# PTA 5/6 OPERATION MANUAL

## Solvent Recovery

**(Document No. RIL_PTA-5/6_OPMAN-007)**

**(Revision 0)**

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## Revision control

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**GLOSSARY:**

1. BD: Bursting disc
2. CTA: Crude Terephthalic Acid
3. CW: Cooling Water
4. DCS: Distributed Control System
5. DM: Demineralised
6. DSS: Duplex Stainless Steel
7. DH: Dehydration
8. FCV: Flow Control Valve
9. FI: Flow indicator
10. FO: Flow Orifice
11. FT: Flow Transmitter
12. FV: Full vacuum
13. HCV: Hand controlled valve
14. HP: High Pressure
15. HS: Hand Switch
16. LCV: Level Control Valve
17. LI: Level indicator
18. LP: Low Pressure
19. LS: Level Switch
20. LT: Level Transmitter
21. MOC: Material of Construction
22. MSDS: Material Safety Data Sheets
23. n-PA: normal Propyl Acetate
24. OD: Outer Diameter
25. P&ID: Process and Instrumentation Diagram
26. PCV: Pressure Control Valve
27. PFD: Process Flow Diagram
28. PI: Pressure indicator
29. PT: Pressure Transmitter
30. PTA: Purified Terephthalic Acid
31. PX: Paraxylene
32. ROVAC: Rotary Vacuum
33. ROSOV: Remotely Operated Shut-Off Valve
34. RPM: Revolutions per minute
35. RV: Relief Valve
36. SS: Stainless Steel
37. TA: Terephthalic Acid
38. TCV: Temperature Control Valve
39. TI: Temperature Indicator
40. TOI: Temperature of Interest
41. TT: Temperature Transmitter

## 1. SCOPE

Scope of this manual covers removal of water from water-rich streams coming from different sections of the plant in Solvent Dehydration Column using azeotropic distillation. This water is produced in the oxidation reaction, while some of it is added in the Atmospheric Scrubber and HP Absorber. Some water also enters the system with process air which is used for oxidation reaction. Some water also ingresses into the system through pump and agitator mechanical seals where seal water is used as the flushing medium in plan 54 system. Extent of that ingress depends on the conditioning of the seals.

This operating manual covers PTA 5 plant. PTA 6 process being identical to PTA 5 with same tag numbers, a separate operation manual shall not be issued PTA 6.

## 2. REFERENCE DOCUMENTS

This manual should be read in conjunction with following documents –

- PFD and Stream Summary (attached in Annexure A)
- Following Process P&IDs

| | |
|---|---|
| 10005-G41-GPZ105-00301 | 10005-G41-GPZ105-00603 |
| 10005-G41-GPZ105-00305 | 10005-G41-GPZ105-00604 |
| 10005-G41-GPZ105-00401 | 10005-G43-GPZ105-00606 |
| 10005-G41-GPZ105-00403 | 10005-G43-GPZ105-00607 |
| 10005-G41-GPZ105-00501 | 10005-G41-GPZ105-00802 |
| 10005-G41-GPZ105-00504 | 10005-G43-GPZ105-00902 |
| 10005-G41-GPZ105-00505 | 10005-G43-GPZ105-02205 |
| 10005-G41-GPZ105-00601 | 10005-G43-GPZ105-02613 Sh. 2 |
| 10005-G41-GPZ105-00602 | 10005-G43-GPZ105-02644 |

- Following Vendor P&IDs:
  - Kirloskar Ebara: 10005-GPZ105-MPPA15-7389080-C02-001
  - Sulzer Pumps: 10005-GPZ105-MPPA14-7388617-C02-001
  - Sulzer Pumps: 10005-GPZ105-MPPA14-7388617-C02-002

- Following P&IDs of Deluge stations and fume suppression system
  - 10005-U20-GPZ105-7003

- Following hazardous area classification drawing
  - 10005-E66-GPT105-001
  - 10005-E66-GPT105-002

- 10005-E66-GPT105-003

- Following Fire & Gas Layout P&IDs:
  - 10005-U23-GPT105-001
  - 10005-U23-GPT105-002
  - 10005-U23-GPT105-003
  - 10005-U23-GPT105-004
  - 10005-U23-GPT105-005

- Standard Operating Conditions(attached in Annexure B)
- Alarm and trip schedule (attached in Annexure C)
- Chemical information sheet / MSDS (attached in Annexure D)
- Inventory Summary in Vessels (attached in Annexure E)

# 3. PROCESS DESCRIPTION

## 3.1 General Introduction

To control the water content of the Oxidation Plant solvent it is necessary to remove the water produced in the oxidation reaction, together with that added in the Atmospheric Scrubber and HP Absorber plus that in the process air. This water removal is carried out in Solvent Dehydration (DH) Column D5-601 by azeotropic distillation. The DH Column is designed to produce a distillate of water containing <0.1% w/w acetic acid and a bottoms product of acetic acid containing 5% water for return to the Oxidation Plant.

There are five "normal" feed streams to the DH Column. These are:

- Vapour from the Solvent Stripper D5-511
- Flash vapour from the Second CTA Crystalliser D5-402
- "Water draw-off" from the Reactor overheads
- Condensate from the First CTA Crystalliser Vent Separator F5-432
- Vapour from the Purge Column D5-651

The overhead vapor from the DH Column contains a significant amount of methyl acetate which must be purged from the Solvent Dehydration system for recycle to the Reactor. Simply cooling the overhead vapour in a water-cooled condenser will lead to significant subcooling and hence methyl acetate trapping. To prevent this "trapping" a portion of the overhead vapour bypasses the DH Column Condenser, E5-608. This vapour is re-mixed with the material that has passed through the Condenser and is then passed over the DH Column Condenser Contactor, F5-608/1, to re-equilibrate. The bypass flow is controlled to maintain a roughly constant vapour flow to the Recovery Column D5-631, and the re-equilibration results in a typical vapour and liquid temperature of 78°C.

Liquid falling from the DH Column Condenser Contactor is collected in the DH Column Decanter F5-608, where the organic entrainer (nPA) and aqueous phases are separated based on density difference. From the DH Column Decanter the organic phase overflows to the Reflux Pumps G5-615A/B which pump it back to the top bed of the DH Column as reflux. The heavier aqueous phase flows over an adjustable weir to a segmental partition, and then flows under gravity to the Recovery Column, D5-631.

Below the DH Column Decanter is an integral Entrainer Storage Vessel F5-609, which is used to provide buffer storage for entrainer as reflux rates vary, and to provide an on-plant inventory for the make-up of process losses. This entrainer vessel is filled from storage facility at PTA 5 tank farm (refer Operation Manual of "Entrainer Receipt, Storage & Distribution").

Boil-up heat for the DH Column is supplied by the DH Column Reboiler E5-602 using LP steam. This is an external vertical thermosyphon reboiler.

Dehydrated acetic acid (5% water) overflows through a segmental weir to the other side of the DH Column sump. This solvent is then withdrawn from the bottom of the DH Column at 120.2°C and cooled to 95°C in the Solvent Bottom Cooler E5-603 prior to distribution to the various solvent users via DH Solvent Pumps G5-606A/B and HP Solvent Pumps G5-607A/B.

In addition to the normal feeds, in order to make up for processing losses, fresh acetic acid is supplied into the bottom section of the DH Column from acetic acid storage facility at PTA 5 tank farm (refer Operation Manual of "Acetic Acid Receipt, Storage & Distribution").

There is a side stream take-off at the bottom of the top packed section of the DH Column. This is necessary in order to purge paraxylene from the DH Column, which would otherwise build up and impair the DH Column performance.

There is a very low concentration of paraxylene in the water draw-off feed to the DH Column. Paraxylene and nPA together can form an azeotrope with a boiling point higher than that of water and nPA (82.4°C). As a consequence, paraxylene begins to build up in the top packing bed. Since paraxylene and acetic acid form an azeotrope with a boiling point lower than the boiling point of acetic acid, the acetic acid concentration of the DH Column vapour outlet will then increase as paraxylene builds up in the Column.

This paraxylene purge contains a large amount of entrainer. To prevent this being lost when the paraxylene purge is returned to the Oxidation Reactor, it is first fed to the Purge Column D5-651.

In the Purge Column the entrainer is recovered and returned to the DH Column as a vapour below the top bed, whilst the paraxylene purge passes from the bottom of the column as a liquid. This is transferred to the Mother Liquor Drum under gravity and subsequent recycle to the Oxidation Reactor. Boil-up heat for the Purge Column is provided by flash vapour from the Second CTA Crystalliser.

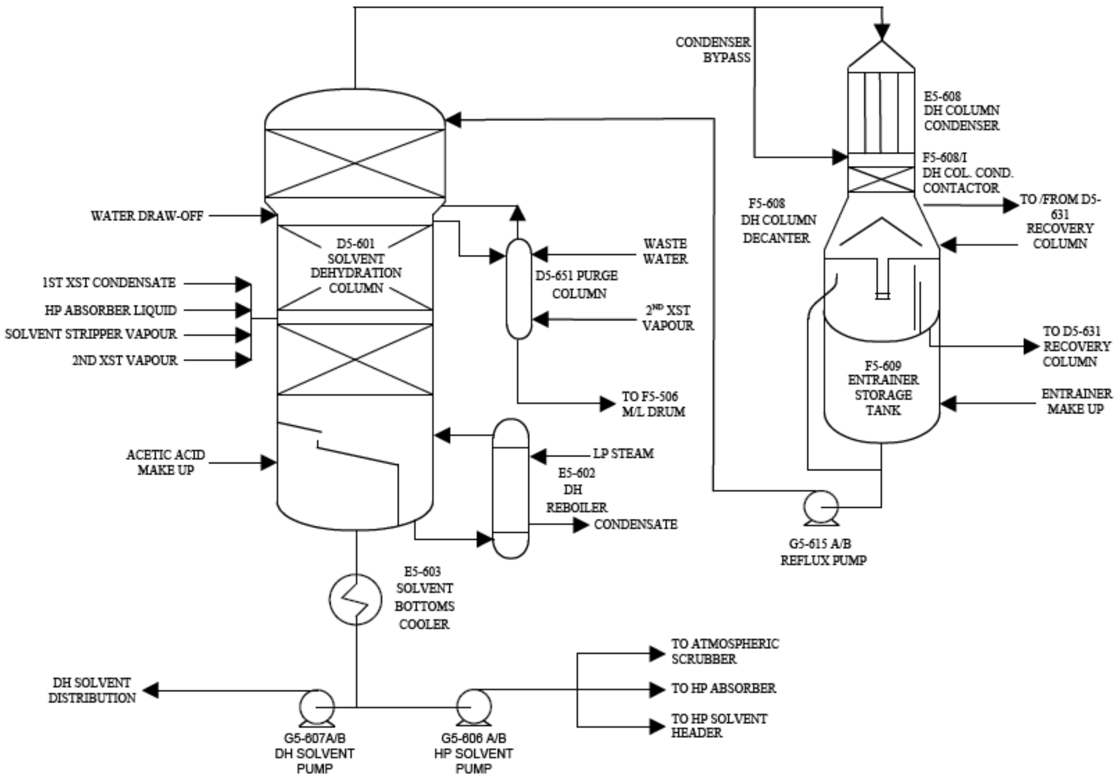



***Figure 3.1 – Solvent Dehydration System Overview***

## 3.2 Process Theory - Distillation

### 3.2.1 Conventional & Azeotropic Distillation

Distillation is the most widely used method for separating components by the Chemical Industry. The process involves successive vaporisation and condensation of countercurrent flows of material in a tower containing contacting apparatus. The process relies on there being a changing composition between the liquid that is flowing down the tower and the vapour (in thermodynamic equilibrium with the liquid) that passes up the tower. Components that have a high vapour pressure/low boiling point migrate to the top of the tower and components having a low vapour pressure/high boiling point sink to the base of the tower. In the majority of cases the separation can be achieved as described above with a simple feed to the centre of the tower and without having to use additional components to achieve the desired separation.

The vapour pressure of a component in a liquid mixture depends on the nature of the molecule in question, the temperature of the mixture and the composition of the mixture. For ideal mixtures, the vapour pressure is given as the vapour pressure of the pure component at the temperature multiplied by the mole fraction of the component in the liquid. However, most mixtures are not ideal and a further multiplying factor, known as the activity coefficient, is required in order to correctly model the behaviour of a component. The activity coefficient is itself a function of temperature and composition. In some cases the combination of ideal vapour pressure, composition and activity coefficient can give rise to a situation where the composition of a vapour phase and the composition of the liquid phase in which it is in equilibrium are identical. This is termed an azeotrope and further contacting of the azeotropic vapour and liquid with each other cannot lead to further separation/concentration of the components.

Azeotropes may be high boiling or low boiling. In a high boiling azeotrope, such as occurs with water and hydrochloric acid, the azeotrope will leave from the base of a simple distillation tower and the overhead product will be either water or hydrogen chloride, depending on which component was in excess of the azeotrope in the feed. In a low boiling azeotrope, such as occurs with water and ethanol, the azeotrope will leave from the top of the tower with water or ethanol leaving from the base of the tower, once again depending which was in excess of the azeotropic composition in the feed.

Low boiling azeotropes can further be separated into homogeneous azeotropes, where the liquid is present as a single phase, and heterogeneous, where the liquid is present as two phases. Homogeneous azeotropes are difficult to break, but heterogeneous azeotropes are easy to break since the condensing azeotrope splits into two liquid phases of different composition to the azeotrope and so can be separated by simple decantation based on density difference. In order to break a homogeneous azeotrope, a further component is sometimes added in order to generate a heterogeneous azeotrope which can then be broken through decantation. The heterogeneous azeotrope produced may be either a binary azeotrope of the entrainer with one of the components of the homogeneous azeotrope, or a ternary azeotrope involving the original components of the homogeneous azeotrope plus the entrainer (the classical method of breaking the water and ethanol azeotrope using benzene or cyclohexane as the entraining agent).
The entraining agent may be introduced with the feed or may be stored in the decanter at the top of the azeotropic tower. The overheads vapour composition will be close to the azeotrope. Reflux to the tower may be either one or both liquid phases depending on the ease of the separation. It is important to control the degree of penetration of the entrainer down the tower and this is achieved through a combination of temperature and inventory control.

### 3.2.2 Acetic Acid/Water Distillation

Water and acetic acid can be separated by simple distillation. Although the components differ in boiling point by 17°C, their separation is difficult because of the non-ideal nature of the vapour-liquid equilibrium. In order to avoid a distillation requiring many theoretical stages and high energy inputs, entrainers are used to generate a low boiling, binary, heterogeneous azeotrope of the water and the entrainer. This approach requires fewer distillation stages and lower energy input. The heterogeneous azeotrope breaks on condensation into two liquid phases which can be separated by decantation.

There are a number of possible candidates for the choice of entrainer to assist in the separation of water and acetic acid. One of the constraints is to use an entrainer that is compatible with the background process. In the case of terephthalic acid production, alkyl esters of acetic acid are the most suitable choice. Normal propyl acetate, iso-butyl acetate and normal butyl acetate are all used commercially for this process.

The patented azeotropic distillation process used by Invista in recovering water from water/acetic acid mixtures uses normal propyl acetate (nPA) as the entrainer. The process operates with a single nPA-rich phase being refluxed to the dehydration tower (normally a packed unit). Use of nPA minimizes the height of the tower compared to iso-butyl acetate (iBA) or normal butyl acetate (nBA) systems for the same feed composition. The main water feed to the tower is condensate derived from the overheads from the oxidation reaction. This is typically 20 - 30% w/w water. With iBA or nBA, the aqueous phase would also have to be refluxed to obtain separation of the water from the acetic acid and to recover the entrainer. Hence the choice of nPA gives the lowest column height and lowest energy usage for these water/acetic acid feed compositions.

A table comparing the azeotropic distillation of water and acetic acid using nPA as entrainer with conventional fractional distillation is shown below.

| | Conventional Distillation | Azeotropic Distillation |
|---|---|---|
| Effective Reflux Ratio | 3.8 | 1.6 |
| Scaled Heat Load | 100% | 54% |
| Theoretical Stages | 52 | 24 |
| Column Height m. approx.) | 59 | 31 |
| Column Diameter m.(approx.) | 4.6 increasing to 6.1 | 4.4 |

***Table 3.2.2 - Comparison of Conventional and Azeotropic Columns for Acetic Acid/Water Separation***

## 4. EQUIPMENT LIST

| **Equipment Number** | **Description** |
|---|---|
| D5-601 | DH Column |
| E5-602 | DH Column Reboiler |
| E5-603 | Solvent Bottoms Cooler |
| G5-606A/B | DH Solvent Pump |
| G5-607A/B | HP Solvent Pump |
| E5-608 | DH Column Condenser |
| F5-608/I | DH Column Condenser Contactor |
| F5-608 | DH Column Decanter |
| F5-609 | Entrainer Storage Tank |

| | |
|---|---|
| G5-615A/B | Reflux Pump |
| D5-651 | Purge Column |

# 5. EQUIPMENT DESCRIPTION

## 5.1 Solvent Dehydration Column, D5-601 (Vendor: L&T)

### 5.1.1 General Information

DH Column D5-601 is a packed fractionating column constructed from 2205 Duplex Stainless Steel. It has an internal diameter of 6500mm (bottom) / 6900mm (top) and a tan/tan height of 38130 mm. The column is separated into three sections: Zone A, Zone B and Zone C.

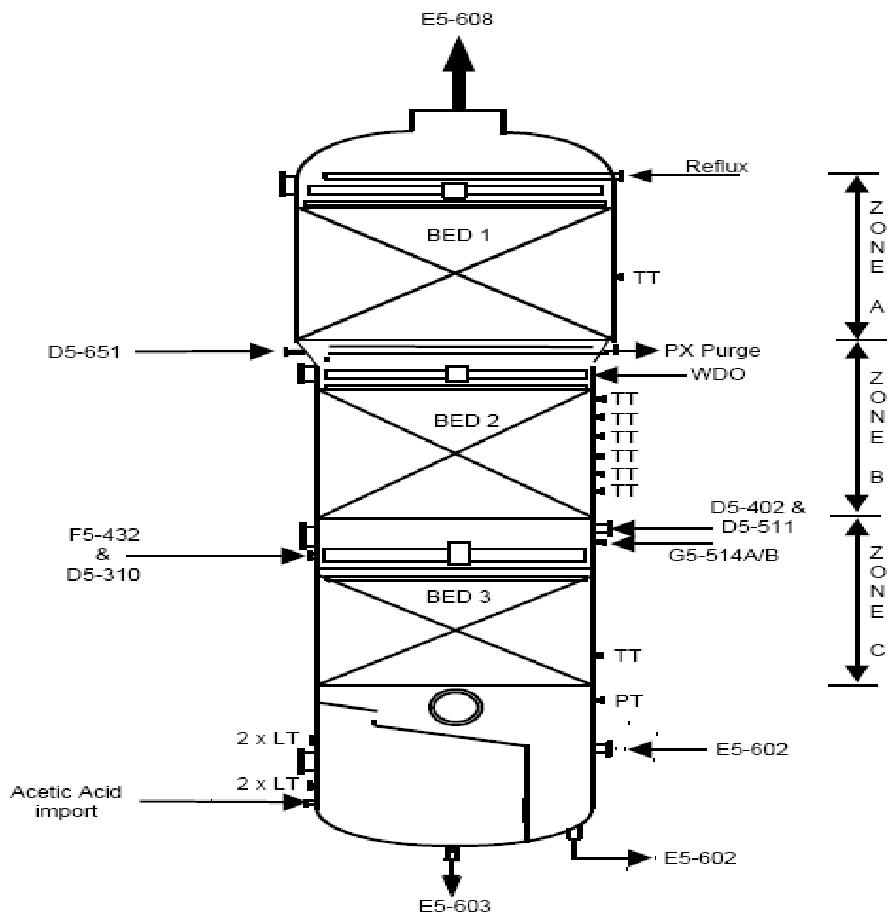



***Figure 5.1.1 – DH Column***

Column internals are supplied by Sulzer.

The top section, Zone A, is packed with one bed (Bed 1) of structured packing (Mellaplus 252Y, 32 layers) constructed from 316L stainless steel.

Above this bed are the following nozzles:-

- Vapour outlet from the top
- Insert nozzle for entrainer reflux return
- Manway

The bed itself is provided with a branch for a duplex type thermowell/thermocouple.

The middle section, Zone B is packed with one bed (Bed 2) of structured packing (Mellaplus 252Y, 37 layers) constructed from 2205 Duplex stainless steel. Above this bed are the following:-

- Insert nozzle for the Water Draw-off feed from the HP Absorber
- Paraxylene purge to D5-651
- Purge Column overhead return
- Manway

The bed itself is provided with six equally spaced branches for duplex type thermowells/ thermocouples.

The bottom section, Zone C is packed with one bed (Bed 3) of structured packing (Mellaplus 252Y, 22 layers and Mellagrid 64Y, 2 layers) constructed from 2205 Duplex stainless steel. Above this bed are the following:-

- Vapour from the Solvent Stripper
- Flash vapour from the Second CTA Crystalliser
- Insert for liquid from the First CTA Crystalliser Vent Separator
- Insert for acid liquor from the HP Absorber
- Atmospheric Scrubber Bottoms (NNF)
- Manway

The bed itself is provided with a branch for a duplex type thermowell/thermocouple.

Below the bed (in the sump) are the following:-

- Pressure transmitter
- Level transmitters (2 off)
- Manway
- Acetic acid make-up
- Liquid feed (on bottom cover) to the Reboiler

- Reboiler vapour return
- DH Solvent outlet (on bottom cover)

A vortex breaker is fitted at the DH Solvent outlet nozzle.

An internal baffle is provided in the column bottom sump to partition the column base. Recovered acetic acid is collected first in the reboiler's liquid feed sump below bed 3, this then overflows through the baffle weir to the second larger partition for storage, make up and supply of DH Solvent. An access panel is fitted on the partition baffle for inspection purposes and a drain hole is also provided at the baffle low point to ensure complete draining during shutdowns.

Fitted on the vapour outlet pipe line from the DH Column, relief valves RV/D5-601A/B/C protect the Solvent Dehydration Column and connected system from overpressure. Although the vessels in the Solvent Recovery area are designed for full vacuum, in the event of low pressure, inert gas is automatically provided to prevent air being drawn in from the Atmospheric Scrubber and forming a flammable atmosphere (see the section on Trips & Alarms for more details).

Additional equipment details are provided below:

- Vessel Contents: Water, Acetic Acid, n-PA, other organics and inerts
- Internal design pressure: 3.5 bara (upper section), 5.38 bara (lower section)
- External design pressure: Full vacuum (upper section), Full vacuum (lower section)
- Design temperature: 150°C (upper section), 225°C (lower section)
- Operating pressure:
  i. Zone A: 1.2 – 1.24 bara
  ii. Zone B: 1.24 – 1.27 bara
  iii. Zone C: 1.27 – 1.3 bara
- Operating Temperature (Top/Bottom): Zone A: 85/94°C; Zone B: 96/111°C; Zone C: 112/116°C
- Insulation Type: Heat Conservation/Fire Hazard Protection Type of thickness 115 mm
- Gross Capacity of Column : 1415 $m^3$
- Tan to tan height: 38130 mm
- Height from BTL to mid-point of Bed 2 (bottom section): 21900 mm
- Height from mid-point of Bed 2 to TTL (top section): 16230 mm
- Zone A: 8730 mm
- Zone B: 11500 mm
- Zone C: 8500 mm

### 5.1.2 Operating Philosophy

The Solvent Dehydration Column is designed to separate water from a number of water-rich liquid and vapour streams from the Oxidation plant, and produce acetic acid containing 5.0 ±0.5% w/w water from the base for redistribution as DH Solvent in addition to water containing 0.1% w/w acetic acid from the top of the Column. The Column is an azeotropic unit using normal propyl acetate (nPA) as an entrainer to reduce the energy required for the acetic acid/water separation by breaking the acetic acid/water azeotrope at high water concentration. The heat input to the Column is provided by the DH Column

Reboiler which is a thermosiphon-type unit using LP steam. The reflux flow to the Column is provided from the Entrainer Storage Tank F5-609 via the Reflux Pumps G5-615A/B.
There are five feed streams to the DH Column in normal plant running. These are:-

- Vapour from the Solvent Stripper D5-511
- Flash vapour from the Second Crystalliser D5-402
- "Water draw-off" from the Reactor overheads
- Condensate from the First CTA Crystalliser Vent Separator F5-432
- Vapour from the Purge Column D5-651

A line from Atmospheric Scrubber Bottoms, D5-508 is also provided just below Bed 2, the control philosophy for which is discussed in Section 5.1.3.1.2.

An HP solvent line from G5-607A/B discharge and a waste water line from G5-632A/B discharge are provided which add on to the “Water Drawoff” flow from D5-310. The flows of these lines are adjusted according to “DH Water Recycle Calculation” block XY-06101. These are normally not used unless demanded by XY-06101.

Also, a line from exit of E5-320 (acid-rich) is provided below Bed 2 of the column.

Flash vapours entering from the Solvent Stripper and Second CTA Crystalliser enter above the lower bed in order to ensure efficient methyl acetate stripping from the bottoms product.

In addition, acetic acid is imported into the Oxidation plant and distributed via the base of the DH Column.

Vapour from the top of the DH Column is fed to the DH Column Condenser and Decanter.

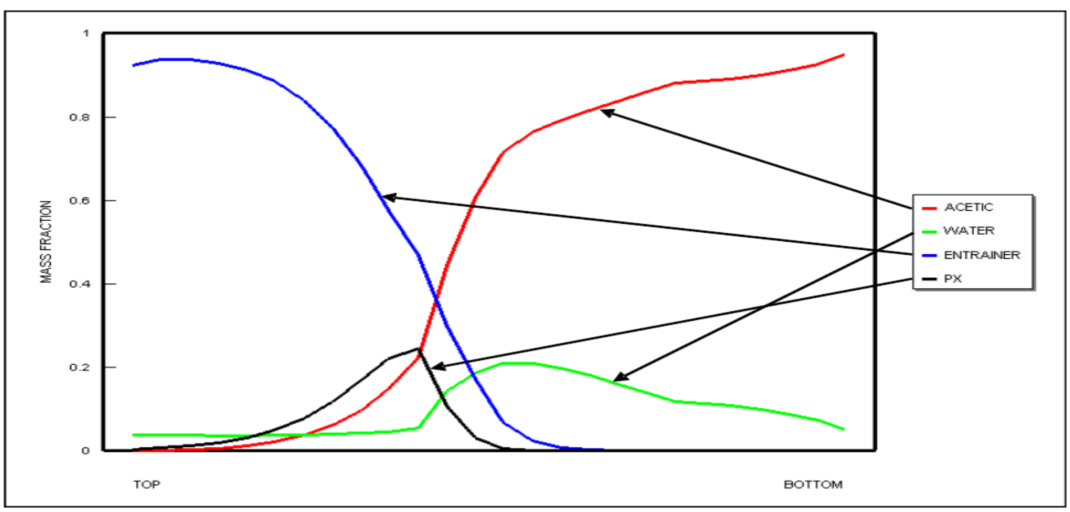



***Fig 5.1.2.1 - A Typical DH Column Composition Profile***

Being an azeotropic column, the composition of the top and bottom products is maintained by controlling the composition profile in the Column. At the point where the nPA composition rises quickly (the nPA "front"), the Column temperature falls sharply (see below). The *position* of the sharp temperature change is therefore controlled, rather than simply monitoring the *value* of a single temperature point, as in conventional columns. The positioning of the nPA front is achieved by controlling the reflux rate to the column.

