total Volume			5896.28

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 5772.04/0.7
  = 8245.77 L

Reactor Sizing and Cost Estimation

- **Stage 5: Jacketed Mixing Vessel (Output)**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
Fluroxypyr Sodium Salt	1250	~1.18	1059.32
unreacted Ester	27.6	~1.14	24.21
NaOH (unreacted)	3.8	1.53	2.48
Acid (HCl)	149.3	1.18	126.53
Water in NaOH	4559.6	1	4559.6
Total Volume			5772.14

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 5772.04/0.7
  = 8245.77 L

Reactor Sizing and Cost Estimation

- **Stage 6: Filter and Dryer**

Volume Estimation:

Component	Mass (kg)	Density (kg/L)	Volume (L)
Fluroxypyr Sodium Salt	1250	~1.18	1059.32
By-products	4552 kg	–	~4500 + (dissolved in water)
Total Volume			5772.14

The dried Fluroxypyr Sodium Salt obtained after filtration and drying has a purity of >95%, ensuring it meets the required quality standards for further processing or use.

Reactor Volume Calculation:

- Total Reactor Volume = (Total Estimated Volume)/0.7
  = 5772.04/0.7
  = 8245.77 L

Reactor Sizing and Cost Estimation

Reactor Volume Estimation Table for all reactors:

Reactor	Estimated Volume (L)	Design Capacity (L) (Based on 70% usage)
Batch Reactor	10,502	15,003.3
Jacketed Mixing Vessel	5896.7	8,500
Jacketed Mixing Cooler	15243.7	21,776.7
2-Layer Seperator	15243.7	21,776.7
Nutsche Filter & Vaccum Dryer 1	10,500	15000
Nutsche Filter & Vaccum Dryer 2	5772.12	8245.77
Equipment for Treatment and recovery of discarded Feeds	Not Calculated	Not calculated

d) Reactor Sizing and Cost Estimation

Capital Cost Estimation

- The cost estimation is based on the Matche Equipment Cost Calculator and we are utilizing Jacketed and Agitated Reactor in each with material as Glass Lined CS and pressure set between atmospheric to 25 psi.

  Capital Cost Table:

Equipment	Design Capacity (Litres)	No. of Units	Cost/unit ($)
Batch Reactor	15,003.3	1	33,000
Jacketed Mixing Vessel	8,500	1	9,800
Jacketed Mixing Cooler	21,776.7	1	20,000
2-Layer Seperator	21,776.7	1	20,000
Nutsche Filter & Vaccum Dryer	15000	2	22,000

Total Cost: $ 1,268,700 = ₹ 1,08,49,008

References:

- https://patents.google.com/patent/CN104844574A/en
- https://www.matche.com/equipcost/Reactor.html

Contribution of each member

Adarsh Raj (230057)

- Developed the comprehensive Process Flow Diagram (PFD) incorporating all key unit operations—including reactors, washers, dryers, crystallizers, and heat exchangers.
- Defined critical process conditions (temperature, pressure, solvent recovery, waste management) to ensure efficient operation.
- Sized reactors to achieve 70% of total volume utilization for the reaction mixtures.
- Led the capital cost estimation, specifically analyzing costs for Glass-Lined Carbon Steel (GLCS) reactors.
- Prepared detailed Gantt charts to outline project timelines and milestones.
- Contributed to reactor costing and overall process economics.

Karan Keer (230531)

- Conducted a thorough material balance for the scaled-up process plant operating at 1000 kg/day.
- Determined input and output flow rates for all process streams and unit operations.
- Established robust assumptions for simplified yet precise mass flow tracking.
- Optimized reactant consumption and process efficiency to reduce material losses and improve yield.

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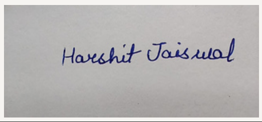
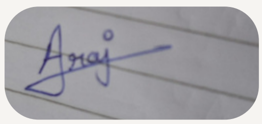
Harshit Jaiswal	230460	
Adarsh Raj	230057	
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