

LAB JOURNAL - Version 2.0

Experiment No. : LBN 19 Aim : To develop product equivalent to reference product
Equipment Details : Glass RBF with stirrer set up
Reaction Scheme : Safety Precautions :

Input Raw Material

Sr.No	Raw Material	Sources	Cas No.	RM No	Quantity gms	Purity	Rm %	Molecular Wt.	No. of Moles	Molar Ratio
1	Vicaspin LUB 1005 Bu- EO-PO HV-58.1/ MW-750	Viswaat			195.0			750	0.26	1.0
2	P2O5	Commercial			10.877				0.077	0.3
3	Epomine SP-018	Nippon			30.7	100				
4	2 ethyl hexyl acrylate				57.1	100		184.3	0.31	
5	Step- B reaction mass (20%)				37.3					
6	Step- A product				154.4					

Experimental Procedure

Sr.No	Time hrs	Temperature °C	Pressure/Vacuum-N2	Process Steps	Remarks
1	2 / 2	45-50 / 65-70	N2	Step- A To main reactor added Vicaspin LUB 1005 Bu-EO-PO HV-58.1/ MW-750 under stirring and nitrogen atmosphere temp. 40 deg increased temp slowly with addition of P2O5	viscous clear mass
2	1 / 1	40-45 / 60-65	N2	Step-B To main reactor added Epomine SP-018- reaction mass held with slow addition of 2 ethyl hexyl acrylate, checked free acid at interval	AV-50.7, free acid 0.8
3	2	65-70	N2	Step-C To main reactor added Step- B reaction mass (20%) and Step- A product, mixed till uniform	AV 48.9 Amine 32.3

Optimized Reaction Conditions: Temperature c: 60-65 | N2/Vac: N2 | Time: 4h

Critical Process Parameter

Sr No	Source	Time	Temp	Observation Details
1	Step-A	2	50	viscous clear mass

Critical Parameters

S.no	Parameter	Reference	Value
1	av	43.2	48.9
2	aminev	30.1	32.3

HPLC Results :
Conclusion: AV 48.9 against target 43.2; batch outside range, rework planned.