CP PROCESS OVERVIEW
Melt Phase – “3 reactor” technology

- PTA & MEG slurry mixing
- Reactor 1 – Esterification
- Catalyst and additive injection
- Reactor 2 - Pre-polymerisation
- Reactor 3 – Polymerisation
- Chip formation
Melt Polymerisation Process Flowsheet

- PTA STORAGE
- PTA SLURRY MAKEUP
- ESTERIFIER
- CATALYST & ADDITIVES
- UFPP
- FINISHER
- POLYMER PUMP AND FILTER
- BASE CHIP
## Melt Polymerisation Process Flowsheet

<table>
<thead>
<tr>
<th></th>
<th>Esterifier</th>
<th>Additives</th>
<th>UFPP (Temp)</th>
<th>Finisher (Pressure)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DP (iv)</td>
<td>4-6</td>
<td>3</td>
<td>25 (0.20 – 0.30)</td>
<td>100-120 (0.55-0.64)</td>
</tr>
<tr>
<td>DE%</td>
<td>85-90</td>
<td>95</td>
<td>99</td>
<td>99.9</td>
</tr>
<tr>
<td>Temp</td>
<td>260-300</td>
<td>270-289</td>
<td>279-298</td>
<td>278-298</td>
</tr>
<tr>
<td>Pressure</td>
<td>1.05 bara</td>
<td>6 bar</td>
<td>30 mbar</td>
<td>1-2 mbar</td>
</tr>
<tr>
<td>Res Time</td>
<td>120-180 mins</td>
<td>30 mins</td>
<td>40 mins</td>
<td>80 mins</td>
</tr>
</tbody>
</table>
SLURRY HANDLING

- Excess glycol to reduce viscosity – simple pumps
- Agitation to handle settling slurry with blockage
- Advanced automatic injection system

PTA SLURRY PREPARATION
ESTERIFIER

- Natural thermosyphon circulation – no mechanical agitator
- Glycol-water separation column
UFPP (UP- FLOW-PRE-POLYMERISER)

- Glycol enhanced mass transfer – no mechanical agitator
- Optimised Chemistry (End group balance) through staged pressure control
- Air exclusion system
POLYMERISER - FINISHER
- Cage-type finisher agitator with no central shaft
- Continuous wiping of finisher vessel wall eliminating degradation
- Air exclusion system
EXTRUSION, FILTER AND CHIPPING

- In line viscosity measurement to directly control key quality parameter via Finisher vacuum control (1.0 → 3.0 mbara)
GLYCOL

- All glycol produced is recycled back to front of plant eliminating distillation for glycol recovery
- Waste water is processed on site and VOCs are used in vapouriser to recover energy (approx 7% fuel saving)
HTF vaporized and sent to the process units for heat transfer. HTF Vaporizer burner used to utilize heat value from the organics gases collected at the Organic Stripping Column (reducing emissions).
Raw Material Consumptions

• Typical Raw Materials Consumption per kg
  – PTA + IPA, kg = 0.860
  – MEG, kg = 0.335
  – DEG, kg = 0.006
  – Catalyst, kg = 0.0003
OVERALL CONTROL PHILOSOPHY — SIMPE
PTA CONVEYING
PTA CONVEYING SYSTEM - Overview
PTA CONVEYING SYSTEM – Process Description

PURPOSE
To feed the process with correct quantity of PTA essential for product quality.

HOW
Feedstock ex trucks is blown into a storage silo (batch mode)
From there the powder is blown to a day bin (CP area) on a continuous basis.

FEEDS
PTA powder ex trucks
Nitrogen

PRODUCTS
Available PTA supply for slurry make up
PTA CONVEYING SYSTEM - Equipment Description

**Off loading station (x2)**
- Each off loading station comprises two rotary valves / buffer hopper and is linked to common delivery line to transport powder into storage silo.

**Storage silo**
- Existing equipment. Capacity : 1500tes
- Vessel has nitrogen blanket
- Two conservation vents are installed
- Vent filter is provided with timer controlled nitrogen pulsation
- Common export line splits into two streams leading to the PTA storage bin.

**PTA Storage bin**
- Capacity : 150tes
- Vessel has nitrogen blanket with aeration rings installed to assist discharge
- Two conservation vents are installed
- Vent filter is provided with timer controlled nitrogen pulsation
- Product exit is via the vessel base through a slide valve and a variable speed rotary valve to slurry mix tank
PTA CONVEYING SYSTEM - Equipment Description

**Nitrogen Blower Package x4**
- Nitrogen flow is achieved by positive displacement blowers
- Three blowers in the system and one spare
- PLC based control system is used to control and monitor the system
- PLC interacts with the DCS via a software link.

**Return Gas Fan**
- Gas returning from the silos and the buffer hoppers is conveyed via filters to suction of compressors by return gas fan
Oxygen measurement

Concentration of Oxygen in return gas is measured by AT-202136 /37. Oxygen analysers are active whenever PLC is active. Gas is drawn from return gas line and filtered before reaching Oxygen sensor. Oxygen analysers are equipped with:

- Oxygen sensor
- Flow switch
- Calibration and span connection
PTA CONVEYING SYSTEM - Equipment Description

Nitrogen make – up unit

Nitrogen feed in and feed out is available to keep a pressure in nitrogen system between 30 – 70 mbar
Make up unit is active whenever PLC is operating
It consists of feed in with control valve PV-202180, feed out with control valve PV-202179 and pressure measurement PT-202138

Nitrogen feed out consists of control valve PV-202179 and is controlled by pressure measurement PT-202138
PTA CONVEYING SYSTEM - Equipment Description

Nitrogen make – up unit

Nitrogen feed in consists of control valve PV-202180 and is controlled by pressure measurement PT-202138
PTA CONVEYING SYSTEM - Process Flow

General

**PTA :**
PTA powder arrives at the plant in bulk containers
PTA unloaded and conveyed with nitrogen gas to a large storage silo
From storage silo, PTA conveyed to a smaller day bin for immediate use slurry preparation

**Nitrogen :**
Nitrogen gas, used for conveying, is filtered & checked for oxygen content and reused
Volume of nitrogen in conveying system is controlled using nitrogen make up and discharge stations
Oxygen content is measured and if detected to be higher than the permissible limit of 4%, additional nitrogen is introduced to dilute the amount of oxygen in the system
The flow of nitrogen is achieved using ‘positive displacement blowers’
Total of 4 blowers are present in the system (3 plus 1 spare)

**PLC :**
PLC based control system is used to control and monitor the conveying system
PLC interacts with the DCS over a software link.
PTA CONVEYING SYSTEM - Quality

Powder quality is critical to the operation of the CP plant and the quality of the final product. Although the powder quality is primarily determined in the PTA manufacturing plant the way that it is transported and stored can make a significant difference.

Transport:
Powder that has been transferred significant distances by road or rail can become compacted in its container and prove difficult to discharge.

Particle Size:
Powder that has been left on silo walls for long periods tends to have a smaller particle size. This leads to ‘frothy’ slurry which in turn leads to rapid esterification and low CEG in the polymer (poor reactivity in SSP).
PTA CONVEYING SYSTEM - SUMMARY

- Powder is imported from containers to silo (1500m³)
  - Conveyed by nitrogen gas
  - Silo has nitrogen blanket
  - Nitrogen is filtered and re-used
  - Oxygen content measured and monitored
    - O₂ content must remain <4%

- Powder is conveyed from silo to storage bin (150m³)
  - Ready for use in PTA slurry preparation

- Conveying system is controlled by a PLC
PTA POWDER & SLURRY PREPARATION
POWDER & SLURRY HANDLING

- Excess glycol to reduce viscosity – simple pumps
- Tank Agitation to minimise settling of slurry
- Automated (DCS) injection system
PTA SLURRY SYSTEM - Process Description

PURPOSE
To feed process with correct quantity of raw materials in critical ratios which are essential for product quality and a successful reaction.

HOW
Takes PTA from day bin and feed the powder at correct ratio and flowrate to match glycol feeds to the system. After mixing, the resulting slurry is then injected into the esterifier.

