

LAB JOURNAL - Version 3.0

Experiment No. LBN 36

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	Surfol R-1400 (R-53/07032024	Sun shield			350					
2	Polyphosphoric acid	Commercial			4					
3	Phosphorous pentaoxide	Commercial			4.35					
4	Epomine SP-018	Nippon			60	100				
4	Toluene	Commercial			190	99				
5	AIBN catalyst	Himedia			0.4	100				
5	2 ethyl hexyl acrylate				140	100				
6	AIBN catalyst				0.64	100				
Total										

Experimental Procedure

Sr.No	Time(hrs)	Temp - (°C)	Pressure/Vacuum - N2(kg/cm2)	Process Steps	Remarks
1	5	58-75	N2	Step 1) Phosphate ester- RBF with Surfol R-1400 heated to 58 deg C and then slowly added Polyphosphoric acid followed by P2O5 into it under N2 atmosphere. Digested this for 3 hrs at 70-75 deg C. Cool to RT and filter through Nylon Mesh AV-34.2	Appearance- yellowish clear
2	5.5	65-72	N2	Step 2) Acrylate intermediate- RBF with Epomin SP-18 with Toluene solvent (Dissolved AIBN- 0.4 gm) heated to 66-72 deg C under N2 atmosphere. Slowly feed of 2-ethyl hexyl acrylate (with dissolved AIBN- 0.64 gm) into it over 1.15 hrs and continue reaction for 3 hrs. Cool to RT removed toluene under reduced pressure. (IV-13.2)	Appearance- yellowish clear
3	3.5	56-61	N2	Step 3) Neutralization - RBF with acrylate intermediate of step-1) (15%) heated to 56 deg C under N2 atmosphere. Slowly feed of step-2) (85%) Phosphate ester into it over 30 min and continue for 3 hrs. Cool to RT and filter through Nylon Mesh. AV- 31.5/ IV-6.22	

Optimized Reaction Conditions

Temperature (c)	N2/Vac	Time

Critical Process Parameter				
Sr No.	Source	Time	Temp	Observation Details

Critical Parameters			
S.no	Parameter	Reference	Value
1	aminev	30.1	
2	av	43.2	31.5

SPDU Conditions					
Date					
Batch No.					
Degasser		spdu I		spdu II	
TEMP (°C)	Vaccum	TEMP (°C)	Vaccum	TEMP (°C)	Vaccum
Actual	mbar	Actual	mbar	Actual	mbar

HPLC Results					
	Crude	Stripped	Liq. Seal	Distilled	Residue

SPDu	
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Conclusion
Application test against violet- 27 / 23 (Resolubility) is good only gloss less.