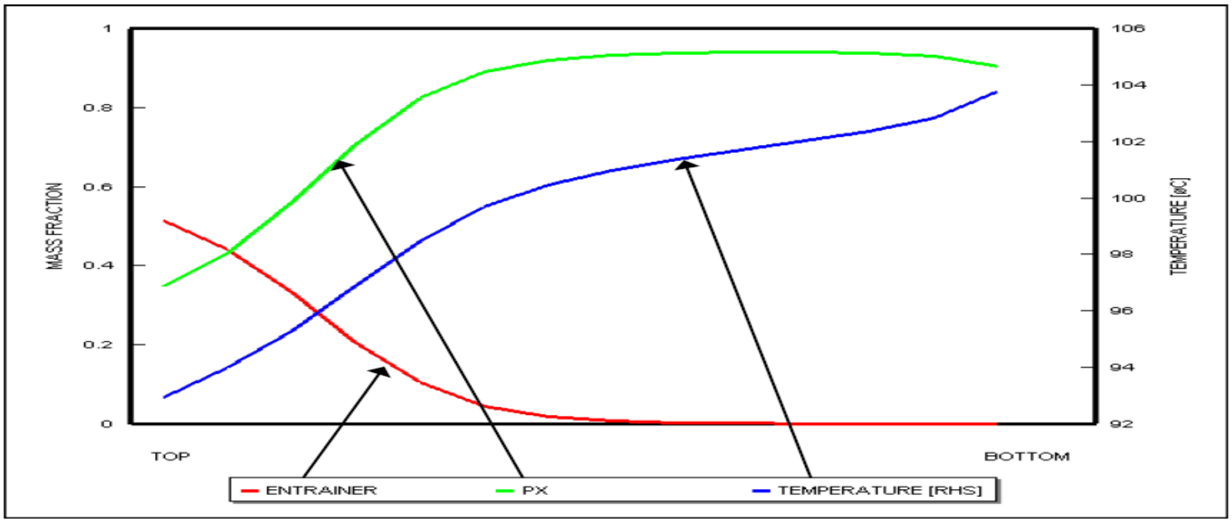



***Fig 5.1.2.2 - A Typical DH Column Entrainer Concentration Vs Temperature Profile***

For further detail on the control philosophy, see section 5.1.3.

### 5.1.3 Instrumentation & Control

This section covers control of the DH Column feeds, DH Column reflux, the bottoms temperature (reboil), acetic import and the solvent distribution system. Consequently this section is also applicable to control of the DH Reboiler, HP Solvent Pumps, DH Reflux Pumps, Solvent Bottoms Cooler and the HP Solvent Cooler.

In addition to the above control loops there is a pressure loop, PICA-06201 to prevent the pressure falling. For details on this loop see Section 5.1.4.3.

#### 5.1.3.1 Column Feed Control

There are five normal feeds to the DH Column. These are:

- Reactor water draw-off (WDO) and scrubbing water as a liquid feed from the HP Absorber
- Flash vapour from the Second CTA Crystalliser
- Condensate from the First CTA Crystalliser Vent Separator
- Vapour from the Purge Column
- Vapour from the Solvent Stripper

##### 5.1.3.1.1 "Water Draw-off" Control

To maintain stable operation of the DH Column it is necessary to have sufficient water feed. During normal operation this water is provided by the water draw off stream from HP Absorber base section. However during low rate, start-up and Reactor hold situations, additional water is required and this is provided by adjusting recycling water flow from the Recovery Column which in turn adjusts HP solvent flow from G5-607A/B through ratio controller, FFY-06136, to maintain same water concentration as in water draw off stream. The details of this complex loop are covered below.

The Reactor WDO flow, containing scrubbing water from the HP Absorber top section, is controlled by the level controller for the HP Absorber, LICA-03509. Control valve LCV-03509 is mounted as close to the DH Column as possible to minimise the possibility of two-phase flow at downstream of LCV and consequent erosion of the inlet nozzle.

Recycle water from the base of the Recovery Column and HP solvent from G5-607A/B join the WDO line from the HP Absorber downstream of LCV-03509.

**Complex Loop FICA-06101: DH Column Recycle Water Control**

When the water draw off feed from the HP Absorber D5-310 to the Solvent Dehydration Column D5-601 is lost, for example on a reactor trip, water and solvent must be added to the column to prevent the nPA entrainer from falling down the column.

Waste Water is added via FICA-06101 and HP Solvent is added via FICA-06136 as a ratio of the measured waste water flow.

Calculation XY-06101 calculates the required flow of waste water and sets the setpoint of FICA-06101. The measured flow of waste water is then used to provide a setpoint to the HP Solvent controller FICA-06136 via ratio block FFY-06136.

When the HP Absorber water draw-off flow (FT-03514) is above a minimum value (X), no additional water/solvent is required via FICA-06101/FICA-06136. This is the normal case.

When the flow indicated by FT-03514 falls below the minimum value (X), XY-06101 calculates the required additional water flow needed to maintain the minimum water flow, based on the measured flow (FT-03514) and the preset mass fraction of water in the water draw off flow (Y). HP Solvent is also added as a ratio of measured added Waste Water.

***Loop Structure and Controller Details***

*FICA-06101*
PI Control
ACTION Reverse
Remote setpoint from XY-06101 (usually 0%)

*XY-06101 Recycle water calculation*
When the water draw off flow (FT-03514) falls below X, FICA-06101 setpoint is moved to keep the total water and solvent flow constant. The HP Solvent flow is ratioed to the waste water flow.

Flow 1 = FT-03514
Flow 2 = FICA-06101 setpoint

X is the value of Flow 1 at which Waste Water flow FICA-06101 is started (initial value 70 t/h, may require adjustment during commissioning).

The mass fraction of water in the water draw off flow from the HP Absorber is 0.3; the make-up Waste Water stream is 100% water; the HP Solvent is 5% water and 95% acetic acid. Hence if the HP Solvent flow is 2.8 times the Waste Water flow the combined flow will contain 30% water and 70% acetic acid to match the lost water draw off flow.

If Flow 1 > X, then Flow2 = 0
If Flow 1 $\le$ X, then Flow 2 = (X-Flow 1)/3.8

*FFY-06136*
Ratio block.
Input 1 is the measured Waste Water flow from FT-06101
Input 2 is the ratio constant 2.8
Output is the product of inputs 1 and 2 and is connected to the setpoint input of FICA-06136.

*FICA-06136*
PI Control
Action Reverse
Remote setpoint from FFY-06136 (usually 0%)

### 5.1.3.1.2 Liquid and Vapour Feed Control

Three feeds normally enter between the middle and bottom packed beds:

- The liquid feed from the First CTA Crystalliser Vent Separator is controlled by the level controller LICA-04309 on the First CTA Crystalliser Vent Separator.
- The vapour feed is the flash from the Second CTA Crystalliser which is controlled by the Second CTA Crystalliser pressure controller PICA-04173.
- The vapour flow from the Solvent Stripper is controlled by boil-up in the Solvent Stripper.

In addition to the feeds described above there is also a liquid feed line from the bottom section of the Atmospheric Absorber via G5-514A/B Pumps. This stream is normally routed to the Mother Liquor Drum but on Reactor trips and start-up it is routed into D5-601. There is also a liquid feed line from the HP Absorber Recycle Pumps, G5-311A/B. This liquid feed only occurs on Reactor trips and start-up when the solvent from the bottom of the DH Column normally used for methyl acetate scrubbing in the HP Absorber has to be returned to the DH Column via LCV-03511.

### 5.1.3.2 DH Column Profile Control

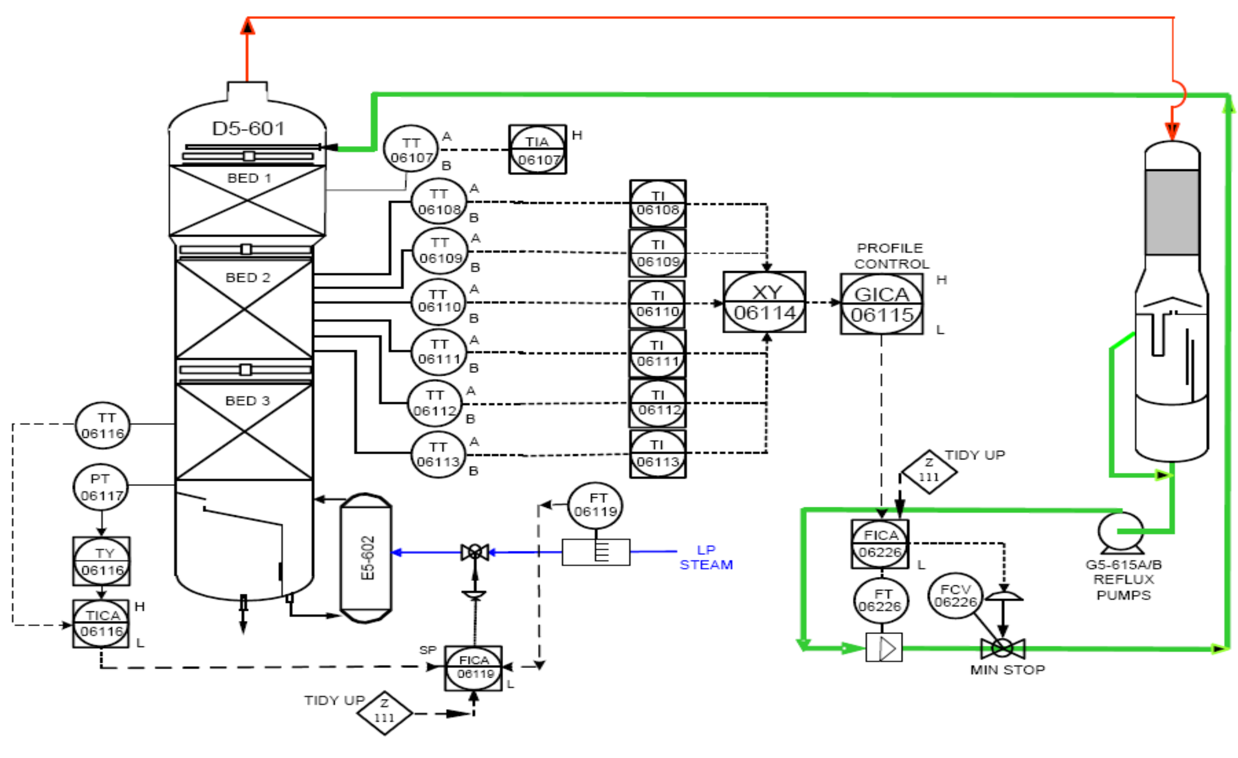



*Fig 5.1.3.2 - DH Column Profile Control*

The temperature in the DH Column rises sharply at the point where the nPA entrainer concentration drops off quickly, and this point is called the "position of the nPA front". XY-06114 calculates the position of the nPA “front” from the six temperatures TI-06108, TI-06109, TI-06110, TI-06111, TI-06112 and TI-06113 located in the middle bed of the Column and provides an input to the Column Profile Controller GICA-06115. GICA-06115 provides an input to FICA-06226 to allow the reflux flow to be trimmed as required in order to maintain the correct location of the “front”. This is the preferred operating scheme where perturbations to the Solvent DH Column are relatively slow and adjustment to the reflux flow alone is capable of providing stable Column operation. Details of GICA-06115 are provided below.

Reflux valve FCV-06226 has a minimum stop in the DCS to prevent running the Reflux Pump against a closed head and set at a flow of 175 TPH. The minimum stop is set only when FICA-06226 is on automatic. To fully close FCV-06226 at start up, FICA-06226 should be put on manual.

The six temperature detection points located within the middle bed are each provided with two thermocouples (duplex type) & the transmitter will automatically select the correct one. This built-in redundancy is required because the DH Column Profile control is critical and maintenance access to the upper part of the column is difficult.

### Complex Loop GICA-06115 DH Column Top Profile Control

In order to control the composition of the top and bottom products, the temperature profile of the column must be maintained. At the point where the nPA entrainer composition rises quickly, the column temperature falls sharply with distance up the column. The position of the sharp temperature change is therefore controlled rather than a single temperature point as in conventional columns.

XY-06114 calculates the position of the nPA "front" from the temperatures in the column.

GICA-06115 controls the position of the nPA "front" by means of a cascade to reflux flow controller, FICA-06226.

The temperature corresponding to the point at which he nPA entrainer concentration falls quickly is called the Temperature of Interest (TOI). The position in the column corresponding to this point is calculated by XY-06114.

The calculation looks at the temperatures measured by TT-06108, TT-06109, TT-06110, TT-06111, TT-06112 and TT-06113, and compares them with the TOI in order to find out at which position the TOI lies.

If TI-06108 equals the Temperature of Interest then the position is 100%
If TI-06109 equals the Temperature of Interest then the position is 80%
If TI-06110 equals the Temperature of Interest then the position is 60%
If TI-06111 equals the Temperature of Interest then the position is 40%
If TI-06112 equals the Temperature of Interest then the position is 20%
If TI-06113 equals the Temperature of Interest then the position is 0%

If the TOI lies between these measurement points then the position is found by linear interpolation.

In response to a change in position of the nPA “front” GICA-06115 will adjust the reflux flowrate by changing the setpoint of flow controller FICA-06226. This causes a change in position of the control valve, FCV-06226.
If one of the outer temperature indicators (TI-06108, TI-06109, TI-06112 and TI-06113) fails (value is outside range of 4-20mA) then the calculation shall be allowed to continue, providing the TOI is not near that temperature indicator.

If any temperature indicator is reading more than 1°C higher than the one below it then the output shall be held at the last value and an alarm raised in the DCS.

The temperature indicators must be equi-spaced and straddle the position in the column where the TOI is found. Experience and dynamic simulation has shown that poor control will result if the probes are not equi-spaced.

***Position algorithm***

The output of this algorithm is the % position of interest. The setpoint is "Temperature of Interest" - not accessible to the operator. The algorithm looks at the temperature signals T1, T2, T3, T4, T5 and T6 and compares them with the temperature of interest.
Linear interpolation is then used to calculate the % position.

| Variable | Temperature | Position |
|---|---|---|
| T1 | TI-06108 | 100% (Top) |
| T2 | TI-06109 | 80% |
| T3 | TI-06110 | 60% |
| T4 | TI-06111 | 40% |
| T5 | TI-06112 | 20% |
| T6 | TI-06113 | 0% (Bottom) |

| | |
|---|---|
| TOI | Temperature of interest, initial value to be 103°C (to be confirmed during commissioning) |
| POSITION | % Position output |

***Algorithm Definition***

a) Input the data:
T1, T2, T3, T4, T5, T6 (measured values) and TOI
b) Calculate position:

If $T1 \geq TOI$, then $POSITION = 100\%$
If $T2 \geq TOI > T1$, then $POSITION = 100 - 20*(TOI-T1)/(T2-T1)\ \%$
If $T3 \geq TOI > T2$, then $POSITION = 80 - 20*(TOI-T2)/(T3-T2)\ \%$
If $T4 \geq TOI > T3$, then $POSITION = 60 - 20*(TOI-T3)/(T4-T3)\ \%$

If T5 $\geq$ TOI > T4, then POSITION = 40 – 20*(TOI-T4)/(T5-T4) %
If T6 $\geq$ TOI > T5, then POSITION = 20 – 20*(TOI-T5)/(T6-T5) %
If T6 $\leq$ TOI, then POSITION = 0%

c) Output data:
OUTPUT = POSITION

***Loop Structure and Controller Details***

*GICA-06115*
PID control
ACTION Direct
SETPOINT 28% (to be confirmed during commissioning)
I expected to be over 30 minutes
Master in cascade to FICA-06226
The process variable is the output of calculation XY-06114

*FICA-06226*

PI control
ACTION Reverse
P and I settings to be tuned for fast response
Slave in cascade from GICA-06115

### 5.1.3.3 DH Bottoms Temperature Control

The Reboiler provides the heat to boil-up the Column bottoms and so maintain the desired temperature. It is important to keep the water content of the DH Solvent at 5%. If the temperature is allowed to fall, the water content will increase, leading to a higher water concentration in the Reactor and thereby poor Reactor efficiency. Too high a Column bottom temperature will cause a low water concentration and will very quickly lead to corrosion of the Column shell, Reboiler tubes and end covers and other pipelines/equipment in the Oxidation Plant. The bottoms water concentration should normally be maintained in the range 4.5-5.5%.

The heat required to maintain the boil-up in the DH Column is provided by LP steam condensing on the shellside of the DH Column Reboiler E5-602. The steam flow rate is measured using FT-06119.

Temperature transmitter TT-06116, located close to the bottom of Bed 3, provides a signal to temperature controller TICA-06116. Pressure transmitter PT-06117, located in the DH Column base, provides a signal to PIA-06117 and to calculation block TY-06116. The process operator enters the desired DH Column base water concentration into TY-06116 and the calculation block generates a set-point for temperature controller TICA-06116 based on the DH Column pressure (i.e. pressure corrected). The output of TICA-06116 automatically adjusts the setpoint of flow controller, FICA-06119, which controls the LP steam flowrate by adjusting FCV-06119 on the steam inlet line to the DH Column Reboiler. FICA-06119 also receives input from FY-06119 which calculates the temperature and pressure corrected steam flow using inputs from FT-06119, PT-06145 and TT-06146 located in the LP steam line to E5-602. The DCS indications for the pressure and temperature of LP Steam are provided as PI-06145 and TI-06146 respectively.

In addition PT-06117 and TT-06116 also provide inputs into calculation block XY-06116 which provides an estimated water content in the base of the Column via AI-06116 located in the DCS.

**Complex Loop TICA-06111: DH Column Bottoms Control**
This loop maintains the concentration of water in the D5-601 Solvent Dehydration Column bottoms at 5.0% +/- 0.5%.

The operator sets the required water concentration as a manual input to TY-06116, which then calculates the required temperature setpoint for TICA-06116, based upon the column pressure from PT-06117. XY-06116 estimates the water concentration in the D5-601 Column base by performing a calculation on PT-06117 and TT-06116.

TICA-06116 maintains the temperature in the base of the D5-601 Solvent Dehydration Column by adjusting the flow of LP Steam to the reboiler, using FICA-06119. The setpoint for FICA-06119 is cascaded from the output of TICA-06116.

***Loop Structure and Controller Details***

*TY-06116*
Calculation block
Inputs: PT-06117 & manual water concentration setpoint (in %)
Output: Setpoint for temperature controller TICA-06116
First, the temperature that corresponds to the required water concentration in acetic acid at 0.25 barg is calculated:
$T_{ref} = 0.0586W^2 - 1.55W + 123.52$

Then this temperature is corrected for the actual pressure in the column (as measured by PT-06117), according to the following equation:
$T = T_{ref} + 16(P-0.25)$

Where:
W is the required water concentration (%w/w) set by the operator
T is the corrected temperature (°C)
P is the pressure measured by PT-06117 (barg)
Tref is the required temperature at 0.25 barg (°C)

*TICA-06116*
PID Control
ACTION Reverse
Remote setpoint from TY-06116
Tuned slower than FICA-06119

*FICA-06119*
PI Control
ACTION Reverse
Tuned fast

*XY-06116*
Calculation Block
Inputs: PT-06117 and TT-06116

Output:
AI-06116 = $(1.55 - (2.4025 - 0.2344(123.52 - TT\text{-}06116 + 16(P - 0.25)))^{0.5})/0.1172$

### 5.1.3.4 The Solvent Distribution System

The solvent distribution system takes solvent from the base of the DH Column and provides HP and DH (LP) Solvent to various users via the HP and DH Solvent Pumps.

Temperature transmitter TT-06404 is provided on the exit line of the DH Solvent Cooler since abnormally high temperatures (> 95°C) could cause cavitation problems in both the HP and DH Solvent Pumps.

The HP Solvent Pumps, G5-607 A/B have the following instrumentation:-

- A common "motor stopped" alarm YA-06415
- Local pressure gauges PI-06411 and PI-06413 on the delivery side of the A and B pumps
- Local motor amps II-06412 & II-06414 on the motors for the A and B pumps

The DH Solvent Pumps, G5-606 A/B have the following instrumentation:-

- A common "motor stopped" alarm YA-06409
- Local pressure gauges PI-06405 and PI-06407 on the delivery side of the A and B pumps
- Local motor amps II-06406 & II- 06408 on the motors for the A and B pumps

The DH Solvent Pump feeds the DH Solvent header and is provided with a flow-controlled kickback system. The flow control loop FICA-06401 has a low alarm set at the minimum flow required for pump protection and is designed to open FCV-06401 in the kickback line to ensure the pump delivery does not fall below this value. The DH Solvent Pumps also provide a route for the export of DH Solvent to off-plot storage at a rate controlled by FO-06410. An inert gas connection is provided to the export route to facilitate emptying out of the long export line to make it free of acetic acid during annual turarounds. Inert gas flow is restricted by FO-06430 and is indicated locally on FI-06431. Export of DH solvent to off-plot storage is measured on FT-06142. Flow is indicated in DCS on FI-06142 and totalized on FQI-06142.

The HP Solvent Pump is also provided with a flow-controlled kickback system. The flow control loop FICA-06416 has a low alarm set at the minimum flow required for pump protection and is designed to open FCV-06416 in the kickback line to ensure the pump delivery does not fall below this value. The bulk of the forward flow from the HP Solvent Pumps passes to the Recycle Heater E5-320 and HP Solvent Cooler E5-321 where the solvent is cooled to ~40°C for methyl acetate scrubbing duties in the Atmospheric Scrubber and the HP Absorber. The HP Solvent pumps are also provided with PT-06420 on their common discharge line which triggers HP Solvent Pumps Autostart interlock, I-139E on detection of low pressure. This is discussed in detail discussed in Section 5.5.4.2.

In addition to supplying solvent to the HP Solvent Cooler, the HP Solvent Pumps supply the HP Solvent header for flushing and start-up duties. HP Solvent flow is controlled locally by the users. Also, HP Solvent is supplied to DH Column when the Water Draw Off flow from HP Absorber falls below a minimum value (X) discussed in Section 5.1.3.1 above.

### 5.1.3.5 Acetic Acid Import Control

The base of the DH Column is provided with level transmitter LT-06105 which provides the signal for level controller LICA-06105. A separate level indicator LT-06106 provides the signals for a DCS indication, LIA-06106A and a local indication LI-06106B. The two loops use different methods of measurement and so provide added security against the base overfilling which could lead to damage of the lower packed bed. The level in the DH Column is used to control the import of fresh solvent to make up for process losses.

Under normal operation, acetic acid import is via a 1½" line and the flow is measured by FT-06102 and adjusted by FCV-06102. The import of acid is controlled by LICA-06105 which adjusts the set point of flow controller FICA-06102 to maintain the DH Column base level.

On start-up, when a large import of acid is required, the import is via a 6" line from the acetic acid start-up pump and the flow is measured by FT-06103 and indicated in DCS as FI-06103. In this situation there is no automatic level control in the DH Column base.

The total quantity of acid imported into the plant is calculated and displayed in the DCS by FQI-06104, which takes flow readings from both FT-06102 and FT-06103.

### 5.1.4 Trips & Alarms

#### 5.1.4.1 Z-140-28: G5-606A/B, G5-607A/B Pump Seals, and D5-601 Flammable Inventory Fire Protection

Pumps G5-606A/B and G5-607A/B handle flammable liquid and are at particular risk of serious fire due to the large inventory of flammable liquid that could escape through a failed pump seal. The logic comprises of remotely operated shut-off valve (ROSOV) XSV-06118 mounted on D5-601 bottoms exit route to E5-603, the related instrumentation of which is explained below.

The P&ID legend drawing 10005-G41-GPZ105-0003 shows generic ROSOV arrangements for both actuated on/off valves and for control valves used as ROSOVs. The tag numbers in this description refer to those in the generic arrangements; actual tag numbers for each system are listed in section below.

Each protection system typically closes the relevant ROSOV(s) and shuts the pump(s) down if a fire is detected; the operator can also isolate the system from a safe distance using a manual push button.

| ROSOV tag | Pump tag | XSV-X | GSS-X (ROSOV) | PT-X | HS-X | HSS-X (C/R) | HSS-X (Field) | XA-X |
|---|---|---|---|---|---|---|---|---|
| Z-140-28 | G5-606A/B<br>G5-607A/B | XSV-06118 | GS-06118 | PT-06118 | HS-06118 | HSS-06118 | HSS-06118 | XA-06118 |

Description of operation (for tag number refer table above)

Initiators

- PT-29256 Low low pressure of instrument air header to PTA 5 ISBL
- GSS-X Limit switch signaling emergency isolation valve (ROSOV) XSV-X closed
- PT-X Low pressure in the instrument air line to isolation valve XSV-X
- HSS-X (C/R) A Hard-wired switch on fire-alarm panel in control room
- HSS-X (Field) Remote-mounted, manual emergency close button
- HS-X DCS hand switch to close XSV-X

Outputs

- XSV-X Close upstream emergency isolation valve (ROSOV), via manually reset solenoid
- XA-X Alarm in plant fire-alarm system
- Stops pumps G5-606 A/B and G5-607 A/B

- Inhibit pump auto-start I-139E

Logic of Operation

- The instrument air supply to each ROSOV (XSV-X) includes two fusible sections (UV ray resistant flexible nylon tubing which typically melts between 170 °C and 260°C), one of which is passed over and clamped in position around each pump seal, the other supported over the gland of the ROSOV (XSV-X). Either fusible section is the weak part of the loop which melts when exposed to fire, venting the instrument air supply and depressurizing ROSOV actuator. Being actuated on/off valve, the ROSOV closes in such scenario and this isolates the source of flammable DH solvent from the fire.
- A pressure switch, PT-X is fitted to the instrument airline to each ROSOV close to the valve. When it detects a loss of air pressure, it sends a signal to the fire alarm system, which closes ROSOV XSV-X and trips the associated pump(s) and inhibits the auto-start system (I-139E) to prevent the standby pump, G5-607A/B from kicking into service on sensing low HP Solvent header pressure. ROSOV, if the fusible section melts, thus automatically closes; either directly or via the trip as narrated above. Stoppage of both G5-606 A/B triggers I-466 and stoppage of both G5-607 A/B triggers I-169. I-466 and I-169 are described later under section 5.4.4.1 and 5.5.4.1 respectively.
- The fire alarm system also receives a signal from instrument air header PT-29256, at the source of instrument air to the affected section of the plant, to inhibit multiple alarms that would otherwise result from a loss of air pressure across the whole plant. An orifice restrictor (FO) is fitted to the air supply to the ROSOV, to provide sufficient pressure drop when the tubing melts to ensure that the pressure switch operates.
- The ROSOVs are fitted with closed limit switch, GSS-X which stops the associated downstream pump(s) if the ROSOV closes.
- An emergency shutdown button, HSS-X (C/R), is installed in the control room, close to the fire alarm system, to allow this complete ROSOV system to be isolated manually.
- A second button, HSS-X (Field), is installed at a safe distance away from ROSOV(approx. 10-15 meters away from the potential leak source, preferably on the likely escape route) with the same function as HSS-X (C/R).
- DCS hand switch HS-X is provided to close ROSOV (via a solenoid valve) and trip the associated pump(s) from DCS..
- Each ROSOV valve is provided with a lockable, manual, 3-way valve on the instrument air supply line for maintenance isolation.
- A bypass facility is provided to allow on-line testing of PT-X. PT-29256 does not require a bypass facility; it may be tested during normal operation.