FEEDS
- PTA – (Terephthalic Acid)
- MEG – (Mono ethylene glycol) from 2 sources
  - Esterifier Column Hotwell (ca 170degC)
  - CP Glycol Feed Tank (ca 40 degC)

\[ \text{Combined as one stream} \]

PRODUCTS
- PTA/MEG Slurry
PTA SLURRY SYSTEM - Equipment Description

**SILO**
- Situated on load cells
- PTA silo capacity approximately 1500 tonnes
- PTA imported via road container

**Day Bin**
- Situated on load cells
- PTA storage bin capacity approximately 150 tonnes
- PTA conveyed by inert gas from PTA silo
PTA SLURRY SYSTEM – Equipment Description

SLURRY MIX TANK
- An agitated tank with a 1 hour residence time to mix glycol and powder into a slurry
- Single speed agitator plus short term reverse facility
- Approx 85°C, heated by recycle glycol from esterifier hotwell
- Mix tank is vented via a scrubber to seal pot (esterifier)
- Nitrogen blanketed to prevent dust explosion

SLURRY TRANSFER PUMPS
- Two centrifugal pumps
- Both operate at 50% of max capacity
- Automatic ramp up if one pump trips
PTA SLURRY SYSTEM – Equipment Description

SLURRY FEED TANK
- A turbine agitated tank with a 4 hour residence time
- Provides buffer storage between slurry preparation and esterification
- Feed tank is vented to the seal pot (esterifier)
- Tank has nitrogen blanket

SLURRY FEED PUMPS
- Two centrifugal pumps
- Both operate at 50% of max capacity
- Feeds slurry at required rate to esterifier injection nozzle
- Automatic ramp up if one pump trips
PTA SLURRY SYSTEM - Glycol Supply

**CP Recycle tank**
- Supplies EG from the UFPP & Finisher hotwells at ca 45degC.
- Flow is measured by 2 correolis type meters operating in sequence
- Tank capacity approximately 45 M³

**Esterifier hotwell**
- Supplies EG evolved from the esterifier at ca 170degC.
- Flow is measured by 2 correolis type meters operating in sequence
- Tank capacity 12 M³

**Glycol flow**
- Interlock to stop powder feed on loss of glycol flow
- Interlock glycol flow on loss of powder feed

Ratio control – CP recycle / Esterifier hotwell glycol ratio is measured, monitored (displayed on DCS) and controlled as it forms part of the slurry composition calculation system
PTA SLURRY SYSTEM – Overview

- TPA STORAGE BIN
- N2 PURGE
- SEALPOT
- FM
- VENT
- OVERFLOW TO GLYCOL FEED TANK
- VENT TO WATER SEAL POT
- FM
- FROM CP RECYCLE TANK
- FROM ESTERIFIER HOTWELL
- SLURRY MIX TANK
- FM
- SLURRY MIX PUMPS
- FM
- FROM ESTERIFIER HOTWELL BOOSTER PUMP
- VIRGIN GLYCOL
- FM
- FROM ESTERIFIER HOTWELL
- RETURN TO SLURRY MIX/FEED TANKS
- SLURRY FEED TANK
- FM
- FM
PTA SLURRY SYSTEM – Slurry Injection

SLURRY PIPES - Designed to eliminate dead zones

- Sloping lines to facilitate draining

- EG flush nozzles
  - hot EG from esterifier hotwell
  - cold virgin glycol

INJECTION NOZZLES

- Slurry enters base of heat exchanger of esterifier

- DCS monitors the pressure to detect any blockages (indicated by low flow & pressure)

- Sequence automatically closes injection nozzle and opens the EG flush nozzle
PTA SLURRY SYSTEM - SUMMARY

- Powder system – explosion hazard!
  - Nitrogen blanket
  - Hazardous area classification

- Slurry density control is critical to product quality & esterifier operation

- Slurry must be kept moving – if pumps or agitators fail then settling will occur very quickly and lines will block

- If the plant is shutdown for >5 days consideration must be given to slurry quality
  - Continual agitation & pumping will start to break down the powder and cause the slurry to become ‘frothy’ – will result in poor esterification
PTA SLURRY

INJECTION
SLURRY CONTROL SEQUENCES

Slurry injection sequence

The slurry injection sequence is used to operate the valves associated with the slurry injection pipework in such an order that the slurry route can be changed from recirculating to injecting into the Esterifier without blocking any pipework. It is also used to control the injection flow of Esterifier EG hold tank glycol into the Esterifier when on 'hold' conditions to maintain a thermosyphon.

Both A and B injection streams have their own associated identical sequences as they operate independently of one another.

Each sequence consists of three main steps displayed on the DCS slurry injection display by selecting either the ‘SLURRY A’ or ‘SLURRY B’ icon, these steps are as follows:

- Off
- EG Flush
- Slurry on
SLURRY INJECTION

The slurry injection sequence allows TPA/EG slurry to be automatically injected into the esterifier to produce oligomer.

Normally, pressure in the slurry injection system is higher than in the esterifier, forcing slurry through the injection nozzle into the esterifier. If pressure falls in the slurry injection system, hot oligomer can flow backwards from the esterifier into the slurry pipework blocking the injection nozzle. This can lead to a shutdown of the CP line. To prevent this, the DCS monitors the slurry injection flow and automatically stops slurry injection if a problem occurs. The DCS also allows the operator to start and stop injection and control flushing of the system with EG.

Slurry injection operation
The injection system is split into 2 identical lines (A&B).
In normal operation one line is in service whilst the other is in standby, ready for operation should a problem occur.
Slurry can be routed to either injection nozzle or recirculated to either the slurry mix or feed tanks.
4 valves control the slurry routing – A,B,C,D. Valves A&C are on the A injection line, valves B&D are on the B injection line. Valves A&B feed slurry to the esterifier whilst valves C&D recirculate slurry to the mix or feed tanks.
The injection system has 4 modes of operation:
SLURRY INJECTION

OFF:
slurry injection nozzle is CLOSED.
Slurry flow is returned to the feed tank.
EG booster pump is not running.
This mode allows pressurised EG to remain in the pipe between the slurry flow valve and the injection nozzle to prevent oligomer backing up past the nozzle when it is first opened.
ESTERIFIER
ESTERIFIER

- Natural thermosyphon circulation – no mechanical agitator
- Glycol-water separation column to remove water
ESTERIFIER SYSTEM - Overview
ESTERIFIER SYSTEM - Process Description

**Purpose** - To react EG, PTA slurry in an atmospheric reactor (typically 260 - 290°C) to produce oligomer and then separate excess glycol and water produced. Residence time typically 120 - 180mins.

**How** - Uses thermosyphon mixed reactor, vapour separator and column (with trays) to split water and glycol.

**Feeds** - Slurry  
- Dowtherm vapour  
- Cooling water

**Products** - Oligomer  
- low molecular weight polymer of approximately 5 repeat units)  
- CEG’s in range 30-50 µG  
- Glycol : excess from feed slurry  
- Water : from reaction and slurry
Thermosyphon Loop
ESTERIFIER SYSTEM - Equipment Description

Heat Exchanger

- Single pass, shell and tube heat exchanger
- Largest heat transfer achieved in CP unit
  - Slurry from 85°C to typically 260 - 290°C
- Largest supply line (diameter) from HTF system

Vapour Separator

- Cross over line runs fully flooded
- Internal “tube” arrangement
- Large diameter to ensure minimal oligomer carry over
- Vapour exists at base of separation column

Recirculation Line

- Large diameter
- Connects vapour separator exit (at base) to heat exchanger
- Line contents has low vapour content
  - More dense than material in heat exchanger
  - Thermosyphon effect
ESTERIFIER SYSTEM - Equipment Description

Separation Column

- 12 sieve cap trays
- Internal spray nozzle (glycol)
  - Desuperheat vapour and knock out oligomer
- Top product
  - Water
  - Acetaldehyde
  - 1,4 dioxane
  - 2 methyl 1,3 dioxalane
- Bottom product
  - Glycol
  - Water
  - DEG
  - Oligomer
- Glycol transfer line steam traced to minimise fouling
ESTERIFIER SYSTEM - Equipment Description

**Column Condenser**
- Condenser is shell and tube heat exchanger
- Column overheads cooled and condensed to approx. 50°C
- Nitrogen supply to esterifier system for “in breathing” provided on line exit condenser

**Reflux Tank**
- Atmospheric tank venting to seal pot (water)
- Liquid overflow to OSC feed tank

**Esterifier Hotwell**
- Glycol storage vessel
- Small capacity (12m3)
- Provide EG input for
  - Column desuperheater spray
  - PTA slurry make-up
Esterifier Heat Exchanger
VAPOUR SEPARATOR WITH COLUMN

FROM HEAT EXCHANGER

TO THERMO-SYPHON LOOP

SEPARATION COLUMN

SIGHT GLASS
ESTERIFIER SYSTEM - Rate Control

Philosophy:

- Plant rate set by Finisher output (Maag speed)
- Process vessel levels (Finisher, UFPP, Esterifier) then cascade to input flows for each vessel
- Slurry mix and feed tank levels cascade to input flows for each tank
- Ultimately, this is PTA input for the slurry mix tank
ESTERIFIER SYSTEM - Control Loops

TO FT-20302 INLET TO SLURRY MIX TANK

SLURRY FEED TANK

ESTERIFIER HOTWELL

ESTERIFIER HEAT EXCHANGER

VAP. SEP.

