INVISTA Performance Technologies

SOLVENT DEHYDRATION AREA OVERVIEW

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DH Area General

- D5-601 – Solvent Dehydration Column
  - Azeotropic distillation with nPA
  - nPA profile control designed to prevent loss of nPA in bottoms and loss of acetic acid in the tops
- D5-651 – Purge Column
  - Removal of PX from column without significant loss of entrainer
- D5-631 – Recovery Column
  - Separation of water/MeOAc and recovery of entrainer

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DH Area Optimisation

- Optimisation Objectives
  - Minimisation of entrainer loss
  - Minimisation of acetic acid loss
  - Control of MeOAc in the Dehydration Column, D5-601 reflux
  - Control of PX concentration in the Dehydration Column, D5-601

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DH Area Optimisation

- Characteristics of optimum operation
  - Stable D5-601 profile control at 30% (Range 25% to 60%)
  - Constant steam rate to DH Column Reboiler, E5-602
  - Constant Dehydration Column pressure
  - Constant Purge Column bottoms temperature of 111degC (TBC)
  - Constant MeOAc concentration in the D5-601 reflux of 8% to 10%, if this is too high it will result in excessive steam consumption and high D5-601 pressure drop.
  - Stable D5-631 profile control at 50% (TBC)
- DH Area Disturbances
  - Change in WDO flow setpoint
  - Cycling of PCV 04173 in response to changes in D5-301 level control valve

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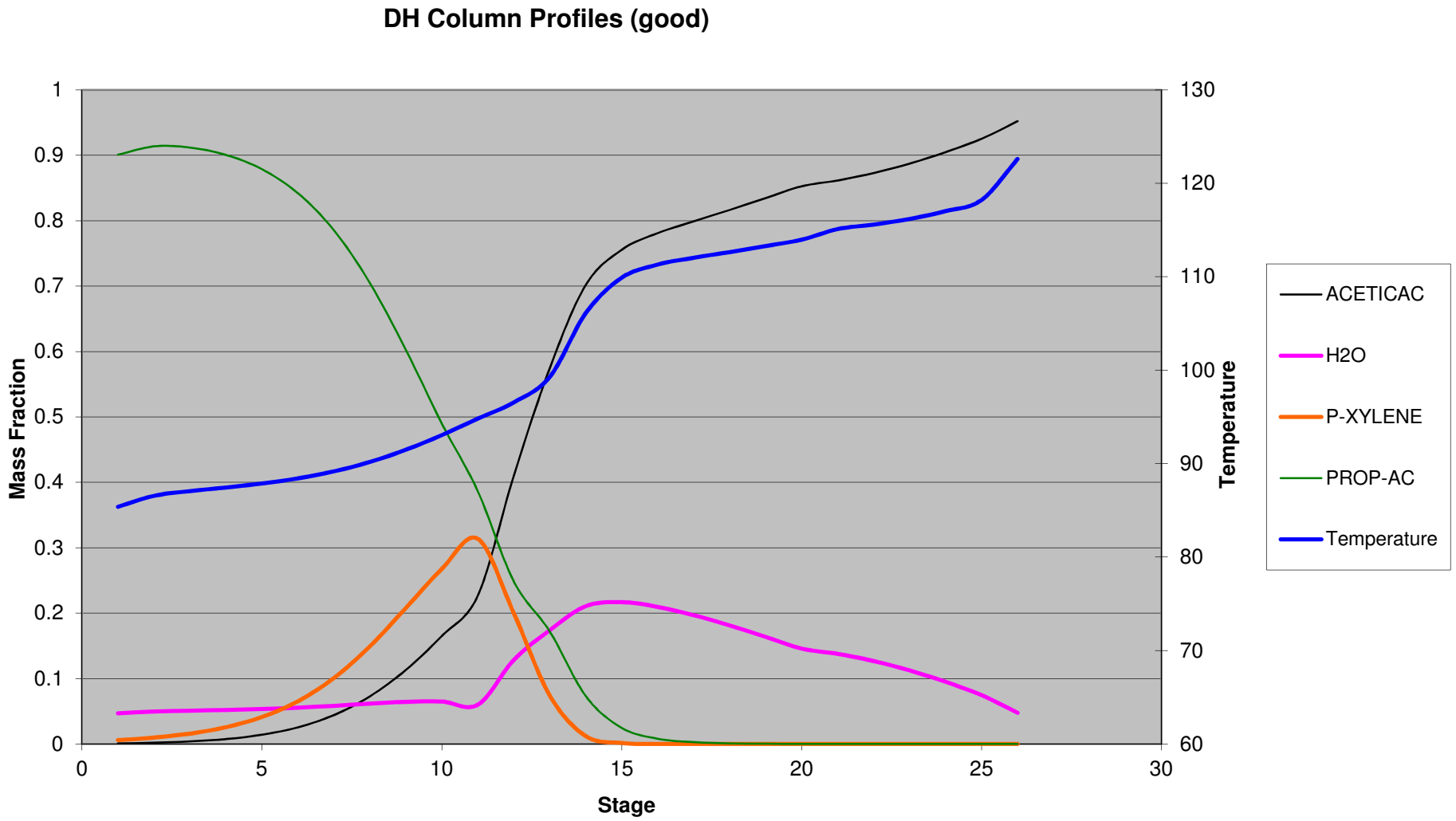
DH Area Optimisation



