NH_3 SCRUBBING SYSTEM

PROCESS ENGINEERING STUDY

Contents

1.0 MANAGEMENT SUMMARY

This study considers the NH_3 scrubbing capability of an existing system comprising two scrubbers in series, namely SC100 and SC101, with the following specifications and operating conditions:

Primary scrubber SC100
Diameter: 450mm
Packed height: 4500mm
Packing type: Polypropylene Pall Rings 40mm
Circulation flow: 60gal/min, subsequently modified to 7.2 m^3/h
Scrubber tank: 1000 gallons of water
Heat exchanger on re-circulation line: 2.4m2 / cooling water (20ºC assumed)
Secondary scrubber SC101
Diameter: 450mm
Packed height: 4500mm
Packing type: Polypropylene Pall Rings 40mm
Circulation flow: 40gal/min, subsequently modified to 7.2 m^3/h
Scrubber tank: 1000 gallons of 13% w/w phosphoric acid
Heat exchanger on re-circulation line: 2.4m2 / cooling water (20ºC assumed)
Fan extraction: 800m3/hr @6"wg

The CHEMCAD simulations and supporting calculations lead to the following conclusions:

- The scrubbing system is more than capable of handling the predicted normal process flows of 87kg NH_3 over 3h and then a further 102 kg NH_3 over 3h.
- Primary scrubber SC100 can handle the Emergency Relief case of 530 kg/h NH_3 and inert flow of 50 kg/h N_2. A column loading of approximately 73% is predicted.
- It has been previous site practice to replace the primary scrubbing liquor with fresh water when the NH_3 concentration reaches 20%. This practice will result in a heavy slippage to the secondary scrubber leading to an unacceptable temperature rise. To mitigate the emergency relief case 4000 l of fresh water should be charged to the primary scrubbing system before each reaction.
- The heat of solution temperature rise in primary scrubber SC100 will be in the range 32 to 36ºC for a 7.2 m^3/h liquor rate. The system should be able to handle a significant increase in liquor rate to at least 15 m3/h and this should be investigated.
- The heat of reaction temperature rise in the secondary scrubber using phosphoric acid is 20ºC for a 7.2 m3/h liquor rate and an NH3 slippage flow of 90 kg/h.
- The scrubbing system cannot handle the bursting disc rupture flow of 3018 kg/h.

For the emergency relief case the water scrubber will initially cope but significant slippage to the secondary scrubber starts as the concentration and temperature rise in the scrubber. Primary scrubber outlet temperature peaks at 68ºC, which is excessive, and measures should be taken to reduce this by increasing the liquor rate or improving heat removal capability.

2.0 PROCESS DESCRIPTION AND CONSIDERATIONS

A HAZOP study has identified the following scenarios:

1 Normal Operation

Vent down : - Release quantity 87 kg ammonia over 3 h. The release will not be linear as it is controlled by an orifice plate, so will be high initially and tail off.
Initial release rate may be approximately 6 times average, approx 170 kg/h. Inert nitrogen concentration at this point in the vessel headspace are expected to be 25 % v/v (36 % w/w) but would be quickly displaced by ammonia desorbed from solution (87 kg represents 114 m^3).

Heat up : Release quantity 102 kg over 3 h. The release will not be linear and is controlled by heat up of the batch at constant rate (0.3 ºC/min). Release rate will be highest at the start of heat up when the batch is most saturated. Initial ammonia rate will be 60 kg/h for this rate of temperature rise. Inerts will be displaced from the vessel headspace and pipework initially but would be replaced by ammonia desorbed from solution

2 Relief Case

Vessel pressure relief valve lifts giving a release rate of 430 kg/hr with calculated temperature rise across the 1st column of 14 ºC (Shasun prediction).

3 Emergency Case

The worst case source is the vessel jacket failing to full heating. An estimated flow rate of 530 kg/h ammonia with total mass release of 275 kg is proposed. This estimate uses a conservative value for the heat of solution of ammonia in IPA (4.7 kcal/mole) which has been extrapolated from hazard evaluation data. Although this flow is higher than the nominal capacity of the relief valve sufficient over pressure would be generated to drive this quantity to the scrubbing system.

4 Vessel bursting disc rupture results in a predicted flow of 3018 kg/h if the vessel is full of IPA with a calculated a temperature rise of 98 ºC.

Based on the above scenarios the study has concentrated on Scenario 3, the Emergency Case, using an NH_3 flow of 530 Kg/h with 50 kg/h N_2 inert flow and a liquor circulation rate of 7200kg/h. The Relief Case and Normal Operation results are also presented.

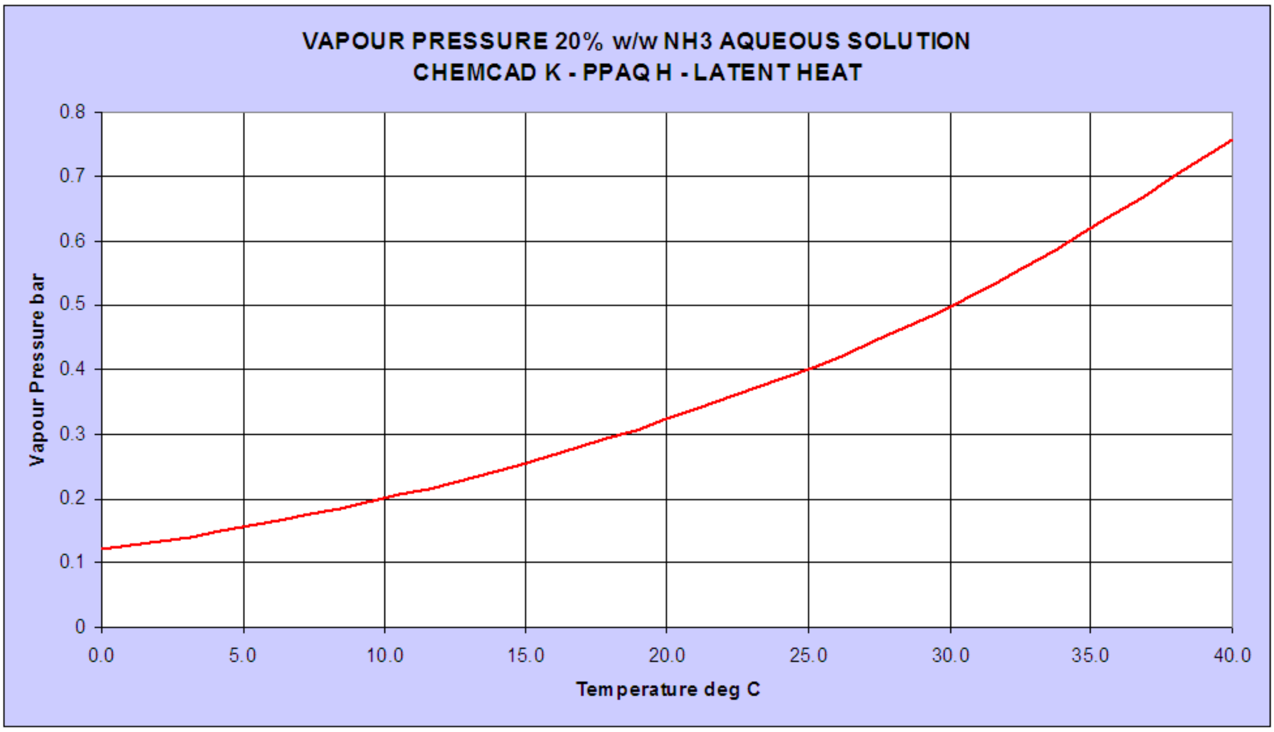
3.0 PHYSICAL PROPERTY DATA

Detailed physical property data for NH_3 and NH_3 aqueous solutions are provided in the Appendices.

CHEMCAD simulations have used K value PPAQ which includes the behaviour of NH_3 in aqueous solutions. The results compare well with the experimental data shown in the Appendices. The vapour pressure of 20% w/w NH_3 solution derived from CHEMCAD is shown below. The H value is set at Latent Heat.

This K-value method is for modelling the vapor-liquid equilibrium of compounds dissolved in water using partial pressures to calculate the equilibrium of the solute. This option is normally used for the modelling of ionic type compounds, such as HCl or HNO_3 , which dissolve in water and disassociate. The equilibrium of such systems is highly concentration dependent, that is, it is largely a function of the relative amounts of the solute and solvent present, and is not much affected by the presence of other components. As such, these mixtures do not lend themselves well to modelling by activity coefficients or other correlated procedures. The partial pressure data for the HCl-Water and NH_3 -Water systems are stored in the program.

