

Technical Team

Applicant: VortexChem

Inventors: Karmanya Goyal, Naveen Godara

Chemical Formula: C₁₄H₁₇Cl₂N₃O

Chemical Name: Hexaconazole

Process Title: Industrial Production of Hexaconazole

Raw materials and chemicals required: Meta Di Chloro Benzene (MDCB), Valeryl Chloride, Aluminium Chloride (AlCl₃), Water, Valerophenone, Dimethyl Sulfate (DMSO₄), Dimethyl Sulfide (DMS), Potassium Hydroxide (KOH), DMF (Dimethylformamide), Triazole, Methanol.

Process Description:

Hexaconazole is synthesized through a multi-step reaction involving alkylation, oxidation, and cyclization. The key steps are as follows

1. Alkylation Reaction (Step-1: Preparation of Valerophenone)

- **Reactants:** Meta Di Chloro Benzene (MDCB), Valeryl Chloride
- **Catalyst:** Aluminium Chloride
- **Reaction:** MDCB reacts with Valeryl Chloride in the presence of Aluminium Chloride as a catalyst to form Valerophenone.
- **Process Control:** The alum layer is separated, and Valerophenone is distilled to obtain the desired purity.

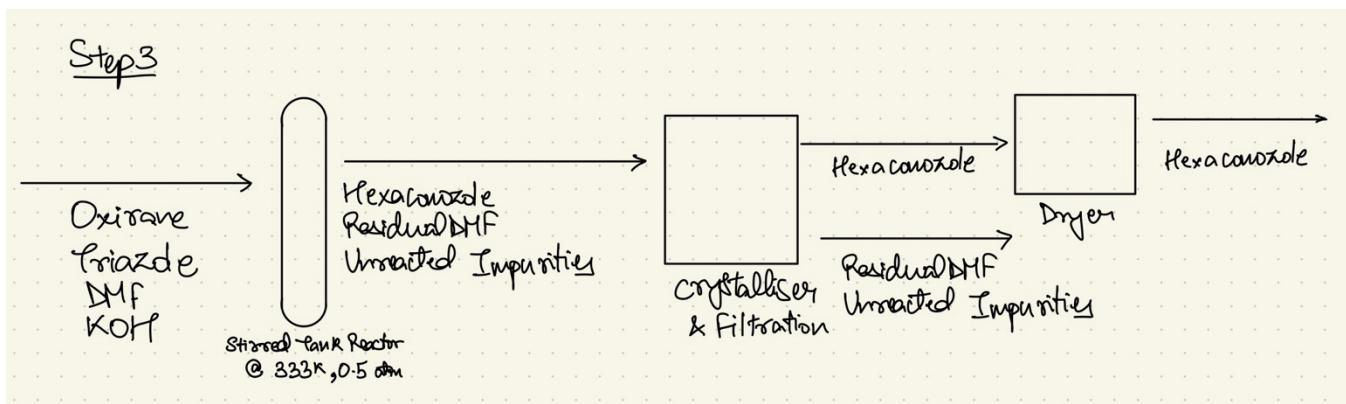
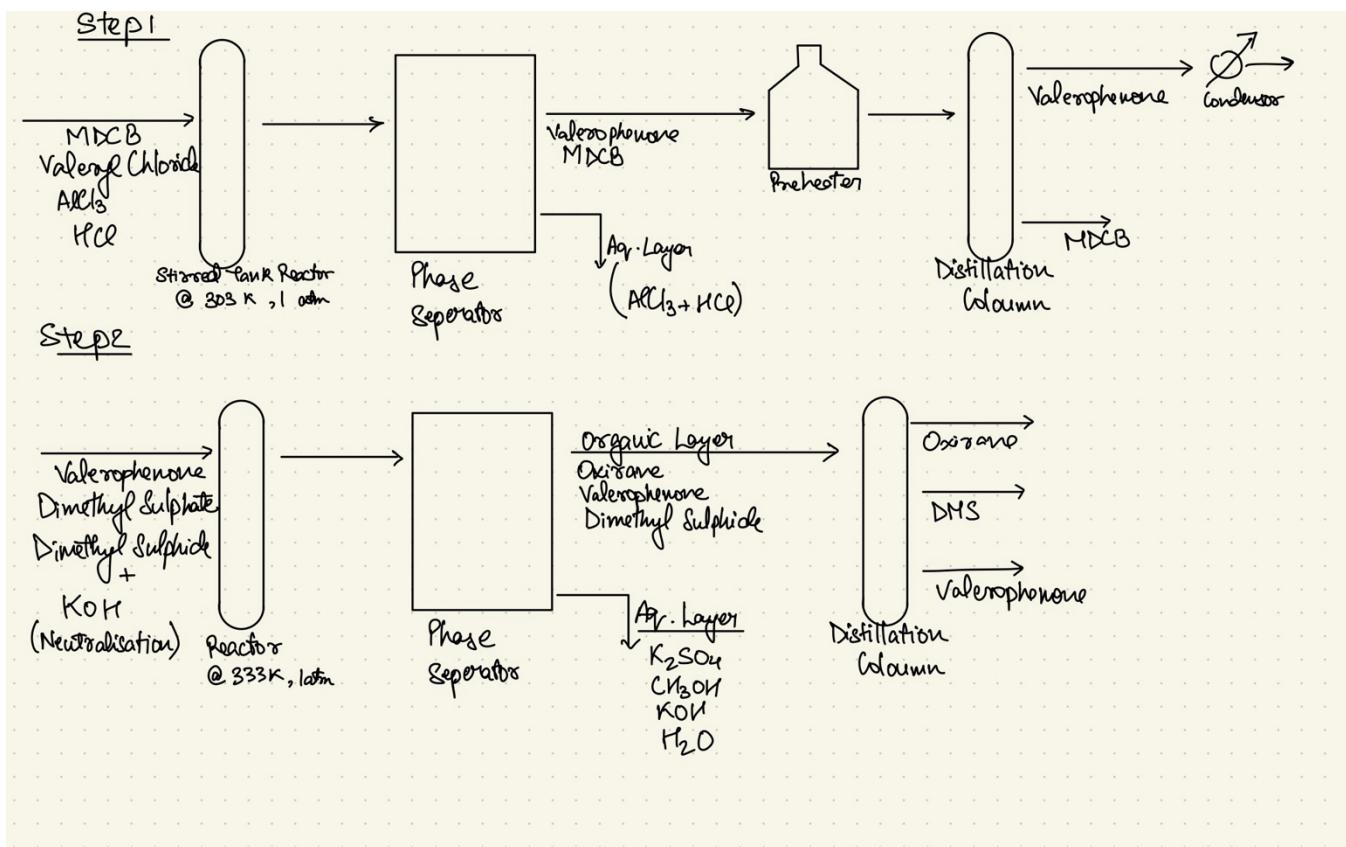
2. Oxidation (Step-2: Preparation of Oxirane)