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D5-601 Optimisation

- Steam rate to the base of the column is controlled by a pressure corrected set point so cycling of the column is reduced
- If the profile is too high then acetic acid will be lost in the overheads.
- If the profile is too low entrainer will be lost in the bottoms
- Optimum position is 30% with a TOI of 103deg C.
- Cycling of PCV 04173 introduces large changes in the heat to D5-601 which introduces cycling of the column profile.
- The objective of the control scheme is to make only small changes to the top reflux flowrate.
- The control scheme aims to keep the profile bottom temperature TI-06113 above 109-111 degC.

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D5-651 Optimisation

- If the base temperature is too low entrainer will be lost in the bottoms.
- If the base temperature is too high the paraxylene purge will be too low.
- A low paraxylene purge results in accumulation in D5-601. The PX reduces the steepness of the profile so that the temperatures near the top become higher. This results in increased acetic loss (normally visible as high COD in the base of D5-631).
- The D5-651 profile control has been replaced by simple temperature control on the base.
- The optimum base temperature is 111 deg C (TBC)
- The PX purge should however be minimised to reduce entrainer loss

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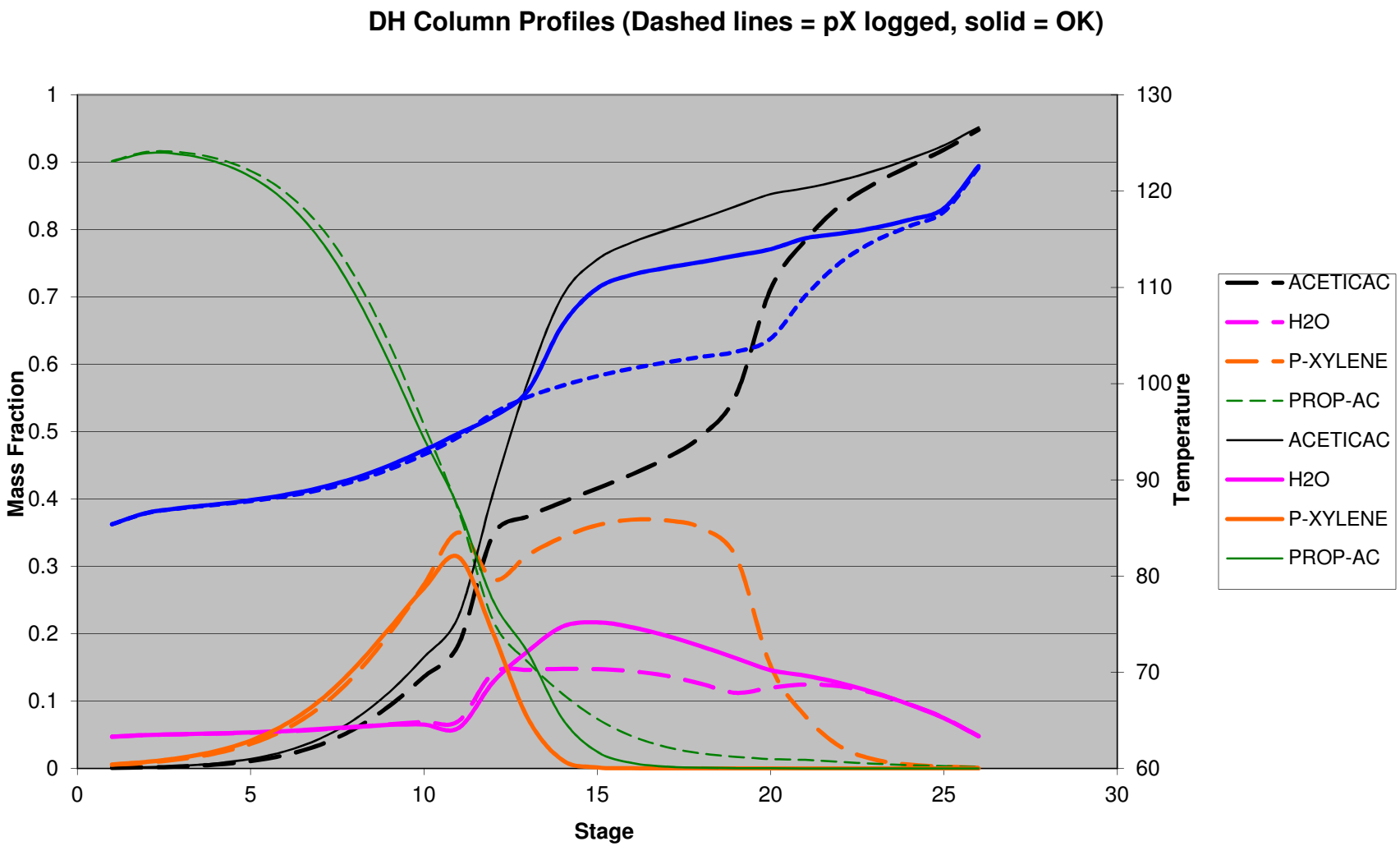
DH Area Optimisation