When using the PPAQ method, the Heat of Solution method is selected for enthalpies. This will permit accounting for the heat of disassociation.

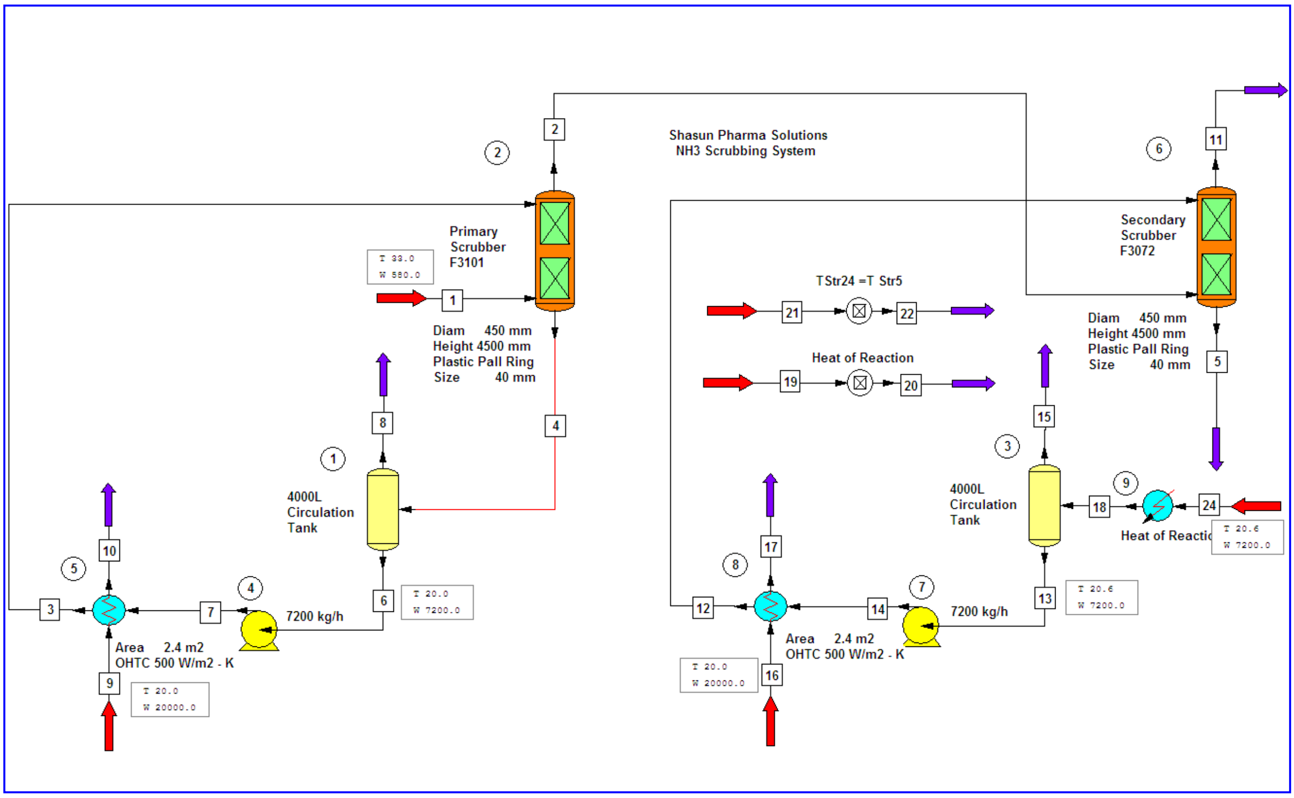


The NH_3 heat of solution in water used was 8.5 kcal / mole, being sourced from the Terra Industries data.

HSE EH40/2005 Workplace exposure limits gives LTEL (8h TWA reference period) for NH_3 as 25 ppm or 18 mg/m^3.

4.0 CHEMCAD SIMULATIONS

The CHEMCAD dynamic flowsheet for the total system is shown below:



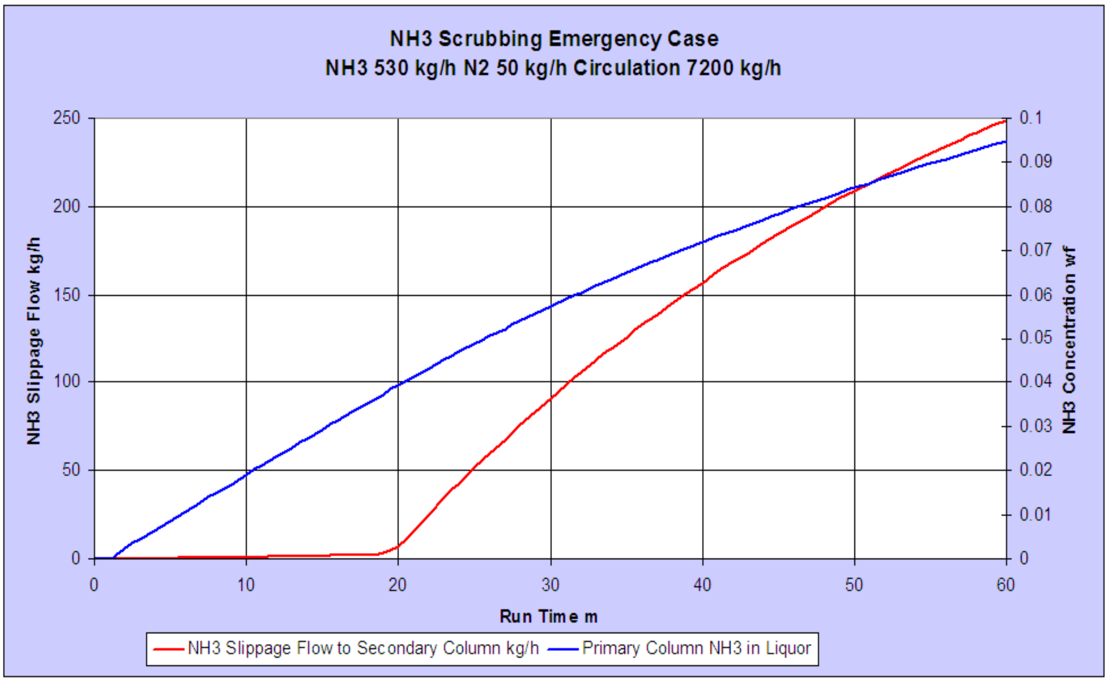
The stream parameters shown are for the initial state.

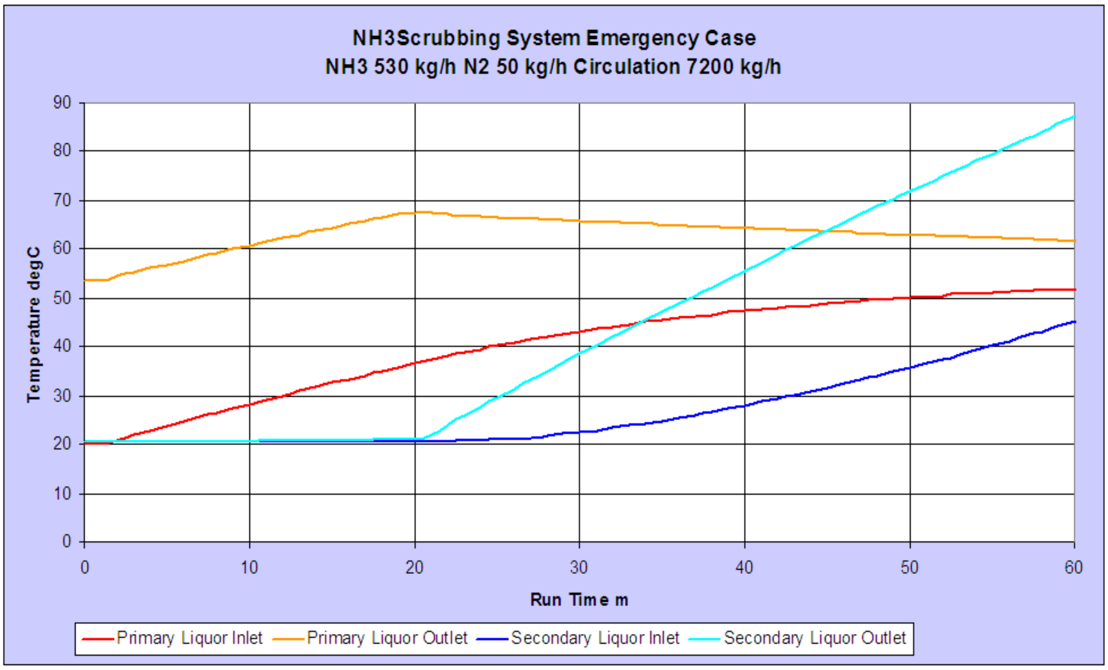
The primary tower is set up with 10 theoretical stages using water re-circulating at 7200 l/h.