The Solvent Dehydration System is protected against the effects of overpressure by Z-196, Z-191 and D5-

601 relief streams, and against the effects of underpressure by Z-190. In addition, PICA-06201 reduces the potential demand on Z-190 by providing a means of controlling pressure above the low pressure trip setting.

Although the Solvent Dehydration system is designed for full vacuum, the system is connected to atmosphere and therefore trip Z-190 is designed to provide protection against air ingress and the possible creation of a flammable atmosphere.

#### 5.1.4.2 Z-196 DH Column Base High Pressure

This trip reduces the risk of over-pressuring the DH Column.

If high high pressure is detected in the base of the DH Column via PT-06117 then Z-196 is initiated and the liquid feeds into the DH column from the HP Absorber and First Crystalliser Vent Separator are stopped to prevent gas breakthrough and a consequent demand on the relief system. Z-196 closes the following valves via solenoids:-

| | |
|---|---|
| LCV-03509 | WDO from D5-310 |
| LCV-03511 | Acid liquor from D5-310 |
| LCV-04309 | Condensate from F5-432 |

***Trip tidy actions***

| | |
|---|---|
| LICA-03509 | Set controller to manual and 0% output |
| HS-03511 | Set DCS hand switch to manual and 0% output |
| LICA-04309 | Set controller to manual and 0% output |

These outputs are executed as “one-shot” actions in the DCS.

#### 5.1.4.3 Z-190 Solvent DH Column Low Pressure Trip

The Solvent Dehydration Column D5-601 is designed for full vacuum. The column and overheads system is connected into the relief header, which is blanketed with inert gas. Negative pressure in the system will initially draw in inert gas from this header, but will ultimately draw in air via the Atmospheric Scrubber, D5-508. This could potentially result in a flammable atmosphere in the column.

Z-190 protects against the creation of a flammable atmosphere due to air ingress. It achieves this by opening the pressure control valve PCV-06201B on the inert gas supply to the column, closing the isolation valve XSV-06315 in the line to the Atmospheric Scrubber and stopping the Reflux Pumps G5-615A/B. The valves will remain in this state until the column pressure rises sufficiently to reset the initiating pressure, PT-06203, at which point the trip will reset.

***Initiators***

| | |
|---|---|
| PT-06203 | Low low pressure in D5-601 Solvent Dehydration Column<br>(PT-06203 is also an initiator for Z-191) |

| | |
|---|---|
| Z-140-29 | G5-615A/B upstream ROSOV trip |

***Outputs***

| | |
|---|---|
| PCV-06201B | Open inert gas supply to Solvent Dehydration Column overheads (via solenoid valve) |
| XSV-06315 | Close isolation valve in line from E5-631 to the Atmospheric Scrubber |
| G5-615A | Stop Reflux Pump G5-615A |
| G5-615B | Stop Reflux Pump G5-615B |
| PSA-06203B | Inhibit autostart of G5-615A/B via interlock I-139C |

***Logic Operation***

On detection of a low low pressure by PT-06203, Z-190 causes the pressure control valve PCV-06201B to open on the inert gas supply to the column, the isolation valve XSV-06315 to the Atmospheric Scrubber to close and the Reflux Pumps G5-615A and G5-615B to stop. Autostart of the pumps via I-139C is also inhibited. The outputs will remain in these states until the column pressure rises sufficiently to reset the initiating pressure, PT-06203, at which point the trip will reset and the outputs will return to normal.

The pump G5-615A or G5-615B will require restarting by the operator.

To reduce the demand on Z-190, pressure control loop PICA-06201 is provided. This loop operates via split range controller PY-06201 which operates as follows:-
- Valve PCV-06201A controls flow from the back up LP Nitrogen supply (0-50% of PICA-06201 output).
- Valve PCV-06201B controls flow of inert gas from the Buffer Vessel F5-925 (50-100% of PICA-06201 output).

As the pressure decreases, valve PCV-06201A (LP Nitrogen) changes from fully closed to fully open, with the B valve (Inert Gas) closed. As the pressure decreases further, the PCV-06201A valve remains open and the PCV-06201B valve changes from closed to open.

#### 5.1.4.4 Z-191 Solvent DH Column High Pressure Trip

The Solvent Dehydration Column D5-601 and overheads system are connected into the Atmospheric Scrubber, D5-508. Overpressure in the column D5-601 is ultimately protected by three relief valves. However, at elevated pressures below the relief setting, a considerable amount of material could be discharged from the column overheads system to the Atmospheric Scrubber, D5-508. This may exceed the design capability of D5-508 and may lead to emission to atmosphere. Z-191 limits this discharge by closing the isolation valve XSV-05519 in the line to D5-508 and the flow is directed to the Relief Scrubber D5-840. The valve will remain closed until the column pressure falls sufficiently to reset the initiating pressure, PT-06203, at which point the trip will reset.

If the high pressure persists for 5 minutes, then the Oxidation Reactor D5-301 is tripped.

***Initiators***

| | |
|---|---|
| PT-06203 | High high pressure in D5-601 Solvent Dehydration Column |

(PT-06203 is also an initiator for Z-190)

***Outputs***

| | |
|---|---|
| XSV-05519 | Close isolation valve in line from E5-631 to the Atmospheric Scrubber |
| Z-111 | Trip D5-301 Oxidation Reactor after a delay of 5 minutes |
| FICA-05121 | MP Steam to Stripper Reboiler E5-513 – set to manual, 0% |

***Logic Operation***

On detection of a high high pressure by PT-06203, Z-191 causes the isolation valve XSV-05519 to the Atmospheric Scrubber to close and sets FICA-05121 to manual mode with 0% output. XSV-05519 will remain closed until the column pressure falls sufficiently to reset the initiating pressure, PT-06203, at which point the trip will reset and the valve will open. If the high pressure persists for 5 minutes, then the Oxidation Reactor D5-301 is tripped.

A bypass facility is provided to permit on-line testing of the initiator. This bypass is also effective on Z-190 as the initiator is common to both trip systems.

### XCV-06315 / XSV-05519 valve action

If XSV-05519 is closed by Z-191, the pressure inside the DH system must be lowered to the trip reset pressure for the valve to re-open. If XCV-06315 were also closed and then were to open quickly, the resultant rapid de-pressurisation could damage the Atmospheric Scrubber and Recovery Column. A bypass is therefore provided around XCV-06315 to allow slow de-pressurisation via the Relief Scrubber.

For pressure testing, the DH system XCV-06315 can be closed by HS-06315, and following pressure testing the system must be de-pressurised using the bypass. When HS-06315 is in the 'Auto' position the valve will remain open unless tripped closed by Z-190.

### 5.1.4.5 Z-195 DH Column Base High Level

This trip reduces the risk of over-filling the DH Column at plant start-up.

***Initiators and Resets***

| | |
|---|---|
| LT-06106 | High high level in the DH Column base |

***Outputs***

| | |
|---|---|
| HCV-06141 | Close acetic acid filling valve, via solenoid |
| HIC-06141 | Set hand controller to 0% output |
| FIC-06102 | Set controller to manual mode, 0% output |

***Logic Operation***

High high level in the DH Column base closes the filling valve HCV-06141, sets its hand controller HIC-06141 to 0% output and closes FCV-06102 by setting its controller to manual mode with 0% output. The controllers are held at 0% output until the trip initiator becomes healthy.

#### 5.1.4.6 Alarms

The following alarms are provided to warn against undesirable and/or unacceptable operating conditions:-

| | |
|---|---|
| FICA-06101 | Waste water to D5-601 from D5-631 High Flow alarm |
| FICA-06136 | HP Solvent to D5-601 High Flow Alarm |
| TICA-06116 | High/Low temperature at the base of the DH Column |
| TIA-06107 | High temperature at the top of the DH Column |
| PIA-06117 | High pressure at the base of the DH Column |
| LICA-06105 | High/Low level in the base of the DH Column |
| LIA-06106A | High level in the base of the DH Column |
| PICA-06201 | High/Low pressure in the DH Column overheads |
| FICA-06226 | Low reflux flow |
| FICA-06416 | Low HP Solvent flow |
| TIA-03101 | High HP Solvent temperature |
| GICA-06115 | High/Low temperature profile controller output |
| FICA-06119 | Low LP Steam flow to DH Column Reboiler |
| PICA-06120 | LP Steam to Reboiler Low Pressure Alarm |
| FICA-06102 | Make-up Acetic Acid Low Flow Alarm |
| YA-06409 | Both DH Solvent Pumps G5-606A/B stopped |
| YA-06415 | Both HP Solvent Pumps G5-607A/B stopped |

In addition, all alarms related to Auto start of pumps, e.g. "Auto start unavailable" will also appear on the DCS.

## 5.2 DH Column Reboiler, E5-602 (Vendor: L&T)

### 5.2.1 General Information

DH Column Reboiler E5-602 is a single pass, vertical, shell-and-tube type heat exchanger which operates on the thermosyphon principle, having LP steam on the shellside and acetic acid/water (i.e. process solvent) in the tubes. The shell is constructed from carbon steel and the tubes (seamless), tubesheet and headers from 2205 duplex stainless steel. The design pressures and temperatures are 5.9 barg/FV and 225°C on the process side and 5.5 barg/FV and 185°C on the steam side.

The shell has steam inlet and condensate outlet nozzles plus an impingement plate at the steam inlet to prevent possible tube damage from the velocity of the incoming steam. The top header has a process side outlet nozzle and a manway. The bottom header has a process side inlet nozzle and a bottom manway, plus a low point drain.

LP Steam line to E5-602 is provided with relief protection via relief valve, RV/E5-602.

Additional equipment details are provided below:

- TEMA Type: NEN (Vertical)
- Tubeside Fluid: Acetic Acid/Water at an inlet pressure of 2.1 bara
- Tubeside Fluid Temperature(In/Out): 120.2°C/ 123°C
- Shellside Fluid: LP Steam at an inlet pressure of 4.15 bara
- Shellside Fluid Temperature(In/Out): 144.9°C/ 144.4°C
- Number of tubes: 8386
- Tube length: 6200 mm (from face to face of tubesheet)
- Tube OD: 25.4 mm
- Tube thickness: 1.65 mm min.
- Pitch: 31.75 mm
- Layout angle: 30°
- Shell inside diameter: 3400 mm
- Effective Heat Transfer Area: 3982 $m^2$
- Number and type of baffles: Horizontal double segmental baffles with 31% cut(9 in number)

### 5.2.2 Operating Philosophy

The DH Reboiler is situated at the base of the DH Column. It provides the heat energy required to drive the azeotropic distillation which separates acetic acid solvent from waste water. The LP Steam flow to the DH Reboiler is controlled to maintain an acetic acid concentration of 95 ± 0.5 wt% in the base of the DH Column. The resultant condensate stream from the Reboiler is exported under pressure control to the ELP Steam Flash Drum, F5-2209.

A butterfly valve is provided on process liquid inlet line to Reboiler. Normally the valve will remain open. It is provided to add in a flexibility should some throttling be required to control the thermosiphon circulation flow.

### 5.2.3 Instrumentation and Control

***Note: Some instrumentation for this equipment has been covered in Section 5.1.3.3.***

The steam flow to the DH Reboiler is adjusted to control the DH Column bottoms product composition and this is described in Section 5.1.3.3.

Temperature controller TICA-06116 controls the LP Steam flow to the reboiler by adjusting FCV-06119 via FICA-06119. LP Steam pressure at E5-602 inlet is measured by PT-06120.

A local pressure indication for process side inlet to E5-602 is provided by PI-06131.

Level and pressure in the Reboiler (tubeside) is controlled by LIC-06121 and PICA-06120. LIC-06121 and PICA-06120 provide input to the low select block PY-06120 which adjusts PCV-06120 in the export line to F5-2209, with the PCV located at minimum distance from F5-2209.

At start-up the Reboiler is primed by filling it from the DH Solvent Pump.

**Complex Loop PICA-06120 DH Column Reboiler Pressure**

The purpose of this loop is to regulate the discharge of steam condensate from DH Column Reboiler E5-602 to the ELP Steam Flash Drum F5-2209 under varying operating conditions.

The setpoint of controller PICA-06120 is set slightly above the pressure that is needed to reliably discharge steam condensate to the ELP Steam Flash Drum F5-2209. The output of PICA-06120 is normally 100% as its measured value is normally above its setpoint.

The setpoint for LIC-06121 is expected to be low and will be adjusted at commissioning to be consistent with the required heating duty of the reboiler.

The outputs of both controllers are input to low select block PY-06120, the output of which controls the condensate discharge valve PCV-06120. Under normal operation while the reboiler pressure is above the setpoint of controller PICA-06120, the condensate discharge valve PCV-06120 is controlled by the output of level controller LIC-06121 via the low select block PY-06120. In the event of a reactor trip the reboiler process duty reduces and the flow of steam is automatically set to a lower value. If the resultant reboiler pressure is below the setpoint of controller PICA-06120, then the output of PICA-06120 will fall and override the level controller signal and tend to close the condensate valve PCV-06120 to maintain the pressure at the setpoint.

***Loop Structure and Controller Details***

*PICA-06120*
PI control
ACTION: Direct
Output to low select block PY-06120

*LIC-06121*
PI control
ACTION: Direct
Output to low select block PY-06120

*PY-06120*
Low select block
Input 1 from PICA-06120
Input 2 from LIC-06121
Output to PCV-06120

### 5.2.4 Trips and Alarms

Z-111, the Reactor D5-301 trip system, controls the tripping of the output to FICA-06119.
When the Reactor trips, the setpoint of FICA-06119 is set to 44 te/hr on Auto (to be confirmed at commissioning). This sudden cut in demand is required to limit the sharp rise in import demand of SHP steam to the plant on oxidation reactor trip.

The LP steam flow controller FICA-06119 has a low flow alarm.

The LP steam pressure controller PICA-06120 has a low pressure alarm.

### 5.2.5 Relief Information

#### 5.2.5.1 RST/E5-602 /E5-602 Shellside Pressure Relief Valve

DH Column Reboiler, E5-602 is provided with a relief valve on its shellside.
Relief is provided by RV/E5-602 which discharges to atmosphere at a safe location. No special means of disposal are considered necessary.

The shell side of E5-602 has a design pressure of 5.5 barg.

Overpressure protection is to restrict pressure attributed to the following cause:
1. SYSTEM BLOCKED IN
A. External fire. Heat transfer is based on the wetted surface area of the exchanger and shell side piping inside the isolations, assuming it is flooded with condensate. Area has adequate drainage as specified by API and prompt fire fighting response available.

Additional details are as under:

- Safety valve set pressure: 5.5 barg
- Safety valve relieving temperature: 163.71°C
- Phase while relieving: Vapor
- Required Relieving Capacity: 2663 kg/h
- Number off: 1

## 5.3 Solvent Bottoms Cooler, E5-603 (Vendor: Hantech)

### 5.3.1 General Information

Solvent Bottoms Cooler E5-603 is a U-tube type (2-tube pass), horizontal, shell-and-tube heat exchanger with acetic acid solvent on the tubeside and cooling water on the shellside. The purpose of the cooler is to reduce the temperature of the DH Solvent below its boiling point, so as to avoid cavitation in both the DH and the HP Solvent Pumps, and to enable 316L SS to be used in the downstream solvent distribution system. The shell is constructed from carbon steel and the tubes (seamless), tubesheet and header from 2205 duplex stainless steel and  the shellside baffles are from 304L SS. The shell has cooling water inlet and outlet nozzles. The header has a process inlet and a process outlet nozzle.

The process side is designed for 20 barg/FV and 215°C. The cooling water side is designed for 7.0 barg/FV and 173°C.

Additional equipment details are provided below:

- TEMA Type: BEU (Horizontal)
- Tubeside Fluid: DH Solvent at an inlet pressure of 2.45 bara
- Tubeside Fluid Temperature(In/Out): 120.2°C/ 95°C
- Shellside Fluid: Cooling Water at an inlet pressure of 5.5 bara
- Shellside Fluid Temperature(In/Out): 34°C/ 44°C
- Number of tubes: 268
- Straight Tube length: 2900 mm
- Tube OD: 19.05 mm
- Tube thickness: 1.65 mm min.
- Pitch: 25.4 mm
- Layout angle: 30°
- Shell inside diameter: 750 mm
- Effective Heat Transfer Area: 91.49 $m^2$
- Number and type of baffles: Single segmental baffles with 24.39% cut(5 in number)

### 5.3.2 Operating Philosophy

The Solvent Bottoms cooler is designed to cool acetic acid (containing 5% water) leaving the base of the Dehydration Column from 120.2°C to 95°C. After cooling, the solvent is distributed to the Oxidation plant acetic acid users by the DH and HP Solvent Pumps.

A bypass line is provided which allows controlled amount of acetic acid from the DH column bottoms to bypass the cooler which is discussed in Section 5.3.3 below.

### 5.3.3 Instrumentation and Control

Temperature transmitter TT-06404 on the process exit line of the Solvent Bottoms Cooler adjusts E5-603 process bypass valve TCV-06404 via TICA-06404 to maintain the exit temperature at the required set-point of around 95°C.

### 5.3.4 Trips and Alarms

A high temperature alarm is provided by TICA-06404 to warn against lack of cooling in E5-603.

### 5.3.5 Relief Information
#### 5.3.5.1 RST/60427/ E5-603 Shellside Pressure Relief Valve

DH Solvent from Solvent Dehydration Column D5-601 is cooled to 95°C in Solvent Bottoms Cooler E5-603 using cooling water.

E5-603 shellside is provided with a pressure relief valve RV/60427 which discharges to chemical drain.
No special means of disposal are considered necessary.

The cold side of E5-603 has a design pressure of 7 barg. The relief valve set pressure is selected based on the elevation of the valve relative to the base of the condenser.

Overpressure protection is to restrict pressure attributed to the following causes:-
1. SYSTEM BLOCKED IN

A. External fire. Heat transfer based on the shell wetted surface area of the exchanger and cooling water piping inside the isolations. Area has adequate drainage as specified by API and prompt fire fighting response available.

B. System blocked in with thermal expansion due to heating by process fluid. Heat transfer based on heat exchanger design duty multiplied by the ratio of clean to fouled heat transfer coefficients, and an appropriate coefficient of thermal expansion is considered to be 0.0006/°C.

Additional details are as under:
- Safety valve set pressure: 7 barg
- Number off: 1

| Case | Req. Relief Rate(kg/h) | Relieving Temperature(°C) | Phase while relieving |
|---|---|---|---|
| 1A | 1080 | 173 | Vapor |
| 1B | 4763 | 120.2 | Liquid |

### 5.4 DH Solvent Pumps, G5-606A/B (Vendor: Sulzer Pumps)
### 5.4.1 General Information

DH Solvent pumps G5-606A/B (1W, 1S) are horizontal, centrifugal pumps each capable of delivering around 120 $m^3/h$ of DH Solvent at a discharge pressure of ~5.66 barg. The pump impeller and casing are constructed from SS316L while the shaft is constructed from 2205 Duplex SS. The design flowrate includes spare capacity for flushing duties. The pumps have double mechanical seals using LP Seal Water (Demin Water) as the seal fluid. Suction and delivery line isolation valves are installed along with suction and delivery drain lines to allow isolation, draining and maintenance. An LP caustic line (normally blinded) is provided at the discharge of G5-606A/B pumps for caustic flushing of the line to DH Solvent Header if the need arises.

The DH Solvent Pumps also provide a route for the export of DH Solvent to off-plot storage at a rate controlled by FO-06410.

Pump Casing Design conditions are 28 barg/FV and 215°C.

Additional equipment details are provided below:

- Normal/Rated capacity : 120 $m^3$/hr
- Rated differential head : 54.55 m
- Rated discharge pressure : 5.66 barg
- Efficiency at rated impeller(rated/maximum flow) : 72.5/75%
- Impeller type : Enclosed
- Impeller diameter (rated) : 208 mm (minimum : 205 mm / maximum : 259 mm)
- Maximum capacity at rated impeller: 172 $m^3$/h
- Minimum continuous flow : 38 $m^3$/hr
- Absorbed power rated impeller(rated /end of curve) : 23.8 KW / 30.9 KW
- Speed : 2950 rpm
- Seal type : Double mechanical seal with plan 54, 61M
- Seal flush fluid : LP seal water @ 0.24-0.36 m3/hr per pump (seal cavity pressure : 5.1 barg)
- Rotor: Overhung
- Casing type : double volute
- Motor rated power : 30 KW

### 5.4.2 Operating Philosophy

The DH Solvent Pumps run continuously with a flow-controlled kickback (to provide pump protection) and supply the DH Solvent header. In addition to flushing duties, the primary users of DH Solvent are the ROVACs, Solvent Stripper, Third CTA Crystalliser and the PTA Mother Liquor Filters. Solvent off-take from the header is controlled locally by the various users.

### 5.4.3 Instrumentation and Control

For details on solvent use and instrumentation see Section 5.1.3.4.

### 5.4.4 Trips and Alarms

Alarm YA-06409 is provided for "Both motors stopped".

The pumps are tripped by Z-140-28, the ROSOV fire protection system at the base of the Solvent DH Column. See section 5.1.4.1 for full details.

#### 5.4.4.1 I -466: DH Solvent Pumps G5-606A/B Backflow Protection

I-466 provides protective logic to prevent backflow of DH solvent in the event of failure of DH solvent pump, G5-606A&B. This is primarily to prevent any DH solvent reverse flow from DH solvent header in case Z-140-28 has become active due to a fire triggered by leaking seal of G5-606 A/B as otherwise it will add fuel to the fire.

***Initiators***

| | |
|---|---|
| YA-06409 | DH Solvent Pumps G5-606A&B both pumps stopped. |

***Outputs***

| | |
|---|---|
| FCV-06401 | Close DH Solvent Pump G5-606A/B kickback Control valve FCV-06401 (via controller). |

***Logic Operation***

When G5-606 A&B are both stopped, FCV-06401 shall be closed via a one-shot input by setting its controller FICA-06401 to manual with 0% output.

The operator may re-open the pump kickback valve FCV-06401 prior to restarting G5-606A/B.

## 5.5 HP Solvent Pumps, G5-607A/B (Vendor: Kirloskar Ebara)

### 5.5.1 General Information

HP Solvent pumps G5-607A/B (1W, 1S) are horizontal, 2-stage centrifugal pumps each capable of delivering around 280 m³/h of DH Solvent at a discharge pressure of ~20.66 barg. The pump impeller and casing are constructed from SS316L while the shaft is constructed from 2205 Duplex SS. The design flowrate includes spare capacity for flushing duties. The pumps have double mechanical seals using IP Seal Water (Demin Water) as the seal fluid. Suction and delivery line isolation valves are installed along with suction and delivery drain lines to allow isolation, draining and maintenance. An LP caustic line (normally blinded) is provided at the discharge of G5-607A/B pumps for caustic flushing of the line to HP Solvent Header if the need arises.

Pump Casing Design conditions are 46.63 barg/FV and 215°C.

Additional equipment details are provided below:

- Normal/Rated capacity : 280 $m^3$/hr
- Rated differential head : 213.44 m
- Rated discharge pressure : 20.66 barg
- Efficiency at rated impeller(rated/maximum flow) : 69/60%
- Impeller type : Enclosed
- Impeller diameter 1$^{st}$ stage(rated) : 277 mm (minimum : 270 mm / maximum : 321 mm)
- Impeller diameter 2$^{nd}$ stage(rated) : 338 mm (minimum : 330 mm / maximum : 392 mm)

- Maximum capacity at rated impeller: 310 $m^3/h$
- Minimum continuous flow : 60 $m^3/hr$
- Absorbed power rated impeller(rated /end of curve) : 234.66 KW / 215.72 KW
- Speed : 2965 rpm
- Seal type : Double mechanical seal with plan 54, 61M
- Seal flush fluid : IP seal water @ 0.36 m3/hr per pump (seal cavity pressure : 20 barg)
- Rotor: Between bearing
- Casing type : double volute
- Bearing housing (DE/NDE) cooling : Emergency cooling water @ 0.84 m3/hr
- Motor rated power : 275 KW

### 5.5.2 Operating Philosophy

The HP Solvent Pumps run continuously with a flow-controlled kickback (to provide pump protection) and supply the HP Solvent header system. The bulk of the forward flow from the HP Solvent Pumps passes to the Recycle Heaters and HP Solvent Coolers, where the solvent is cooled to ~40°C for methyl acetate scrubbing duties in the Atmospheric Scrubber and HP Absorber.

The HP Solvent Pumps also supply the HP Solvent header for solvent dilution to the CTA crystallisers plus flushing and start-up duties. The flow of HP Solvent is controlled locally by the various users. The pumps are fitted with an auto-start system due to the critical duty the pumps provide. On detection of a low pressure in the common discharge line from the pumps the spare (standby) pump will automatically start.

When the Water Draw Off flow from HP Absorber falls below a minimum value(X), waste water is added from D5-631 and HP solvent is also added by G5-607A/B according to the ratio decided by FFY-06136. This has been discussed in detail earlier in Section 5.1.3.1.1.

### 5.5.3 Instrumentation and Control

G5-607A is provided with motor winding temperature and motor bearing temperature indications in the DCS as TT-06435/36/37 and TT-06438/39 respectively.

G5-607B is provided with motor winding temperature and motor bearing temperature indications in the DCS as TT-06440/41/42 and TT-06443/44 respectively.

For details on solvent use and instrumentation see Section 5.1.3.4.

### 5.5.4 Trips & Alarms

Alarm YA-06415 is provided for "Both motors stopped", which also initiates interlock I-169.

High winding and bearing temperature alarms are provided for the pumps G5-607A/B in the DCS.

The pumps are tripped by Z-140-28, the ROSOV fire protection system at the base of the Solvent DH Column. See section 5.1.4.1 for full details.

### 5.5.4.1 I-169

I-169 provides back flow protection logic for G5-607A/B.