REFLUX DRUM

WATER CONDENSER

TO SLURRY TANK

Simpe - Oct 2008 - Esterifier
ESTERIFIER SYSTEM - Control

Vapour Separator:

**Temperature control**
- Critical for control of CEG
- Critical for control of colour

**Level control**
- Critical for control of CEG
  - Polycondensation reaction (plant IV lift)
  - SSP reactivity

**Plant rate change**
- Must consider level constraints
  - Potential foaming versus column control
  - All key process vessels!
- Must consider HTF capability
  - Is enough “heat” available?
- Must consider “oligomer” quality (chemistry) and consistency
ESTERIFIER SYSTEM - Control

Separation Column / EG Hotwell:

**Temperature control**
- Critical for control of EG carry over from column top

**Level control**
- Limited capacity of EG hotwell
- “tight” level control of EG hotwell essential

**Flow control to Column**
- “no flow”
  - Rapid loss of temperature and pressure control
  - Glycol carry over from column top
  - Potential rupture of relief system
ESTERIFIER SYSTEM - Quality (Oligomer)

CEG
– Critical to downstream processing
  - Affects Finisher vacuum performance
  - Affects SSP reactivity
  - Level and temperature control critical

B colour
– Is adversely affected by:
  - Operating temperature too high
  - Operating level too high

May require increased colour enhancer addition to maintain control
ESTERIFIER SYSTEM – Control Interlocks

I 23A - Standby esterifier hotwell glycol circulation pump will start when in remote if delivery low pressure switch PSL is activated.

I 23A - Standby esterifier separation column reflux pump will start when in remote if delivery low pressure switch PSL is activated.

I 43A - Esterifier pressure high high - setting to be determined during commissioning Action is to shut esterifier HTF supply valve. Will also activate interlock I 56A

I 56A - Slurry injection flow low - setting to be determined during commissioning Action is to close (in sequence) slurry injection valve to esterifier

I 50A - Esterifier Dow supply valves will close if:
- Hardwired Esterifier high pressure switch PSH (set at 0.5bar) is activated
- Condenser exit temperature high high (80 degC)

Some of the interlocks (I43A) associated with the esterifier are part of a hardwired system which close the dow supply valves to the esterifier when high pressure is detected in the vessel or high temperature in the condenser exit.
ESTERIFIER SYSTEM - SUMMARY

- First key process vessel making oligomer from slurry
- Thermosyphon must be carefully established
- Significant load on HTF vapour system
  - heat slurry and boil off EG / Water
- Water removed in separation column to promote reaction
- Level control is critical but affected by many factors
  - eg slurry density, rate changes
- Maintaining a **consistent** esterification product is **critical** to the entire process and final product quality
OLIGOMER LINE & ADDITIVES INJECTION
OLIGOMER LINE & ADDITIVES INJECTION

- Oligomer is pumped to UFPP using positive displacement pumps (range of 7 - 3.5 barg)
- SMEG is injected into oligomer line
- Catalyst (to promote further reaction) is injected
- IPA is injected into oligomer line
- Premix additives (to control product quality) are injected into oligomer line
OLIGOMER SYSTEM - Process Description

PURPOSE
To transfer oligomer from the esterifier to the UFPP.
To add necessary additives to the oligomer during transfer
To complete esterification within the Line Reactor

HOW
Oligomer exit esterifier is pumped by a pair of positive displacement pumps through an HTF jacketed pipe
Four pairs of injection nozzles for additive addition
- One prior to the line reactor
- Three after line reactor
Oligomer passes through a Line Reactor (hold up vessel)
- Allowing completion of esterification

FEEDS
PTA Oligomer (1-6 polymer units long)
SMEG, DEG, IPA monomer, catalyst, toner and inhibitor.

PRODUCTS
PTA/IPA oligomer (with additives incorporated)
OLIGOMER SYSTEM – Equipment Description

Oligomer Pumps
- 2 positive displacement pumps
  - Both run at equal capacity
  - Gear pump heated with HTF vapour jacket
- Speed of pumps based on oligomer line pressure
- Run as a “pair” with auto-ramp if one fails

Oligomer Line
- Range of pressure from approx. 7 to 3.5barg pressure from oligomer pumps to FCV
- FCV to UFPP inlet pressure is negative (i.e. vacuum)
- HTF vapour to line jacket (oligomer approx 285degC)
- Oligomer flow linked to UFPP level

SMEG Injection
- Provides additional virgin glycol to give “chemistry” control
  - Ensure esterification is complete
    (titrate unreacted CEG’s)
- Provides additional EG to increase vapour loading for UFPP
  (improve mass transfer efficiency of the column)
OLIGOMER SYSTEM - Equipment Description

Line Reactor
- Tank with 5.65m³ with special internal design
- HTF vapour to jacket
- Provides residence time between SMEG addition and UFPP vessel

Additive injection
- Two pairs of injection nozzles utilised
  - Catalyst (antimony) addition before IPA addition
  - Toner and phosphoric acid addition after IPA addition

IPA
- Pair of injection nozzles utilised
  - Between catalyst and toner/inhibitor addition

Static Mixers
- Provided immediately after each set of injection nozzles
- Promote homogeneous mixing after addition of each material
OLIGOMER SYSTEM - Additive Injection Sequence

1st Injection Nozzle (pre line reactor)
  • “SMEG” Glycol (from SMEG Feed Tank – virgin EG)

2nd Injection Nozzle (post line reactor)
  • Antimony (Catalyst)
  • DEG

3rd Injection Nozzle (post line reactor)
  • IPA (Monomer)

4th Injection Nozzle (post line reactor)
  • Toner / Phosphoric acid
  • Stability Glycol (from Stability EG tank – process EG)
OLIGOMER SYSTEM - Additive Injection Sequence
## OLIGOMER SYSTEM - Additive Injection Sequence

<table>
<thead>
<tr>
<th>Additive</th>
<th>Source Tank</th>
<th>PID</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMEG</td>
<td>SMEG Feed Tank (2244-TO2)</td>
<td>503207</td>
</tr>
<tr>
<td>Antimony (Sb)</td>
<td>Poly Catalyst Feed Tank (1245-TO1)</td>
<td>503183</td>
</tr>
<tr>
<td>DEG</td>
<td>DEG Feed Tank (1241-TO1)</td>
<td>503167</td>
</tr>
<tr>
<td>IPA</td>
<td>IPA Solution Feed Tank (2216-TO3)</td>
<td>503274</td>
</tr>
<tr>
<td>Premix</td>
<td>Premix Feed Tank (1219-TO3)</td>
<td>503171</td>
</tr>
<tr>
<td>Stability EG</td>
<td>Stability EG Tank (2244-TO1)</td>
<td>503244</td>
</tr>
<tr>
<td>Dye (if required)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

![Diagram of the OLIGOMER SYSTEM](image-url)
OLIGOMER SYSTEM – Line Reactor

0.1 INCH GAP BETWEEN DISTRIBUTOR PLATE AND VESSEL WALL.
4 HOLES ON 7 INCH DIAMETER
16 HOLES ON 26 INCH DIAMETER
32 HOLES ON 26 INCH DIAMETER
64 HOLES ON 52 INCH DIAMETER
128 HOLES ON 65 INCH DIAMETER
HOLES ARE 3/8 INCH DIAMETER
OLIGOMER SYSTEM – Rate Control

**Oligomer**
- Pressure
- Flow (UFPP level)

**Additives**
- Additive flow retains correct ratio with oligomer flow
OLIGOMER SYSTEM- Control of Line Flow

THERMO-SYPHON LOOP

PIC

HIC

HIC

THERMO-SYPHON LOOP

ADDITIVE INJECTION

LINE REACTOR

UFPP

SMEG INJECTION

Simpe - Oct 2008 - Oligomer & Adds Inj
OLIGOMER SYSTEM - Quality

Oligomer
- Carboxly End Groups (C’s)EG
  - SMEG addition impacts downstream reactivity
- Temperature
  - Too hot
    - Impact on colour of final product
  - Too cold (excess SMEG)
    - Potential to freeze oligomer prior to UFPP

Additives
- Injection rate
  - Loss of catalyst injection will result in plant hold
  - Variable flow of other additives will result in product quality issues
OLIGOMER SYSTEM - Control Interlocks

For oligomer pumps:

**I-52** Hardwired trip that trips oligomer pump
- If motor temperature is high
- If discharge pressure is high high

**I-57A** Permissive to start
- If pump temperatures are NOT low low and
- If discharge pressure is NOT high high
OLIGOMER SYSTEM - Control Interlocks

For additive nozzles injecting into oligomer line:

See details in Additives section
OLIGOMER LINE SUMMARY

- Free flowing, hot liquid under pressure which releases significant fumes in air – potential for fire
- Additive injection rates are critical for product quality and UFPP / Finisher operation
- Potential for oligomer line to solidify causing blockages – potential for oligomer degradation leading to increase in pressure
- Degrading polymer is hazardous so EXTREME CAUTION is required
- Transition from pressure to vacuum occurs at flow control valve
UFPP
(Upflow Pre-polymeriser)
UFPP (PRE-POLYMERIZER)

- PREPOLYMERIZER
- POLYMERIZER
- CATALYST & ADDITIVES
- BASE CHIP
- POLYMER PUMP AND FILTER
**UFPP (UP-FLOW PRE-POLYMERISER)**

- Glycol enhanced mass transfer – no mechanical agitator
- Optimised Chemistry (End group balance) through staged pressure control (vacuum)
- Air exclusion system
**UFPP SYSTEM - Process Description**