The secondary tower is set up with 10 stages and uses a once through water configuration. In practice the absorption process is a fast chemical reaction, which assumes that the NH_3 concentration in the liquor is zero. CHEMCAD does not have a fast chemical reaction model so the closest approximation is a once through water set up which will give a conservative simulation. The heat balance is based on the phosphoric acid and NH_3 heat of reaction and the fresh water make up temperature is set equal to the column exit liquor temperature.

4.1 Emergency Case

These plots have been derived for the Emergency Venting scenario with the primary scrubbing system liquor concentration starting at 0.0 wf. These runs have been done with a circulation rate of 7200 kg/h. Primary and secondary heat exchanger service temperature at 20ºC, U 500 W/m^2 ºK and heat transfer area 2.5 m^2.

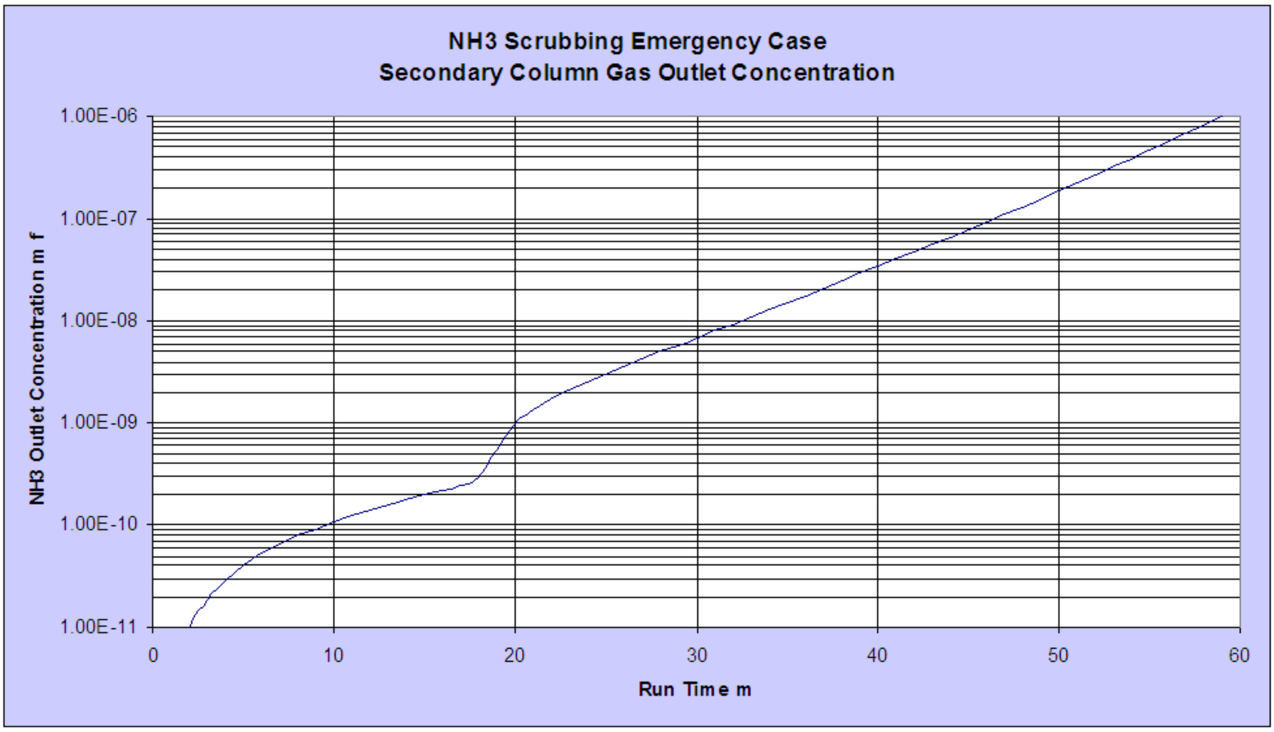




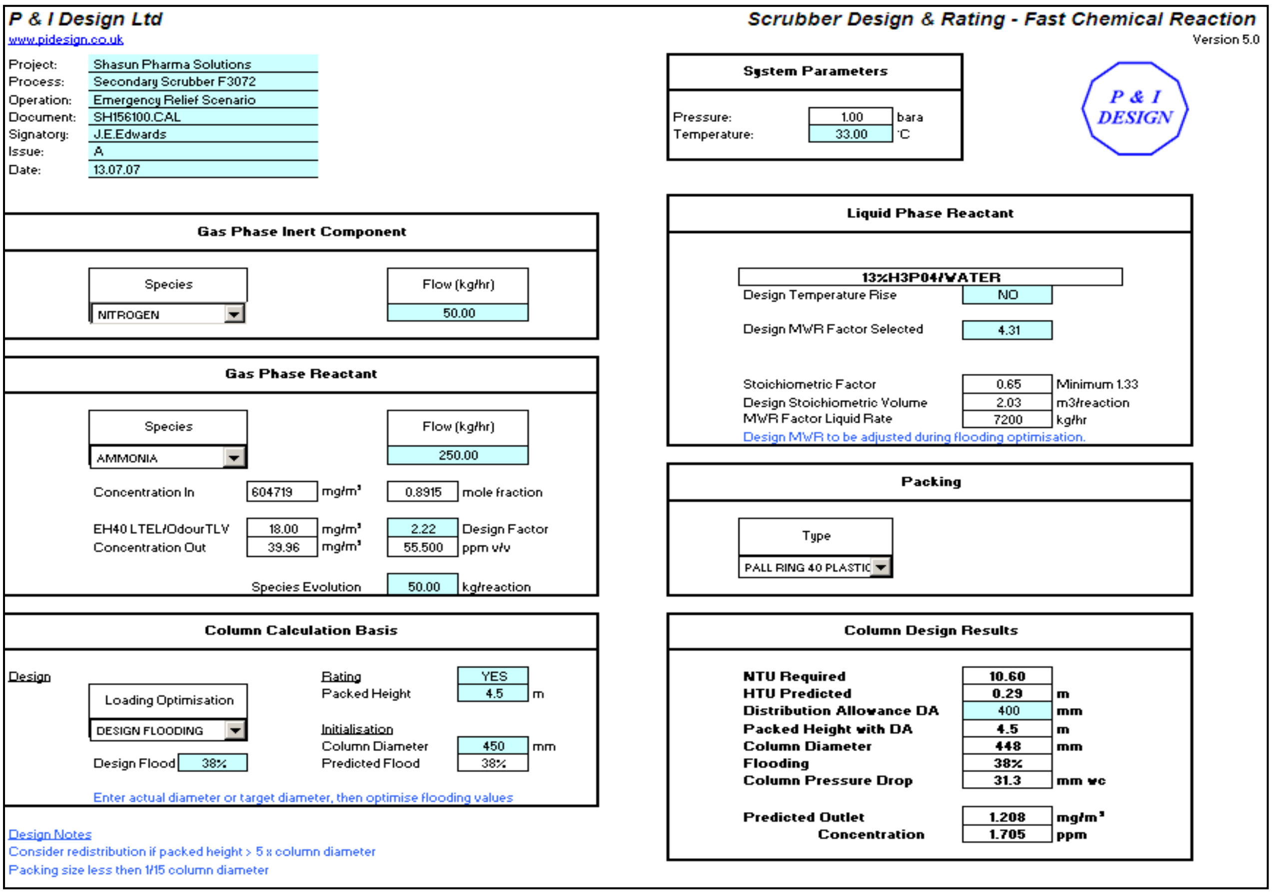
For an NH_3 charge of 275 kg the release should be complete after 31m where the slippage has reached 100 kg/h. However primary liquor outlet temperature peaks at 68ºC after 20min. This temperature can only be reduced by increasing the liquor rate, lowering the service temperature to the heat exchanger or increasing the heat exchanger HTA.