- **Reactants:** Valerophenone, Dimethyl Sulfate (DMSO₄), Caustic Flakes (KOH)
- **Catalysts:** Dimethyl Sulfide (DMS)
- **Reaction:** Valerophenone undergoes oxidation with Dimethyl Sulfate in the presence of Dimethyl Sulfide to form Oxirane.
- **Process Control:** The reaction mass is neutralized with caustic flakes, and Oxirane is recovered.

3. Cyclization & Purification (Step-3: Preparation of Hexaconazole)

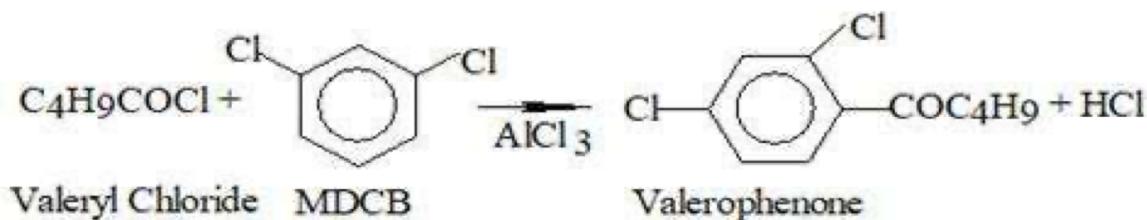
- **Reactants:** Oxirane, DMF (Dimethylformamide), Triazole, Methanol
- **Catalysts:** KOH
- **Reaction:** Oxirane reacts with Triazole in the presence of DMF and KOH catalyst to form Hexaconazole.
- **Purification Steps:**
 - Crystallization enhances product purity.
 - Filtration removes residual solids.
 - Drying ensures complete solvent removal.