<table>
<thead>
<tr>
<th><strong>Purpose</strong></th>
<th>- To initiate polycondensation process in a vacuum reactor at above 285degC to produce pre-polymer (average of 30 repeat units) and removes excess glycol</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>How</strong></td>
<td>- Uses a counter current, trayed reactor linked to an overhead vacuum system to remove glycol and promote the polycondensation reaction</td>
</tr>
<tr>
<td><strong>Feeds</strong></td>
<td>- PTA/IPA oligomer (with additives incorporated)</td>
</tr>
</tbody>
</table>
| **Products**| - Pre-polymer (average of 30 repeat units)  
             - Glycol (water content typically 8%) |
UFPP SYSTEM - Equipment Description

UFPP Preheater

- Single pass, vertical shell and tube heat exchanger
- Final large heat input to process
UFPP SYSTEM - Equipment Description

UFPP Vessel:

UFPP Vessel - Counter current
- 16 trays
- Plug flow
- Residence time = approx. 40 mins

Trays
- Provide residence time for the polymer and vapour to reach equilibrium
- Account for the majority of vessel hold-up
- Laminar flow regime

Risers
- Provide pressure drop to transfer polymer and vapour between trays
- 2-phase bubbly flow regime
- Spray over onto trays

Bubble cap
- Disentrainment device to remove liquid from vapour and prevent carryover
- Slots allow escape of vapour
- Vapour off-take is slightly off-centre
UFPP SYSTEM - Equipment Description

Primary Spray Condenser

- EG and water vapour extracted from UFPP (under vacuum) via vapour line to primary spray condenser

- Vapour enters at top and is cooled / condensed by EG sprays (co-current)

- Gravity drainage of liquid to hotwell via barometric leg

- Hydraulic probe attached to top of condenser to minimise solids build up (non continuous operation)

- Basket filters upstream of spray condensers remove entrained solids

- Shell and tube heat exchangers maintain EG temperature ca 45degC (cooling water on shell side)
HYDRAULIC PROBE
FROM UFPP
DOWNTHERM
NOZZLE RING
(SEE DETAIL ABOVE)
TO HOT WELL

TOP VIEW OF NOZZLES (9)
SPRAY NOZZLE (9 NOZZLES FROM SPRAY RING)

TO SECONDARY SPRAY CONDENSER

NOZZLE DETAIL
**Secondary spray condenser** (containing NEW equipment)

- Vapour enters at the bottom
- Chilled, counter current liquid EG spray (using chilled water)
- Gravity drainage of liquid to coldwell via barometric leg
- Coldwell level controlled by export to
  - CP recycle EG hold tank (1253-TO2)
- Secondary glycol loop circulation rate fixed
  - by pump capacity
- Glycol coolers are plate heat exchanger
  - using chilled water system

Coldwell tank (2253-TO1), coolers (2253-HO1A/B) and pumps (2253-PO1A/B) are all NEW equipment
UFPP SYSTEM - Equipment Description

**Air Exclusion:**

Nitrogen is provided to the UFPP system for the air exclusion system (segmented gaskets)

**Glycol Chillers:**

Chilled water is used as a heat transfer medium for the secondary condensing system to enable a low glycol temperature (typically 15 deg C) to be achieved (assisting vacuum performance)
SEGMENTED GASKET - Diagram
UFPP SYSTEM - Control Philosophy

**LEVEL** - Top tray level on UFPP is maintained by flow controller in the oligomer line

**PRESSURE** - UFPP top pressure is controlled by butterfly valve between spray condensers and steam ejectors

- Pressure profile through the vessel is controlled by the stability EG flowrate (added in the oligomer line)

**TEMPERATURE** - Exit temperature from the preheater is controlled by adjusting the HTF vapour pressure to the preheater shell
UFPP SYSTEM - Quality (pre-polymer)

There are no samples / tests made on material exit the UFPP

Final product quality is the ultimate measure of the polymer

However, it is vital that all parameters associated with the UFPP are operated at Standard Operating Conditions (SOC):

**Temperature:**
- High can result in colour issues and CEG dropping
- Low can result in poor IV lift

**Level:**
- High can result in colour issues and CEG dropping
- Low can result in poor IV lift

**Pressure** (vacuum):
- Hard vacuum can increase IV
- Soft vacuum can decrease IV

Air exclusion system must be monitored regularly – air leakage can have significant impact on final product colour.
UFPP SYSTEM - Control Interlocks

UFPP EG Condenser system

**I 27 A - Standby Glycol Circulation Pump**

Automatically starts whichever UFPP glycol circulation pump is in standby mode if low pressure is detected by the PSL in the glycol circulation line between the UFPP hot well and the UFPP pot filter. This interlock is active only if the hand switch on the DCS is set to the AUTO mode.

**I 13 - UFPP Glycol Hot Well Overflow Tank Sump Pump**

Activates the sump pump on the overflow tank according to high- and low-level switch. The interlock starts the pump at level high and stops the pump at level low. When activated by the interlock, the pump transfers glycol to the crude glycol tank in the tank farm.
UFPP SYSTEM - Control Interlocks

Secondary Spray Condenser system

I 29A - Glycol circulation pumps 2253 PO1A/B trip on low suction pressure. In Auto mode low discharge pressure will start the standby pump
* the low suction pressure trip will override the auto start of the standby pump on low discharge pressure.

Hydraulic probe

I 14 - Probe sequencing – in Auto mode probe cycles up / down one cycle

I 15 - Open hydraulic relief device on high discharge pressure of hydraulic pump, close hydraulic relief device when pressure returns to normal.

I 30 - Trip motor for hydraulic unit on high oil temperature
UFPP SYSTEM - SUMMARY

- Second key process vessel converting oligomer into pre-polymer
  - average of 30 repeat units
- Preheater is used to “re-heat” cooled PTA / IPA oligomer with additives incorporated
- Glycol must be removed from the oligomer to promote polycondensation reaction
  - achieved by using vacuum
- Four stage steam ejectors (linked to the UFPP via a primary and secondary condenser) create the required vacuum
- Polymeric material carried over into the spray condensers and the circulating glycol system make them prone to blockages, requiring careful monitoring and cleaning
FINISHER
FINISHER
- Unique cage-type finisher agitator with no central shaft
- Continuous wiping of finisher vessel wall eliminating degradation
- Air exclusion system
FINISHER SYSTEM - Process Description

**Purpose**
- To continue polycondensation reaction in a vacuum reactor at above 280degC to produce polymer (average of 100 repeat units) and removes excess glycol. Residence time typically 80mins.

**How**
- Uses a “wipe wall” screen reactor to promote mass transfer via a large surface area. Reactor is linked to an overhead vacuum system to remove glycol and promote polycondensation reaction.

**Feeds**
- Pre-polymer (average of 30 repeat units) with IV typically 0.20

**Products**
- Polymer (average of 100 repeat units) with typical IV 0.60
- Glycol (water content typically 1-2%)
FINISHER SYSTEM - Equipment Description

- Horizontal, cylindrical vessel

- Dowtherm jacketed

- Complex shaftless agitator
  - Glycol cooled seals at both ends of agitator shaft
  - Driven from outlet end of vessel

- Screens perpendicular to vessel axis
  - Three screen types that vary in size and construction through vessel as polymer viscosity increases:
    - Increased mesh size
    - Increased screen spacing
FINISHER SYSTEM - Glycol Overview
Primary Spray Condenser

- EG and water vapour extracted from Finisher (under vacuum via vapour line to primary spray condensor)

- Vapour enters at top and is cooled / condensed by EG sprays (co-current)
  - Gravity drainage of liquid to hotwell via barometric leg
  - Hydraulic probe attached to top of condenser to minimise solids build up (non continuous operation)
  - Basket filters upstream of spray condensers remove entrained solids
  - Shell and tube heat exchangers maintain EG temperature ca 45degC (cooling water on shell side)
SECONDARY SPRAY CONDENSER

- Vapour enters at bottom
- Chilled, counter current liquid EG spray (using chilled water)
- Gravity drainage of liquid to coldwell via barometric leg
- Finisher coldwell level maintained via an overflow to UFPP coldwell
- Circulation rate fixed by pump capacity
- Plate heat exchanger using a chilled water system
FINISHER SYSTEM – Equipment Description

Ejectors

Pressure (vacuum) within individual reactors is controlled by pressure control valves on individual secondary spray condensers

Two 4 stage steam ejectors are installed (one on line/ one standby)

1\textsuperscript{st} Stage :

- HTF vapour jacket
- Steam supply is superheated
- Finisher vapour enters
- Vacuum capability typically <2mbar

2\textsuperscript{nd} Stage :

- HTF vapour jacket
- UFPP vapour enters
- Vacuum capability typically <8mbar

Interstage condensing after the second and stages is achieved by a shell and tube, water condensing heat exchanger
FINISHER SYSTEM – Equipment Description

Ejectors

3rd Stage :
   - Vacuum capability typically <20mbara
Interstage condensing after the third stages is achieved by a shell and tube, water condensing heat exchanger
4th Stage :
   - Vacuum capability typically <200mbara
Condensing heat exchanger after the fourth stage minimises emission of volatile organics to atmosphere

All condensing heat exchangers are linked by baromateric legs to a condenser water hotwell
This liquid is then sent to OSC feed tank prior to OSC processing to remove VOC’s
FINISHER SYSTEM – Equipment Description

Air Exclusion:

Nitrogen is provided to the Finisher system for the air exclusion system (segmented gaskets).