For a 7200 kg/h liquor rate CHEMCAD predicts, using PPAQ thermodynamics, a primary column temperature rise of 32ºC compared with an ENVIROKIT prediction of 36ºC.

4.1 Emergency Case (Cont.)



For an NH_3 slippage flow of 250 kg/h CHEMCAD is predicting a secondary column NH_3 outlet concentration of 1 ppm whereas ENVIROKIT is predicting 1.7 ppm. For a 7200 kg/h liquor rate CHEMCAD predicts a temperature rise of 42ºC compared with an ENVIROKIT prediction of 51ºC.



P & I Design Ltd
www.pidesign.co.uk

Scrubber Design & Rating - Fast Chemical Reaction
Version 5.0

Project:	Shasun Pharma Solutions
Process:	Secondary Scrubber F3072
Operation:	Emergency Relief Scenario
Document:	SH156100.CAL
Signatory:	J.E.Edwards
Issue:	A
Date:	13.07.07

System Parameters

Pressure:	1.00	bara
Temperature:	33.00	°C

P & I DESIGN

Gas Phase Inert Component

Species	Flow (kg/hr)
NITROGEN	50.00

Gas Phase Reactant

Species	Flow (kg/hr)
AMMONIA	250.00

Concentration In	604719	mg/m³	0.8915	mole fraction
EH40 LTEL/OdourTLV	18.00	mg/m³	2.22	Design Factor
Concentration Out	39.96	mg/m³	55.500	ppm v/v
Species Evolution			50.00	kg/reaction

Liquid Phase Reactant

13%H3PO4/WATER

Design Temperature Rise	NO	
Design MWR Factor Selected	4.31	
Stoichiometric Factor	0.65	Minimum 1.33
Design Stoichiometric Volume	2.03	m3/reaction
MWR Factor Liquid Rate	7200	kg/hr

Design MWR to be adjusted during flooding optimisation.

Packing

Type: PALL RING 40 PLASTIC

Column Calculation Basis

Design
Loading Optimisation: DESIGN FLOODING
Design Flood: 38%

Rating: YES
Packed Height: 4.5 m

Initialisation
Column Diameter: 450 mm
Predicted Flood: 38%

Enter actual diameter or target diameter, then optimise flooding values

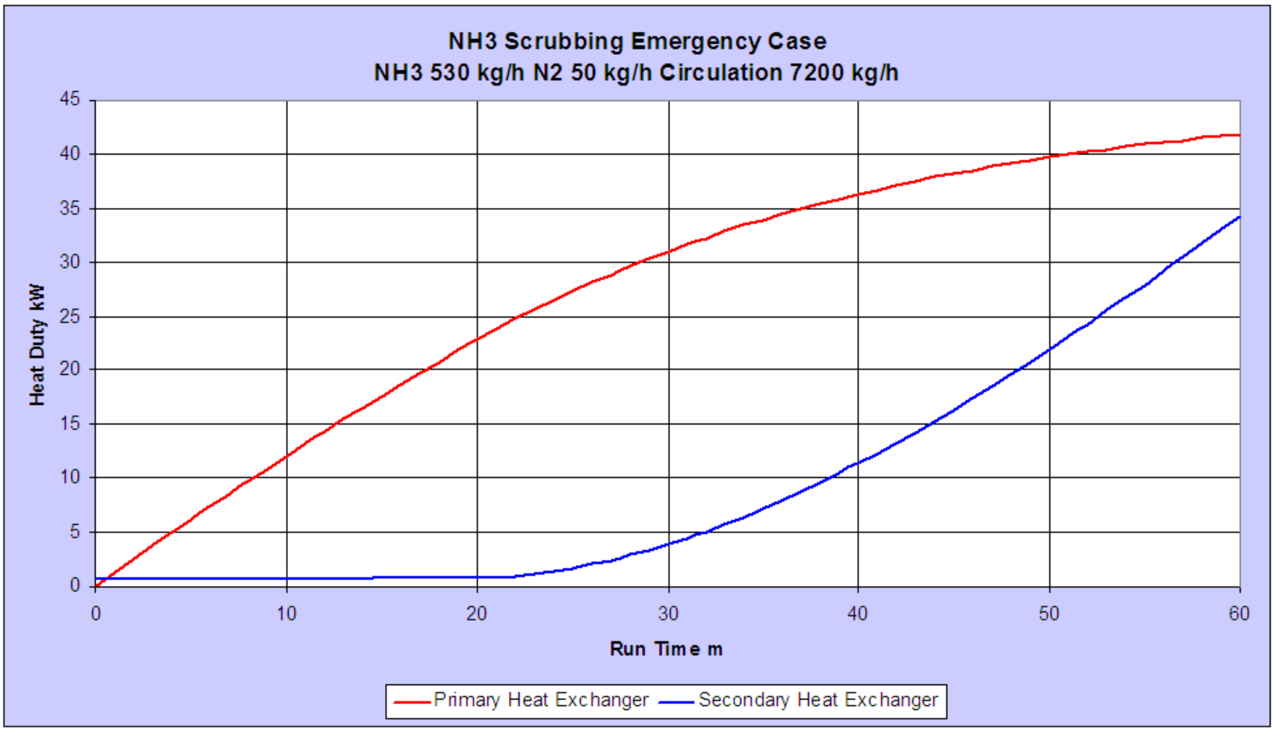
Column Design Results

NTU Required	**10.60**	
HTU Predicted	**0.29**	**m**
Distribution Allowance DA	400	**mm**
Packed Height with DA	**4.5**	**m**
Column Diameter	**448**	**mm**
Flooding	**38%**	
Column Pressure Drop	**31.3**	**mm wc**
Predicted Outlet	**1.208**	**mg/m³**
Concentration	**1.705**	**ppm**

Design Notes
Consider redistribution if packed height > 5 x column diameter
Packing size less then 1/15 column diameter

4.1 Emergency Case (Cont.)

The circulating system heat duties are shown in the plots below. Primary and secondary heat exchanger service inlet temperature at 20ºC, and flow of 20000 kg/h. The heat exchanger parameters were set at U 500 W/m^2 ºK and heat transfer area 2.5 m^2.



4.1 Emergency Case (Cont.)

The primary tower column loading was checked in CHEMCAD using the Billet-Schultes correlation as follows:

```
Primary Tower Hydraulic Data
Safety Case
Gas Input:   NH3 530 kg/h N2 50 kg/h
Liquor Rate: Water 7200 kg/h
Job Name:    NH3SCRUBRXCLIENTCASE
By:          J.E.Edwards

Billet-Schultes Correlation

Packing Parameters Plastic Pall Rings
Size                =      35.0 mm
A                   =     151.1
Void Fraction       =       0.906
Cs           =       2.72500
Cfl          =       1.74200
Ch           =       0.71800
Cp0          =       0.92700
Cl           =       0.85600
Cv           =       0.38000

Packed Tower Design for Tower 2
```