***Initiator***
YA-06415

***Outputs***

| | |
|---|---|
| FCV-03252 | HP Solvent to D5-301 to D5-401 transfer line (via I-137) |
| FCV-04180 | HP Solvent to D5-401 to D5-402 transfer line (via I-137) |
| FCV-03505 | HP Solvent exit E5-321 to D5-310 (via controller) |
| FCV-06416 | HP Solvent pump G5-607A/B kickback (via controller) |

***Logic Operation***
After a 20 second delay (adjustable 0-30 s) to allow the standby pump to autostart, initiator YA-06415 shall close valves FCV-03252, FCV-03505, and FCV-04180 by setting their controllers to manual and holding the outputs at 0%. FCV-06416 shall also close via a one-shot input to set its controller to manual, 0%.

The operator may re-open the pump kickback valve FCV-06416 prior to re-starting G5-607A&B; the remaining valves are available to the operator after the pump has been started.

### 5.5.4.2 I-139E

G5-607 A/B are provided with auto start facility to bring in the standby pump when the combined discharge line pressure drops below a set pressure due to snag developed in the in-line pump or the in-line pump trips. Combined discharge line pressure is monitored on PT-06420 which is used for the auto start logic I-139E.

Following alarms are associated with auto start logic which appear on DCS :

YA-06425 : Auto start unavailable : If either motor is electrically unavailable e.g. the stop button is latched in or the motor is de-energised at substation

YA-06426 : Both motors running : If both motors are running as a result of auto start action or manual operation

YA-06415 : Both motors stopped : If both motors are stopped. The alarm is delayed for 5 secs (adjustable 0-10 secs) to allow the standby pump to auto start when low pressure is detected.

PT-06420 : Low discharge pressure

Auto start logic is inhibited when the related ROSOV system (Z-140-28) gets activated (refer section 5.1.4.1 for details).

## 5.6 DH Column Condenser, Condenser Contactor, Decanter and Entrainer Storage Tank, E5-608, F5-608/I, F5-608, F5-609 (Vendor: Godrej)

### 5.6.1 General Information

These four main plant items (MPIs) are enclosed in a single unit and their operation is intimately linked, therefore the description and operation of the total system is described in this section.

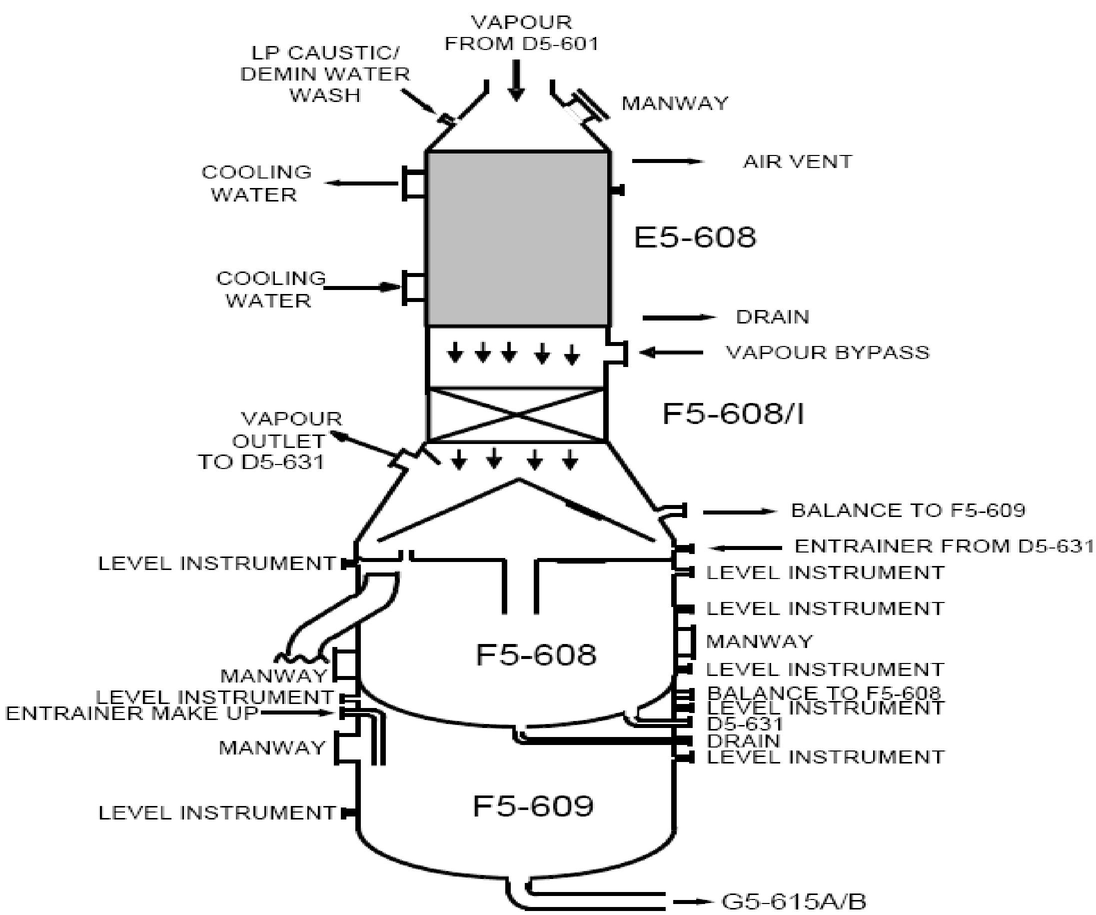



*Fig 2.6.1 - DH Column Condenser, Condenser Contactor, Decanter & Entrainer Storage Vessel*

### 5.6.1.1 DH Column Condenser E5-608

DH Column Condenser E5-608 is a single pass, downflow, vertical, shell-and-tube heat exchanger with cooling water on the shellside. The shell is constructed from carbon steel, the tubes from 2205 duplex stainless steel, the tubesheet from 304L stainless steel-clad carbon steel and the header from 304L stainless steel. The shell has cooling water inlet and exit nozzles, a vent nozzle and an inspection nozzle. The top header has a vapour inlet nozzle, a manway and a nozzle for Demin Water/Caustic flush. The bottom of the exchanger is bolted directly onto the DH Column Condenser Contactor F5-608/1. Inert gas connections are provided at upstream as well as downstream of GCV-06208 in the vapor line from D5-601 for purging purposes.

The design pressures are 3.5 barg/FV for the tubeside, and 7 barg/FV for the shellside. The design temperatures are 173°C for both shell and tubeside.

Additional equipment details are as provided below:

- Tubeside Fluid: DH Column Overhead Vapor at an inlet pressure of 1.2 bara
- Tubeside Fluid Temperature(In/Out): 86°C/ 78°C
- Shellside Fluid: Cooling Water at an inlet pressure of 5.5 bara
- Shellside Fluid Temperature(In/Out): 34°C/ 44°C
- Heat duty: 101600 kW
- Tube length: 6383 mm from reference to top face of tubesheet
- Shell inside diameter: 3100 mm
- Effective Heat Transfer Area: 2725 $m^2$
- Number and type of baffles: Single segmental baffles with 17.7% cut (2 in number)

### 5.6.1.2 DH Column Condenser Contactor F5-608/I

DH Column Condenser Contactor F5-608/1 is connected directly to the base of the DH Column Condenser and is an integral part of the DH Column Decanter F5-608. The Contactor has a bed of 304L SS random column packing IMTP 50 supplied by Koch, above which there is a vapour/liquid re-distributor to ensure efficient re-mixing and equilibration of the cooled and partially condensed stream coming from E5-608 and the hot and uncondensed stream bypassing E5-608. An orifice deck distributor positioned above the packed bed distributes the condensed liquid coming from E5-608 across the cross section of packing.Two nozzles for the vapour bypass lines and a manway are provided in the section between the top of the Contactor and the base of the DH Column Condenser.

Column bed height is 1000 mm and bed diameter is 3100 mm.

### 5.6.1.3 DH Column Decanter F5-608

DH Column Decanter F5-608 is a 304L SS vessel which is connected directly to the DH Column Contactor by a conical section.

The Decanter has the following branches in the conical section:-

- Vapour outlet to D5-631
- Vapour balance line to the Entrainer Storage Tank

In the side wall there are the following branches:-

- Two level transmitters (organic and aqueous phase transmitters)
- Manways (two off)
- Liquid inlet from the Recovery Column
- Organic phase takeoff to G5-615A/B suction

In the dished base, and exiting through the wall of the vessel below, are the following nozzles:-

- Drain (normal no flow)
- Aqueous phase outlet to the Recovery Column

Inside the vessel there are hatches for inspection access on the impingement cone roof and internal collection plate. An internal overflow pipe is also provided on the internal collection plate. The baffle plates have access ways arranged to allow entry to the aqueous chamber for weir adjustment. The width clearance between the two baffle plates is 65 mm and they differ in height by 450 mm.

Additional equipment details are as under:

- Design Pressure: 3.5 barg @150°C/ FV @46°C
- Design Temperature: 150°C
- Operating pressure (max/norm/min): 1.2/1.15/1.02 barg
- Operating Temperature (max/norm/min): 78/78/72°C
- Gross capacity (combined for F5-608/F5-609): 374.8 $m^3$
- Operating Fluid: n-propyl acetate + water + organics
- Insulation type: Heat Conservation + Fire Hazard Protection type of thickness 115 mm
- Shell diameter: 6400 mm
- Bottom dished end of F5-608 is spherical

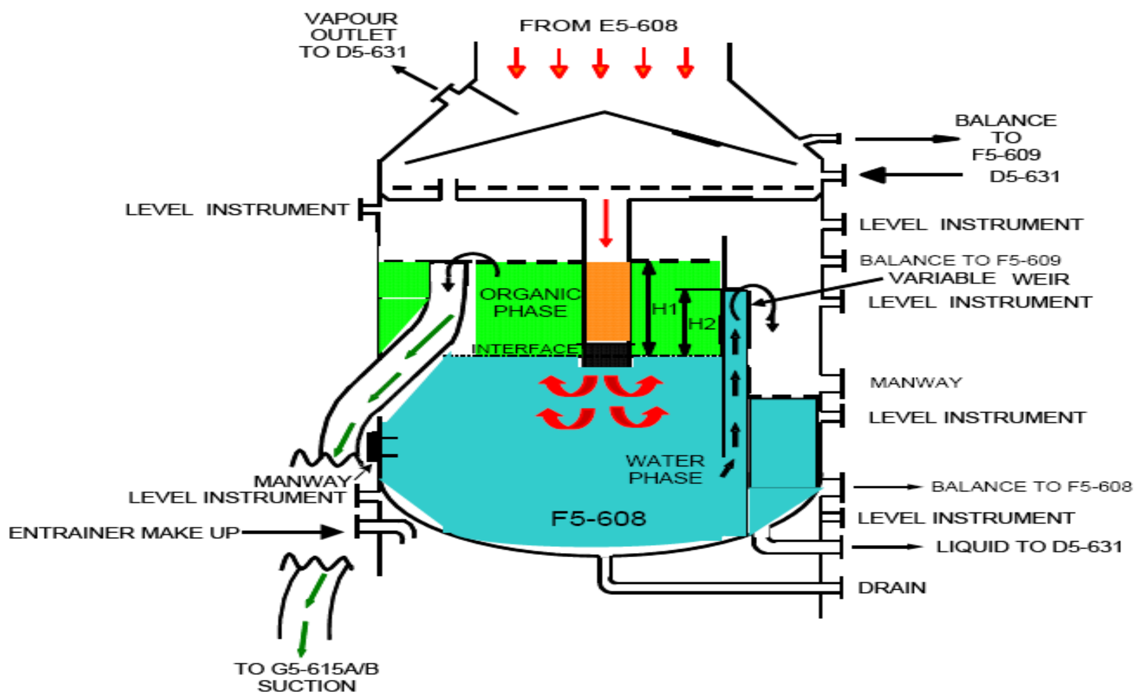



*Figure 5.6.1.3 - DH Column Decanter*

### 5.6.1.4 Entrainer Storage Tank F5-609

The Entrainer Storage Tank is a 304L SS vessel coupled directly to the base of the DH Column Decanter.

The following branches are provided in the side wall of the vessel:-

- Two level transmitters
- Vapour balance line to the Decanter
- Entrainer make-up dip pipe
- G5-615A/B Kickback dip pipe
- Aqueous phase outlet from the Decanter
- Entrainer drainage from the Decanter (normal no flow)
- Manway

In the bottom of the vessel, there is a nozzle for the entrainer outlet (with vortex breaker).

The entrainer import line and Reflux Pump circulation line are both routed into the vessel via dip-legs which are fitted with anti-syphon holes. This protects against a splash filling effect.

Additional equipment details are as under:

- Design Pressure: 3.5 barg @150°C/ FV @46°C
- Design Temperature: 150°C

- Operating pressure (max/norm/min): 1.15/1.10/1.02 barg
- Operating Temperature (max/norm/min): 86/40-78/40°C
- Gross capacity (combined for F5-608/F5-609): 374.8 $m^3$
- Operating Fluid: n-propyl acetate + water + organics
- Insulation type: Heat Conservation + Fire Hazard Protection type of thickness 115 mm
- Shell diameter: 6400 mm
- Overall height of (F5-608+F5-609): 7150 mm from tan line to tan line
- Bottom dished end of F5-609 is 2:1 ellipsoidal

### 5.6.2 Operating Philosophy

#### 5.6.2.1 DH Column Condenser and Contactor

Overhead vapours from the DH Column enter the Condenser at 86°C and it is necessary to control the degree of cooling to manipulate the flow of methyl acetate through the DH System. Since cooling water side fouling constraints (at low flow) in E5-608 make it undesirable to alter the flow of cooling water, the temperature is instead controlled by allowing some of the vapour to bypass the Condenser and then re-mixing it in the DH Column Condenser Contactor with the vapour which has passed through the Condenser.

In the DH Column Condenser Contactor the vapour and liquid from the DH Column Condenser, plus the vapour from the bypass stream, are brought into intimate contact. This mixing results in an equilibrium mixture of vapour and liquid passing out of the bottom at typically 78°C. Any variation in the DH System condensing load is handled by the DH Column Condenser, with the intention of providing a relatively steady vapour feed to the Recovery Column over a wide range of plant operating rates. The bypass flow is therefore controlled to maintain a stable flow of vapour from the DH Column Condenser Contactor to the Recovery Column as described in Section 5.6.3.1.

#### 5.6.2.2 DH Column Decanter

The two-phase mixture leaving the DH Column Contactor is directed onto the internal cone deflector plate in the DH Column Decanter where the phases separate. The vapour is vented to the Recovery Column, whilst the liquid, together with the liquid from the rectification section of the Recovery Column, passes down onto an internal collection plate, and then drains into a downcomer pipe which has a KnitMesh-type demister fitted at the outlet to promote droplet coalescence, so enhancing separation of the two liquid phases.

In the lower section of the DH Decanter, the organic phase is separated from the aqueous phase by specific gravity difference (organic phase on top, aqueous phase below). The organic layer overflows through an internal pipe and passes to the Reflux Pumps for return to the DH Column. The aqueous phase passes under and over a double baffle arrangement before being taken off at the bottom of vessel to the Recovery Column. The level of the aqueous/organic interface depends on the level of the take-off position. The level of the interface between the two phases depends on the weir height of the overflow baffle plate.

The interface level should be at the midpoint of the coalescence pad to maximise the efficiency of separation. The water overflow baffle plate has an adjustable weir that has been set to give the correct interface level. On the sketch Fig 5.6.1.3, H1 is the height of entrainer above the interface and H2 is the level of water. H1/H2=density of water/density of entrainer.

If the weir height is too low, the interface will be lower, leading to a potential for the organic phase to pass through the baffles to the aqueous take-off. If the weir height is too high, the interface will be higher, leading to a potential for the aqueous phase to pass through the organic take-off.

The weir can also be adjusted for width as well as height. The width must be correct to ensure that rate changes in the DH Column do not upset the liquid interface level too much. These rate changes cause the liquid levels above the entrainer take-off and above the water weir to vary.

#### 5.6.2.3 Entrainer Storage Tank

The Entrainer Storage Tank provides limited, on-plot storage for entrainer and a buffer for coping with fluctuations in usage in the Solvent Dehydration Column. The Tank is on a continuous import system based on a supply from an off-plot metering pump.

The discharge flow from the tank bottom alongwith the organic phase take-off line from DH Column Decanter provides the suction for G5-615A/B pumps.

### 5.6.3 Instrumentation and Control

#### 5.6.3.1 Control of Condensation and Separation

The function of this group of main plant items is to control the condensation of DH Column overheads in a way that prevents significant subcooling (which could lead to the trapping of methyl acetate in the DH Column and reflux loop), then to separate vapour and aqueous liquors for further treatment in the Recovery Column and to reflux the organic phase back to the DH Column.

Controlling the cooling water flow is undesirable as high cooling water return temperatures lead to corrosion problems and low cooling water flow rates lead to rapid exchanger fouling. The preferred method of control is to bypass a proportion of the DH Column overheads around the condenser then re-mix to equilibrate as a means of controlling condensation.

The condensation of overheads is controlled to achieve a steady vapour flow to the Recovery Column D5-631. The flowrate of vapour to the Recovery Column is measured by low pressure drop flow meter FT-06209, and flow controller FICA-06209 adjusts flow control valve FCV-06209 located in the vapour bypass line as necessary in order to maintain a steady flow. The operation of this loop is slow as over-condensation of overhead vapours only leads to a slow build-up of methyl acetate in the Solvent Dehydration Column loop and rapid changes in the vapour flowrate to the Recovery Column are undesirable.

GCV-06208 in the main overheads line to the Condenser is provided to balance the pressure drops around the bypass and through the DH Column Condenser E5-608, and to ensure that the bypass flow control valve FCV-06209 has an adequate pressure drop in order to function correctly and on the overall ensures that the pressure drop between D5-601 and E5-608 is kept minimum and thereby minimizing pressure in D5-601 itself. If GCV-06208 is set too far closed, the Solvent Dehydration Column D5-601 runs at a higher than necessary pressure; if it is set too far open, then the flow control valve cannot function correctly. In order to achieve the optimum operation, the flow controller FICA-06209 also inputs the position of control valve FCV-06209 into valve position controller GIC-06208, which modulates valve GCV-06208 in the process flow to the Condenser. Setpoint of GIC-06208 should be the maximum valve position that still allows adequate response to disturbances (initial value 90%). However once the system has been balanced it is anticipated that the position of GCV-06208 will remain largely unchanged. The flow controller (FICA-06209) should be tuned first to give good flow control. The valve position controller (GIC-06208) should then be tuned to be slow enough to avoid disturbing the flow controller.

The temperature of the vapour flow to the Recovery Column is recorded by TIA-06210. This temperature is not used for control as the temperature vs. degree of condensation curve is very steep in this area.

Following condensation, the two liquid phases are separated in the DH Column Decanter F5-608, with the organic phase draining by gravity to the Reflux Pumps G5-615A/B and the aqueous phase draining by gravity to the Recovery Column D5-631. To provide process monitoring, the flow of the aqueous phase to the Recovery Column is measured by FT-06217 and recorded by FI-06217 on the DCS. The temperature of the reflux returning to the DH Column is measured by TT-06223 and recorded by TIA-06223 on the DCS. Level measurement on the aqueous overflow and organic phase in the decanting section of the DH Decanter is provided by LICA-06212 and LIA-06211 respectively which receive their signals from LT-06212 and LT-06211 respectively. Level control of the aqueous phase compartment is achieved by LICA-06212 which adjusts LCV-06212 in the liquid outlet line to D5-631.

In the overheads line to the DH Column Condenser are temperature measurement TI-06204 via TT-06204, and pressure trips, PSA-06203A (Z-191) and PSA-06203B (Z-190). For further information on the pressure trips see Section 5.1.4.

Cooling water return line from E5-608 is provided with a local pressure indication, PI-06240. This line is also provided with flow (FT-06206) and temperature (TT-06207) transmitters which indicate in the DCS as FI-06206 and TI-06207 respectively.

#### 5.6.3.2 Entrainer Storage Instrumentation

Instrumentation is provided to warn of high and low entrainer levels in the Entrainer Storage Tank and to monitor the transfer of entrainer into and out of the tank.

Level transmitter LT-06213 provides level measurement for the Entrainer Storage Tank which indicates in DCS as LIA-06213 and in the field as LI-06213A. The tank is made up from an OSBL supply via XV-06229.

The level is allowed to fluctuate in an acceptable range and Entrainer is imported batch-wise into the vessel.

Level transmitter LT-06215 provides a signal to LIA-06215, which measures over a different (higher) range than LT-06213.

Entrainer is imported to the Entrainer Storage Tank from OSBL Entrainer Storage F5-2641 on a continuous basis via a metering pump with adjustable flowrate, and is measured by flowmeter FT-06228, totalized on FQI-06228 and indicated on DCS as FI-06228. A bypass line of 4” is provided across flowmeter FT-06228 to fill the Entrainer Storage Tank on start-up, the flow of which is adjusted by FO-06230. Entrainer can also be exported to OSBL Entrainer Storage using the same line. A pump-out line is provided from the discharge of the reflux pumps via FO-06214 to OSBL Entrainer Storage which is used at shutdown for deinventorying the Entrainer from the system. An inert gas connection is provided to the pump-out line to facilitate complete deinventorying of Entrainer from the pump out line and make the line Entrainer free. Inert gas flow is adjusted by FO-06245 and measured locally by FI-06246.

The Entrainer import/export flow is also measured using a bi-directional flow measurement, FT-06247 which displays in the DCS as FIA-06247. There is provision to totalize the flow on separate flow totalizers depending on which way the flow happens – FQI-06247A : for import of Entrainer to ISBL and FQI-06247B for export to off-plot Entrainer storage tank. Switching between totalizers is done using DCS switch XI-06247 which toggles between Import and Export based on operator selection. When Import option is selected, import totalizer functions based on flow. Export totalizer counts when Export option is selected on XI-06247

### 5.6.4 Trips and Alarms

#### 5.6.4.1 I-166 High Level in Entrainer Storage Tank F5-609

In the event of a high level being detected in the Entrainer Storage Tank F5-609, this interlock closes the Entrainer transfer valve XV-06229 and stops Entrainer Transfer Pump (G5-2642) and Entrainer Start-up Pump (G5-2644) motors.

***Initiators***

| | |
|---|---|
| LT-06213 | High high level in the Entrainer Storage Tank F5-609 |
| LT-06215 | High high level in the Entrainer Storage Tank F5-609 |

***Outputs***

| | |
|---|---|
| XV-06229 | Close valve via solenoid valve SOV-06229 |
| G5-2642 | Stop Entrainer Transfer Pump |
| G5-2644 | Stop Entrainer Start-up Pump |

***Logic Operation***

If a high high level is detected in the Entrainer Storage Tank F5-609 by level transmitter LT-06213 or LT-06215, the Entrainer transfer valve, XV-06229, shall close via solenoid SOV-06229, and Entrainer Transfer Pump (G5-2642) and Entrainer Start-up Pump (G5-2644) motors shall stop.

DCS hand switch HS-06229 allows the operator to close XV-06229 manually at any time.

#### 5.6.4.2 Z-111

Z-111, the Reactor D5-301 trip system, controls the tripping of the outputs to FICA-06209 and FICA-06226 in the event of a Reactor trip as follows:-

FICA-06209 – controls the flow of vapour from F5-608 to D5-631 Recovery Column.
FICA-06226 – controls the flow of reflux from G5-615A/B to D5-601.

On a Reactor trip, the set-point on FICA-06209 is set to 14 t/h on auto and the set-point on FICA-06226 is set to 170 t/h. (All figures to be confirmed at commissioning).

#### 5.6.4.3 Alarms

The following alarms are provided to warn against undesirable and/or unacceptable operating conditions:

| | |
|---|---|
| FICA-06209 | High/Low vapour flow from the DH Column Condenser |
| TIA-06210 | High vapour temperature exit the DH Column Condenser |
| LIA-06211 | High organic phase level in the DH Column Decanter |
| LICA-06212 | High aqueous phase level in the DH Column Decanter |
| LIA-06213 | High/Low Entrainer Storage Tank level |
| LIA-06215 | High Entrainer Storage Tank Level |
| FICA-06226 | Low reflux flow from G5-615A/B to D5-601. |
| TIA-06223 | High reflux temperature exit G5-615A/B. |
| FIA-06247 | Low Entrainer import/export flow |

### 5.6.5 Relief Information

#### 5.6.5.1 RST/60209/E5-608 Shellside Pressure Relief Valve

Overhead vapour from Solvent Dehydration Column D5-601 is partially condensed in DH Column Condenser E5-608 using cooling water.

E5-608 is provided with a pressure relief valve, RV/60209 on its shellside which discharges to chemical drain. No special means of disposal are considered necessary.

The cold side of E5-608 has a design pressure of 7 barg. The relief valve set pressure is selected based on the elevation of the valve relative to the base of the condenser.

Overpressure protection is to restrict pressure attributed to the following cause:-

1. SYSTEM BLOCKED IN
B. System blocked in with thermal expansion due to heating by process fluid. Heat transfer based on heat exchanger design duty multiplied by the ratio of clean to fouled heat transfer coefficients, and an appropriate coefficient of thermal expansion is considered to be 0.0006/°C.

Additional details are as under:

- Safety valve set pressure: 7 barg
- Relieving Temperature: 86°C
- Relieving Capacity Required: 96830 kg/h
- Phase while relieving: Liquid
- Number off: 1

## 5.7 Reflux Pumps, G5-615A/B (Vendor: Sulzer Pumps)
### 5.7.1 General Information

Reflux Pumps, G5-615A/B (1W, 1S) are horizontal, centrifugal pumps each capable of delivering around 700 $m^3$/h at a discharge pressure of ~5.94 barg. The pump impeller, casing and shaft are constructed from SS316L. The pumps have double mechanical seals using LP Seal Water (Demin Water) as the seal fluid.

The Reflux Pumps discharge also provides a route for the export of Entrainer to off-plot storage at a rate controlled by FO-06214.

Pump Casing Design conditions are 31 barg/FV and 150°C.

Additional equipment details are provided below:

- Normal/Rated capacity : 700 $m^3$/hr
- Rated differential head : 62.2 m
- Rated discharge pressure : 5.94 barg
- Efficiency at rated impeller(rated/maximum flow) : 80.5/75%
- Impeller type : Enclosed
- Impeller diameter (rated) : 465 mm (minimum : 415 mm / maximum : 514 mm)
- Maximum capacity at rated impeller: 700 $m^3$/h
- Minimum continuous flow : 140 $m^3$/hr
- Absorbed power rated impeller(rated /end of curve) : 122.3 KW / 143.2 KW
- Speed : 1485 rpm
- Seal type : Double mechanical seal with plan 54, 61M
- Seal flush fluid : LP seal water @ 0.24-0.36 m3/hr per pump (seal cavity pressure : 5.9 barg)
- Rotor: Overhung
- Casing type : double volute

- Motor rated power : 160 KW

### 5.7.2 Operating Philosophy

The Reflux Pumps take suction from the base of the Entrainer Storage Tank F5-609 and provide reflux to the top of Solvent Dehydration Column D5-601. The flowrate is determined by DH Column Profile Controller GICA-06115 (discussed earlier in Section 5.1.3.2). The Pumps also provide a means of transferring entrainer from the Entrainer Storage Tank to off-plot storage, if required. An inert gas connection is provided to the pump-out line for de-inventorying purposes the flow of which is adjusted by FO-06245 and measured locally by FI-06246.