Process Flowsheet for the production of Hexaconazole :



Reactions Involved:

Reaction 1:

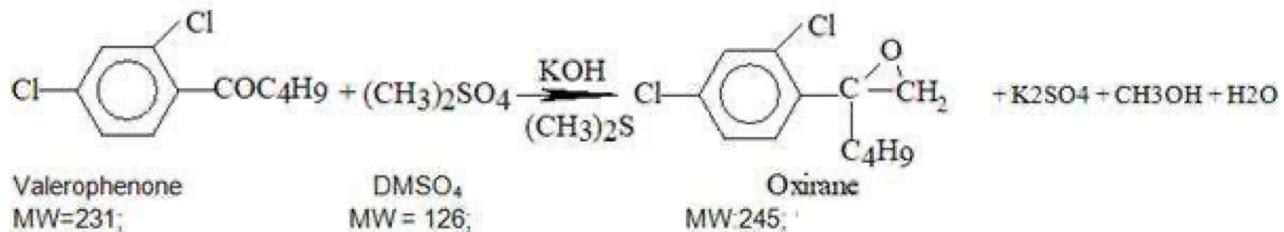


Reactor Type : Stirred Tank Reactor

Operating Temperature : 303K

Product Yield : 95.8 %

Reaction 2 :

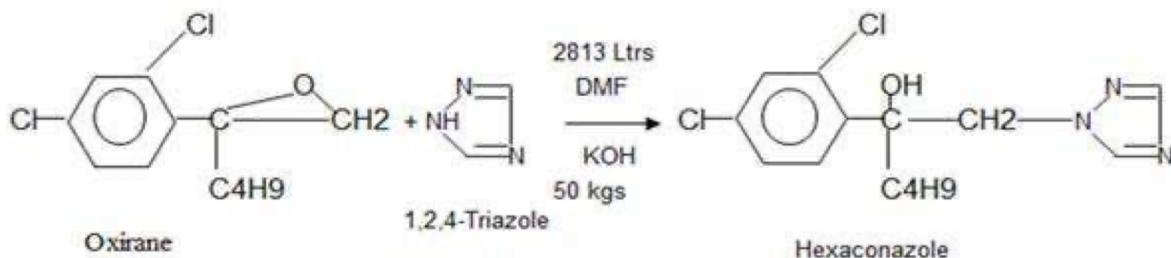


Reactor Type : Stirred Tank Reactor

Operating Temperature : 333K

Product Yield : 95.6 %

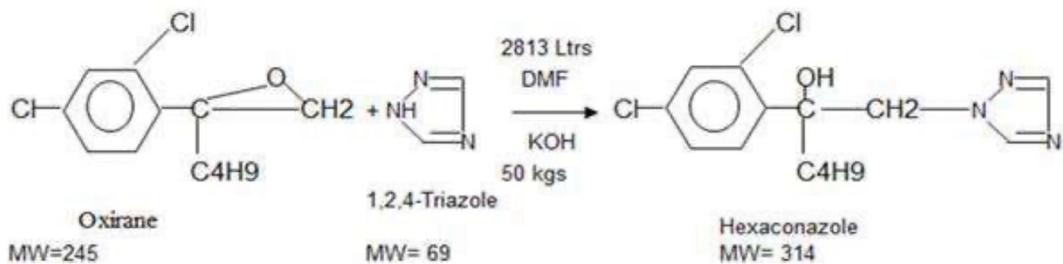
Reaction 3:



Reactor Type : Stirred Tank Reactor

Operating Temperature : 333K

Product Yield : 95.6 %

Material Balance:Stoichiometric CalculationsBasis: 1000 kg/day Hexaconazole \equiv 3.18 kmol/dayStep 3:

Assuming 90% yield and inputs are added in stoichiometric amounts

Oxirane: set x kmoles/day of input of Oxirane

$$\left(\frac{90}{100}\right)x = 3.18 \text{ kmol/day}$$

$$\Rightarrow x = 3.53 \text{ kmol/day of Oxirane}$$

$$\Rightarrow 866.94 \text{ kg/day of Oxirane}$$

Triazole: set x kmoles/day of input of Triazole

$$\left(\frac{90}{100}\right)x = 3.18 \text{ kmol/day}$$

$$\Rightarrow x = 3.53 \text{ kmole/day of Triazole}$$

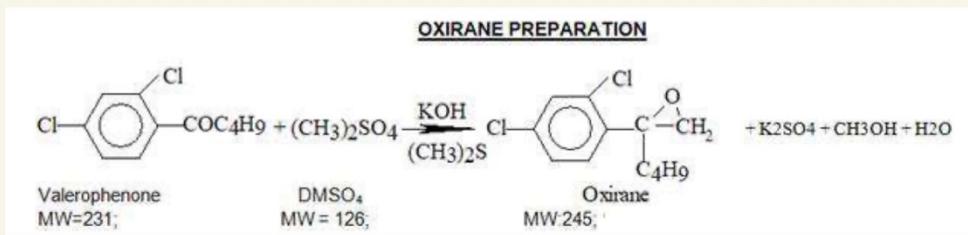
$$= 243.57 \text{ kg/day of Triazole}$$

Required Amount of Inputs

Input	Amount
Oxirane	866.94 kg/day
Triazole	243.57 kg/day
Total	1110.51 kg/day

Amount of Output formed

Output	Amount
Hexaconazole	1000 kg/day
Unreacted Reactants	110 kg/day
Total	1110.51 kg/day

Step 2:

Basis: 3.53 kmol/day of Oxirane

Assuming 90% yield and inputs are added in stoichiometric amounts

Valerophenone: det x kmole/day of input of Valerophenone

$$\left(\frac{90}{100}\right)x = 3.53 \text{ kmol/day}$$

$$x = 3.92 \text{ kmole/day of Valerophenone}$$

$$= 906 \text{ kg/day of Valerophenone}$$

DMSO₄: det x kmole/day of input of DMSO₄

$$\left(\frac{90}{100}\right)x = 3.53 \text{ kmol/day}$$

$$x = 3.92 \text{ kmole/day of DMSO}_4$$

$$= 494.2 \text{ kg/day of DMSO}_4$$

K₂SO₄: det x kmole/day of output of K₂SO₄

$$\left(\frac{90}{100}\right)x = 3.53 \text{ kmol/day K}_2\text{SO}_4$$

$$x = 3.92 \text{ kmole/day of K}_2\text{SO}_4$$

$$= 682.08 \text{ kg/day of K}_2\text{SO}_4$$

CH₃OH: det x kmole/day of output of CH₃OH

$$\left(\frac{90}{100}\right)x = 3.53 \text{ kmol/day}$$

$$x = 3.92 \text{ kmole/day of DMSO}_4$$

$$= 125.59 \text{ kg/day of DMSO}_4$$

H₂O: det x kmole/day of output of H₂O

$$\left(\frac{90}{100}\right)x = 3.53 \text{ kmol/day H}_2\text{O}$$

$$x = 3.92 \text{ kmole/day of H}_2\text{O}$$

$$= 70.56 \text{ kg/day of H}_2\text{O}$$

KOH: det x kmole/day of input of KOH

Moles of KOH = 2 * Moles of K₂SO₄ + Moles of KOH unreacted
 (Assuming 90% yield, ∴ 10% KOH will be left)

$$\frac{90}{100}x = 2(3.92)$$

$$\Rightarrow x = 8.71 \text{ kmole/day}$$

$$= 487.76 \text{ kg/day}$$

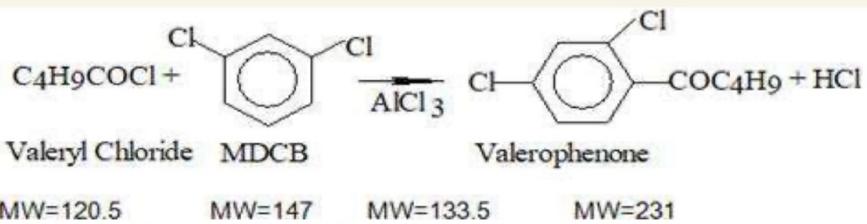
Required Amount of Inputs

Input	Amount
Valerophenone	906 kg/day
DMSO ₄	494.2 kg/day
KOH	487.76 kg/day
Total	1887.96 kg/day