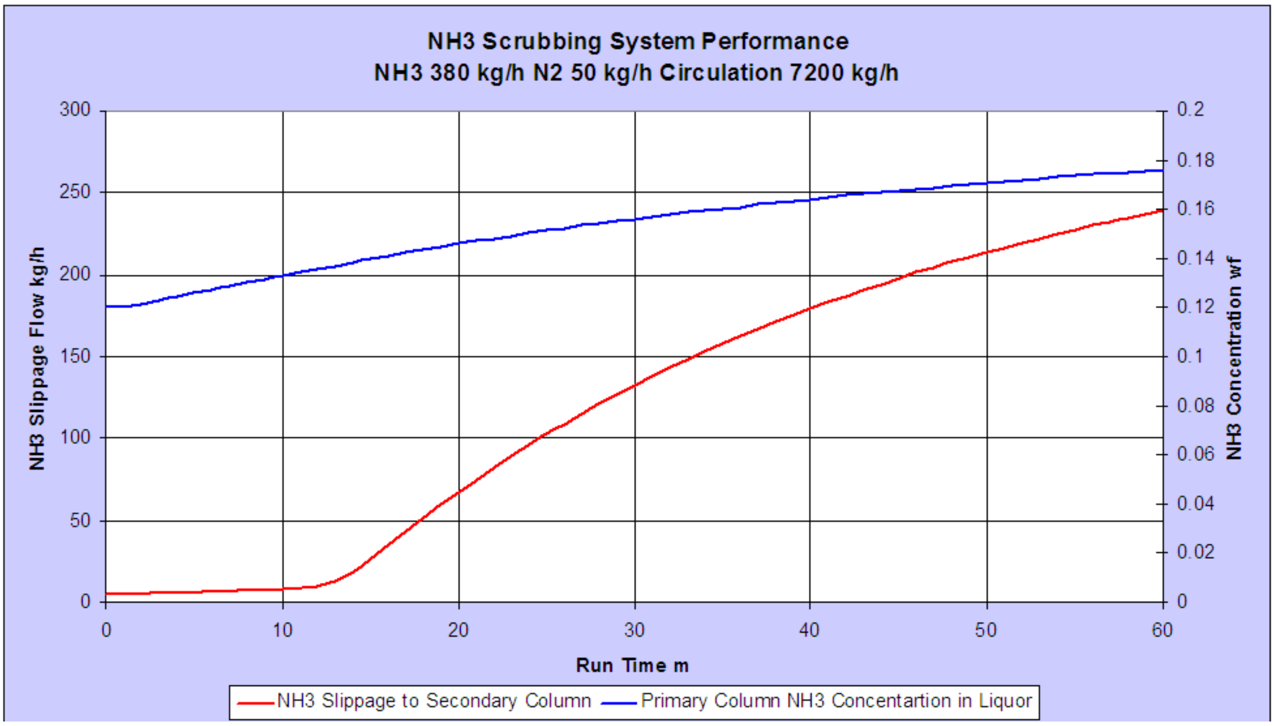
Stg	PDrop bar	% Flood	VapLoad kg/(m2*sec)	LiqLoad kg/(m2*sec)	Diam m	HTUov m
1	0.001	62.55136	0.867	12.778	0.450	0.275
2	0.001	69.01852	0.968	12.879	0.450	0.280
3	0.001	71.54134	1.007	12.918	0.450	0.275
4	0.001	72.42947	1.021	12.932	0.450	0.275
5	0.001	72.72841	1.026	12.937	0.450	0.275
6	0.001	72.82115	1.027	12.938	0.450	0.275
7	0.001	72.82972	1.027	12.938	0.450	0.274
8	0.001	72.76358	1.025	12.936	0.450	0.274
9	0.001	72.53972	1.020	12.931	0.450	0.271
10	0.001	72.61346	1.020	12.924	0.450	0.268

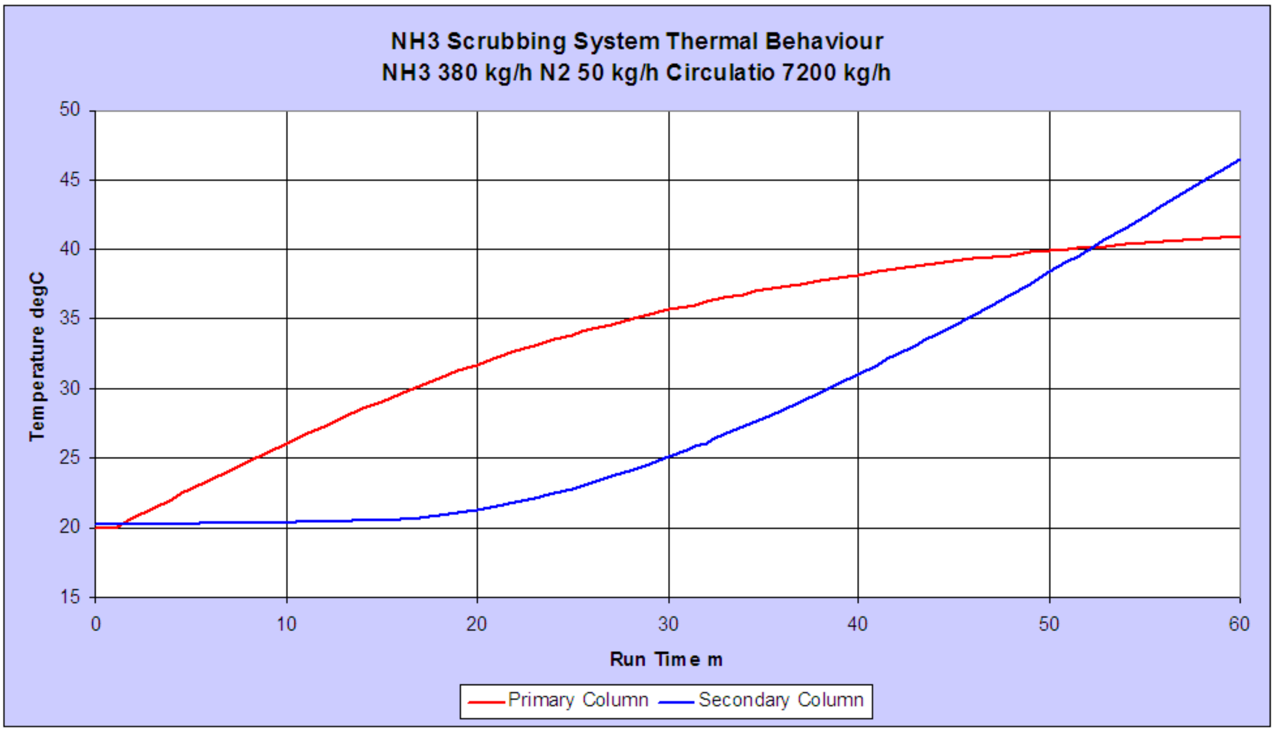
```
Overall :
 Height  m                          ................      4.000
 HETP  m                            ................      0.400
 Pressure drop  bar                 ................      0.010
 Vapor Load at Loading  kg/(m2*sec)      .......          0.955
 Vapor load at Flooding  kg/(m2*sec)      ......          1.406
 Liquid Load at Loading  kg/(m2*sec)      ......         12.343
 Liquid Load at Flooding  kg/(m2*sec)      .....         18.172
 Diameter at Loading  m                 ............      0.461
 Diameter at Flooding  m                 ...........      0.380
 Design pressure  bar               ................      1.013
```

It can be seen that the column loading peaks at 73% flood at the column inlet which is considered acceptable.

4.2 Relief Case

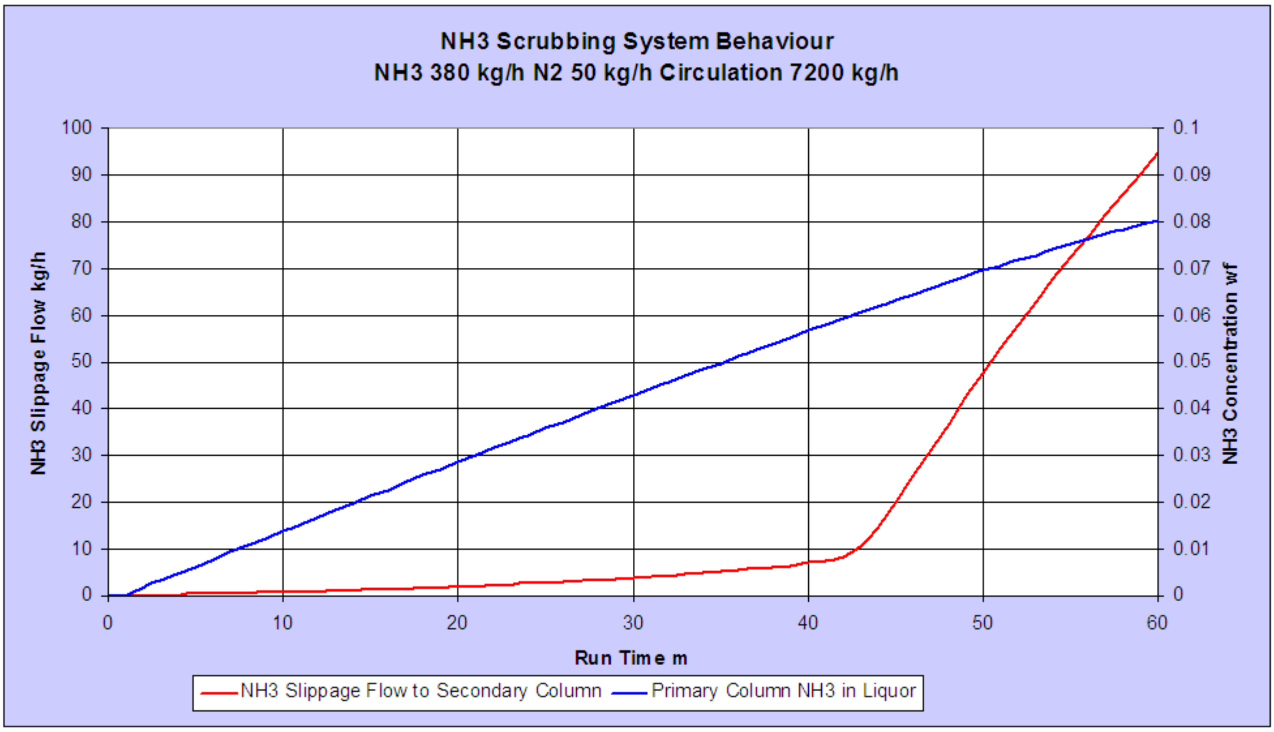
These plots have been derived for the reactor relief scenario with the primary scrubbing system liquor concentration starting at 0.12 wf. It can be seen that slippage to the secondary system takes off when the primary scrubber temperature reaches 30ºC and an NH_3 concentration of 0.15 wf. These runs have been done with a circulation rate of 7200 kg/h. Primary and secondary heat exchanger service temperature 20ºC, U 500 W/m^2 ºK and A 2.5 m^2. Primary system temperature can be seen to asymptote at 42ºC as the slippage increases.