Glycol Chiller:

Chilled water is used as a heat transfer medium for the secondary condensing system to enable a low glycol temperature (typically 15 degC) to be achieved (assisting vacuum performance).
FINISHER SYSTEM - Overview Diagram

- FINISHER
- MOTOR AND GEARBOX
- FINISHER LCV
- POLYMER PUMP
- PRIMARY CONDENSER
- TO SPRAY RING
- HYDRAULIC PROBE
- HEAT EXCHANGERS
- BASKET FILTERS
- BAROMETRIC LEG
- POLYMER FILTERS
- HOTWELL
- WASTE DISPOSAL UNIT
- CATCH TANK
- EG CIRCULATION PUMP
- A EJECTOR
- B EJECTOR
- SECOND CONDENSER
- TO CHIPERS
FINISHER SYSTEM - Control Principles

Level

- Measured by HTF jacketed Nitrogen bubble tubes
  - two at each end of the vessel
- Inlet level is measured by pressure drop down tubes
  - controls the butterfly valve between
    UFPP and the Finisher

Pressure

- Finisher pressure (vacuum) controlled by butterfly valve between
  secondary spray condenser and ejectors
- Cascade loop from IV measurement (TOV’s) sets Finisher pressure

Temperature

- Minor changes to temperature made by adjustment of HTF
  vapour pressure to Finisher vessel jacket
- Principle heat input existing from the UFPP preheater
FINISHER SYSTEM - Control Principles

Viscosity

- Torsional Oscillatory Viscometer (TOV) installed in transfer line to measure polymer viscosity
  - Viscometer sends signal to the Finisher pressure (vacuum) controller

Agitator Speed

- Set by hand
  - Speed set for
    - Viscosity requirement
    - Minimising overhead fouling (entrainment)
FINISHER SYSTEM - Glycol Control

STABILITY EG TANK (2244-TO1) → BASKET FILTERS → CIRCULATION PUMPS → WDU → CATCH TANK → TO OVERFLOW TANK (1252-TO2)

FINISHER PRIMARY SPRAY CONDENSER

TO OVERFLOW TANK (1252-TO2)

CATCH TANK

[Level] FROM CP RECYCLE EG HOLD TANK (1253-TO2) LIC

FINISHER HOTWELL (1262-TO1) → TO SUMP PUMP (IMPURE GLYCOL – CEG TANK)

VIRGIN EG
FINISHER SYSTEM - Quality (Polymer)

There are no samples / tests made on material exit the Finisher.

Final product quality is the ultimate measure of the polymer.

However, it is vital that all parameters associated with the Finisher are operated at S.O.C.

**Temperature:**
- high can result in colour issues and CEG dropping
- low can result in poor IV lift

**Level:**
- high can result in colour issues and CEG dropping
- low can result in poor IV lift

**Pressure** (vacuum):
- hard vacuum can increase IV
- soft vacuum can decrease IV

**Agitator speed:**
- high speed can increase IV (excess carryover of entrained solids)
- low speed can reduce IV

Air exclusion system must be monitored regularly – air leakage can have significant impact on final product colour.
FINISHER SYSTEM - Control Interlocks

I 1  - Trip inverter on motor winding temperature high high

I 21A - Permissive to start finisher agitator if seal flow not low

I 14  - Probe sequencing (in auto mode probe cycles up-down one cycle)

I 15  - Open hydraulic relief device on high discharge pressure of hydraulic pump
        close hydraulic relief device when pressure returns to normal

I 30  - Trip motor for hydraulic unit on high oil temperature

I 29A - Trip SSC circulation pump on low suction pressure
        (in auto mode on low discharge pressure start standby pump)
        Low suction pressure shall override auto start of standby pump
FINISHER SYSTEM - SUMMARY

- Third and final key process vessel converting pre-polymer to polymer
  - Average of 100 repeat units
- No additional, significant heat input is provided from the HTF system
- Glycol must be removed from the pre-polymer to promote polycondensation reaction
  - Achieved by using vacuum
- Four stage steam ejectors (linked to Finisher via primary and secondary condenser) create the required vacuum
- Polymeric material carried over into the spray condensers and the circulating glycol system make them prone to blockages, requiring careful monitoring and cleaning.
- Finisher controls the final IV for the polymer by adjusting reactor pressure (vacuum). The IV is measured using a TOV which must be carefully maintained & calibrated.
- Loop seal allows the UFPP and Finisher to operate under different pressures
- Increased viscosity of the process material in Finisher means the internal agitator is mechanically complex
  - Special care of the seals to prevent damage or air ingress
EXTRUSION
& CHIPPING
EXTRUSION, FILTER & CHIPPING

CATALYST & ADDITIVES

BASE CHIP

POLYMER PUMP AND FILTER
Extrusion line

Finisher

Gear pump

TOV

Polymer Filter

Polymer Filter

To chip cutters
Extrusion & Chip Cutting - Diagram
EXTRUSION SYSTEM - Process Description

Purpose
- To convert molten polymer leaving Finisher into filtered and cooled polymer chips

How
- Positive displacement gear pump (Maag) transfers the molten material though an HTF jacketed pipe
- Molten material filtered (60 micron)
- Extruded through a die plate prior to cutting
- Pre-determined size of chip:
  - cutter speed and die head orifice size
- Pair of Torsional Oscillatory Viscometers (TOV) used to measure viscosity of melt
- TOV’s cascade the signal back to the controlling Finisher vacuum system
EXTRUSION SYSTEM - Process Description

**Feeds**
- Polymer leaving Finisher (average of 100 repeat units) with IV typically 0.60

**Products**
- Polymer in chip form as per product specification
- Cool (70 degC) and non crystalline (amorphous)
EXTRUSION SYSTEM - Equipment

Maag polymer pump

- Positive displacement gear pump
- Typically operating with discharge pressure range 75 – 125 bar.
- Pump jacket heated by vapour HTF

Jacketed transfer line

- HTF vapour heated line incorporating 4 sets of static mixers
  - 3 prior to polymer filtration and 1 after
- Torsional Oscillatory Viscometers (TOV's)
- Two TOV's are installed on the discharge side of the Maag polymer pump
- Measure melt viscosity
EXTRUSION SYSTEM - Equipment

Duplex polymer filter

- Pair of stainless steel polymer filters
  - one in service / the other on standby
- Maximum pressure drop across the filter is typically 70barg
- Filters remove gels and other impurities that could impact product quality
- Filters are changed frequently and the candles are cleaned for re-use

Die heads

- Transfer line splits into 3 streams after filtration
  - Each line terminates in a die head
  - Molten polymer is extruded as laces

Chip cutters

- Three USG 600 Reiter chip cutters (each typically capable of 8.33t/hr)
- Molten laces are cooled in a water spray before entering the cutting head
EXTRUSION SYSTEM - Equipment

Driers & Classifiers

- Cut chips are conveyed in a water stream to the driers
- Chips pass through screens and blown by compressed air
- Classifiers segregate non standard size chips
- Only correct size chips pass into the chip buffer tank

Materials of construction

- Carbon steel used from Maag polymer pump forwards
  - Due to relatively low quantities of “free” glycol
EXTRUSION SYSTEM - Transfer Line Control

Viscosity

- Measured by two torsion oscillatory viscometers (TOV’s)
- Located in series in the Transfer Line
  (downstream of the Maag pump)
- Both TOV’s equipped with temperature and pressure compensation
- Viscosity signal is transmitted via a selector switch to the Finisher pressure controller

Pressure

- Total of five pressure transmitters:
  - Three pressure transmitters are located in first section of transfer line
    (after Maag polymer pump)
  - Two pressure transmitters are located after the polymer filter

- First pressure transmitter is linked to the high pressure trip interlock
  (Maag polymer pump)
- Other four transmitters used to monitor differential pressure and form pressure control loop feeding back to Maag polymer pump inverter
EXTRUSION SYSTEM - Transfer Line Control

Temperature

- Transfer line heating system primarily to maintain the polymer temperature between the reaction vessels and extrusion
- No significant additional heat input is provided
- Pressure controller maintains the HTF vapour at a set pressure
EXTRUSION SYSTEM - Transfer Line Control Diagram
Transfer Line – Pressure Control Diagram
Transfer Line – Viscosity Control Diagram
EXTRUSION SYSTEM  - Quality

Product is sampled in chip form exit the classifiers

The following parameters are tested :

- IV (for TOV calibration)
- B*
- L*
- CEG
- DEG
- Retained Antimony
- Retained Cobalt
- Retained Phosphorous
- Chip size

Each parameter has a target value and acceptable ranges

Control of the quality parameters largely depends on operating the plant at Standard Operating Conditions (SOC) however fine tuning is typically required
EXTRUSION SYSTEM - Interlocks

