LAB JOURNAL - Version 3.0

Experiment No.	LBN 34 - B
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			290					
2	Phosphorous pentaoxide	Commercial			10.8					
3	Epomine SP-018	Nippon			30	100				
4	Toluene	Commercial			94	99				
5	AIBN catalyst	Himedia			0.2	100				
5	2 ethyl hexyl acrylate				70	100				
6	AIBN catalyst				0.32	103				
Total										

Critical Process Parameter

Sr No.	Source	Time	Temp	Observation Details

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 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- 38.5	Appearance- yellowish clear
2	5.5	65-72	N2	Step 2) Acrylate intermediate- RBF with Epoimin SP-18 with Toluene solvent (Dissolved AIBN- 0.2 gm) heated to 66-72 deg C under N2 atmosphere. Slowly feed of 2-ethyl hexyl acylate (with dissolved AIBN- 0.32 gm) into it over 1.15 hrs and continue reaction for 3 hrs. Cool to RT removed toluene under reduced pressure. (IV-10.5)	Appearance- yellowish clear
3	3.5	56-61	N2	Step 3) Neutralization - RBF with acrylate intermediate of step-1) (20%) heated to 56 deg C under N2 atmosphere. Slowly feed of step-2) (80%) Phosphate ester into it over 30 min and continue for 3 hrs. Cool to RT and filter through Nylon Mesh. AV-31.7	AV-31.7

Optimized Reaction Conditions

Temperature (c)	N2/Vac	Time

Critical Parameters			
S.no	**Parameter**	**Reference**	**Value**
1	**aminev**	**30.1**	
2	**av**	**43.2**	**31.7**

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	

Conclusion
Application test against violet- 27 / Black is good only re-solubility failed for violet 23 & Blue