The Reflux Pumps are provided with an auto-start facility to ensure a reliable reflux feed to the Solvent Dehydration Column.

The Pumps are protected against cavitation at high turndown by a flow-controlled kickback which opens when the forward flow from the on-line Pump is insufficient to meet its minimum flow requirements.

### 5.7.3 Instrumentation and Control

The delivery flow to D5-601 control scheme for the Reflux Pumps is described in Section 5.1.3.2. Pump delivery is at a rate determined by the DH Column Profile Controller GICA-06115. The control valve in the reflux line to the top of the Column, FCV-06226, has a minimum stop for pump protection during normal operation.

During plant de-burdening, entrainer is pumped to off-plot storage at a rate limited by FO-06214.

Each pump has local delivery pressure indication PI-06219/06221 and pump motor amps indication II-06220/06222. The delivery temperature is displayed in the DCS by TIA-06223.

The Reflux Pumps are protected against cavitation due to excessive turndown by a flow-controlled kickback. In the event of the forward flow measured by FT-06226 falling below the minimum flow required for Pump protection, FY-06225 is designed to start opening FCV-06225 and keep opening it in proportion to the reduction of forward flow until, when the forward flow is zero, FCV-06225 will be sufficiently far open to pass a flow slightly in excess of the minimum required. This ensures that the minimum flow through the pump is maintained at all times.

The pumps have an auto-start initiated by low delivery pressure at PT-06224.

**Complex Loop FY-06225: G5-615A/B Kickback to F5-609**

Pumps G5-615A/B must maintain a minimum flow rate for pump protection. FY-06225 in conjunction with FT-06226 maintains this flow by setting the kickback flow through FCV-06225.

In the event of the forward flow measured by FT-06226 falling below the minimum flow required for pump protection FY-06225 is designed to start opening FCV-06225 and keep opening it in proportion to the reduction of forward flow until when the forward flow is zero FCV-06225 will be sufficiently far open to pass a flow slightly in excess of the minimum required. This ensures that the minimum flow through the pump is maintained at all times.

If the kickback valve remains closed when the forward flow has fallen below the minimum required an alarm, GIA-06225 is initiated after a short delay to allow for valve travel.

***Loop Structure and Controller Details***
*FY-06225*
Positions valve FCV-06225 according to the profile shown in the table below.

| FT-06226 | FCV-06225 |
|---|---|
| >Minimum | 0% |
| Minimum | 0% |
| Zero | 100% |

The profile must be specified, taking into account the kickback valve characteristic, such that the kickback flow increases approximately linearly as the forward flow decreases so that the total flow through the pump never falls below the minimum allowed.

The operator will have access to auto/manual switching on HS-06225.
The operator will not be able to alter the pre-determined value at which the valve FCV-06225 begins to open.

### 5.7.4 Trips and Alarms

There are two trips relevant to the operation of the Reflux Pumps:

#### 5.7.4.1 Z-140-29: ROSOV Fire Protection

Pumps G5-615A/B handle flammable liquid and are at particular risk of serious fire due to the large inventory of flammable liquid that could escape through a failed pump seal. The logic comprises of remotely operated shut-off valve (ROSOV) XSV-06218 mounted on F5-609 bottoms exit route to G5-615A/B suction, the related instrumentation of which is explained below.

The P&ID legend drawing 10005-G41-GPZ105-0003 shows generic ROSOV arrangements for both actuated on/off valves and for control valves used as ROSOVs. The tag numbers in this description refer to those in the generic arrangements; actual tag numbers for each system are listed in section below.

Each protection system typically closes the relevant ROSOV(s) and shuts the pump(s) down if a fire is detected; the operator can also isolate the system from a safe distance using a manual push button.

| ROSOV tag | Pump tag | XSV-X | GSS-X (ROSOV) | PT-X | HS-X | HSS-X (C/R) | HSS-X (Field) | XA-X |
|---|---|---|---|---|---|---|---|---|
| Z-140-29 | G5-615A/B via Z-190 | XSV-06218 | GS-06218 | PT-06218 | HS-06218 | HSS-06218 | HSS-06218 | XA-06218 |

Description of operation (for tag number refer table above)

Initiators

- PT-29256 Low low pressure of instrument air header to PTA 5 ISBL
- GSS-X Limit switch signaling emergency isolation valve (ROSOV) XSV-X closed
- PT-X Low pressure in the instrument air line to isolation valve XSV-X
- HSS-X (C/R) A Hard-wired switch on fire-alarm panel in control room
- HSS-X (Field) Remote-mounted, manual emergency close button
- HS-X DCS hand switch to close XSV-X

Outputs

- XSV-X Close upstream emergency isolation valve (ROSOV), via manually reset solenoid
- XA-X Alarm in plant fire-alarm system
- Stops pumps G5-615 A/B
- Set FICA-06119 to manual and 0% output (one shot action)
- Inhibit pump auto-start I-139C

Logic of Operation

- The instrument air supply to each ROSOV (XSV-X) includes two fusible sections (UV ray resistant flexible nylon tubing which typically melts between 170 °C and 260°C), one of which is passed over and clamped in position around each pump seal, the other supported over the gland of the ROSOV (XSV-X). Either fusible section is the weak part of the loop which melts when exposed to fire, venting the instrument air supply and depressurizing ROSOV actuator. Being actuated on/off valve, the ROSOV closes in such scenario and this isolates the source of flammable DH solvent from the fire.
- A pressure switch, PT-X is fitted to the instrument airline to each ROSOV close to the valve. When it detects a loss of air pressure, it sends a signal to the fire alarm system, which closes ROSOV XSV-X and trips the associated pump(s) and inhibits the auto-start system (I-139C) to prevent the standby pump, G5-615A/B from kicking into service on sensing low common discharge pressure. Also, FICA-06119 is set to manual with 0% output in an one shot action.

ROSOV, if the fusible section melts, thus automatically closes; either directly or via the trip as narrated above.

- The fire alarm system also receives a signal from instrument air header PT-29256, at the source of instrument air to the affected section of the plant, to inhibit multiple alarms that would otherwise result from a loss of air pressure across the whole plant. An orifice restrictor (FO) is fitted to the air supply to the ROSOV, to provide sufficient pressure drop when the tubing melts to ensure that the pressure switch operates.
- The ROSOVs are fitted with closed limit switch, GSS-X which stops the associated downstream pump(s) if the ROSOV closes.
- An emergency shutdown button, HSS-X (C/R), is installed in the control room, close to the fire alarm system, to allow this complete ROSOV system to be isolated manually.
- A second button, HSS-X (Field), is installed at a safe distance away from ROSOV(approx. 10-15 meters away from the potential leak source, preferably on the likely escape route) with the same function as HSS-X (C/R).
- DCS hand switch HS-X is provided to close ROSOV (via a solenoid valve) and trip the associated pump(s) from DCS.
- Each ROSOV valve is provided with a lockable, manual, 3-way valve on the instrument air supply line for maintenance isolation.
- A bypass facility is provided to allow on-line testing of PT-X. PT-29256 does not require a bypass facility; it may be tested during normal operation.

#### 5.7.4.2 I-139C

G5-615 A/B are provided with auto start facility to bring in the standby pump when the combined discharge line pressure drops below a set pressure due to snag developed in the in-line pump or the in-line pump trips. Combined discharge line pressure is monitored on PT-06224 which is used for the auto start logic I-139C.

Following alarms are associated with auto start logic which appear on DCS :

YA-06235 : Auto start unavailable : If either motor is electrically unavailable e.g. the stop button is latched in or the motor is de-energised at substation

YA-06236 : Both motors running : If both motors are running as a result of auto start action or manual operation

YA-06237 : Both motors stopped : If both motors are stopped. The alarm is delayed for 5 secs (adjustable 0-10 secs) to allow the standby pump to auto start when low pressure is detected.

PT-06224 : Low discharge pressure

Auto start logic is inhibited when the related ROSOV system (Z-140-29) gets activated (refer section 5.7.4.1 for details).

#### 5.7.4.3 Alarms

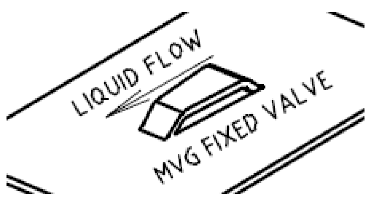
To warn against undesirable and/or unacceptable operating conditions, a high reflux temperature alarm is provided by TIA-06223. To warn of a low delivery flow from G5-615A/B which could damage the pumps, a low flow alarm is provided by FICA-06226.

The kickback line flow control valve FCV-06225 is fitted with a limit switch, GS-06225. An alarm will sound if the kickback FCV is closed and at the same time a low flow is detected on FT-06225 reflux delivery flow to D5-601.

## 5.8 Purge Column, D5-651 (Vendor: Hantech)

### 5.8.1 General Information

The Purge column D5-651 is a vertical, trayed column, constructed from 2205 Duplex stainless steel, having design conditions of 225°C and 9.5 barg/FV. The Column has 25 single pass MVG fixed valve trays supplied by Sulzer. In this type of valve tray, vapour is released through punched rectangular slots parallel to tray surface and at 90° with the liquid flow.




There are the following nozzles on the column :-

- DH Column purge / Oxidation Waste Water (above the trays)
- Vapour inlet from D5-402 Crystalliser
- Temperature transmitters (6 off)
- Level transmitter
- Level switch
- Temperature transmitter
- Pressure gauge
- Vapour outlet at top
- Liquid out at bottom

3 manways are also provided in the side wall for inspection purposes.

Additional equipment details are as provided under:

- Gross capacity: 11.372 $m^3$
- Fluid handled: Water + Acetic Acid + n-PA + PX and minor organics
- Operating Pressure (top/bottom): 1.2/1.5 bara
- Operating Temperature (top/bottom): 93/109°C
- Insulation Type: Heat conservation type of thickness 115 mm
- Shell inner diameter: 1200 mm
- Column Height: 10450 mm (from BTL to TTL)

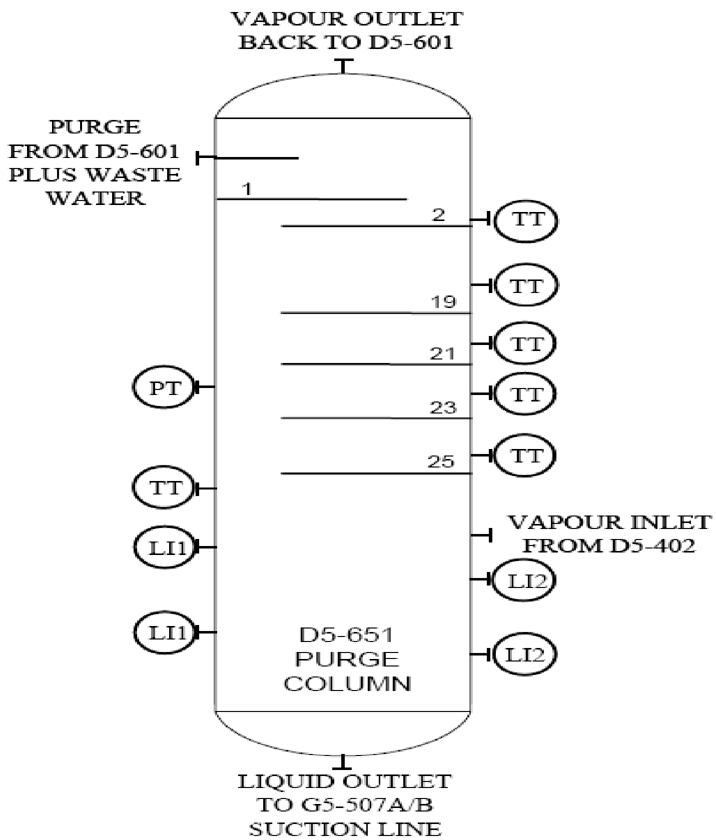



***Figure 5.8.1 – Purge Column***

### 5.8.2 Operating Philosophy

The Purge Column D5-651 removes entrainer from the paraxylene purge of the Solvent Dehydration Column D5-601 and returns the recovered entrainer to the Solvent Dehydration Column, thereby reducing the potential losses of entrainer from the paraxylene purge when it is recycled to the Oxidation Reactor. However, as the concentration of paraxylene takes time to build up in the Solvent Dehydration Column, on start-up the Purge Column will not need to be commissioned until the Reactor has been online for a number of hours.

The purge from the Solvent Dehydration Column and a small flow of Oxidation waste water from Recovery Column D5-631 are added to the top tray. The entrainer is recovered and returned to the Solvent Dehydration Column as a vapour. Along with the recovered entrainer, a substantial portion of paraxylene fed into Purge Column through paraxylene purge stream also gets recycled through the purge column vapour stream. In the process, effectively approx. 15% of paraxylene fed to Purge Column through paraxylene purge stream is purged out through Purge Column bottom stream. The paraxylene-rich bottoms stream from the Purge Column gravity flows to the Mother Liquor Drum F5-506 under level control. The level control valve is located close to F5-506 at minimum distance to prevent two phase flow in the line due to flashing that happens across the LCV.

Boil-up in the Purge Column is provided by flash vapour from Second CTA Crystalliser D5-402, with the flow being controlled to maintain a constant temperature in the base of the Column.

The separation of paraxylene and entrainer in the presence of acetic acid requires the addition of water to break a paraxylene/entrainer azeotrope and, in order to control separation, the temperature profile of the Column must be maintained. At the point where the entrainer composition falls quickly, the Column temperature rises sharply. The position of the sharp temperature change is therefore controlled, rather than a single temperature point in standard columns, through manipulation of the waste water flow.

In event of a plant upset or Reactor hold, the vapour for boil-up from the Second CTA Crystalliser will be lost and the Column will dump. After such an event the Purge Column will be taken off-line until steady operation of the Oxidation plant is restored and the column can then be re-commissioned.

An LP Steam connection is provided to D5-651 for “steaming out” at shutdown and for purging the Column during a Reactor hold. The LP Steam flow is restricted by flow orifice FO-06134.

In event of problems with the Purge Column, it is possible to purge from the Solvent Dehydration Column directly to the Solvent Dehydration Column base discharge with the flow measured by local flowmeter FI-06402. However this mode of operation should not be required as it is possible to operate for short periods with no purge because it takes time for the paraxylene concentration in the Solvent Dehydration Column to build up.

### 5.8.3 Instrumentation and Control

Temperature transmitters (TT-06126/27/28/29/30) are provided so that the Purge Column temperature profile and ultimately the position of the nPA “front” can be monitored. These are indicated in the DCS as TI-06126/27/28/29/30. An additional temperature transmitter, TT-06125 is provided for further temperature monitoring, which shows in DCS as TI-06125.

The Solvent DH Column purge flow to the Purge Column is measured by FT-06123 and controlled by FIC-06123 and the waste water flow from the Recovery Column is measured by FT-06122 and controlled by FICA-06122. In order to provide sufficient water to the Purge Column to maintain an effective azeotrope, the waste water flow is fed *pro rata* with the purge flow from the DH Column via ratio controller FFY-06122, which takes an input from purge flowmeter FT-06123.

When the Column is initially commissioned, FICA-06122 will be put on auto rather than ratio control, and the flow of water adjusted independently based on Column bottoms analysis (An601/2). Once the relationship between temperature profiles and Column bottoms composition has been established, FFY-06122 will be allowed to automatically adjust the set point of FICA-06122.

The liquid level in the base of the Purge Column is measured by LT-06135 and controlled automatically by LICA-06135, with excess liquid being discharged to the Mother Liquor Drum. LCV-06135 is located close to the Mother Liquor Drum to prevent flashing in the line.

TICA-06130 in the base of the Purge Column provides an input to FICA-06133 to control the flash vapour flow from the Second CTA Crystalliser and maintain the correct Column profile. However, in the event of falling pressure in the Second Crystalliser, low select relay FY-06133 will start to close FCV-06133 to maintain the Crystalliser pressure at the expense of the Purge Column temperature.

A local pressure indication, PI-06124 is provided for measurement of Purge Column pressure. A level switch LS-06137 is provided which gives an input to I-136 on detection of high high level in Purge Column bottom.

### 5.8.4 Trips and Alarms

#### 5.8.4.1 I-136

This protects the Purge Column from tray damage. On high high level in the base of the Purge Column, level switch LS-06137 initiates interlock I-136 which closes FCV-06133, the Second CTA Crystalliser flash vapour flow to the Purge Column, to prevent tray damage. Once the level in the Purge Column sump reduces and the level switch is reset the flash steam flow can be recommissioned.

#### 5.8.4.2 Z-111

Z-111, the D5-301 Reactor trip system, controls the tripping of the output to FICA-06123 in the event of a Reactor trip as follows:-

FICA-06123 – controls the flow of vapour from D5-601 to D5-651 Purge Column.
On a Reactor trip, the set-point on FICA-06123 is set to manual mode with 0% output.

#### 5.8.4.3 Alarms

The following alarms are provided to warn against undesirable and/or unacceptable operating conditions:-

FICA-06122 High water flow to the Purge Column
TICA-06130 High/Low temperature in the base of the Purge Column

FICA-06133 Low vapour flow from D5-402 to the Purge Column
LICA-06135 High/Low level in the base of the Purge Column

## 6. Additional Pressure Relief Systems

## 6.1 RST/D5-601 Dehydration Column System Pressure Relief Valves

D5-601 is used to separate water from acetic acid. The liquid feeds are Water Draw-off from HP Absorber D5-310 and First Crystalliser overheads condensate from F5-432. There are vapour feeds from Second Crystalliser D5-402 and Solvent Stripper D5-511. DH Column Reboiler E5-602 and DH Column Condenser E5-608 provide reboil and reflux for D5-601. A sidestream to remove paraxylene passes to Purge Column D5-651 which recovers the n-PA.

The relief system discussed provides pressure relief for the following plant equipment:

| | |
|---|---|
| D5-601 | Solvent Dehydration Column |
| E5-602 | DH Column Reboiler (tubeside) |
| D5-651 | Purge Column |

Vapour relief is provided by RV/D5-601A/B/C which discharge via the Relief Header. The back pressure from the relief header is significant and requires the use of a balanced bellows valve.

Overpressure/underpressure protection is to restrict pressure attributed to the following causes:-

1. SYSTEM BLOCKED IN

A. External Fire and full flame envelopment up to a height of 15m. Heat transfer based on the total wetted surface areas of the protected equipment up to HLL and associated process piping inside the isolations. Area has adequate drainage as specified by API and prompt fire fighting response available.

2. RESTRICTED OUTLETS

B. Process Abnormality

i) Failure of the condensing system or reflux flow to D5-601.

ii) Breakthrough of offgas through the Water Draw Off line from HP Absorber D5-310 to D5-601 through FO-03545. The flow through this orifice is calculated assuming an upstream maximum operating pressure of 14.5 barg and downstream system at relieving pressure.

iii) Backflow due to failure of HP Solvent Pump G5-607A/B with following cases.

(a) Restricted outlet with backflow of offgas from D5-310 at 44°C through fully open FCV-03505. The flow through the valve is calculated assuming an upstream maximum operating pressure of 14.5 barg and downstream system at relieving pressure.

(b) Restricted outlet with backflow of flashing slurry from D5-301 at 200°C and maximum operating pressure of 16 barg through twin 4" NRVs (dissimilar types) in line SV-30262, and downstream system at relieving pressure. It is assumed that the NRVs behave as a sharp-edged orifice with an area equivalent to 10% of the pipe cross-sectional area, assuming "frozen flow" i.e. no vaporisation of liquid across the NRVs.

(c) Restricted outlet with backflow of flashing slurry from D5-401 at 189°C and maximum operating pressure of 14.5 barg through twin 2" NRVs (dissimilar types) in line SV-40138, and downstream system at relieving pressure. It is assumed that the NRVs behave as a sharp-edged orifice with an area equivalent to 10% of the pipe cross-sectional area, assuming "frozen flow" i.e. no vaporisation of liquid across the NRVs.

iv) Maximum flow of nitrogen / inert gas through purge line CG-60202 via PCV-06201A and PCV-06201B (both fully open). Note that the selected Cv for both valves is no more than 20% greater than that required for the stated flow. The flow of nitrogen / inert gas through PCV-06201A and PCV-06201B is calculated assuming upstream design pressures of 10.3 barg and 20.9 barg respectively, and downstream system at relieving pressure. Maximum flow through RV/D5-601A/B/C combined is assumed to be 50% of the total flow through the valves. Relief for the remaining 50% is provided by RV/D5-631.

v) Breakthrough of vapour from First CTA Crystalliser Vent Separator F5-432 via fully open valve LCV-04309. The flow through the valve is calculated assuming an upstream maximum operating pressure of 14.5 barg and downstream system at relieving pressure.

vi) Breakthrough of offgas through the Acid Liquor line from HP Absorber D5-310 to D5-601 through LCV-03511. The flow through this valve is calculated assuming an upstream maximum operating pressure of 14.5 barg and downstream system at relieving pressure.

vii) Failure of G5-311A/B and backflow of flashing slurry from D5-301 at 200°C and maximum operating pressure of 16 barg through twin 6" NRVs (dissimilar types) in line SV-30104, and downstream system at relieving pressure. It is assumed that the NRVs behave as a sharp-edged orifice with an area equivalent to 10% of the pipe cross-sectional area, assuming "frozen flow" i.e. no vaporisation of liquid across the NRVs.

C. EQUIPMENT FAILURE

Power failure causing loss of G5-607A/B and G5-311A/B, see Cases 2B(iii) and 2B(vii). It is not considered necessary to take the full flowrate from these cases simultaneously.

- In Case 2B(vii) loss of G5-311A/B initiates Z-113 which closes FCV-03102. The flow enters D5-601 via LCV-03511 which will be closed by Z-196 on high pressure.
- Interlock I-169 which closes control valves and additional NRVs should reduce the flowrates for Case 2B(iii).
- RV/D5-631 A/B are available to provide additional capacity if required.

Inflow from D5-402 at relief valve set pressure with PCV-04173 fully open is disregarded. The flow could not be sustained and the volume of D5-601 is much higher than the normal vapour volume in D5-402. The maximum expected flow is less than GCV-06208 minimum stop flow. As E5-608 is assumed to continue operating in this case the relief requirement due to this event is insignificant. RV/D5-631 A/B prevent E5-608 becoming vapour blanketed and protect E5-608, F5-608, F5-609 from overpressure along with D5-631 and E5-631. They are set at 3.5 barg and covered in detail in Operation Manual of "Entrainer Recovery".

Liquid relief is disregarded as it is non-governing by inspection.

Additional details are as under:

- Safety valves set pressure: 3.5 barg
- Number off: 3

| Case | Req. Relief Rate(kg/h) | Relieving Temperature(°C) | Phase while relieving |
|---|---|---|---|
| 1A | 46037 | 172 | Vapor |
| 2B.i | 82481 | 160 | Vapor |
| 2B.ii | 32092 | 44 | Vapor |
| 2B.iii.a | 47800 | 44 | Vapor |
| 2B.iii.b | 15619 | 166 | Vapor |
| 2B.iii.c | 2520 | 164 | Vapor |
| 2B.iv | 55350 | 40 | Vapor |
| 2B.v | 9285 | 143 | Vapor |
| 2B.vi | 16508 | 44 | Vapor |
| 2B.vii | 31260 | 166 | Vapor |
| 2B.iii.a+b+c+ D5-402 flow | 148419 | 116 | Vapor |
| 2B.iv + D5-402 Flow | 137831 | 129 | Vapor |

## 6.2 RST/60428 Solvent Bottoms Cooler Process Side Bursting Disc and Pressure Relief Valves

Solvent Bottoms Cooler E5-603 cools the flow of solvent from the base of Solvent Dehydration Column D5-601 to the DH Solvent Pump G5-606A/B and the HP Solvent Pump G5-607A/B.

The relief system discussed here provides pressure relief for the following plant equipment:

E5-603 Solvent Bottoms Cooler (process side)
G5-606A/B DH Solvent Pump

Relief is provided through bursting discs BD/60428A/B and associated relief valves RV/60428A/B which discharge to atmosphere via Relief Header R-80202 and Relief Scrubber D5-840. The back pressure from the relief valve header is significant and will require the use of balanced bellows valves.

Overpressure/underpressure protection is to restrict pressure attributed to the following causes:-

1. SYSTEM BLOCKED IN

A. External fire. It is assumed that E5-603 tubeside wetted surface area and associated pipework up to a height of 15m does not exceed 74 $m^2$. It is assumed that the vessel is in an adequately well-drained area as defined by API.

C. Equipment /Services Failure. Tube rupture and breakthrough of cooling water at its design pressure. Broken tube assumed to be equivalent to two orifices equal to the tube ID of 15.75 mm, limiting the flow to no more than 21.073 te/h.

2. RESTRICTED OUTLETS

A. External Fire

i) 1) Restricted outlet with maximum backflow of DH solvent at 40°C and RV/D5-310 relieving pressure (at fire conditions) through fully open FCV-03505. It is assumed that valve FCV-03505 can pass a maximum of 683.2 te/h of DH solvent at 40°C with an upstream pressure of 20.46 barg and a downstream pressure of 4.675 barg.

i)2) Restricted outlet with maximum backflow of offgas at 215°C and RV/D5-310 relieving pressure through fully open FCV-03505. It is assumed that valve FCV-03505 can pass a maximum of 50.352 te/h of offgas at 215°C with an upstream pressure of 20.46 barg and a downstream pressure of 4.675 barg.

B. Process Abnormality

i) Backflow due to failure of HP Solvent Pump G5-607A/B.

a)1) Restricted outlet with maximum backflow of DH solvent at 40°C and D5-310 maximum operating pressure through fully open FCV-03505. It is assumed that valve FCV-03505 can pass a maximum of 538.811 te/h of DH solvent at 40°C with an upstream pressure of 14.5 barg and a downstream pressure of 4.675 barg

a)2) Restricted outlet with maximum backflow of offgas at 44°C and D5-310 maximum operating pressure through fully open FCV-03505. It is assumed that valve FCV-03505 can pass a maximum of 45.321 te/h of offgas at 44°C with an upstream pressure of 14.5 barg and a downstream pressure of 4.675 barg

b) Restricted outlet with maximum backflow of flashing slurry at 200°C and D5-301 maximum operating pressure through twin 4” NRV’s (dissimilar types) in line SV-30262 and twin 6” NRV’s (dissimilar types) in line SV-60430. It is assumed that the NRV’s behave as a sharp-edged orifice with an area equivalent to 10% of the pipe cross-sectional area, and can pass a maximum of 92.415 te/h of flashing slurry at 200°C, assuming “frozen flow” i.e. no vaporisation of liquid across the NRV’s, with an upstream pressure of 17 barg and a downstream pressure of 4.675 barg

c) Restricted outlet with maximum backflow of flashing slurry at 193°C and D5-401 maximum operating pressure through twin 2” NRV’s (dissimilar types) in line SV-40138 and twin 6” NRV’s (dissimilar types) in line SV-60430. It is assumed that the NRV’s behave as a sharp-edged orifice with an area equivalent to 10% of the pipe cross-sectional area, and can pass a maximum of 23.901 te/h of flashing slurry at 193°C, assuming “frozen flow” i.e. no vaporisation of liquid across the NRV’s, with an upstream pressure of 15.5 barg and a downstream pressure of 4.675 barg.