Polymer Pump

I 19A - Temperature permissive for start if polymer temp and pump temp not low low
- If vent temp not less than 280degC

I 2 - Trip inverter if motor winding temp high high or pump discharge pressure high high

I 20A - Close HTF supply valve on high temperature

Chippers

PLC Stop - E stop activated
- Light barrier of the strand section is blocked
- Low water flow
- Low instrument air pressure
- Cutter frame not located
- Cutter cover is open
- Dryer/classifier not running
- Buffer tank level high

I 20A - Close HTF supply valve on high temperature
EXTRUSION SYSTEM SUMMARY

- This area has potential to expose personnel to:
  - Hazardous molten polymer
  - Sharp rotating equipment
  - Water droplets with bacterial growth
- Risk assessment, formal work control methods and personnel protection are essential
- Polymer is pumped at very high pressures through a filter to remove any contamination
  - Line & equipment are protected by a high pressure trip
- Polymer pump has a complex seal arrangement to prevent air ingress & lubricate the bearings
- Polymer filters get dirty and must be changed for cleaning
  - Requires venting & draining the filter housing
- Degrading polymer is hazardous so EXTREME CAUTION is required
EXTRUSION SYSTEM SUMMARY

- Chippers & dryers are complex, continuously rotating machines
  - Essential that regular maintenance is planned and completed
  - Essential that a defined set of critical spares is available for use
  - Electrical / Instrument as well as Mechanical work load will be required

- Chip cut must be monitored for quality problems
  - Indicative of blunt rotors, “gaps” incorrectly set etc
  - Sampling frequency must be followed (maximum hourly on start up)
GLYCOL
GLYCOL SYSTEM – Overview Drawing (Virgin EG)
GLYCOL SYSTEM – Overview Drawing (Crude EG)
• GLYCOL SYSTEM - Process Description

• Mono ethylene glycol (EG)
  • Required for fundamental chemical reaction

• But also has many other uses:
  • Used as a carrier for powders (PTA and IPA)
  • Used to promote natural thermosyphon in esterifier
  • Desuperheater medium for esterifier column
  • Used as a carrier for all additives
  • Used to enhance mass transfer in UFPP
  • Condensing medium for vapour evolved from polymerisation vessels

•
• GLYCOL SYSTEM - Process Description

• Di ethylene glycol (DEG)
  - Injected into oligomer line as part of additive sequence
  - To facilitate chip crystallisation (SSP)

• Tri ethylene glycol (TEG)
  - Can be used for cleaning at plant shutdown
  - Can be used for polymer filter cleaning
  - Typically utilised near its boiling point
  - Extreme fire risk!
GLYCOL SYSTEM – Process Overview

- Virgin EG only added at the “end” of the process (Finisher hotwell)
  - This volume replaces EG used to make monomer by esterifier
  - Virgin EG (low water content) added to assist vacuum performance
GLYCOL SYSTEM – Overview Drawing (internal process EG)

- SMEG Feed Tank (2244-TO2)
- Stability EG Tank (2244-TO1)
- Esterifier EG Tank (2223-TO2)
- CP Recycle EG Hold Tank (1253-TO2)
- TEG Circ. Pump
- Catalyst Mix Tank
- Inhibitor Mix Tank
- Toner Mix Tank
- Finisher Agitator Seal
- Slurry Mix Tank (2213-TO1)
- Finisher Hotwell (1262-TO1)
- UFPP Coldwell (2253-TO1)
- Finisher Coldwell (1253-TO1)
- UFPP Hotwell (1262-TO1)
- IPA Slurry Mix Tank (2216-TO1)
- SMEG Feed Tank (2244-TO2)
- Stability EG Tank (2244-TO1)
- Esterifier EG Tank (2223-TO2)
- CP Recycle EG Hold Tank (1253-TO2)
- TEG Circ. Pump
- Catalyst Mix Tank
- Inhibitor Mix Tank
- Toner Mix Tank
- Finisher Agitator Seal
- Slurry Mix Tank (2213-TO1)
- Finisher Hotwell (1262-TO1)
- UFPP Coldwell (2253-TO1)
- Finisher Coldwell (1253-TO1)
- UFPP Hotwell (1262-TO1)
- IPA Slurry Mix Tank (2216-TO1)

- NC (SU&SD only)
- NC (SU&SD only)
GLYCOL SYSTEM - Process Description

The following table indicates where glycol is used on the plant, and under what condition it is used:

<table>
<thead>
<tr>
<th>System</th>
<th>Type</th>
<th>Supplemented</th>
<th>Condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Additives preparation</td>
<td>Virgin EG</td>
<td>Virgin EG</td>
<td>Ambient temp</td>
</tr>
<tr>
<td>SMEG injection</td>
<td>Circulating EG</td>
<td>UFPP/Finisher hotwells</td>
<td>Ambient temp</td>
</tr>
<tr>
<td>Slurry make up</td>
<td>1-Circulating EG</td>
<td>CP recycle</td>
<td>Ca 40deg C</td>
</tr>
<tr>
<td></td>
<td>2-Est hotwell EG</td>
<td>Est hotwell</td>
<td>Ca 165deg C</td>
</tr>
<tr>
<td>UFPP 1° Condenser</td>
<td>Circulating EG</td>
<td>SMEG head tank</td>
<td>Ca 40 deg C</td>
</tr>
<tr>
<td>Finisher 1° Condenser</td>
<td>Circulating EG</td>
<td>Virgin EG</td>
<td>Ca 40 deg C</td>
</tr>
<tr>
<td>UFPP 2° Condenser</td>
<td>Circulating EG</td>
<td>Overflow from Finisher coldwell</td>
<td>Chilled : ca 15 deg C</td>
</tr>
<tr>
<td>Finisher 2° Condenser</td>
<td>Circulating EG</td>
<td>From Finisher agitator seal tank</td>
<td>Chilled : ca 15 deg C</td>
</tr>
<tr>
<td>Finisher agitator seal tank</td>
<td>Circulating chilled EG</td>
<td>Finisher coldwell</td>
<td>Chilled : ca 15 deg C</td>
</tr>
<tr>
<td>Esterifier separation column</td>
<td>Est hotwell EG</td>
<td>Est hotwell</td>
<td>Ca 165 deg C</td>
</tr>
<tr>
<td>DEG injection</td>
<td>Clean DEG</td>
<td>DEG storage</td>
<td>Ambient temp</td>
</tr>
</tbody>
</table>
GLYCOL SYSTEM – Equipment Description

**Bulk storage (external)**

- MEG tank (S11) – capacity 640M³ (bund capacity 659M³)
  Distribution – centrifugal pumps x 2 (from tank to plant)

- DEG tanks (2) – capacity 30M³ each – no bund
  Distribution – centrifugal pumps x 2 (from tank to plant)

- CEG tank (S35 - used material for export) – capacity 200 M³
  (bund capacity 60M³)
  Distribution – centrifugal pumps x 2 (to feed distillation)

- TEG tanks (used material for export) – capacity 30M³ +100M³ in CP3
  Distribution – centrifugal pumps x 1

- TEG tanks (clean material) – capacity 30M³+100M³ in CP3
  Distribution – centrifugal pumps x 1
  No bunds around 30M³ tanks but CP3 has 271M³ bund
GLYCOL SYSTEM – Equipment Description

Intermediate storage (internal - process)  total volume = 80M³

Esterifier hotwell :
Capacity – 12M³
Circulation – centrifugal pumps x 2 (1 running and 1 standby – auto c/o)
Filtration – not installed

CP recycle tank:
Capacity - 45M³
Circulation – centrifugal pumps x 2
Filtration – not installed

UFPP hotwell:
Capacity – 6.5M³
Circulation – centrifugal pumps x 2
Filtration – basket strainers 10 mesh
Heat exchanger – shell & tube (x6 pass) cooling medium : cooling water
GLYCOL SYSTEM – Equipment Description

Finisher hotwell:
- Capacity – 6.5M³
- Circulation – centrifugal pumps x 2
- Filtration – basket strainers 20 mesh
- Heat exchanger – shell & tube, cooling medium – cooling water

UFPP coldwell (old):
- Capacity - 5M³
- Circulation – centrifugal pumps x 2
- Filtration – none installed
- Heat exchanger – plate and frame, cooling medium - chilled water

Finisher coldwell:
- Capacity - 5M³
- Circulation – centrifugal pumps x 2
- Filtration – none installed
- Heat exchanger – plate and frame, cooling medium - chilled water
GLYCOL SYSTEM - Control

Esterifier Hotwell

Temperature
- Defined by the temperature of glycol evolved from the esterification reaction (typically 165degC)
- Hotwell and some of the pipework is steam traced to minimise solids separation

Flow
- Provides desuperheat spray flow to separation column
  - Flow is maintained at a constant rate by means of a flow transmitter and FCV located in the pump discharge line
- Provides flow to PTA slurry mix / feed system
  - Flow is dependant on plant rate and is described in the ‘Slurry ‘section