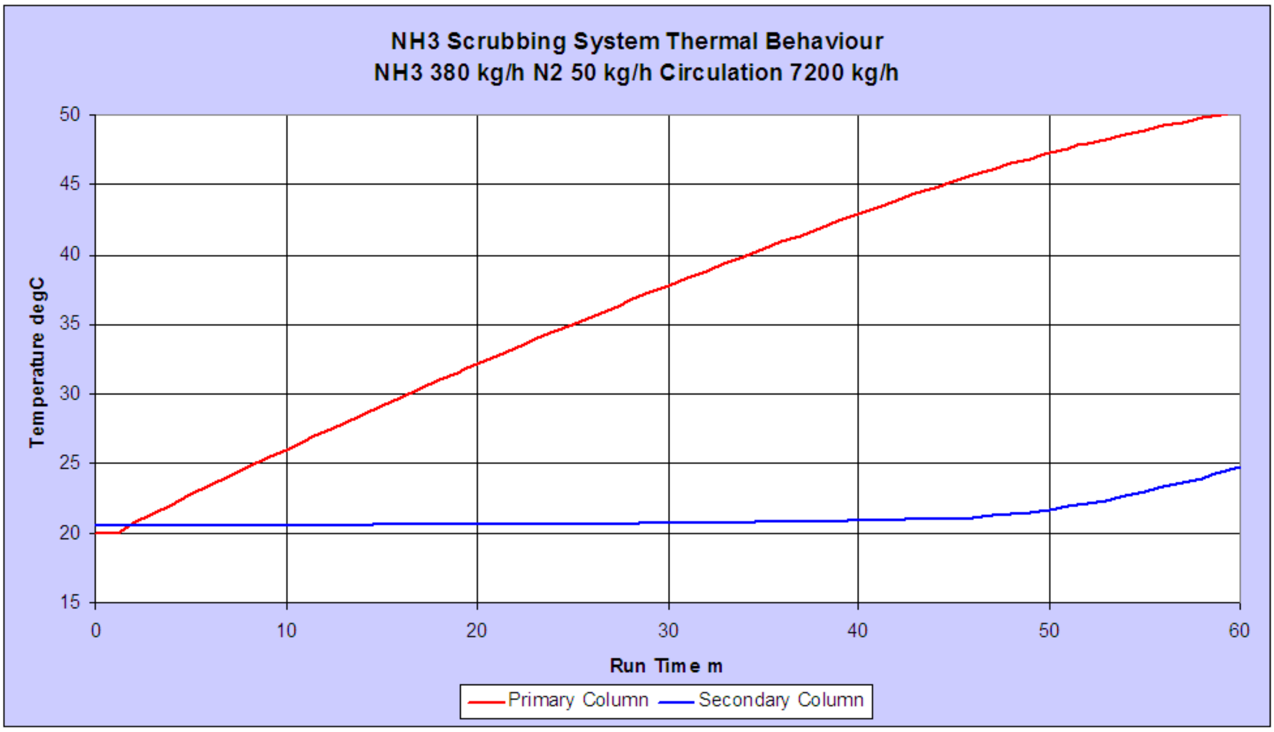




4.2 Relief Case (Cont.)

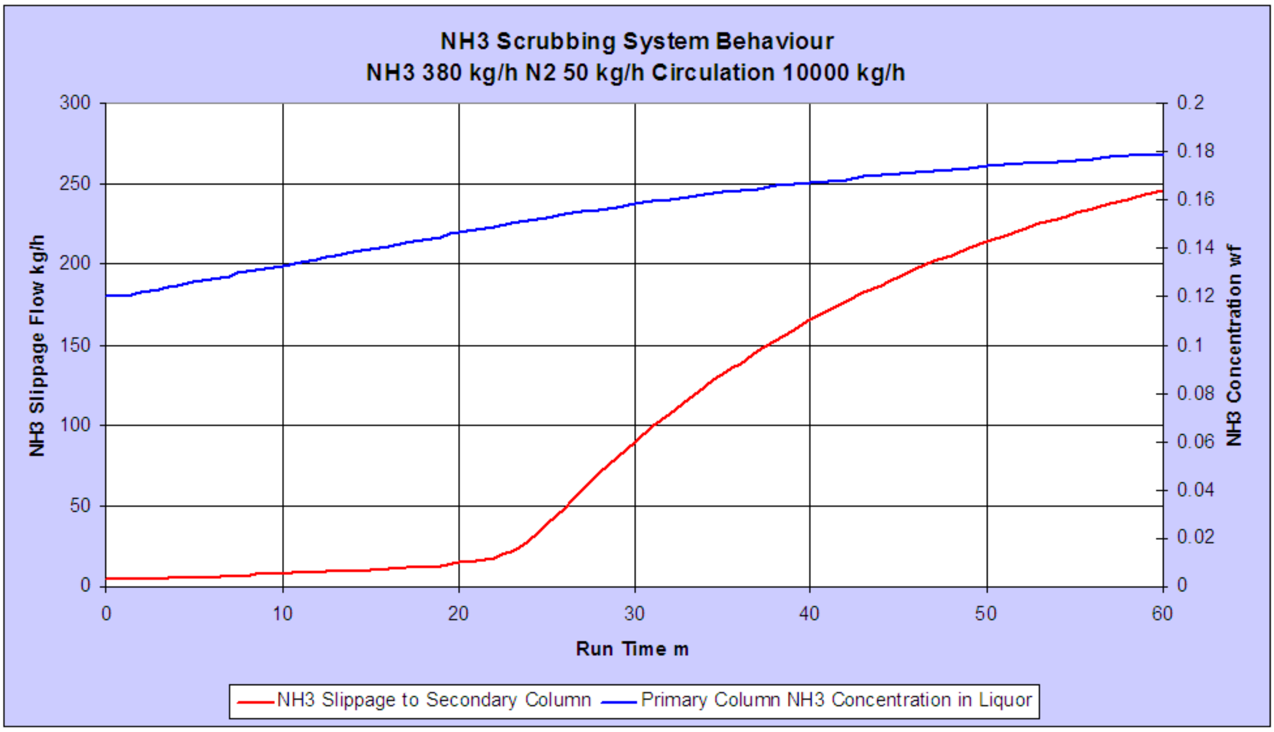
These plots have been derived for the reactor relief scenario with the primary scrubbing system liquor concentration starting at 0.0 wf. It can be seen that slippage to the secondary system is now very much reduced. However the temperature rise in the primary column is now more severe. These runs have been done with a circulation rate of 7200 kg/h. Primary and secondary heat exchanger service temperature 20ºC, U 500 W/m^2 ºK and A 2.5 m^2.