Additional details are as provided under:

- Bursting Disc BD/60428A Set Pressure: 5.6 barg
- Bursting Disc BD/60428B Set Pressure: 5.85 barg
- Relief Valve RV/60428A Set Pressure: 4.0 barg
- Relief Valve RV/60428B Set Pressure: 4.25 barg

The difference between respective BD and RV setting comes from the hydraulic head as the BDs are located at grade level whereas the RVs are located at much higher elevation to facilitate gravity draining of RV discharge (mix of vapour and liquid) toward relief header.

| **Case** | **Req. Relief Rate(kg/h)** | **Phase while relieving** |
|---|---|---|
| 1A | 25300 | Vapor |
| 2A(i)2 | 50352 | Vapor |
| 2B(i)a2 | 45321 | Vapor |
| 1C | 21073 | Liquid |
| 2A(i)1 | 683204 | Liquid |
| 2B(i)a1 | 538811 | Liquid |
| 2B(i)b | 93415 | Vapor + Liquid |
| 2B(i)c | 23910 | Vapor + Liquid |
| 2B(i)a2 + 2B(i)b + 2B(i)c | 142310 | Vapor + Liquid |

## 7. Safety Aspects

### 7.1 Flammability Hazards

The entrainer (normal propyl acetate) and methyl acetate contained within the Solvent Dehydration area are well above their flash points (10° and -9° respectively) and some flashing will occur if liquid is released to atmosphere. To minimise the loss of containment in the event of a fire or similar, ROSOVs are installed on the lines from the base of the DH Column and DH Column Decanter. In addition, the bases of the support structures are protected by an automatic water deluge system.

The acetic acid solvent contained in the Oxidation purge and Mother Liquor treatment section of solvent recovery is well above its flash point of 40°C, meaning there is a risk of a pool fire resulting from a major spillage/leak of acetic acid. However, in practice there is no record of a major pool fire ever having occurred on this type of plant.

It is good operating practice to ensure that any loss of containment is minimised as this impacts upon both the environmental and variable cost performance of the plant. In the event of minor losses or leaks occurring then an adequate water supply should be available around the base of the structures to enable any spillages/drainings to be washed away into the drain gullies.
The solvent dehydration system is protected against over-pressure by relief valves. All relief valves that discharge toxic material are vented to the Relief Header, which discharges to the Relief Scrubber.

In addition to the relief system, the DH system has a protective inert gas system to prevent the ingress of air in the event of underpressure. This protection is not required for vessel protection as the system is designed for full vacuum, but to prevent the formation of a flammable atmosphere inside the DH system.

### 7.2 Hazardous Area Classification

The following areas described are classified as Zone 2(II A, T2).

a) Full area covering solvent dehydration section equipment + 16 m radius (upto 8 m of elevation)
b) Full area covering solvent dehydration section equipment + 8 m radius (above 8 m of elevation till 8 m above the top of respective equipment)

### 7.3 Deluge and Fume Suppression Systems

Following types of fire water deluge and fume suppression systems are in place –

- Deluge rings around vessels to cool the structure / supports : Following vessels are provided with such arrangement
    - D5-601
    - E5-602
    - F5-609

  D5-601 is provided with multi-layer fire water deluge rings (3 spray rings outside vessel skirt and 2 spray rings inside vessel skirt) at different elevations. The rings outside the skirt have 12 spray nozzles each while the rings inside the skirt have 9 spray nozzles each.

  E5-602 and F5-609 are each provided with a single fire water deluge ring.

  The fire water sprays discussed above are used to quench equipment supports should there be any fire and prevent mechanical damage (buckling / collapse) of the equipment due to heat and flame impingement. The deluge system is controlled through a deluge station. D5-601 and E5-

602 have a common deluge control station whereas F5-609 has a separate one shared with D5-631, D5-511 and F5-516. The deluge system operates automatically based on quartzoid bulb thermal detectors placed around the respective equipment. These are all connected to an instrument air header which also supplies instrument air to the deluge valve. When any of thermal detectors melt under heat (should there be a fire), it depressurizes the instrument air header and opens the deluge valve. Deluge valve can also be operated remotely from F&G panel located in central control room. Following alarms are triggered on F&G panel on activation of the deluge system to warn the operator –

- Low instrument air header pressure alarm of that particular deluge system
- High deluge water pressure alarm (measured downstream of the deluge valve)

- Water curtain : Provided around D5-601 base in two layers covering upto 11 m elevation. Each ring is fitted with multiple jumbo water curtain nozzles. This ring serves the purpose of fume suppression in case of an accidental release of hydrocarbon vapor by creating a water curtain around which scrubs down acetic acid vapour.

- Fume suppression sprays : it is provided for following pumps primarily to suppress the fumes should there be a severe failure of mechanical seal leading to large spillage of acetic acid. The deluge is operated manually through a motor operated valve from the deluge station.
  - G5-606A
  - G5-606B
  - G5-607A
  - G5-607B

The fume suppression deluge system of G5-606 A/B and G5-607 A/B are controlled from a common deluge control station which is also shared with G5-512 and G5-517.

## 7.4 Fire and Gas Detection System

A flame detector is provided in the vicinity of DH Solvent Pumps.

Acetic Acid vapor detectors are provided near each of the DH Solvent Pumps (G5-606A/B), HP Solvent Pumps (G5-607A/B), DH Column Reboiler (E5-602) and Purge Column (D5-651).

Propyl Acetate vapor detectors are provided near each of the Reflux Pumps (G5-615A/B) and Purge Column (D5-651).

Several manual alarm call points are provided at different elevations the details of which can be seen from the reference P&IDs mentioned earlier.

Multi-tone sounders are provided to alert personnel about various situations.

## 7.5 Fire Hazard Protection (FHP) insulation

Following vessels located in Solvent Recovery section that have large inventories of hot acetic acid and n-propyl acetate and have a potential to get engulfed in flame due to their proximity to grade should there be an incident of pool fire, are provided with fire resistant (FHP) insulation.

- D5-601
- E5-602
- F5-608
- F5-609

Typically FHP insulation comprises of fire resistant mineral fibre slabs for operating temperatures above 150°C and cellular glass slabs for operating temperatures of 150°C and less. The design and application of the insulation system is such that it does not dislodge by fire hose stream impingement. There is stainless steel type 304 (ASTM A240) weatherproof cladding 0.8 mm thick over the insulation.

## 7.6 Secondary containment

A kerbed area is provided around G5-615 A/B pumps to contain any Entrainer should there be a spillage and prevent its escape into the open effluent drainage system of the plant. With this kerbing, any spilled material is directed to an oil-water separator (multi chamber underflow / overflow arrangement) where Entrainer, being lighter than water, is separated through decantation. Separated Entrainer which floats on top is removed from the oil / water separator manually using a mobile dewatering pump. Effluent from the oil-water separator is routed through an underground sealed drain to Oxidation Effluent Pit, A5-856. The intent of having this arrangement is to prevent contamination of plant drainage system with Entrainer should there be any spillage as Entrainer being ligheter, will float on surface and these drains being open, pose a great risk of fire and if ignited, can trigger a fire ring across the plant.

As the entriner system comprises of multiple lines at different elevations and provided with drain points scattered around, it is not possible to have similar system like that around G5-615 A/B to contain any spilled Entrainer. Therefore it is absolutely essential to contain any drained material whenever drained from any of the drain points into drum / container.

## 8. Troubleshooting

### 8.1 Solvent Dehydration Column

| **Problem** | **Probable Causes** | **Actions to be carried out.** |
|---|---|---|
| D/H Column unstable.<br><br>- High Reboiler steam rate. | | 1. Check operating parameters against standard operating conditions<br>2. Check for high water ingress (high water content in Reactor, too much WDO etc).<br>3. Check overall DH Column loading - possible over cooling of overheads. Adjust CW to Condenser as required.<br>4. Look for high levels of Methyl acetate in entrainer - check operation of Recovery column - check Reactor conditions. |
| Base Water levels too high / low<br><br>- High water levels will cause problems with Reaction<br><br>- Low water levels can lead to corrosion of the column base | | 1. Check on-line analyser results against manual samples<br>2. Check Column base pressure is "normal" – changes in pressure alter the temperature / water concentration relationship.<br>3. Reset column base to automatic temperature control – Ensure Column base pressure is normal |
| High pressure | | 1. Check operating parameters against standard operating conditions<br>2. Is the base pressure and overheads pressure high -adjust steam flow and plant rate as required to prevent flooding, - check column differential pressure.<br>3. Check Base level.<br>4. Check DH Column loading eg high reboiler steam rates, high reflux rates.<br>5. Check for excessive Crystalliser flash vapour rate – high crystalliser PCV output. |

| | | |
|---|---|---|
| High temperature profile. | | 1. Check Column loading , eg high reboiler steam rates, high reflux rates - reduce load as required<br>2. Check for build-up of paraxylene - consider increasing paraxlene purge rate.<br>3. Check Crystalliser flash vapour control. – high crystalliser PCV output |
| High base level<br><br>- High levels in the base in normal operation can cause internal damage to packing<br><br>- After loss of LP Steam (e.g. boiler trip) column may dump | | 1. Check / calibrate instruments to confirm reading - pump out solvent to OSBL storage to reduce level.<br>2. Check Plant solvent inventory control.<br>3. Check base Pressure & Temperature - increase in base pressure also indicates higher level.<br>4. Consider reducing column loading (see above). |
| Decanter level unstable.<br><br>- High level (water side) | | Check for high Recovery column loading - this can restrict flow from Decanter into Recovery column.<br><br>Consider reducing Recovery column load. |
| High water in solvent | - base temperature too low | Increase reboiler steam flow by increasing bottom temperature setpoint |
| High PX in reflux | - D5-651 bottom temperature too high<br><br>- Water reflux flowrate of D5-651 too low<br><br>- PX in D5-651 feed stream is too low | Optimise bottom operating temperature setpoint for D5-651. Higher temperature gives low n-PA loss but low PX purge rate. Lower temperature gives high PX purge rate but high n-PA loss<br><br>1. Optimise water reflux flowrate. It should be approx 1/3rd of feed flow<br>2. Check D5-601 profile is correct<br><br>Ensure D5-601 collector which supplies feed to D5-651 is correctly installed. |
| High Methyl acetate in reflux | - Reflux temperature too low | Increase E5-608 bypass flow to increase vapor flowrate to D5-631 |

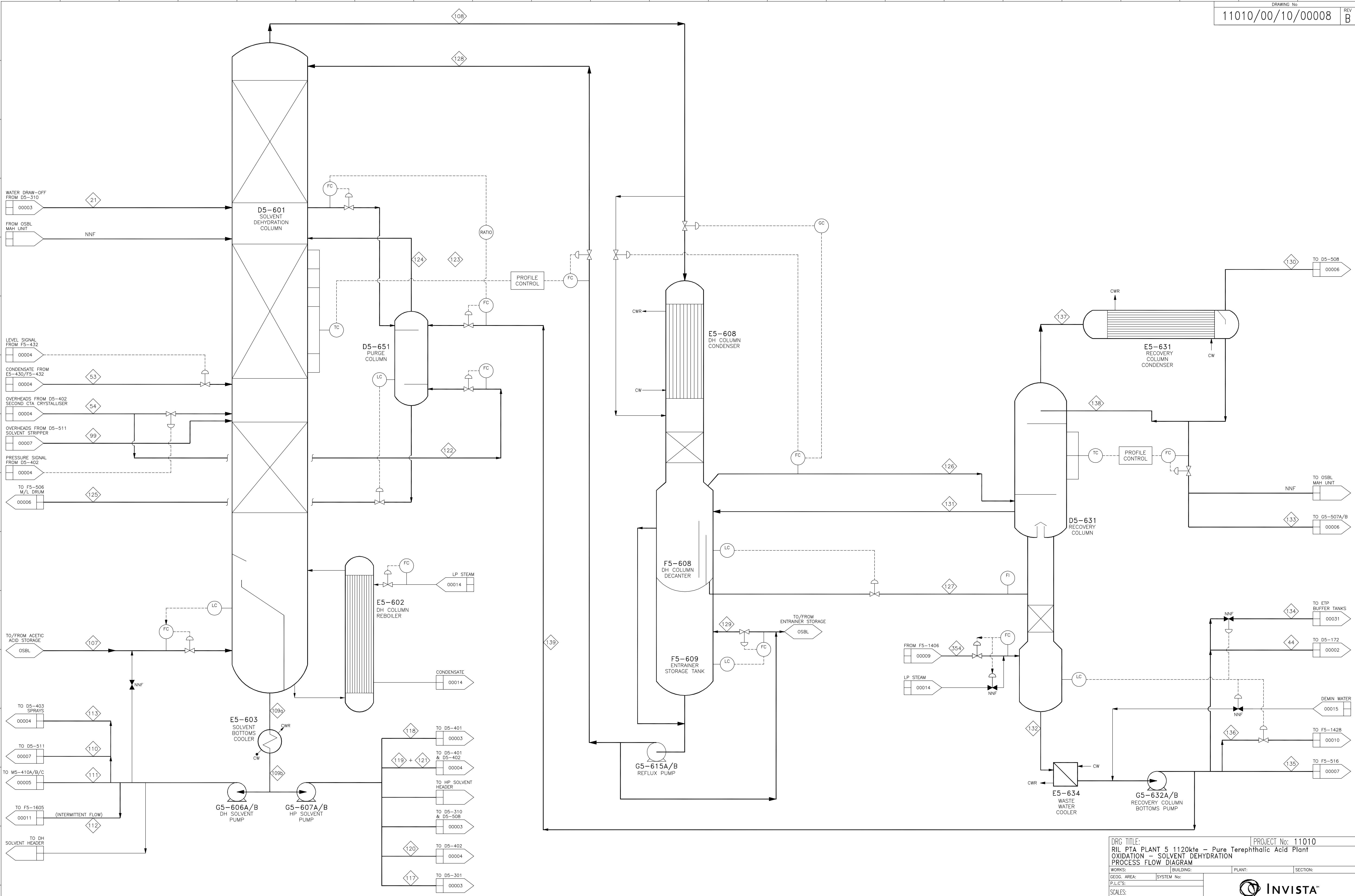
| | | |
|---|---|---|
| High water in reflux | - Decanter malfunction: temperature profile changes rapidly with reflux flowrate change | 1. Check setting of height of decanter overflow weir<br>2. Check if knitmesh pad in central down-comer is installed or blocked |
| High Acetic Acid in Waste Water | - D5-601 profile too high<br><br>- D5-601 profile unstable<br><br>- PX in reflux too high | Reduce profile controller setpoint - optimise profile setting<br><br>1. Eliminate possible column disturbances, e.g, from D5-511 or D5-401<br>2. Tune profile controller<br><br>See high PX in reflux above |
| High NPA usage | - High PX in reflux<br><br>- D5-601 profile too low, NPA slips into base<br><br>- D5-631 profile too high, n-PA slips into methyl acetate<br><br>- D5-651 bottom temperature too low<br><br>- WDO % water composition | See high PX in reflux<br><br>Correct D5-601 profile controller setpoint<br><br>Decrease D5-631 profile controller setpoint<br><br>Increase D5-651 bottom temperature controller setpoint<br><br>Check % water concentration in WDO is as per design – insufficient water will result in n-PA penetration further down the column |
| High propanol in reflux | - Hydrolysis of n-PA caused due to high AA in reflux<br><br>- Insufficient propanol draining | See high AA in waste water<br><br>Check water in reflux – normally high water = high propanol also. Check decanter performance.<br><br>1. Increase propanol purge from D5-631 chimney tray.<br>2. Reduce D5-631 bottom operating temperature until COD of waste water increases to around 5000-6000ppm. |
| | | |

| | | |
|---|---|---|
| High bromide in DH Solvent at base of D5-601<br><br>- Bromide can result in higher corrosion rates | | 1. Check for carryover from D5-511 or D5-402.<br><br>2. Analyse for Co/Mn to identify possible source<br><br>3. Increase DH solvent flow to D5-511 top tray |
| Unstable reboiler flow in thermosyphon loop | | Partly close the butterfly valve at inlet to E5-602 to stabilise flow |

### 8.2 Purge Column

| Problem | Probable Causes | Actions to be carried out. |
|---|---|---|
| High base level<br><br>- High levels in the base in normal operation can cause internal damage to internals<br><br>- After loss of D5-402 vapour column may dump | | Check / calibrate instruments to confirm reading<br><br>- pump out to reduce level. Will result in a loss of n-PA. |

# Annexure A: PFD and Stream Summary




| ISSUE | REVISION | GRID | DRN | APP | DATE | FILMED |
|---|---|---|---|---|---|---|
| P8 | FLAGS MODIFIED, MASS BALANCE INFO ADDED | A4,A13 H14,V13 U6,U7 | PR | | 22.12.10 | |
| P7 | REVISED | | GM | | 16.09.10 | |
| P6 | FLAG CORRECTED. | | APJL | | 06.09.10 | |
| P5 | REVISED FOLLOWING RIL TELECON. | | JO'B | | 02.08.10 | |
| P4 | REVISED TO REFLECT P7 DEVELOPMENT. | | JO'B | | 16.06.10 | |
| B | FEEP ISSUE. FLAGS CORRECTED. DIAMOND DELETED. ARROW ADDED. | A14.H14 S9 | BR | | 21.06.11 | |

NOTES:

DRG TITLE: RIL PTA PLANT 5 1120kte – Pure Terephthalic Acid Plant
OXIDATION – SOLVENT DEHYDRATION
PROCESS FLOW DIAGRAM

PROJECT No: 11010

WORKS: | BUILDING: | PLANT: | SECTION:

GEOG. AREA: | SYSTEM No:

P.L.C'S:

SCALES:

DRG TYPE: | DO LOC:

DRAWN BY: | DATE:

CHECKED: | DATE:

| APPROVED | SECTION | DATE |
|---|---|---|
| J.LIMBACH | PROCESS | 21.06.11 |
| A.GOODRUM | PROJECTS | 21.06.11 |

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DRG SIZE A0

DRAWING No 11010/00/10/00008 REV B

CONFIDENTIAL TO INVISTA

| Stream Number | 21 | 53 | 54 | 99 | 107 | 108 | 109a | 109b | 110 | 111 | 112 | 113 | 117 | 118 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Description** | WDO to DH Column | Combined 1st Crystalliser Vent Liquid | 2nd Crystalliser Vapor | Stripper Vapor | Acetic Acid Makeup | DH Column Overheads | DH Column Total Bottoms | DH Column Bottoms Exit E5-603 | DH Solvent to Stripper | DH Solvent Wash to Filters | Solvent to Pure ML Slurry via F5-1605 | 3rd Crystalliser Spray | HP Solvent Spray to Reactor | First Crystalliser Fresh Dilution Solvent |
| ACETIC ACID | 89063.1 | 34864.9 | 45754.8 | 51445 | 4455.4 | 115.1 | 219639 | 219639 | 9491.1 | 46506.5 | 2513.4 | 1898.2 | 4745.6 | 35185 |
| WATER | 42730.5 | 5690.6 | 6617.1 | 9034.9 | 6.2 | 72490.2 | 11676 | 11676 | 504.8 | 2473.3 | 133.7 | 101 | 252.4 | 1871.2 |
| OXYGEN | 6.9 | 4 | 74.1 | 0.1 | 0 | 85.6 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| NITROGEN | 109.7 | 48.2 | 1142.7 | 1.3 | 0 | 1307 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CARBON MONOXIDE | 0.5 | 0.4 | 9.4 | 0 | 0 | 10.3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CARBON DIOXIDE | 52.2 | 30.8 | 137 | 0.9 | 0 | 242.3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| HYDROGEN | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| PARAXYLENE | 287.9 | 0.1 | 0.1 | 0 | 0 | 438.5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| TA SOLIDS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CBA SOLID | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P- TOLUIC ACID | 0.2 | 1.1 | 0.8 | 0 | 0 | 0 | 2.1 | 2.1 | 0.1 | 0.4 | 0 | 0 | 0 | 0.3 |
| BENZOIC ACID | 0.1 | 22.2 | 17.6 | 0.2 | 0 | 0 | 38.1 | 38.1 | 1.6 | 8.1 | 0.4 | 0.3 | 0.8 | 6.1 |
| METHYL ACETATE | 9206.2 | 903.7 | 1230.7 | 274 | 0 | 66984.9 | 0.4 | 0.4 | 0 | 0.1 | 0 | 0 | 0 | 0.1 |
| PROPYL ACETATE | 2.2 | 1.6 | 2.4 | 5.5 | 0 | 337119 | 2.6 | 2.6 | 2.3 | 11.4 | 0.6 | 0.5 | 1.2 | 8.6 |
| COBALT | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| MANGANESE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| BROMIDE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYL BROMIDE | 0.5 | 0.1 | 0.5 | 0 | 0 | 3.1 | 0 | 0 | 0 | 0.2 | 0 | 0 | 0 | 0.1 |
| PROPANOL | 0 | 0 | 0 | 0 | 0 | 8429.9 | 1.3 | 1.3 | 0 | 0 | 0 | 0 | 0 | 0 |
| METHANOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 6 | 0 | 0 | 0 | 0 | 0 | 0 |
| BYPRODUCTS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| OXALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| SODIUM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CARBONATES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | | | | | | | | | | | | |
| TOTAL | 141460 | 41567.7 | 54987.1 | 60762 | 4461.6 | 487226 | 231359 | 231359 | 10000 | 49000 | 2648.1 | 2000 | 5000 | 37071.4 |
| MEAN MOL. WT. | 35.5 | 45.6 | 46.1 | 44.6 | 59.9 | 57.8 | 53.7 | 53.7 | 53.7 | 53.7 | 53.7 | 53.7 | 53.7 | 53.7 |
| TEMPERATURE | 86 | 129 | 160 | 117 | 25 | 85 | 120 | 95 | 95 | 95 | 95 | 95 | 95 | 95 |
| PRESSURE | 13.34 | 13.55 | 4.3 | 1.3 | 5 | 1.2 | 1.3 | 1.15 | 7.5 | 7.5 | 7.5 | 7.5 | 17.29 | 17.29 |
| DENSITY | 952.3 | 917.4 | 7.4 | 2.4 | 1042 | 2.4 | 936.6 | 965.9 | 965.9 | 965.9 | 965.9 | 965.9 | 965.9 | 965.9 |
| ENTHALPY | -397730 | -102140 | -119910 | -140350 | -10054 | -795520 | -531080 | -535110 | -23144 | -113410 | -6129 | -4629 | -11572 | -85798 |
| SOLIDS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| VISCOSITY | 0.0196/0.6525 | 0.0193/0.4449 | 0.0126/ | 0.0111/0.5016 | /1.1325 | 0.0090/ | /0.4575 | /0.5988 | /0.5988 | /0.5988 | /0.5988 | /0.5988 | /0.5988 | /0.5988 |

| Stream Number | 119 | 120 | 121 | 122 | 123 | 124 | 125 | 126 | 127 | 128 | 129 | 131 | 139 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Description** | First Crystalliser Spray | 2nd Crystalliser Dilution Solvent | Second Crystalliser Spray | 2nd Crystalliser Vapor to Purge Column | Paraxylene Purge | Purge Column Overheads | Purge Column Bottoms | Vapor from E5-608 | Decanter Water | Main organic reflux | Entrainer Make-up | Organics from D5-631 to F5-608 | Water to Purge Column |
| ACETIC ACID | 3796.5 | 3796.5 | 3796.5 | 5075.8 | 1176.1 | 332.6 | 5920.2 | 0.9 | 25.3 | 91.1 | 0 | 2.1 | 0.8 |
| WATER | 201.9 | 201.9 | 201.9 | 734.1 | 482.2 | 1245.8 | 1763.5 | 4136.2 | 56872.8 | 20057.1 | 0 | 8576.4 | 1792.9 |
| OXYGEN | 0 | 0 | 0 | 8.2 | 0 | 8.2 | 0 | 85.1 | 0 | 0.5 | 0 | 0 | 0 |
| NITROGEN | 0 | 0 | 0 | 126.8 | 0 | 126.7 | 0 | 1301.8 | 0.1 | 5.2 | 0 | 0.2 | 0 |
| CARBON MONOXIDE | 0 | 0 | 0 | 1 | 0 | 1 | 0 | 10.3 | 0 | 0 | 0 | 0 | 0 |
| CARBON DIOXIDE | 0 | 0 | 0 | 15.2 | 0 | 15.1 | 0.1 | 221.1 | 0.4 | 21.5 | 0 | 0.8 | 0 |
| HYDROGEN | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| PARAXYLENE | 0 | 0 | 0 | 0 | 1886.9 | 1598.7 | 288 | 11.1 | 0 | 438.4 | 0 | 11.1 | 0 |
| TA SOLIDS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CBA SOLID | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| P- TOLUIC ACID | 0 | 0 | 0 | 0.1 | 0 | 0 | 0.1 | 0 | 0 | 0 | 0 | 0 | 0 |
| BENZOIC ACID | 0.7 | 0.7 | 0.7 | 2 | 0 | 0 | 2 | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYL ACETATE | 0 | 0 | 0 | 136.5 | 58.1 | 156.1 | 38.5 | 11883.7 | 2268.2 | 55409.2 | 0 | 2571.9 | 0 |
| PROPYL ACETATE | 0.9 | 0.9 | 0.9 | 0.3 | 2395.5 | 2396 | 0.1 | 16993.7 | 1074.8 | 337158 | 70 | 18041 | 0 |
| COBALT | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| MANGANESE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| BROMIDE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| METHYL BROMIDE | 0 | 0 | 0 | 0.1 | 0 | 0.1 | 0 | 1.2 | 0 | 2 | 0 | 0.1 | 0 |
| PROPANOL | 0 | 0 | 0 | 0 | 1.2 | 1.4 | 0 | 553.6 | 500.4 | 8431 | 0 | 1042.8 | 0.3 |
| METHANOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 1 |
| BYPRODUCTS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| OXALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| SODIUM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| CARBONATES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | | | | | | | | | | | | | |
| TOTAL | 4000 | 4000 | 4000 | 6100 | 6000 | 5881.8 | 8012.6 | 35198.6 | 60742.1 | 421614 | 70 | 30246.5 | 1794 |
| MEAN MOL. WT. | 53.7 | 53.7 | 53.7 | 46.1 | 67.9 | 48.8 | 40.1 | 56.7 | 18.9 | 79.4 | 102.1 | 42.9 | 18 |
| TEMPERATURE | 95 | 95 | 95 | 160 | 94 | 93 | 109 | 78 | 78 | 79 | 25 | 79 | 90 |
| PRESSURE | 17.29 | 17.29 | 17.29 | 4.3 | 1.24 | 1.24 | 1.44 | 1.17 | 1.17 | 1.3 | 1.3 | 1.17 | 1.28 |
| DENSITY | 965.9 | 965.9 | 965.9 | 7.4 | 840 | 2.1 | 939.1 | 2.3 | 958.2 | 829.7 | 882.2 | 858.2 | 959.2 |
| ENTHALPY | -9258 | -9258 | -9258 | -13302 | -7962 | -8387 | -20651 | -55605 | -253130 | -635190 | -95 | -66769 | -7774 |
| SOLIDS | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| VISCOSITY | /0.5988 | /0.5988 | /0.5988 | 0.0126/ | / | 0.0095/ | 0.0108/0.5433 | 0.0094/ | /0.3669 | /0.4272 | /0.5542 | / | /0.3137 |