Level
- Controlled as part of the slurry make up sequence
- Tank has a relatively small inventory (12m3) and must be carefully controlled
GLYCOL SYSTEM - Control

UFPP Hotwell

Temperature
- Maintained at a constant 45 degC.
- Circulating glycol passes through a shell and tube heat exchanger using tempered water on the shell side

Flow
- Provides flow of glycol to spray condenser
- Flow measured by an orifice plate in the lines to each of the two spray rings
  - no flow control function
- Constant flow rate is delivered by the fixed speed centrifugal pumps

Level
- Hotwell level is maintained constant by exporting excess volume to the CP recycle tank
- Level must never be allowed to fall below the bottom of the barometric leg
  - Would allow air to be sucked into the vessel and create a potentially explosive atmosphere
GLYCOL SYSTEM - Control

Finisher Hotwell

Temperature
- Maintained at a constant 45 degC
- Circulating glycol passes through a shell and tube heat exchanger using tempered water on the shell side

Flow
- Provides flow of glycol to the spray condenser
- Flow measured by an orifice plate in the lines to each of the two spray rings
  - No flow control function
- Constant rate delivered by the fixed speed centrifugal pumps

Level
- Hotwell level is maintained constant by exporting excess volume to the stability EG tank
- Level must never be allowed to fall below the bottom of the barometric leg
  - Would allow air to be sucked into the vessel and create a potentially explosive atmosphere
GLYCOL SYSTEM - Control

UFPP Coldwell

Temperature
- Maintained at a constant 15deg C
- Circulating glycol cooled in a Plate & Frame heat exchanger
- Cooling medium is chilled water

Flow
- Glycol flow is measured before the secondary condenser
- Hand valves are used to set desired constant flow rate
- No control function

Level
- Coldwell level is supplemented by the overflow from the Finisher coldwell
- Excess level is exported to the crude glycol tank
GLYCOL SYSTEM - Control

Finisher Coldwell

Temperature
- Maintained at a constant 15deg C
- Circulating glycol cooled in a Plate & Frame heat exchanger
- Cooling medium is chilled water

Flow
- Glycol flow is measured before the secondary condenser
- Hand valves are used to set desired constant flow rate
- No control function

Level
- Coldwell level is supplemented by the return from the Finisher agitator seal tank
- Excess level overflows to the UFPP coldwell
GLYCOL SYSTEM - Control

CP Recycle Tank

Feeds in – Export from UFPP coldwell
Export from UFPP hotwell
Export from Overflow tank (special circumstance)

Exports – To slurry mix tank for slurry preparation
To CEG tank (special circumstance)

Temperature – not controlled but should always be ca 30degC
Flow – export flow will be determined by slurry make up requirement
Level – recycle tank level controls the VEG flow into the Finisher hotwell
GLYCOL SYSTEM - Quality

MEG & DEG are supplied with certificates of analysis when delivered confirming product is within the agreed specification.
Lab checks may be required for MEG (GC absorbance and pH)

Esterifier hotwell glycol is not typically routinely tested due to the operating temperature. The following table indicates the typical routine testing carried out on other glycol streams:

<table>
<thead>
<tr>
<th></th>
<th>Water</th>
<th>HTF</th>
<th>pH</th>
<th>DEG</th>
<th>Sb</th>
<th>Co</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP recycle tank</td>
<td>Daily</td>
<td>Daily</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
</tr>
<tr>
<td>UFPP hotwell</td>
<td>Daily</td>
<td>Daily</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
</tr>
<tr>
<td>Finisher hotwell</td>
<td>Daily</td>
<td>Daily</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
<td>3 times per week</td>
</tr>
</tbody>
</table>
GLYCOL SYSTEM – Control Interlocks

I 13 – Stops sump pump on low level and starts pump on high level

I 27A – (UFPP) Trips glycol circulation pumps on low suction pressure
         on low discharge pressure standby pump starts
         low suction pressure trip overrides auto start of standby pump

I 1 - Trips inverter on motor winding temperature high

I 29A – Same as I 27A (Finisher)

I 25A – In auto mode on low pressure start standby CP recycle pump

I 31A – Open DEG supply valve to feed tank on low level and close valve on high level

I 28 – Trip permissive to start impure TEG transfer pump if temperature is above 100degC.

I 29 – Trip permissive to start respective transfer pump if grounding connection for respective tanker / tank is not done or broken.
GLYCOL SYSTEM - SUMMARY

- Glycol is used in 3 forms: MEG, DEG, TEG (for cleaning only)

- Potential for fire & explosion as process temperatures are in excess of MEG flash point

- Pipework designed to minimise leak potential

- Virgin MEG is added at the end of the process (Finisher hotwell)

- Glycol temperature in polymeriser condensing systems carefully controlled for optimum vacuum performance

- Regular testing required to ensure no breakthrough of other process materials into the glycol system
Heat Transfer Fluid
HEAT TRANSFER FLUID (HTF)

- HTF vaporized and sent to the process units for heat transfer
- HTF Vaporizer burner used to utilize heat value from the organics gases collected at the Organic Stripping Column (reducing emissions)
HEAT TRANSFER FLUID (HTF)

- HTF vaporized and sent to the process units for heat transfer
- HTF Vaporizer burner used to utilize heat value from the organics gases collected at the Organic Stripping Column (reducing emissions)
HTF SYSTEM - Process Description

Split into two key parts:

Primary (liquid):
Consisting of
- Storage tanks
- Liquid circulating pumps
- Vaporisers / flash tanks
- Vapour header
- Condensate tanks (x3)

Secondary (vapour):
Consisting of
- Multiple “users” that all have
  - Desuperheater loops
  - Condensate traps
  - Common vent header
  - Common vent collection tank
HTF SYSTEM - Overview (Process)
HTF SYSTEM - Overview (Process)

New Equipment

Esterifier HTF Condensate Tank

Tank receives HTF condensate from
   Esterifier
   IPA solution processor (liquid)
   Oligomer drain line
   Hot condensate tank

HTF is pumped from tank primarily back to vapourisers
IPA solution processor and oligomer drain line take a feed from this stream

Why a new tank?

Due to the differing pressures of users for operation
   e.g. difficult to return condensate from chipper system (typically operating at 1.8barA) to
   esterifier HTF condensate tank where the user is operating at ca 3.2 barA.
   Condensate would not be able to return to the tank - would back up into the user resulting
   in loss of temperature control

Overpressure Relief:

Relief system located on top of tank set at 5.3kg/cm²g
Relieves to HTF containment tank
HTF SYSTEM - Overview (Process)

New equipment

IPA Solution Processor

IPA slurry heated to ca 180degC in solution processor
Vessel is equipped with internal coils around which liquid HTF is pumped
HTF is pumped from and returns to the esterifier HTF condensate tank
Heating time typically 3-4 hours
Temperature control is via control valve on the HTF return line from processor
  Adjusts the valve position depending on temperature of material inside processor

Overpressure Relief :

Relief valve on inlet line to processor set at 4.7 kg/cm²g
Relieves to HTF containment tank
HTF SYSTEM – Process Description

Primary (liquid) :

Purpose  To generate and then recover (closed loop) HTF vapour that is utilised to provide heat to various different users (key plant items)

How  Liquid HTF is heated rapidly (using natural gas) to approx 340degC at a pressure of approx 3.5bara generating a vapour. “Used” vapour is collected as a condensate (liquid) and then returned to the start of this process (closed loop)

Feeds  Liquid HTF (from storage tank initially)
Natural gas
OSC gas stream (VOC’s from esterifier column)

Products  Vapour HTF (at required temperature and pressure)
HTF SYSTEM – Process Description

Secondary (vapour):

Consisting of multiple “users” that all have
- Desuperheater loops
- Condensate traps
- Common vent header
- Common vent collection tank
HTF SYSTEM – Equipment Description

**Typical vapour system**
Primary HTF vapour desuperheated by liquid spray of HTF condensate
Saturated vapour supplies the user
Inerts removed from system by common vacuum / vent system
Condensate returns to either hot or cold condensate tanks via trapping arrangement

**Liquid system**
Primarily used for cooling the process
Flow is counter current to the process
Centrifugal pumps convey HTF around the system
Mixing valves blend hot and cooled HTF to achieve the correct temperature
HTF SYSTEM - Basic Description

HTF vapour systems are essentially identical. Primary HTF vapour is fed to a desuperheater where it is reduced in pressure, desuperheated and then supplies the user. Condensate is returned via trap sets to either the hot or cold condensate tanks (depending on temperature). The condensate is then returned to the HTF generation system. The HTF users are linked to a common vent system used to remove inerts from the HTF.

The relationship between pressure and temperature for HTF vapour is such that excellent control of temperature can be achieved by controlling the pressure. Typical points in the relationship are:

<table>
<thead>
<tr>
<th>Pressure Bar (psig)</th>
<th>Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (15)</td>
<td>292</td>
</tr>
<tr>
<td>2 (30)</td>
<td>314</td>
</tr>
<tr>
<td>3 (45)</td>
<td>332</td>
</tr>
</tbody>
</table>

These temperatures are typical of those used in a CP polymer process. They are achieved by letting down the pressure of the primary HTF (ca 4 bar) to the desired figure by means of control valves. However, doing this inevitably adds some superheat to the vapour.
**Desuperheaters**

Superheat is undesirable for two reasons:

1. Temperature control is not so good because some parts of the process can get hotter than specified.
2. Superheated vapour reduces heat transfer coefficient because it can prevent condensation locally.