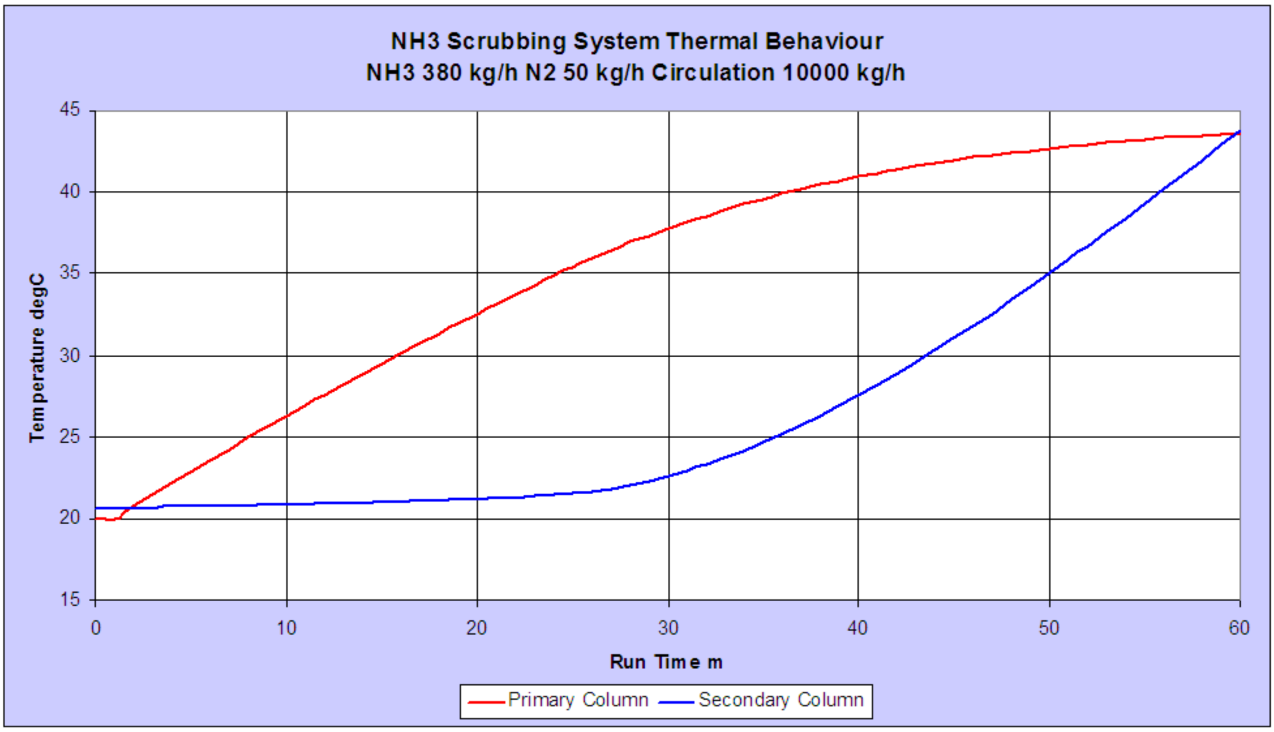




4.2 Relief Case (Cont.)

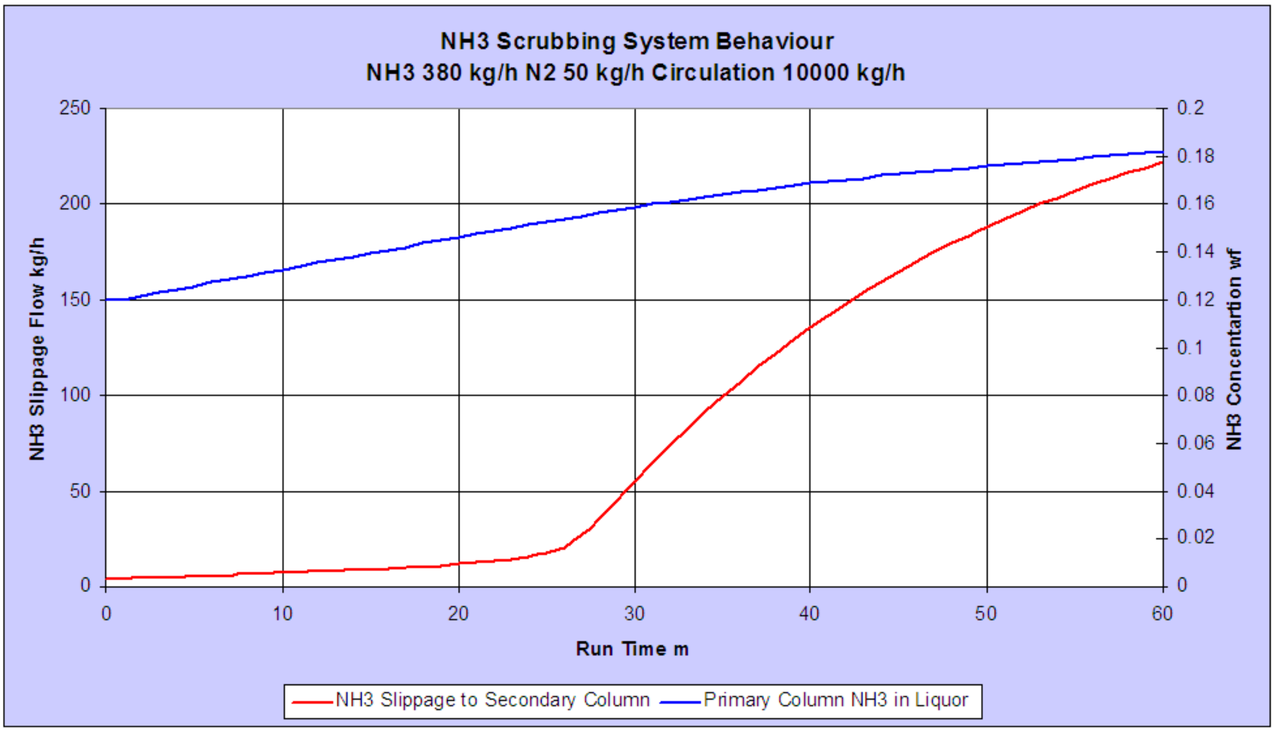
These plots have been derived for the reactor relief scenario with the primary scrubbing system liquor concentration starting at 0.12 wf. It can be seen that slippage to the secondary system takes off when the primary scrubber temperature reaches 30ºC and an NH_3 concentration of 0.15 wf. These runs have been done with a circulation rate of 10000 kg/h. Primary and secondary heat exchanger service temperature 20ºC, U 500 W/m^2 ºK and A 2.5 m^2.