Amount of Output formed

Output	Amount
Oxirane	866.94 kg/day
K ₂ SO ₄	682.08 kg/day
C ₁₂ H ₁₁ ON	125.59 kg/day
H ₂ O	70.56 kg/day
Unreacted Reactants	142.79
Total	1887.96 kg/day

Step 1:



Basis: 3.92 kmol/day of Valerophenone

Assuming 90% yield and inputs are added in stoichiometric amounts

Valeryl Chloride: let x kmol/day of input of Valeryl Chloride

$$\left(\frac{90}{100}\right)x = 3.92 \text{ kmol/day}$$

$$x = 4.35 \text{ kmol/day of Valeryl Chloride} \\ = 524.175 \text{ kg/day of Valeryl Chloride}$$

MDCB: let x kmol/day of input of MDCB

$$\left(\frac{90}{100}\right)x = 3.92 \text{ kmol/day}$$

$$x = 4.35 \text{ kmol/day of MDCB} \\ = 639.45 \text{ kg/day of MDCB}$$

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HCl : let x mmole/day of input of HCl

$$\left(\frac{90}{100}\right) x = 3.92 \text{ mmole/day}$$

$$x = 4.35 \text{ mmole/day of HCl}$$

$$= 524.175 \text{ kg/day of HCl}$$

$$= 158.6 \text{ kg/day of HCl}$$

Required Amount of Inputs (calculated by assuming 90% yield of each reaction)

Input	Amount
MDCB	639.45 kg/day
Valenyl Chloride	524.175 kg/day
Total	1163.625 kg/day

Amount of Output formed

Output	Amount
Valerosphenone	906 kg/day
HCl	158.6 kg/day
Unreacted Reactants	99.625 kg/day
Total	1163.625 kg/day

The previous calculations were based on stoichiometric assumptions. However, in large-scale industrial production, inefficiencies such as side reactions, incomplete conversions, and material losses must be considered. Based on data from existing patents and research papers, the actual material balance accounts for these factors, ensuring a more realistic estimation of reactant consumption and product yield. These adjustments help optimize raw material usage, minimize waste, and improve overall process efficiency.

Additionally, the market analysis and other teams have relied on this industrial material balance for their assessments, as it provides a more accurate representation of real-world production conditions compared to theoretical stoichiometric data.

Step-1: Preparation of Valerophenone:

Material Balance:

Input	Kg		Output	Kg
Meta Di Chloro Benzene	1500	Step-1	Valerophenone	924
Velaryl Chloride	503		Meta Di Chloro Benzene rec.	574
Aluminium Chloride	733		Aq. Waste to ETP	1452
Water	2637		Al(OH)3	2393
			Residue	30
	5373			5373

Step-2: Preparation of Oxirane:

Material Balance:

Input	Kg		Output	kg
Valerophenone	924	Step-2	Oxirane	937
KOH	531		DMS Rec.	1455
DMSO4	602		K2SO4	898
DMS	1476		EDC Rec	1182
EDC	1250		Aq. Waste to ETP	2195
Water	1884			
	6667			6667

Step-3 Preparation of Hexaconazole:

Material Balance:

Input	Kg		Output	Kg
Oxirane	937	Step-3	Hexaconazole	1000
DMF	3562		DMF Rec	3289
KOH	67		KOH	71
Triazole	299		Methanol Rec	1405
Methanol	1560		Residue	440
Water	2000		Aq. Waste to ETP	2220
	8425			8425