For this reason, as soon as the HTF vapour has been reduced in pressure, it passes through a desuperheater. This is a small vessel in which the vapour enters near the bottom, and flows upwards against a spray of hot liquid which is introduced from the top. Enough of the liquid evaporates to remove the superheat, and the vapour which emerges from the desuperheater is slightly wet, and so will give good heat transfer and accurate temperature. Adjustment of the rate of spray is manual: the regulating valve is opened until the temperature of the vapour corresponds to saturation temperature at the pressure to which it has been controlled.

**Condensate Return**

After the vapour has performed its heating duty, it condenses, and has to be removed from the system. This is done with traps, which are similar to conventional steam traps. The trap allows the system to drain out liquid but retains the vapour. This does not apply to the Esterifier heat exchanger. Because of the high heat load and condensate flow, an extremely large trap would be needed. Instead, the condensate is returned to a level pot which is discharged by a control valve which maintains a constant level.
Desuperheater system
IPA SYSTEM - Hazards

- Static electricity
- Potential for IPA dust explosion
- Potential for Nitrogen asphyxiation
- Potential for Slips, trips and falls from spilling IPA powder on the floor
- Potential for personnel exposure to respiratory irritation due to IPA dust in the air
- Potential for thermal burns when IPA is in oligomeric/solution state
IPA SYSTEM – Overview Drawing

• IPA FEED HOPPER
• CONVEYOR
• IPA SLURRY MIX TANK
• SEPARATION COLUMN
• IPA SOLUTION PROCESSOR
• 10 BAR STEAM
• IPA SOLUTION FEED TANK
• CONDENSATE
• TO INJECTION SYSTEM
IPA SYSTEM – Process Description

PURPOSE
To feed the process with correct quantity of IPA essential for product quality.

HOW
IPA powder mixed as a slurry with EG. The slurry is then reacted under heat to form a monomer which is injected to the oligomer line.

FEEDS
IPA – (isophthalic acid) combined as one stream
MEG – (mono ethylene glycol)

PRODUCTS
IPA/MEG monomer
• IPA SYSTEM – Equipment Description

• Feed Silo
• - Existing equipment
• - Capacity : 80tes
• - Vessel is nitrogen blanketed and equipped with nitrogen aeration rings

• Tubular Chain Conveyor
• - New equipment–
• - See web site (www.schrage.de)

• IPA Slurry Mix Tank
• - New equipment
• - Equipped with single speed agitator
• - Vent scrubber mounted on top of mix tank linked to a seal pot

• EG Heater
• - Shell and tube heat exchanger heated by 3.5 bar steam
• - Will raise glycol temperature to 100degC.
IPA SYSTEM – Equipment Description

Slurry Transfer Pumps
- 2 centrifugal pumps transfer slurry to the solution processor
- Combined flow / density meter located in common delivery line from the pumps

Basket Strainers
- 2 basket strainers located between the slurry mix tank and transfer pumps

IPA Feed Hopper (temporary)
- Alternate IPA discharge source provided should IPA chain conveyor fail
- IPA feed hopper attached to overhead beam and can be moved in/out of position as required
- Transition piece connects the hopper to mix tank via an 8” spare nozzle located on the mix tank top

IPA Solution Processor
- Vessel equipped with a single speed agitator
- Separation column is mounted directly on top of the processor vessel
- Knock back condenser located on top of separation column
  - temperature controlled using cooling water
- Vessel heated to 180degC by means of an internal HTF coil
- Vessel overpressure protection by 2 rupture discs set at 1 kg/cm².
IPA SYSTEM – Equipment Description

**Water Collection Tank**
- Water from the knock back condenser collected in water collection tank before transferring to OSC feed tank

**IPA Solution Feed Tank**
- Material from IPA solution processor gravity fed to the feed tank
- Feed Tank equipped with single speed agitator
- Vent condenser located on top of feed tank connected to seal pot by 4” line and temperature controlled by cooling water

**IPA Solution Feed Tank Pumps**
- 2 centrifugal pumps transfer material from solution feed tank to additive injection nozzles
- Kick back line on discharge of pumps available if injection is stopped
  (to return material back to solution feed tank)
- Injection flow measured by pair of mass flow meters.
• IPA SYSTEM - Process Flow

• IPA Batch Preparation
  - Tubular chain conveyor transports powder from existing silo to slurry mix tank
  - Back up system (hopper & big bags) available should conveyor fail
  - EG preheated to 100degC & charged in 4 stages to slurry mix tank
  - Powder added until mole ratio of 4.5:1 achieved (EG to IPA)
  - Continual density monitoring by combined mass flow / density meter

• IPA Solution Processor
  - Prepared slurry is pumped into solution processor
  - HTF coils in the processor heat the solution to 180degC (3-4 hrs)
  - Water produced in the reaction removed by separation column
  - Solution transferred by gravity to IPA feed tank

• IPA Feed Tank
  - IPA solution pumped into the oligomer line by centrifugal pumps
  - Flow monitored by mass flow meters
ORGANIC STRIPPING COLUMN (O.S.C.)
O.S.C. SYSTEM
O.S.C. SYSTEM - Process Description

**PURPOSE**
Removal of by-product volatile organic compounds from process wastewater

**HOW**
VOC’s are stripped out of the wastewater by steam

**FEEDS**
- Reaction water from esterifier
- Overflow from the vent seal pot
- Waste water flow from IPA water collection tank
- Waste stream from existing facility

**PRODUCTS**
- Vapour stream (ca 18% by weight VOC) fed directly to vaporiser
- Stripped waste water pumped to effluent pit
O.S.C. SYSTEM – Equipment Description

Stripping Column (packed)

- Column is 480mm in diameter
- 3 packed beds of pall rings
- 16.48m tall (tan line to tan line)
- Wastewater fed to the top of the column
- Steam fed to the bottom of the column
  - Temperature range 100 – 111degC
- Steam is flow controlled
  - Set point received from ratio controller linked to waste water feed rate
- VOC stripped from the water by steam
- Vapour leaving OSC fed to vaporiser combustion chamber
- Off gas leaving column passes through demister pad to remove entrained water droplets

Vent Seal Pot (& scrubber)

- Vent seal pot is fed from 3 sources:
  - Slurry mix tank
  - Slurry feed tank
  - Eterifier reflux tank
- Scrubber mounted directly on top of seal pot equipped with water spray (industrial water)
- Scrubber outlet connected to flame arrestor (venting to safe location)
O.S.C. SYSTEM – Equipment Description

OSC Feed Tank
Feed tank maintains supply for the stripper feed pump
Streams feeding tank from esterifier reflux tank & vent seal pot
Caustic added to incoming stream (a ‘T’ joint is provided to ensure proper mixing)
To mix the tank contents:
- Portion of flow from feed pump discharge recirculated back to feed tank through an eductor nozzle
pH analyser provided on discharge of feed pump to control pH of stream
Level transmitter measures tank level
Flow control valve in feed line allows wastewater to flow to OSC under normal operation
Tank is fitted with conservation vent

OSC Feed Preheater
Plate and frame heat exchanger used to maintain temperature
- 96degC inlet to the column
Material at column bottom is maintained at 100 – 111 degC by steam injection
This material is used to control inlet feed temperature as it is pumped via the heat exchanger to the effluent pit
• O.S.C. SYSTEM - Process Flow

OSC removes by product volatile compounds (VOC’s) from the process wastewater.

Principal VOC’s are

- Acetaldehyde
- 2-methyl-1,3 – dioxolane
- 1,4-dioxane

Wastewater is fed to top of OSC and steam to bottom
VOC’s stripped from water by steam
Vapour leaving OSC is fed to combustion chamber of HTF vapouriser

Normal operating temperature of OSC is 100 – 111degC.

Steam flow controller gets set point from ratio controller which
- Adjusts steam flow based on wastewater feed rate
- Ratio set point must be adjusted to get required VOC removal
1,4-dioxane is most difficult component to strip
- Relatively non-biodegradable
Column performance is measured by amount of 1,4-dioxane remaining in column bottoms
O.S.C. SYSTEM - Process Flow

VOC feed to OSC (approx. % by weight):
- Acetaldehyde (50%)
- 2-methyl-1,3 dioxolane (46%)
- 1,4-dioxane (4%)

Normal VOC generation is 0.005kg/kg polymer

Vapour stream leaving OSC contains approx. 18% (by weight) VOC

VOC’s have potential to form explosive mixture in air
- DCS based hazard management system provided
- Monitors off-gas mixture fed to the vapouriser
- Will shutdown the OSC in the event of an abnormal condition
- Will inhibit starting of system until purging and correct operating conditions are established
O.S.C. SYSTEM - Detailed Process Diagram