

LAB JOURNAL - Version 2.0

Experiment No. : LBN 51 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 1010 (R225100845) Bu- EO-PO HV-62.3/ MW-900	Viswaat			199.0			900	0.22	1.0
2	P2O5	Commercial			10.736				0.076	0.35
3	Epomine SP-012	Nippon			31.2	100				
4	2 ethyl hexyl acrylate				68.5	100		184.3	0.37	
5	Step- B reaction mass (20%)				33.0					
6	Step- A product				154.1					

Experimental Procedure

Sr.No	Time hrs	Temperature °C	Pressure/Vacuum-N2	Process Steps	Remarks
1	1-2	45-50	N2	Step- A To main reactor added Vicaspin LUB 1010 (R225100845) Bu- EO-PO HV-62.3/ MW-900 under stirring and nitrogen atmosphere temp. 40 deg increased temp slowly with addition of P2O5	clear mass
2	0.5 / 1	50-55 / 65-70	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-51.3, free acid 0.4
3	1-2	60-65	N2	Step-C To main reactor added Step- B reaction mass (20%) and Step- A product, mixed till uniform	AV 37.6 Amine 28.7

Optimized Reaction Conditions: Temperature c: 65-70 | N2/Vac: N2 | Time: 4h

Critical Process Parameter

Sr No	Source	Time	Temp	Observation Details
1	Step-A	1-2	50	clear mass
2	Step-B	1	65	free acid < 0.5 endpoint check

Critical Parameters

S.no	Parameter	Reference	Value
1	av	43.2	37.6
2	aminev	30.1	28.7

HPLC Results :
Conclusion: AV 37.6 against target 43.2; batch outside range, rework planned.