Energy Balance:Energy balance in reaction 1

the first reaction occurring with the temperature condition from 303K (for reaction itself) afterward, need to heat to 350K so the sensible heat is now for ΔT . Assuming the input stream at room temperature we have,
 $\Delta T = 303K - \cancel{300}K = \cancel{3}^{\underline{29}}8K$

we will use the heat capacities and the mass flow rates same as before, then combine for sensible energy balance

Species	mass flow (kg/dy)	C_p (kJ/kgK)
Valero phenone (product)	906.00	1.8
MDCB	639.45	1.3
Valeryl Chloride	524.17	1.5
HCl	158.60	0.9

Sensible heat calculations for the reaction.

for each species i , we have $Q_i = m_i C_i \times \Delta T$

a) Valero phenone

$$Q_{\text{valero phenone}} = 906 \frac{\text{kg}}{\text{day}} \times 1.8 \frac{\text{kJ}}{\text{kgK}} \times \frac{30 \text{K}}{6} = \frac{48924 \text{ kJ/day}}{6} \\ = 8154 \text{ kJ/day}$$

b) MDCB

$$Q_{\text{MDCB}} = 639.45 \frac{\text{kg}}{\text{day}} \times 1.3 \frac{\text{kJ}}{\text{kgK}} \times \frac{30 \text{K}}{6} = \frac{629.45 \times 39}{6} \\ = \frac{24939 \text{ kJ/day}}{6} \\ = 4156.5 \text{ kJ/day}$$

c) Valeryl Chloride

$$Q_{\text{valeryl chloride}} = 524.175 \frac{\text{kg}}{\text{day}} \times 1.5 \frac{\text{kJ}}{\text{kg K}} \times \frac{30 \text{K}}{6}$$

$$= \frac{524.175 \times 45}{6} = \frac{23,588 \text{ kJ/day}}{6} = 3931.3 \text{ kJ/day}$$

d) HCl

$$Q_{\text{HCl}} = 158.6 \frac{\text{kg}}{\text{day}} \times 0.4 \frac{\text{kJ}}{\text{kg K}} \times \frac{30 \text{K}}{6}$$

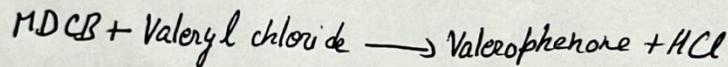
$$= \frac{4,282 \text{ kJ/day}}{6} = 713.6 \text{ kJ/day}$$

by summing up,

$$\text{Total sensible heat} = (48,924 + 24,939 + 23,588 + 4282)/6$$

$$= \frac{101,733 \text{ kJ/day}}{6} = 16955.5 \text{ kJ/day}$$

for the Friedel Crafts acylation between MDCB and Valeryl chloride,



the reaction enthalpy is $-70 \text{ kJ/mol} \Rightarrow \Delta H = -70 \text{ kJ/mol}$

number of moles, $n_{\text{mole}} = \frac{639.45}{0.147} = 4350 \text{ mol/day}$

thus heat released in this rxn.

$$Q_{\text{rxn}} = n_{\text{mole}} \times \Delta H$$

$$= 4350 \text{ mol/day} \times -70 \text{ kJ/mol} = -304,500 \text{ kJ/day}$$

Combining both sensible and reaction enthalpy

$$Q_{\text{net}} = Q_{\text{sensible}} + Q_{\text{reaction}}$$

$$= 16955 \text{ kJ/day} + (-304,500 \text{ kJ/day})$$

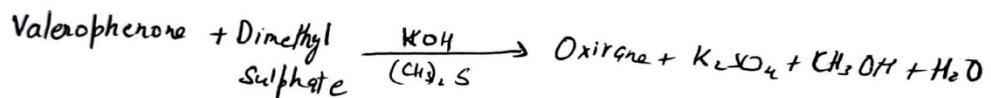
$$= -287,767 \text{ kJ/day}$$

Energy balance for reaction step 2

for this reaction using the data of experimental heat capacities -

<u>species</u>	<u>mass (kg/day)</u>	<u>C_p</u>
Valerophenone	90.6 kg/day	1.8
Dimethyl sulphate	494.2 kg/day	1.4
Oxirane	864.94 kg/day	1.7
K ₂ SO ₄	494.2 kg/day	1.0
CH ₃ OH	688.1 kg/day	2.5
H ₂ O	70.56 kg/day	4.18

reaction :