# Annexure B: Standard Operating Conditions

| Tag No | Fluid | State | min. | normal | max. | Units | Remarks | **Consequences of going above max or below min value** (only provided for parameters having impact on safety and equipment integrity) | **Protection provided** (only provided for parameters having impact on safety and equipment integrity) | **Corrective Actions** (only provided for parameters having impact on safety and equipment integrity) |
|---|---|---|---|---|---|---|---|---|---|---|
| PT-06117 | Acetic acid, water, trace components | Gas/Liquid | - | 0.3 | - | bar g | | High pressure may lead to overpressurization of column beyond design window | 1. High pressure alarm provided.<br>2. High high pressure trip (Z-196) provided which cuts off liquid feed streams to D5-601.<br>3. High high pressure trip (Z-191) based on overhead pressure (PT-06203) provided<br>4. Overpressure protection provided through relief valves (RV/D5-601A) | 1. Reduce solvent load to column before Z-196 gets triggered.<br>2. If required, reduce plant throughput to reduce column hydraulic load. |
| PT-06120 | LP steam | Vapour | 1.7 | 2.9 | 3.25 | bar g | | | | |
| PI-06124 | Acetic acid, water, paraxylene & traces | Gas/Liquid | - | 1.5 | - | bar g | | | | |
| PI-06131 | DH solvent | Liquid | - | 1.1 | 5.7 | bar g | | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| PT-06145 | LP steam | Vapour | | 3.5 | 5.5 | barg | | | | |
| PT-06201 | n-PA, water, methyl acetate + traces | Vapour | - | 0.2 | 3.5 | bar g | | High pressure may lead to overpressurization of column beyond design window | 1. High pressure alarm provided.<br>2. High high pressure trip (Z-191) based on overhead pressure (PT-06203) provided<br>4. Overpressure protection provided through relief valves (RV/D5-601A) | 1. Reduce solvent load to column before Z-196 gets triggered.<br>2. If required, reduce plant throughput to reduce column hydraulic load. |
| PT-06203 | n-PA, water, methyl acetate + traces | Vapour | - | 0.2 | 3.5 | bar g | | High pressure may lead to overpressurization of column beyond design window | 1. High pressure alarm provided on PT-06201.<br>2. High high pressure trip (Z-191) based provided on PT-06203)<br>4. Overpressure protection provided through relief valves (RV/D5-601A) | - |
| PI-06219 | n-PA, methyl acetate, water + traces | Liquid | - | 5.9 | 12 | bar g | | | | |
| PI-06221 | n-PA, methyl acetate, water + traces | Liquid | - | 5.9 | 12 | bar g | | | | |
| PT-06224 | n-PA, methyl acetate, water + traces | Liquid | - | 5.9 | 12 | bar g | | | | |
| PI-06240 | Cooling water | Liquid | - | 0.3 | 7 | bar g | | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| PI-06405 | DH Solvent | Liquid | - | 5.6 | 12 | bar g | | | | |
| PI-06407 | DH Solvent | Liquid | - | 5.6 | 12 | bar g | | | | |
| PI-06411 | HP Solvent | Liquid | - | 20.6 | 31 | bar g | | | | |
| PI-06413 | HP Solvent | Liquid | - | 20.6 | 31 | bar g | | | | |
| PT-06419 | DH solvent | Liquid | - | 0 | - | bar g | | | | |
| PT-06420 | HP Solvent | Liquid | - | 20.6 | 31 | bar g | | Low pressure may lead to reverse flow of hot slurry and solvent from D5-301 / D5-401 and may lead a overpressure relief case from D5-601 | 1. Low pressure auto start provided for standby pump G5-607 A/B<br>2. Interlock I-169 provided which prevents reverse flow in case both G5-607 A/B pump stops<br>3. Relief calculation of D5-601 has factored in reverse flow of hot slurry and solvent | - |
| PT-06432 | DH solvent | Liquid | - | 0 | - | bar g | | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| LT-06105 | DH Solvent | Liquid | 1000 | - | 3800 | mm | D5-601 Bottom tan line | Overfilling coupled with vapour slug flow from E5-602 may lead to damage to bottom packed bed | 1. Two independent level measurements.<br>2. High level alarm on both the LTs.<br>3. High high level trip (Z-195). | 1. Chcek fresh acetic acid make-up valve for passing.<br>2. Export out solvent from D5-601 sump back to Acetic Acid Storage Tank F5-2611 A/B in Tank Farm. |
| LT-06106 | DH Solvent | Liquid | 1000 | | 3800 | mm | D5-601 Bottom tan line | Overfilling coupled with vapour slug flow from E5-602 may lead to damage to bottom packed bed | 1. Two independent level measurements.<br>2. High level alarm on both the LTs.<br>3. High high level trip (Z-195). | 1. Chcek fresh acetic acid make-up valve for passing.<br>2. Export out solvent from D5-601 sump back to Acetic Acid Storage Tank F5-2611 A/B in Tank Farm. |
| LT-06121 | LP steam / condensate | Gas/Liquid | 400 | - | 4000 | mm | E5-602 Bottom tube sheet | | | |
| LT-06135 | Solvent | Liquid | 270 | 380 | 480 | mm | D5-651 Bottom tan line | Overfilling coupled with vapour / steam flow may lead to damage to bottom tray | 1. High level alarm<br>2. Interlock I-136 which closes vapour flow from D5-402 | 1. Check whether LCV-06135 is functioning / sticking<br>2. Reduce liquid feed to D5-651 from D5-601 |
| LT-06211 | n-PA, methyl acetate, water + traces | Liquid | - | 1900 | - | mm | F5-608 bottom tan line | | | |
| LT-06212 | Water + organic traces | Liquid | - | 1000 | - | mm | F5-608 bottom tan line | | | |

| | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| LT-06213 | n-PA, methyl acetate, water + traces | Liquid | 600 | - | 1600 | mm | F5-609 bottom tan line | | | | |
| LT-06215 | n-PA, methyl acetate, water + traces | Liquid | 600 | - | 1600 | mm | F5-609 bottom tan line | | | | |
| FT-06101 | Water + trace organics | Liquid | 3000 | - | 30000 | kg/h | | | | | |
| FT-06102 | Acetic Acid | Liquid | 800 | 4460 | 7000 | kg/h | | | | | |
| FT-06103 | Acetic Acid | Liquid | 0 | 150000 | 200000 | kg/h | | | | | |
| FT-06119 | LP steam | Vapour | 40000 | 110000 | 137000 | kg/h | | | | | |
| FT-06122 | Water + trace organics | Liquid | 750 | 1800 | 2300 | kg/h | | | | | |
| FT-06123 | Propyl acetate, paraxylene, acetic acid traces | Liquid | 2500 | 6000 | 7500 | kg/h | | | | | |
| FT-06133 | Acetic acid, water & inerts | Vapour | 3000 | 6100 | 7700 | kg/h | | | | | |
| FO-06134 | LP steam | Vapour | - | 200 | - | kg/h | | | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| FT-06136 | HP Solvent | Liquid | 7000 | - | 84000 | kg/h | | | | |
| FT-06142 | HP Solvent | Liquid | | 100000 | | kg/h | | | | |
| FO-06205 | Demin water | Liquid | - | 50000 | - | kg/h | | | | |
| FT-06206 | Cooling water | Liquid | 6000 | 7000 | 8760 | t/h | | | | |
| FT-06209 | n-PA, methyl acetate, water + traces | Vapour | 23000 | 35200 | 44000 | kg/h | | | | |
| FO-06214 | n-PA, methyl acetate, water + traces | Liquid | - | 50000 | - | kg/h | | | | |
| FT-06217 | Water + organic traces | Liquid | 15000 | 60800 | 76000 | kg/h | | | | |
| FT-06226 | n-PA, methyl acetate, water + traces | Liquid | 175 | 422 | 581 | t/h | | Low / loss of reflux flow may lead to overpressurization of column and eventually lead to release of acetic acid / n-PA vapours to relief scrubber | 1. Low flow alarm<br>2. Auto start of standby reflux pump<br><br>3. Minimum stop on valve set in DCS | 1. In case auto start of standby reflux pump fails immediately shutdown column by cutting steam to E5-602 and trip oxidation reactor. |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| FT-06228 | n-PA | Liquid | 20 | 70 | 200 | kg/h | | | | |
| FO-06230 | n-PA | Liquid | - | 50000 | - | kg/h | | | | |
| FO-06245 | Inert gas | Gas | - | 24 | - | m3/h | | | | |
| FI-06246 | Inert gas | Gas | | 24 | | m3/h | | | | |
| FT-06247 | n-PA | Liquid | | | 50000 | kg/h | | | | |
| FT-06401 | DH Solvent | Liquid | 36000 | 65000 | 116000 | kg/h | | Flow lower than minimum continuous flow requirement of G5-606 A/B may damage the pump | 1. Low flow alarm<br>2. Flow controlled spillback | - |
| FI-06402 | Propyl acetate, paraxylene, acetic acid traces | Liquid | - | 0 | 1400 | kg/h | | | | |
| FO-06410 | DH Solvent | Liquid | - | 100000 | - | kg/h | | | | |
| FT-06416 | HP Solvent | Liquid | 59000 | 167000 | 271000 | kg/h | | Flow lower than minimum continuous flow requirement of G5-607 A/B may damage the pump | 1. Low flow alarm<br>2. Flow controlled spillback | - |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| FO-06430 | Inert gas | Gas | | 44 | | m3/h | | | | |
| TT-06107 | Acetic acid, water, trace components | Gas/Liquid | 85 | - | 94 | DegC | | | | |
| TT-06108 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06109 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06110 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06111 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06112 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06113 | Acetic acid, water, trace components | Gas/Liquid | 96 | - | 111 | DegC | | | | |
| TT-06116 | Acetic acid, water, trace components | Gas/Liquid | 112 | - | 116 | DegC | | High temperature not commensurate to pressure corresponding to 4.5-5.5% water concentration may lead to corrosion of E5-602 tubes | 1. Pressure compensated temperature control. 2. High temperarure alarm. | 1. Cross check pressure compensation correctness with laboratory analysis of sample on regular basis. |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| TT-06125 | Acetic acid, water, paraxylene & traces | Gas/Liquid | - | 109 | - | DegC | | | | |
| TT-06126 | Acetic acid, water, paraxylene & traces | Gas/Liquid | 93 | - | 109 | DegC | | | | |
| TT-06127 | Acetic acid, water, paraxylene & traces | Gas/Liquid | 93 | - | 109 | DegC | | | | |
| TT-06128 | Acetic acid, water, paraxylene & traces | Gas/Liquid | 93 | - | 109 | DegC | | | | |
| TT-06129 | Acetic acid, water, paraxylene & traces | Gas/Liquid | 93 | - | 109 | DegC | | | | |
| TT-06130 | Acetic acid, water, paraxylene & traces | Gas/Liquid | 93 | | 109 | DegC | | | | |
| TT-06146 | LP Steam | Vapor | | 148 | 185 | DegC | | | | |
| TT-06204 | n-PA, water, methyl acetate + traces | Vapour | - | 85 | 150 | DegC | | | | |
| TT-06207 | Cooling water | Liquid | - | 44 | 60 | DegC | | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| TT-06210 | n-PA, methyl acetate, water + traces | Vapour | - | 78 | 150 | DegC | | | | |
| TT-06223 | n-PA, methyl acetate, water + traces | Liquid | - | 79 | 150 | DegC | | | | |
| TT-06404 | DH Solvent | Liquid | - | 95 | 120 | DegC | | High temperature may lead to corrosison of downstream DH solvent distribution system | 1. High temperature alarm. | 1. Check for valve passing / sticking |
| TT-06435/36/37 | G5-607A Winding Temperature | | TBD | TBD | TBD | DegC | To be decided at time of commissioning | | | |
| TT-06438/39 | G5-607A Bearing Temperature | | TBD | TBD | TBD | DegC | To be decided at time of commissioning | | | |
| TT-06440/41/42 | G5-607B Winding Temperature | | TBD | TBD | TBD | DegC | To be decided at time of commissioning | | | |
| TT-06443/44 | G5-607B Bearing Temperature | | TBD | TBD | TBD | DegC | To be decided at time of commissioning | | | |
| II-06220 | G5-615A Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |
| II-06222 | G5-615B Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| II-06406 | G5-606A Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |
| II-06408 | G5-606B Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |
| II-06412 | G5-607A Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |
| II-06414 | G5-607B Pump Amperage | | | TBD | | A | To be decided at time of commissioning | | | |
| AI-06116 | Water Content in D5-601 base | Liquid | 4.5 | 5.0 | 6.5 | % | | Lower water will lead to corrosion of column sump and reboiler, especially reboiler tubes | 1. Pressure compensated temperature control of column bottom.<br>2. High temperarure alarm on TT-06116. | 1. Cross check pressure compensation correctness with laboratory analysis of sample on regular basis. |

# Annexure C: Alarm and Trip Schedule

| Tag No. | Title | Low Alarm | High Alarm | Low Trip | High Trip | Units | Remarks |
|---|---|---|---|---|---|---|---|
| FT-06101 | WATER TO D5-601 FROM D5-631 | | 32000 | | | | |
| FT-06102 | Acetic Acid Makeup to D5-601 | 720 | | | | | |
| LT-06105 | D5-601 BOTTOMS | 1000 | 3800 | | | | D5-601 Bottom tan line |
| LT-06106 | D5-601 BOTTOMS LEVEL | | 3800 | | 4700 | | D5-601 Bottom tan line |
| TT-06107A/B | D5-601 ZONE "A" TEMPERATURE | | 95 | | | | |
| TT-06116 | D5-601 ZONE "C" TEMPERATURE | 115 | 123 | | | | |
| PT-06117 | PRESSURE IN D5-601 | | 0.6 | | 1.5 | | |
| FT-06119 | LP STEAM TO E5-602 | 70000 | | | | | |
| PT-06120 | LP STEAM TO E5-602 | 2.5 | | | | | |
| FT-06122 | WATER TO D5-651 FROM D5-631 | | 2500 | | | | |
| TT-06130 | D5-651 TEMPERATURE CONTROL | 100 | 130 | | | | |
| FT-06133 | FLASH VAPOUR FROM D5-402 TO D5-651 | 5000 | | | | | |
| LT-06135 | D5-651 BOTTOM LEVEL | 270 | 480 | | | | D5-651 Bottom tan line |
| FT-06136 | HP SOLVENT TO D5-601 TOP | | 90000 | | | | |
| LS-06137 | D5-651 BASE | | | | 650 | | D5-651 Bottom tan line |
| PT-06201 | D5-601 OVERHEADS PRESSURE | 0.05 | 0.4 | | | | |
| PT-06203 | D5-601 OVERHEADS PRESSURE | | | -0.02 | 0.8 | | |
| FT-06209 | VAPOUR FROM F5-608 TO D5-631 | 26000 | 50000 | | | | |
| TT-06210 | VENT GAS FROM F5-608 TO D5-631 | | 83 | | | | |
| LT-06211 | F5-608 LEVEL | | 2050 | | | | F5-608 bottom tan line |
| LT-06212 | F5-608 LEVEL | | 1500 | | | | F5-608 bottom tan line |
| LT-06213 | F5-609 LEVEL | 600 | 1600 | | 1900 $ | | F5-609 bottom tan line, $ High high trip |
| LT-06215 | F5-609 LEVEL | | 1600 | | 1900 | | F5-609 bottom tan line |
| TT-06223 | F5-609 REFLUX TO D5-601 TOP | | 86 | | | | |
| PT-06224 | G5-615A/B DISCHARGE LOW PRESSURE AUTO-START | | | 4 | | | |
| FT-06226 | F5-609 REFLUX TO D5-601 TOP | 270 | | | | | |
| FT-06247 | Entrainer to F5-609 | 61 | | | | | |
| FT-06401 | DH SOLVENT FLOW | 30000 | | | | | |
| TT-06404 | E5-603 OUTLET TEMPERATURE | | 100 | | | | |
| FT-06416 | HP SOLVENT FLOW | 70000 | | | | | |
| PT-06419 | E5-603 Outlet | | 0.2 | | | | |
| PT-06420 | G5-607A/B DISCHARGE (AUTO-START) | | | 16 | | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| PT-06432 | E5-603 Outlet | | 0.2 | | | | |
| TT-06435/36/37 | G5-607A Winding Temperature | | 115 | | | | |
| TT-06438/39 | G5-607A Bearing Temperature | | 95 | | | | |
| TT-06440/41/42 | G5-607B Winding Temperature | | 115 | | | | |
| TT-06443/44 | G5-607B Bearing Temperature | | 95 | | | | |

# Annexure D: Material Safety Data Sheets

## a) Concentrated Acetic Acid(Conc.>90%)

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**1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION**

Name: Acetic Acid (concentrated)

Alternative Names: Ethanoic Acid, Methane carboxylic acid, Glacial acetic acid

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**2. COMPOSITION/INFORMATION ON INGREDIENTS**

| HAZARDOUS INGREDIENT(S) | Classification | % weight | CAS No. | EC-No. |
|---|---|---|---|---|
| Acetic Acid | C; R35 | >90 | 64-19-7 | 200-580-7 |

R35 causes severe burns

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**3. HAZARDS IDENTIFICATION**

Main Hazards: Causes severe burns. Flammable

Health effects: May cause permanent damage if eye is not immediately irrigated. Causes severe chemical burns to the skin, mouth throat and digestive tract.

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**4. FIRST-AID MEASURES**

Inhalation : Remove patient from exposure, keep warm and at rest. Obtain immediate medical attention.

Skin Contact : Wash skin with water. Use safety shower if available. Remove contaminated clothing. If symptoms (irritation or blistering) occur obtain medical attention.

Eye Contact : Immediately irrigate with eyewash solution or clean water, holding the eyelids apart, for at least 30 minutes. Obtain immediate medical attention. Continue irrigation until medical attention can be obtained.

Ingestion : Do not induce vomiting. Wash out mouth with water and give 200-300 ml (half a pint) of water to drink. Obtain immediate medical attention.

**Further Medical Treatment**

Administer oxygen if necessary.

Following exposure the patient should be kept under medical review for at least 48 hours as delayed pulmonary oedema may develop

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**5. FIRE-FIGHTING MEASURES**

Extinguishing Media : polar resistant foam, water spray, dry powder or carbon dioxide.
Fire Fighting Protective Equipment : A self-contained breathing apparatus and suitable protective clothing should be worn in fire conditions.
Further information : Combustion or thermal decomposition will evolve toxic and irritant vapours.

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**6. ACCIDENTAL RELEASE MEASURES**

Ensure suitable personal protection (including respiratory protection) during removal of spillages.
Eliminate all sources of ignition.
Prevent entry into drains.
Contain and adsorb large spillages onto an inert, non-flammable adsorbent carrier.
Transfer to a container for disposal.
Spillages or uncontrolled discharges into watercourses, drains or sewers must be IMMEDIATELY alerted to the National Rivers Authority or other appropriate regulatory body.

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**7. HANDLING AND STORAGE**

**7.1 HANDLING**

Avoid contact with skin and eyes. Do not breathe vapour.
Use only in well ventilated areas.
Atmospheric levels should be controlled in compliance with the occupational exposure limit.

**7.2 STORAGE**

Keep container tightly closed, in a cool, well ventilated place.
Keep away from strong oxidising agents, strong bases.
Attacks most common metals liberating hydrogen, which can form explosive mixtures with air.
Unsuitable containers: mild steel
Suitable containers: stainless steel, high density plastic
Storage Temperature : above 20 Deg C

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**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Wear suitable respiratory protective equipment if exposure to levels above the occupational exposure limit is likely.
Wear suitable protective clothing, gloves and eye/face protection.
PVC gloves and goggles are the minimum protection.
Atmospheric levels should be controlled in compliance with the occupational exposure limit.
Wear full PVC suit, hood and rubber boots if splashing is likely.
Occupational Exposure Limits:

| OES (EH40/2000 | LTEL 8hr TWA | | STEL 15 min. | |
|---|---|---|---|---|
| | ppm | mg/m³ | ppm | mg/m³ |
| Acetic Acid | 10 | 25 | 15 | 37 |

---

**9. PHYSICAL AND CHEMICAL PROPERTIES**

Colour : colourless
Odour : characteristically pungent
pH (Value) : No data.
Melting Point (Deg C) : 16.66
Boiling Point (Deg C) : 117.9
Flash Point (Deg C) : 42 (closed cup)
Flammable Limits (Lower) (%v/v) : 4.0
Flammable Limits (Upper) (%v/v) : 16.0
Auto Ignition Temperature (Deg C) : 463
Explosive Properties : Not an explosive.
Oxidising Properties : No data.
Vapour Pressure (kPa) : 1.52 at 20°C
Density (g/ml) : 1.05 at 20°C
Solubility (Water) : miscible
Solubility (Other) : miscible with alcohols , glycerol, ether
Partition Coefficient : practically non-volatile log P -0.17
Vapour Density (Air= 1) : 2.07

---

**10. STABILITY AND REACTIVITY**

Hazardous Reactions : Attacks most common metals liberating hydrogen, which can form explosive mixtures with air.
Materials to avoid : Oxidising agents, Phosphorous tri-chloride, Alkalis.

---

**11. TOXICOLOGICAL INFORMATION**

Inhalation : Atmospheric concentrations in excess of the occupational exposure limit may lead to irritation of the eyes and respiratory tract, headache and dizziness.
Higher concentrations will produce severe damage to eyes and respiratory system.
Skin Contact : Corrosive. Causes burns. Both the vapour and the liquid cause irritation of the skin. Unlikely to cause skin sensitisation except in a small proportion of individuals.
Eye Contact : Corrosive. May cause permanent damage if eye is not immediately irrigated.
Opacity may develop over a period of days.
Ingestion : Will cause corrosion of and damage to the gastrointestinal tract. May cause nausea, vomiting and diarrhoea.

Long Term Exposure : Repeated exposure by inhalation to high levels may produce respiratory effects.

---

**12. ECOLOGICAL INFORMATION**

**Environmental Fate and Distribution**

Liquid with high volatility.

The substance is soluble in water.

The substance has no potential for bioaccumulation.

**Persistence and Degradation**

The substance is substantially biodegradable in water.

Biological Oxygen Demand (BOD 5 DAY) 76%

**Toxicity**

LC50 (fathead minnow) (96 hour) (static) 88 mg/l

---

**13. DISPOSAL CONSIDERATIONS**

Disposal should be in accordance with local, state or national legislation.

---

**14. TRANSPORT INFORMATION**

UN No. : 2789 UN Pack. Group : II

**AIR**

| | | |
|---|---|---|
| ICAO/IATA Class : 8 | Subsidiary risk : 3 | |
| Packing Group : II | Label: Corrosive, Flammable Liquid | |

Proper Shipping Name : Acetic Acid Solution

**SEA**

| | | |
|---|---|---|
| IMDG Class : 8 | Subsidiary risk : 3 | |
| Packing Group : II | Label: Corrosive, Flammable Liquid | |
| IMDG-Page: 8100 | EmS: 8-04 | MFAG: 700 |

Proper Shipping Name : Acetic Acid Solution

**ROAD/RAIL**

| | |
|---|---|
| ADR/RID Class : 8 | ADR/RID Item No : 32(b)2 |
| Label(s)(Packages) : 8 | Label(s)(Tankers) : 8+3 |
| HI/UN-No : 83/2789 | TremCard: 614 |

Proper Shipping Name: Acetic Acid Solution, 8, 32(b)2, ADR

UK TANKER REGULATIONS - DANGEROUS GOODS

Emergency Action Code : 2P

SI-No (UN) : 2789

---

**15. REGULATORY INFORMATION**

EC Classification : Flammable and Corrosive

Index No. : 607-002-00-6

EC-Label

Hazard Symbol : C

Content : Acetic Acid

| | |
|---|---|
| Risk Phrases : | R10 Flammable |
| | R35 causes burns |
| Safety Phrases : | S23 Do not breathe vapor |
| | S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. |
| | S45: In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible). |

## b) Acetic Acid (Conc. 25-90%)

---

**1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION**

Name: Acetic Acid (concentration 25-90%)

Alternative Names: Ethanoic Acid, Methane carboxylic acid

---

**2. COMPOSITION/INFORMATION ON INGREDIENTS**

| HAZARDOUS INGREDIENT(S) | Classification | % weight | CAS No. | EC-No. |
|---|---|---|---|---|
| Acetic Acid | C; R34 | 25-90 | 64-19-7 | 200-580-7 |

R34 causes severe burns

---

**3. HAZARDS IDENTIFICATION**

Causes burns to skin, eyes and respiratory system.

May cause permanent damage if eye is not immediately irrigated. Combustible.

---

**4. FIRST-AID MEASURES**

Inhalation : Remove patient from exposure, keep warm and at rest. Obtain immediate medical attention.

Skin Contact : Wash skin with water. Use safety shower if available. Remove contaminated clothing. If symptoms (irritation or blistering) occur obtain medical attention.

Eye Contact : Immediately irrigate with eyewash solution or clean water, holding the eyelids apart, for at least 30 minutes. Obtain immediate medical attention. Continue irrigation until medical attention can be obtained.

Ingestion : Do not induce vomiting. Wash out mouth with water and give 200-300 ml (half a pint) of water to drink. Obtain immediate medical attention.

**Further Medical Treatment**

Administer oxygen if necessary.

Following exposure the patient should be kept under medical review for at least 48 hours as delayed pulmonary oedema may develop

---

**5. FIRE-FIGHTING MEASURES**

Combustible.

Comsution or thermal decomposition will evolve toxic and irritant vapours.

Extinguishing Media : polar resistant foam, water spray, dry powder or carbon dioxide.

Fire Fighting Protective Equipment : A self-contained breathing apparatus and suitable protective clothing should be worn in fire conditions.