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MeOAc Control

- Under steady state operation MeOAc concentration is determined by 2 factors
  - Vent rate from F5-608 as controlled by FICA -06209
  - Position of D5-631 profile
- If the vent rate from F5-608 is not sufficient then the Dehydration column reflux will be subcooled which condenses all of the MeOAc. This MeOAc is then returned to D5-601 in the reflux and accumulates within the system.
- A constant purge rate of approx 35te/hr is required to control the MeOAc reflux concentration in the optimum range of 8% to 10%.
- Stable operation of the system cannot be achieved with FICA 06209 in cascade as the D5-631 profile is very sensitive to changes in the vent flow.
- If GCV 06208 (upstream of E5-608) were left fully open then there is not enough pressure drop across FCV 06209 and the vent rate will be too low.
- If GCV 06208 is too far closed then the pressure in D5-601 is too high which inhibits the separation of water from acetic acid.

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MeOAc Control

- D5-631 Profile
  - If the profile is too high then entrainer will be lost in the overheads
  - If the profile is too low then the MeOAc concentration in the return to F5-608 will be too high and MeOAc will accumulate in the D5-601 reflux.
  - If the vent rate from F5-608 is constant then the control should be good.

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Control of Propanol

- Propanol increases the solubility of water in the reflux.
  - Additional water in reflux significantly increases the load on DH Column.
  - Effect has been demonstrated in laboratory and on plant.
- Need to control levels of propanol in reflux to less than **2% w/w.**
  - Use purge from D5-631 bottom if necessary i.e. by lowering the temperature of D5-631 bottoms.