Sensible heat calculations

The sensible heat required to raise temperature of each stream.

$$Q_i = m_i \times C_p \times \Delta T$$

using $\Delta T = 33K - 298$ (room temperature) = 35 K

$$Q_{\text{Valerophenone}} = 90.6 \frac{\text{kg}}{\text{day}} \times 1.8 \frac{\text{kJ}}{\text{kgK}} \times 35 \text{K}$$

$$= 57078 \text{ kJ/day}$$

$$Q_{\text{Dimethyl Sulphate}} = 494.2 \times 1.4 \times 35$$

$$= 24215 \text{ kJ/day}$$

total sensible heat, $Q_{\text{sensible}} = 57078 + 24215$

$$= 81293 \text{ kJ/day}$$

$$n_{\text{ono}} = \frac{0.6 \text{ kg/day}}{0.231 \text{ kg/mol}} = 3422 \text{ mol/day}, n_{\text{reac}} = 3422 \times 0.9 \\ = 3530 \text{ mol/day}$$

$$n_{\text{oxirane}} = \frac{866.94}{0.245} = 3539 \text{ mol/day}$$

using the experimental reaction enthalpy

$$\Delta H_{\text{rxn}} = -60 \text{ kJ/mol}$$

$$Q_{\text{rxn}} = 3530 \text{ mol/day} \times (-60 \text{ kJ/mol}) = -211800 \text{ kJ/day}$$

$$Q_{\text{net}} = (81293 - 211800) \text{ kJ/day} \\ = -120507 \text{ kJ/day}$$

Energy Balance for third step

Oxirane :

$$\text{mass flow rate} = 866.94 \text{ kg/day}$$

$$\text{MW} = 245 \text{ g/mol}$$

$$C_p, \text{oxirane} = 1.7 \text{ kJ/kgK}$$

Triazole

$$\text{mass flowrate} = 243.57 \text{ kg/day}$$

$$\text{MW} = 69 \text{ kg/mol}$$

$$C_p, \text{triazole} = 1.5 \text{ kJ/kgK}$$

using the temperature change from the room temperature 298 K to the temperature required for the given reaction that is 333 K we have,

$$\Delta T = 333 \text{ K} - 298 = 35 \text{ K}$$

Sensible heat calculation.

$$\begin{aligned} Q_{\text{oxirane}} &= m_{\text{oxirane}} \times C_{p,\text{oxirane}} \times \Delta T \\ &= 866.94 \frac{\text{kg}}{\text{day}} \times 1.7 \frac{\text{kJ}}{\text{kg K}} \times 35 \text{K} \\ &= 51583 \text{ kJ/day} \end{aligned}$$

$$\begin{aligned} Q_{\text{triazole}} &= m_{\text{triazole}} \times C_{p,\text{triazole}} \times \Delta T \\ &= 244.26 \frac{\text{kg}}{\text{day}} \times 1.5 \frac{\text{kJ}}{\text{kg K}} \times 35 \text{K} \\ &= 12823 \text{ kJ/day} \end{aligned}$$

total sensible heat for the reaction $Q_{\text{sensible}} = 12823 + 51583$

$$= 64406 \text{ kJ/day}$$

using the stoichiometric basis, $n_{\text{react}} = 3540 \text{ mol/day}$
 using experimental reaction enthalpy we get $\Delta H = -65 \text{ kJ/mol}$

$$\begin{aligned} Q_{\text{rxn}} &= n_{\text{react}} \times \Delta H \\ &= 3540 \times -65 = -230100 \text{ kJ/day} \end{aligned}$$

$$\begin{aligned} Q_{\text{net}} &= 64406 \text{ kJ/day} + -230100 \text{ kJ/day} \\ &= -165694 \text{ kJ/day} \end{aligned}$$

List of Contributions of each author:

Karmanya Goyal : Designed the process flow sheet and did the stoichiometric mass balance and drafted the technical patent.

Naveen Godara : Identified reactor types and other unit operations, Energy balance.

Name	Roll No	Signature
Karmanya Goyal	230538	
Naveen Godara	230685	
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