---

**6. ACCIDENTAL RELEASE MEASURES**

Ensure suitable personal protection (including respiratory protection) during removal of spillages.
Prevent entry into drains.
Contain and adsorb large spillages onto an inert, non-flammable adsorbent carrier.
Transfer to a container for disposal.
Spillages or uncontrolled discharges into watercourses, drains or sewers must be IMMEDIATELY alerted to the National Rivers Authority or other appropriate regulatory body.

---

**7. HANDLING AND STORAGE**

**7.1 HANDLING**

Avoid contact with skin and eyes. Do not breathe vapour.
Use only in well ventilated areas.

**7.2 STORAGE**

Keep container tightly closed, in a cool, well ventilated place.
Keep away from strong oxidising agents, strong bases.
Attacks most common metals liberating hydrogen, which can form explosive mixtures with air.
Unsuitable containers: mild steel
Suitable containers: stainless steel, high density plastic
Storage Temperature : above 20 Deg C

---

**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Wear suitable respiratory protective equipment if exposure to levels above the occupational exposure limit is likely.
Wear suitable protective clothing, gloves and eye/face protection.
PVC gloves and goggles are the minimum protection.
Atmospheric levels should be controlled in compliance with the occupational exposure limit.
Wear full PVC suit, hood and rubber boots if splashing is likely.
Occupational Exposure Limits:

| OES (EH40/99 | LTEL 8hr TWA | | STEL 15 min. | |
|---|---|---|---|---|
| | ppm | mg/m³ | ppm | mg/m³ |
| Acetic Acid | 10 | 25 | 15 | 37 |

---

**9. PHYSICAL AND CHEMICAL PROPERTIES**

Form: mobile liquid

Colour : colourless
Odour : characteristically pungent
pH (Value) : No data.
Melting Point (Deg C) : 16.66
Flash Point (Deg C) : 42 (closed cup) (pure acetic acid)
Flammable Limits (Lower) (%v/v) : 5.4 (pure acetic acid)
Flammable Limits (Upper) (%v/v) : 16.0 (pure acetic acid)
Auto Ignition Temperature (Deg C) : no data
Explosive Properties : No data
Oxidising Properties : No data.
Vapour Pressure (kPa) : 11.9 at 20°C
Density (g/ml) : 1.05
Solubility (Water) : miscible
Solubility (Other) : miscible with alcohols , glycerol, ether
Partition Coefficient : practically non-volatile log P -0.17
Vapour Density (Air= 1) : 2.07

---

**10. STABILITY AND REACTIVITY**

Hazardous Reactions : Attacks most common metals liberating hydrogen, which can form explosive mixtures with air.
Materials to avoid : Oxidising agents, Phosphorous tri-chloride, Alkalis.

---

**11. TOXICOLOGICAL INFORMATION**

Inhalation : Atmospheric concentrations in excess of the occupational exposure limit may lead to irritation of the eyes and respiratory tract, headache and dizziness.
Higher concentrations will produce severe damage to eyes and respiratory system.
Skin Contact : Corrosive. Causes burns. Both the vapour and the liquid cause irritation of the skin. Unlikely to cause skin sensitisation except in a small proportion of individuals.
Eye Contact : Corrosive. May cause permanent damage if eye is not immediately irrigated.
Opacity may develop over a period of days.
Ingestion : Will cause corrosion of and damage to the gastrointestinal tract. May cause nausea, vomiting and diarrhoea.
Long Term Exposure : Repeated exposure by inhalation to high levels may produce respiratory effects.

---

**12. ECOLOGICAL INFORMATION**

**Environmental Fate and Distribution**

Liquid with high volatility.
The substance is soluble in water.

The substance has no potential for bioaccumulation.

**Persistence and Degradation**

The substance is substantially biodegradable in water.

Biological Oxygen Demand (BOD 5 DAY) 76%

**Toxicity**

LC50 (fathead minnow) (96 hour) (static) 88 mg/l

---

**13. DISPOSAL CONSIDERATIONS**

Disposal should be in accordance with local, state or national legislation.

---

**14. TRANSPORT INFORMATION**

**Acetic Acid > 80%**

UN No. : 2789 UN Pack. Group : II

**AIR**

ICAO/IATA Class : 8 Subsidiary risk : 3

Packing Group : II Label: Corrosive, Flammable Liquid

Proper Shipping Name : Acetic Acid Solution

**SEA**

IMDG Class : 8 Subsidiary risk : 3

Packing Group : II Label: Corrosive, Flammable Liquid

IMDG-Page: 8100 EmS: 8-04 MFAG: 700

Proper Shipping Name : Acetic Acid Solution

**ROAD/RAIL**

ADR/RID Class : 8 ADR/RID Item No : 32(b)2

Label(s)(Packages) : 8 Label(s)(Tankers) : 8+3

HI/UN-No : 83/2789

Proper Shipping Name: Acetic Acid Solution, 8, 32(b)2, ADR

**Acetic Acid 10-50% and 50-80%**

UN No. : 2790 UN Pack. Group : II

**AIR**

ICAO/IATA Class : 8 Subsidiary risk : n/a

Packing Group : II Label: Corrosive

Proper Shipping Name : Acetic Acid Solution

**SEA**

IMDG Class : 8 Subsidiary risk : n/a

Packing Group : II Label: Corrosive

IMDG-Page: 8100 EmS: 8-05 MFAG: 700

Proper Shipping Name : Acetic Acid Solution

**ROAD/RAIL**

ADR/RID Class : 8 ADR/RID Item No : 32(b)1 (50-80%)
32(c) (10-50%)

Label(s)(Packages) : 8 Label(s)(Tankers) : 8

HI/UN-No : 80/2790

Proper Shipping Name: Acetic Acid Solution, 8, 32(b)2, ADR
Or Acetic Acid Solution, 8, 32(c) , ADR

UK TANKER REGULATIONS - DANGEROUS GOODS

Emergency Action Code : 2R

SI-No (UN) : 2790

---

**15. REGULATORY INFORMATION**

EC Classification : Corrosive

Index No. : 607-002-00-6 EC-Label

Hazard Symbol : C

Content : Acetic Acid

Risk Phrases : R34 causes burns

Safety Phrases : S23 Do not breathe vapor

S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

S45: In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

## c) Caustic Solution (2-5%)

**1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION**

Name: Caustic Soda(2-<5%)

Alternative Names: Sodium Hydroxide Solution

**2. COMPOSITION/INFORMATION ON INGREDIENTS**

| HAZARDOUS INGREDIENT(S) | Classification | % weight | CAS No. | EC-No. |
|---|---|---|---|---|
| Sodium Hydroxide | C; R35 | 2-<5 | 1310-73-2 | 215-185-5 |

R35 causes severe burns

**3. HAZARDS IDENTIFICATION**

Causes burns to all parts of the body

**4. FIRST-AID MEASURES**

Inhalation: Remove patient from exposure, keep warm and at rest. Administer oxygen if necessary.

Skin Contact: Remove contaminated clothing. After contact with skin, wash immediately with plenty of water.

Eye Contact: Immediately irrigate with eyewash solution or clean water, holding the eyelids apart, for at least 10minutes. Continue irrigation until medical attention can be obtained.

Ingestion: Do not induce vomiting. Provided the patient is conscious, wash out mouth with water and give 200-300 ml(half a pint) of water to drink.

Further Medical Treatment

Symptomatic treatment and supportive therapy as indicated. Administer oxygen if necessary.

**5. FIRE-FIGHTING MEASURES**

Non-combustible. Contact with some metals can produce flammable hydrogen gas. Contact with some organic chemicals can produce violent or explosive reactions.

Extinguishing Media : As appropriate for surrounding fire.

Fire Fighting Protective Equipment: A self-contained breathing apparatus and suitable protective clothing must be worn in fire conditions.

**6. ACCIDENTAL RELEASE MEASURES**

Ensure suitable personal protection during removal of spillages.
Drench spillages with water and wash to drain.
Spillages or uncontrolled discharges into watercourses must be alerted to the appropriate regulatory body.

---

**7. HANDLING AND STORAGE**

**7.1 HANDLING**

Keep away from acids and chlorinated hydrocarbons.
Avoid contact with skin and eyes. Avoid inhalation of high concentrations of mists. Atmospheric levels should be controlled in compliance with the occupational exposure limit.

**7.2 STORAGE**

Keep container tightly closed. Storage vessels should be made of mild steel. Where temperatures exceed 60 Deg C, tanks must be stress relieved.

---

**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Wear close fitting goggles or full face shield.
Wear suitable protective clothing and gloves. PVC is recommended.
Occupational Exposure Limits:

| OES (EH40/99 | LTEL 8hr TWA | | STEL 15 min. | |
|---|---|---|---|---|
| | ppm | $mg/m^3$ | ppm | $mg/m^3$ |
| Sodium Hydroxide | - | - | - | 2 |

---

**9. PHYSICAL AND CHEMICAL PROPERTIES**

Form : Clear or slightly turbid liquid
Colour : colourless
Solubility (Water) : very soluble
Solubility (Other) : very soluble in: alcohol and glycerine
insoluble in: acetone and ether
Specific Gravity : 1.021 - 1.054 (at 20 Deg C)

---

**10. STABILITY AND REACTIVITY**

Hazardous Reactions: Can react violently if in contact with acids.
Can react with chlorinated hydrocarbons to form toxic and explosive chloroacetylenes.

Highly reactive with aluminium, zinc, tin and alloys of these metals producing flammable hydrogen gas.

Can react with sugar residues to form carbon monoxide.

---

**11. TOXICOLOGICAL INFORMATION**

Inhalation: Mist is severely irritant to the respiratory tract.

Skin Contact: Causes burns. Repeated or prolonged contact may cause dermititis.

Eye Contact: Severe/very severe irritant. May cause severe permanent impairment of vision.

Ingestion: Will cause corrosion of and damage to the gastrointestinal tract.

Long Term Exposure: Chronic effects are unlikely.

---

**12. ECOLOGICAL INFORMATION**

**Environmental Fate and Distribution**

Liquid with moderate volatility.

The substance does not bioaccumulate

**Persistence and Degradation**

Chemical Oxygen Demand (COD) zero.

Biological Oxygen Demand (BOD 5 DAY) zero

Sodium hydroxide degrades readily by reaction with the natural carbon dioxide in the air.

**Toxicity**

Concentrations greater than 10ppm, especially in fresh water, or a pH value equal to or greater than 10.5 may be fatal to fish and other aquatic organisms.

Can cause damage to aquatic plants.

Can cause damage to vegetation.

**Effect on Effluent Treatment**

Concentrations sufficient to render effluent alkaline may cause damage to effluent treatment organisms.

---

**13. DISPOSAL CONSIDERATIONS**

Disposal should be in accordance with local, state or national legislation.

---

**14. TRANSPORT INFORMATION**

UN No. : 1824 UN Pack. Group : III

**AIR**

| | |
|---|---|
| ICAO/IATA Class : 8 | Subsidiary risk : n.a. |
| Packing Group : III | Label: Corrosive |

Proper Shipping Name : Sodium Hydroxide Solution

**SEA**

| | | |
|---|---|---|
| IMDG Class : 8 | Subsidiary risk : n.a. | |
| Packing Group : III | Label: Corrosive | |
| IMDG-Page: 8226 | EmS: 8-06 | MFAG: 705 |

Proper Shipping Name : Sodium Hydroxide Solution

**ROAD/RAIL**

| | |
|---|---|
| ADR/RID Class : 8 | ADR/RID Item No : 42(c) |
| Label(s)(Packages) : 8 | Label(s)(Tankers) : 8 |

HI/UN-No : 80/1824
Proper Shipping Name: 1824 Sodium Hydroxide Solution, 8, 42(c), ADR
UK TANKER REGULATIONS - DANGEROUS GOODS
Emergency Action Code : 2R
SI-No (UN) : 1824

---

**15. REGULATORY INFORMATION**

EC Classification : Corrosive
Index No. : 011-002-00-6
EC-Label
Hazard Symbol : C
Content : Sodium Hydroxide(2-<5%)
Risk Phrases : R34 causes burns
Safety Phrases : S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S37/39: Wear suitable gloves and eye/face protection.
S45: In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

---

## d) Nitrogen

### 1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION

Name: Nitrogen

Alternative Names: N2

### 2. COMPOSITION/INFORMATION ON INGREDIENTS

| HAZARDOUS INGREDIENT(S) | Classification | % weight | CAS No. | EC-No. |
|---|---|---|---|---|
| Nitrogen | -- | >99.0 | 7729-37-9 | 231-783-9 |

### 3. HAZARDS IDENTIFICATION

Simple Asphyxiant. Liquid splashes or spray may cause freeze burns

### 4. FIRST-AID MEASURES

Inhalation : Remove patient from exposure. Apply artificial respiration if breathing has ceased or shows signs of failing. Obtain immediate medical attention.

Skin Contact : Immerse the affected area in comfortably warm water to defrost.

Eye Contact : Immediately irrigate with eyewash solution or clean water, holding the eyelids apart, for at least 10 minutes Obtain immediate medical attention.

Ingestion : Do not induce vomiting. Wash out mouth with water and give 200-300 ml (half a pint) of water to drink. Obtain immediate medical attention.

Further Medical Treatment: Symptomatic treatment and supportive therapy as indicated. Administer oxygen if necessary

### 5. FIRE-FIGHTING MEASURES

Extinguishing Media : Keep fire exposed cylinders cool by spraying with water

### 6. ACCIDENTAL RELEASE MEASURES

Ensure the area is well ventilated. If it is desirable to evaporate the liquefied nitrogen spill quickly, water spray may be used to increase the rate of evaporation.

### 7. HANDLING AND STORAGE

#### 7.1 HANDLING

Use only in well ventilated areas. Avoid contact with skin and eyes. (when handling the liquid)

### 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Occupational Exposure Limits: simple asphyxiant. The oxygen content of the air in the work place should never be allowed to fall below 19%.

Personal protection: In case of insufficient ventilation, wear suitable respiratory equipment. Insulating gloves and goggles are suitable when handling the liquid.

---

**9. PHYSICAL AND CHEMICAL PROPERTIES**

Form : gas or liquefied gas
Colour : colourless
Odour : odourless
pH (Value) : not applicable
Melting Point (Deg C) : -209.9
Boiling Point (Deg C) : -195.8
Flash Point (Deg C) : none
Flammable Limits (Lower) (%v/v) : none
Flammable Limits (Upper) (%v/v) : none
Auto Ignition Temperature (Deg C) : none
Explosive Properties : none
Oxidising Properties : no data available
Vapour Pressure (mm Hg) : no data available
Density (g/ml) : no data available
Solubility (Water) : 2.3% vol/vol
Solubility (Other) : no data available
Partition Coefficient : no data available
Vapour Density (Air= 1) : 0.97

---

**10. STABILITY AND REACTIVITY**

Hazardous Reactions: Stable

---

**11. TOXICOLOGICAL INFORMATION**

Inhalation : A simple asphyxiant. High atmospheric concentrations may lead to headache, drowsiness and in extreme cases unconsciousness and death.
Skin Contact : Liquid splashes or spray may cause freeze burns.
Eye Contact : Liquid splashes or spray may cause freeze burns.
Ingestion : Highly unlikely - but should this occur freeze burns will result.
Long Term Exposure : No known hazards are associated with the use of this material.

---

**12. ECOLOGICAL INFORMATION**

Environmental Fate and Distribution: No data available
Persistence and Degradation: No data available
Toxicity: No data available

---

**13. DISPOSAL CONSIDERATIONS**

Do not attempt to dispose of residual waste or unused quantities. Return in the shipping container properly labelled, with any valve outlet plugs or caps secured and valve protection cap in place to BOC gases or authorized distributor for proper disposal

---

**14. TRANSPORT INFORMATION**

UN No. : 1066 UN Pack. Group : n.a.

**AIR**

| | |
|---|---|
| ICAO/IATA Class : 2.2 | Subsidiary risk : n.a. |
| Packing Group : n.a. | Label: Non-flammable gas |

Proper Shipping Name : Nitrogen, compressed

**SEA**

| | | |
|---|---|---|
| IMDG Class : 2.2 | | Subsidiary risk : n.a. |
| Packing Group : n.a. | | Label: Non-flammable gas |
| IMDG-Page: 2163 | EmS: 2-04 | MFAG: none |

Proper Shipping Name : Nitrogen, compressed

**ROAD/RAIL**

| | |
|---|---|
| ADR/RID Class : 2 | ADR/RID Item No : 3°A |
| Label(s)(Packages) : 2 | Label(s)(Tankers) : 2 |
| HI/UN-No : 20/1066 | Tremcard: 20G39-A |

Proper Shipping Name: Nitrogen, compressed

UK TANKER REGULATIONS - DANGEROUS GOODS

Emergency Action Code :

SI-No (UN) :

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**15. REGULATORY INFORMATION**

EC Classification : Not classified as dangerous for supply/use.

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## e) n-Propyl Acetate

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**1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION**

Name: n-propyl acetate
Alternative Names: n-propyl ester, 1-Acetoxypropane

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**2. COMPOSITION/INFORMATION ON INGREDIENTS**

| HAZARDOUS INGREDIENT(S) | Classification | % weight | CAS No. | EC-No. |
|---|---|---|---|---|
| n-propyl acetate | | 100 | 109-60-4 | 203-686-1 |

---

**3. HAZARDS IDENTIFICATION**

Highly flammable.
Irritating to eyes.
Vapor/air mixture are explosive
Vapors may travel considerable distance to a source of fire and flash back.
High vapour concentrations may cause drowsiness and irritation of the eyes or respiratory tract.
Prolonged or repeated skin contact may cause drying, cracking, or irritation due to the defatting properties of the substance.

---

**4. FIRST-AID MEASURES**

Inhalation : Move to fresh air. Treat symptomatically. Get medical attention if symptoms persist
Skin Contact : Wash skin with soap and water immediately. Remove contaminated clothing.
Eye Contact : Immediately irrigate with clean water for several minutes and seek medical advice.
Ingestion : Do not induce vomiting without medical advice. Obtain medical attention immediately.

---

**5. FIRE-FIGHTING MEASURES**

Extinguishing Media : water spray, dry chemical, carbon dioxide, alcohol foam
Fire Fighting Protective Equipment : A self-contained breathing apparatus and suitable protective clothing should be worn in fire conditions.

---

**6. ACCIDENTAL RELEASE MEASURES**

Remove all source of ignition. Dam up and absorb with vermiculite or other inert material, then place in a container for chemical waste. For large spills use water spray to disperse vapours and dilute spill to a non-flammable mixture. Prevent from entering sewers or streams.

---

**7. HANDLING AND STORAGE**

**7.1 HANDLING**

Avoid breathing high vapour concentrations. Avoid prolonged or repeated contact with skin. Use only with adequate ventilation. Wash thoroughly after handling.

**7.2 STORAGE**

Keep away from heat and sources of ignition in a cool and well-ventilated place. Keep away from oxidising materials. Store in original container.

---

**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Keep container tightly closed. Provide for adequate ventilation especially in confined areas. Wear safety glasses or coverall chemical splash goggles face shield when the possibility exists for eye and face contacts. Wear impervious clothing, such as gloves, apron, boots or whole body suit as appropriate.

Occupational Exposure Limits:

| OES (EH40/99 | LTEL 8hr TWA | | STEL 15 min. | |
|---|---|---|---|---|
| | ppm | mg/m³ | ppm | mg/m³ |
| n-propyl acetate | 200 | 849 | 250 | 1060 |

---

**9. PHYSICAL AND CHEMICAL PROPERTIES**

Form : liquid
Colour : colourless
Odour : sweet, ester (pear-like)
pH (Value) : no data available
Melting Point (Deg C) : -92
Boiling Point (Deg C) : 102
Flash Point (Deg C) : 13 (TCC)
Flammable Limits (Lower) (%v/v) : 1.7
Flammable Limits (Upper) (%v/v) : 7.9
Auto Ignition Temperature (Deg C) : 457
Explosive Properties : none
Oxidising Properties : none
Vapour Pressure (kPa at 20° C) : 3.5
Density (g/ml) : 0.9
Solubility (Water) : moderate
Partition Coefficient : 1.24 as log Pow
Vapor density: 3.5

---

**10. STABILITY AND REACTIVITY**

Hazardous Reactions : Stable at normal temperature and storage conditions. Can react violently with oxidizers.

Incompatibility : oxidizers, bases, acids moisture, heat

Decomposition products: carbon monoxide, carbon dioxide

Polymerization: Polymerization will not occur.

---

**11. TOXICOLOGICAL INFORMATION**

Animal data: LD50/oral/rat = 9370 mg/kg.

LC50/inhalation/4h/rat = 33.4 mg/l.

Inhalation : High vapour concentrations may cause drowsiness and irritation.

Skin Contact : Prolonged or repeated contact may cause drying, cracking or irritation

Eye Contact : High vapour concentrations may cause irritation.

Ingestion : Expected to be low ingestion hazard.

Long Term Exposure : no data available

---

**12. ECOLOGICAL INFORMATION**

The product has, a high biochemical oxygen demand and a potential to cause oxygen depletion in aqueous systems, a low potential to affect aquatic organisms and a low potential to bioconcentrate. When diluted with large amounts of water, this material released directly or indirectly into the environment is not expected to have a significant impact.

---

**13. DISPOSAL CONSIDERATIONS**

Incinerate under approved controlled conditions, using incinerators suitable for the disposal of noxious chemical waste. Disposal should be in accordance with local, state or national legislation.

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**14. TRANSPORT INFORMATION**

UN No. : 1276 UN Pack. Group : II

**AIR**

| | |
|---|---|
| ICAO/IATA Class : 3 | Subsidiary risk : n.a. |
| Packing Group : II | Label: Flammable Liquid |

Proper Shipping Name : n-propyl acetate

**SEA**

| | | |
|---|---|---|
| IMDG Class : 3.2 | Subsidiary risk : n.a. | |
| Packing Group : II | Label: Flammable Liquid | |
| IMDG-Page: 3274 | EmS: 3-07 | MFAG: 330 |

Proper Shipping Name : n-propyl acetate

**ROAD/RAIL**

| | |
|---|---|
| ADR/RID Class : 3 | ADR/RID Item No : 3(b) |
| Label(s)(Packages) : 3 | Label(s)(Tankers) : 3 |
| HI/UN-No : 33/1276 | TremCard: 519 |

Proper Shipping Name: n-propyl acetate, 3, 3(b), ADR
UK TANKER REGULATIONS - DANGEROUS GOODS
Emergency Action Code : 3YE
SI-No (UN) : 1276

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**15. REGULATORY INFORMATION**

EC Classification : Highly Flammable and Irritating
Index No. : 607-024-00-6
EC-Label
Hazard Symbol : F-Highly Flammable, Xi-Irritating
Content : n-propyl acetate

| | |
|---|---|
| Risk Phrases : | R11 Highly Flammable |
| | R36 Irritating to eyes |
| | R66 Repeated exposure may cause skin dryness or cracking |
| | R67 Vapors may cause drowsiness and dizziness |
| Safety Phrases : | S16 Keep away from sources of ignition- No smoking |
| | S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. |
| | S29: Do not empty into drains |
| | S33: Take precautionary measures against static discharges |

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## f) TA Oxidation Waste Water

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**1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION**

TA Oxidation Waste Water

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**2. COMPOSITION/INFORMATION ON INGREDIENTS**

Contains also trace amounts of :
Acetic acid
Methanol
Methylacetate
Paraxylene

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**3. HAZARDS IDENTIFICATION**

This is essentially hot water. Unlikely to cause harmful effects under normal conditions of handling and use. Hot water will scald.

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**4. FIRST-AID MEASURES**

Inhalation : Remove patient from exposure. Obtain medical attention if ill effects occur.
Skin Contact : Remove contaminated clothing. Wash skin with water. If symptoms develop, obtain medical attention.
Eye Contact : Irrigate with eyewash solution or clean water, holding the eyelids apart, for at least 10 minutes. Obtain medical attention.
Ingestion : Do not induce vomiting. Wash out mouth with water and give 200-300 ml (half a pint) of water to drink. Obtain medical attention if ill effects occur.

**Further Medical Treatment**

Unlikely to be required but if necessary treat symptomatically.

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**5. FIRE-FIGHTING MEASURES**

Non-combustible

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**6. ACCIDENTAL RELEASE MEASURES**

Drench spillages with water and wash to drain

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**7. HANDLING AND STORAGE**

**7.1 HANDLING**

Extra care should be taken to prevent burns from contact with hot material. Avoid contact with skin and eyes.

**7.2 STORAGE**

No special requirements

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**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

Good working practice suggests gloves and goggles should be worn.
No exposure limits assigned

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**9. PHYSICAL AND CHEMICAL PROPERTIES**

Form : liquid
Colour : colourless
Odour : odourless
pH (Value) : 7 approx
Boiling Point (Deg C) : 100 approx
Melting Point (Deg C) : 0 approx
Flash Point (Deg C) : Not applicable.
Auto Ignition Temperature (Deg C) : Not applicable.
Explosive Properties : Not applicable.
Oxidising Properties : Not applicable.
Density (g/ml) : 1.0 approx
Solubility (Water) : miscible
Partition Coefficient : Not applicable.

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**10. STABILITY AND REACTIVITY**

Hazardous Reactions: None known

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**11. TOXICOLOGICAL INFORMATION**

Inhalation : Unlikely to be hazardous by inhalation.
Skin Contact : Repeated or prolonged skin contact may result in mild irritation. Hot material may scald.
Eye Contact : Unlikely to cause eye irritation. Hot material may scald.
Ingestion : Unlikely to be hazardous if swallowed.

Long Term Exposure : Chronic effects are unlikely.

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## 12. ECOLOGICAL INFORMATION

No adverse effects would be expected.

**Environmental Fate and Distribution**

Medium tonnage material produced in partially contained systems. Liquid with moderate volatility. The product is soluble in water. The product has high mobility in soil.

**Persistence and Degradation**

no data available

**Toxicity**

no data available

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## 13. DISPOSAL CONSIDERATIONS

Disposal in accordance with local, state or national legislation.

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## 14. TRANSPORT INFORMATION

Not classified as dangerous for transport

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## 15. REGULATORY INFORMATION

EC Classification : Not classified as dangerous for supply/use.

# Annexure E: Inventory Summary in Vessels

| Equipment Tag No. | Description | Contents | Normal Working Volume($m^3$) | Maximum Working Volume($m^3$) | Minimum Working Volume($m^3$) |
|---|---|---|---|---|---|
| D5-601 | DH Column Bottom Sump (excludes the partitioned part) | Acetic Acid, Water, n-PA, methyl acetate, traces of other components | Varies between high and low level (note) | Approx.. 83 m3 (high level alarm) | Approx.. 34 m3 (low level alarm) |
| F5-609 | Entrainer Storage Tank | n-PA + traces of other components | Varies between high and low level (note) | 85.7 $m^3$ (High level alarm) | 53.6 $m^3$ (Low level alarm) |

Note : Level can be maintained anywhere between high and low alarm level, typically midway +/- 20%.