

LAB JOURNAL - Version 3.0

Experiment No. LBN 48

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			208					
2	P2O5	Commercial			10.93					
3	Epomine SP-018	Nippon			30	100				
4	2 ethyl hexyl acrylate				70	100				
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 Vicaspin LUB 1010 (R225100845) Bu- EO-PO HV-62.3/ MW-900 heated to 50 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 AV-51.87	Mono-22.56 / Di- 48.93/ Free acid-1.096
2	6.5	78-82	N2	Step 2) Acrylate intermediate- RBF with Epomine SP- 018 heated to 78-81 deg C under N2 atmosphere. Slowly feed of 2-ethyl hexyl acrylate over 2 hrs and continue reaction for 5 hrs. Monitoring of reaction done by IV. IV-26.1 @ 5.5 hrs Cool to RT	Appearance- yellowish clear AV-0.45 IV-26.1 @ 5.5 hrs
3	3.5	56-61	N2	Step 3) Neutralization - RBF with acrylate intermediate of step-2) (13%) heated to 56 deg C under N2 atmosphere. Slowly feed of step-1) (87%) Phosphate ester into it over 30 min and continue for 3 hrs. Cool to RT and filter through Nylon Mesh.	AV-46.5/ HV-49.4/SAP-61.98 /Amine-20.2/ IV-2.58 Tested over pigment Black, Blue, Violet-23/27 etc All having matching solubility in black, blue & violet 27 while slight reduced in gloss found in all pigments

Optimized Reaction Conditions

Temperature (c)	N2/Vac	Time

Critical Parameters			
S.no	Parameter	Reference	Value
1	aminev	30.1	20.2
2	av	43.2	46.5

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
Tested over pigment Black, Blue, Violet-23/27 etc All having matching resolubility in black, blue & viloet 27 while slight reduced in gloss found in all pigments

