

## 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    |         |       | 178            |        |      |               |              |             |
| 2       | P2O5                                                     | Commercial |         |       | 9.357          |        |      |               |              |             |
| 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--52.52                   | Mono-24.44 / Di- 51.76/ Free acid-0.24                                                                                                                             |
| 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) (15%) heated to 56 deg C under N2 atmosphere. Slowly feed of step-1) (85%) Phosphate ester into it over 30 min and continue for 3 hrs. Cool to RT and filter through Nylon Mesh.  | AV-44.36/ HV-46.58/SAP-62.14 /Amine-36.25/ IV-5.8 Tested over pigment Black, Blue, Violet-23/27 etc All having matching resolubility while reduced gloss in violet |

## Optimized Reaction Conditions

| Temperature (c) | N2/Vac | Time |
|-----------------|--------|------|
|                 |        |      |

| Critical Parameters |           |           |       |
|---------------------|-----------|-----------|-------|
| S.no                | Parameter | Reference | Value |
| 1                   | aminev    | 30.1      | 36.25 |
| 2                   | av        | 43.2      | 44.36 |
|                     |           |           |       |

| 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                                                                                                       |
|------------------------------------------------------------------------------------------------------------------|
| Tested over pigment Black, Blue, Violet-23/27 etc All having matching resolubility while reduced gloss in violet |