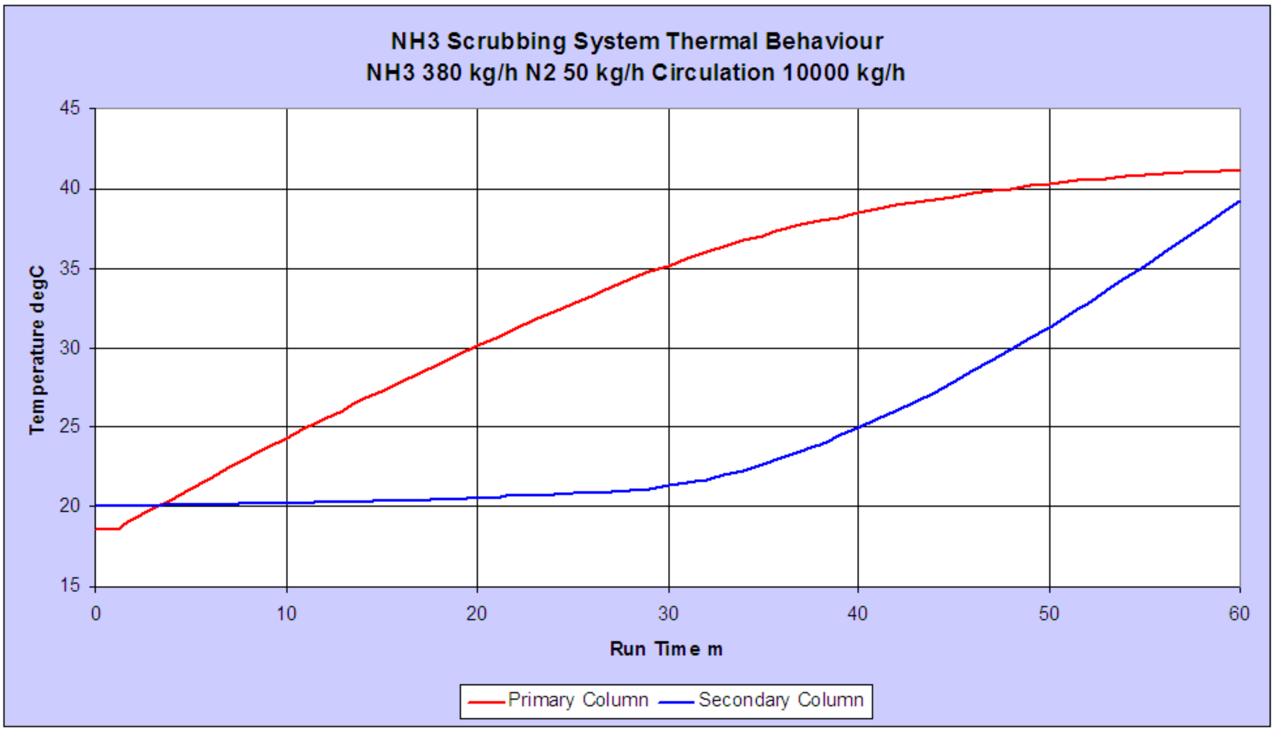




4.2 Relief Case (Cont.)

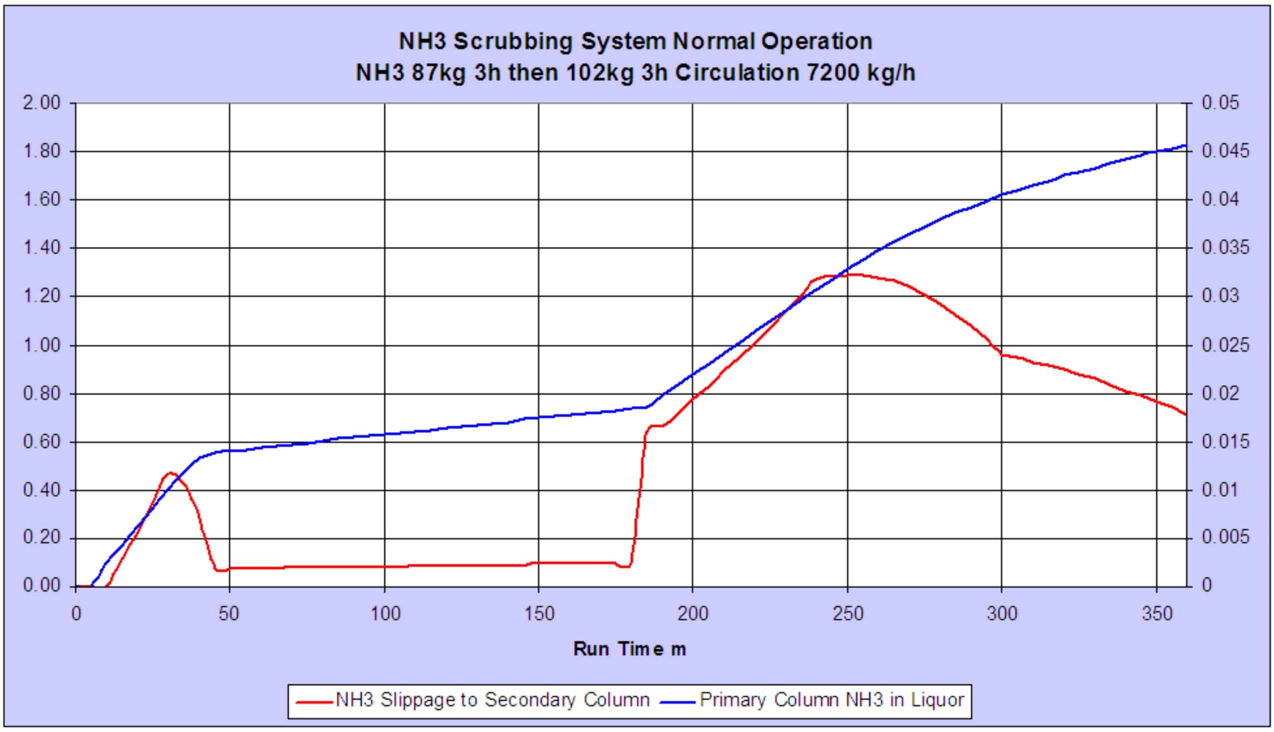
These plots have been derived for the reactor relief scenario with the primary scrubbing system liquor concentration starting at 0.12 wf. It can be seen that slippage to the secondary system takes off when the primary scrubber temperature reaches 35ºC and an NH_3 concentration of 0.16 wf. These runs have been done with a circulation rate of 10000 kg/h. Primary heat exchanger service temperature 5ºC, U 500 W/m^2 ºK and A 2.5 m^2. Secondary service temperature 20ºC.

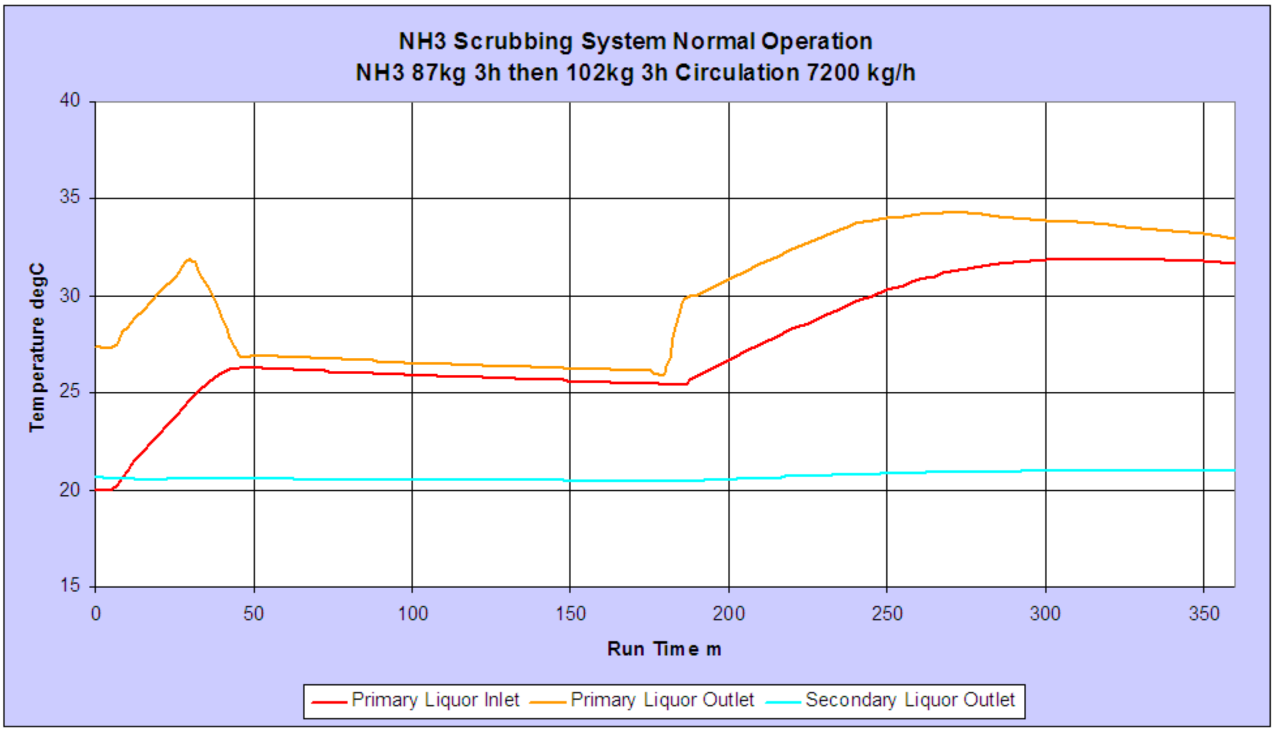




4.3 Normal Operation Case

These plots have been derived for normal operations with the primary scrubbing system liquor concentration starting at 0.0 wf. These runs have been done with a circulation rate of 7200 kg/h. The primary and secondary heat exchanger service inlet temperature 20ºC, U 500 W/m^2 ºK and A 2.5 m^2.





4.3 Normal Operation Case (Cont.)

The plot below shows the secondary scrubber gas outlet concentration reaches a peak at 2.0E-09 weight fraction equivalent to 0.002 ppm or 0.0014 mg/m^3. The second plot shows the primary column gas inlet flow.