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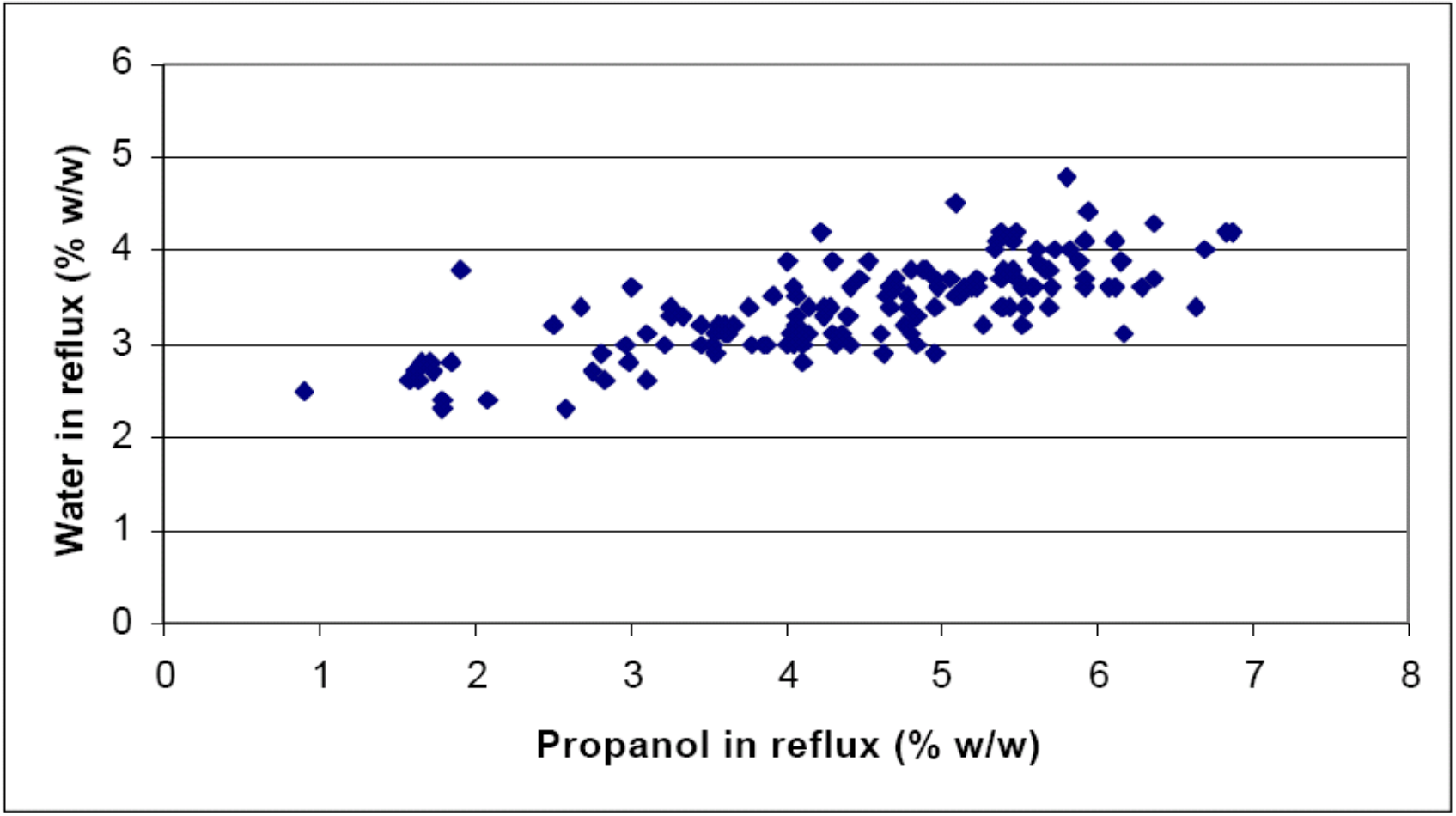
Control of Propanol



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DH Area Control Based On Analysis

Analysis	Normal Range	Actions if out of range
601 Bottoms		
Water	4.5 – 5.5%	Check 601 bottoms temperature if water content is too high or too low.
Br	<10ppm	If high may be due to 511 or 402 carryover
Iron	<1ppm	Indicative of corrosion if high
601 Reflux		
Px	< 2%	If high check 651 operation
MeOAc	8 – 12%	See notes on control of vapour flow from 608 to 631. Check loading of 631 (maybe too much stripping steam)
Water/ Propanol	1 – 3%	If high need to purge Propanol i.e. reduce 631 stripping steam.
Acetic Acid	<0.1%	If high see control of Px in 601.

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DH Area Control Based On Analysis

651 Bottoms		
n-PA	<0.05%	Should be negligible.
631 Bottoms (Waste Water)		
Acetic Acid	<0.1%	If high check 601 Reflux composition (high Px) and column profile control stability.
MeOAc	<0.005%	Should always be <0.005%
COD		If high check 631 bottoms temperature. See actions above for control of high Acetic Acid in 631 bottoms.

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