

LAB JOURNAL - Version 3.0

Experiment No. : LBN 3 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 1020 (R226011203) Bu- EO-PO HV-66.0/ MW-1100	Viswaat			156.0			1100	0.14	1.0
2	P2O5	Commercial			7.881				0.056	0.4
3	Epomine SP-012	Nippon			33.0	100				
4	2 ethyl hexyl acrylate				65.4	100		184.3	0.35	
5	AIBN catalyst				0.28					
6	Step- B reaction mass (20%)				52.5					
7	Step- A product				170.3					

Experimental Procedure

Sr.No	Time hrs	Temperature °C	Pressure/Vacuum-N2	Process Steps	Remarks
1	0.5 / 3 / 0.5	45-50 / 55-60 / 70-75	N2	Step- A To main reactor added Vicaspin LUB 1020 (R226011203) Bu- EO-PO HV-66.0/ MW-1100 under stirring and nitrogen atmosphere temp. 40 deg increased temp slowly with addition of P2O5	viscous clear mass
2	1 / 2	40-45 / 55-60	N2	Step-B To main reactor added Epomine SP-012- reaction mass held with slow addition of 2 ethyl hexyl acrylate, checked free acid at interval	AV-45.4, free acid 0.2
3	1	65-70	N2	Step-C To main reactor added Step- B reaction mass (20%) and Step- A product, mixed till uniform	AV 41.4 Amine 31.2

Optimized Reaction Conditions: Temperature c: 55-60 | N2/Vac: N2 | Time: 4h

Critical Process Parameter

Sr No	Source	Time	Temp	Observation Details
1	Step-A	0.5	50	viscous clear mass

Critical Parameters

S.no	Parameter	Reference	Value
1	av	43.2	41.4
2	aminev	30.1	31.2

SPDu :
Conclusion: AV 41.4 against target 43.2; batch outside range, rework planned.