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# 1 Fundamentals

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## 1.1 Process description

### 1.1.1 General

This process description of the KRUPP UHDE vacuum neutralization process is valid for 100% plant load when 56% nitric acid is used.

Reference drawings are:

PFD:                    D 80575-0            Process flow diagram (Vacuum Neutralization)

P & I diagram:        D 80749-0            P&I no. 301 (Vacuum Neutralization)

Ammonium nitrate solution is formed by a reaction of nitric acid and ammonia gas.

As the ammonia is available in a liquid state it first has to be vaporised and superheated in ammonia vaporizer/superheater E-10 (OSBL). After passing the safety pot it will then be fed to the reactor 31R001.

The 56 %wt. nitric acid from battery limits is fed to the reactor after being preheated in acid preheater 31E002.

In the reactor 31R001 ammonium nitrate is formed by an exothermic reaction between ammonia and nitric acid according to the following equation:




$$H_R = -1345 \text{ kJ/kg} \quad (= 578 \text{ BTU/lb})$$

The formed AN solution is then flashed in vapour separator 31D001. The product stream of the resulting solution of 91 %wt. AN is fed to the AN tank (OSBL), but most of the solution is recirculated to the reactor by circulation pump 31P001.

The flashed process vapours from vapour separator 31D001 fed to vapour scrubber 31C002 A where they are cleaned from the entrained HNO<sub>3</sub> and AN with recirculated clean condensate while passing the Thorman-trays. An additional vapour stream from battery limits is fed to the bottom part of the scrubber (31C002 B) where first the free ammonia is neutralised while passing the wetted packing before it is fed to the tray section of the scrubber.

The bottom condensate of the scrubber is mainly recirculated to the 31C002 B by scrubber pump 31P005, the surplus is sent to battery limits. The cleaned vapours are partly used to evaporate and heat the ammonia gas in ammonia vaporizer/superheater.

The main part of the vapours are totally condensed in condenser 31E001. The resulting process condensate is collected in the clean condensate tank. Part of this condensate is pumped to the top tray

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of vapour scrubber 31C002 A, another part is sent directly to battery limits. The main part of this condensate is used as make-up water in the nitric acid plant after being mixed with the bottom condensate from 31C002 B.

Spillages from the pH-measurements are fed to the AN tank, plant drains are discharged to the chemical sewer system.

## 1.1.2 Ammonia feed

### 1.1.2.1 Ammonia evaporation

**10-5**

The ammonia is available outside battery limits in a liquid state at approx. 11.5 bar abs. It enters the ammonia vaporizer/superheater E-10 via control valve PV 3100. The flashed ammonia is fed to the superheating section at the top, the remaining liquid ammonia to the bottom of the vaporizer/superheater. Here it is evaporated and superheated by use of clean process vapour from vapour scrubber 31C001. The pressure in the vaporizer is controlled at approximately 7 bar abs by PIC 3100/PV 3100 by adjusting the level in the vaporizer. **6**

The impurities (i. e. water and oil) in the liquid ammonia are drained continuously to an ammonia stripper (OSBL). By this the water content in the ammonia vaporizer is kept constant at approx. 15 %wt. The stripped off ammonia gas from ammonia stripper may be recycled into the ammonia gas line just behind ammonia vaporizer/superheater E-10 .


The ammonia vapour from the vaporizer/superheater passes through **PV 3101**, which is controlling the constant ammonia pressure at approx. 4 bar abs before the flow measurement. **3**

### 1.1.2.2 Ammonia feed gas

The heated ammonia gas at a pressure of 7 bar abs and a temperature of 60 °C is now available for the production of ammonium nitrate. To ensure stable reaction conditions the ammonia gas pressure must be kept as constant as possible. **6**

The actual ammonia flow to the reactor is measured by controller FIC 3130 and controlled by control valve FV 3130. The measurement is compensated with the pressure from PIC 3101 and the temperature from TI 3110 by FY 3130. The actual signal is compared with the set value by the controller FIC 3130 and the position of the control valve is changed accordingly. As the ammonia flow serves as master for the nitric acid flow controller FIC 3131, the set value of the ammonia flow determines the actual plant capacity.

( During normal operation a low ammonia flow trips the plant, at start-up conditions a low pressure initiates the plant trip. )

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In normal operation the consumption of ammonia is equivalent to the feed of nitric acid due to ratio control, even at acidic operation of the reaction loop. However, when the plant stops, the surplus acid of the loop will consume ammonia in the ammonia feed line, resulting in pressure reduction inside of the ammonia feed system. The loss of gas volume is replaced by ammonium nitrate solution. In order to avoid any ammonium nitrate solution flowing into the ammonia flow valve and to be able to inject steam into the reaction loop without any risk of contamination a safety pot is installed after the flow control valve FV 3130.

During a plant trip the pH of the reaction loop can be changed to neutral/alkaline by operating push button HS 3150 which opens valve HV 3150. The injection time is limited by the timer HKC 3150. 16<sup>u</sup>

### 1.1.3 Nitric Acid

Nitric acid is made available at battery limits at a pressure of 5 bar abs and a temperature of 40 °C. The design concentration is 56 %wt.

Before being used in the plant the nitric acid has to be heated to 66 °C in acid preheater 31E002. This is done by waste steam from the ejector 31J001 and fresh steam from battery limits. The acid is then fed to reactor 31R001, but may also be used for pH control of the clean condensate tank (if required by MCFI).


Nitric acid is fed as slave-flow in ratio to the ammonia gas. The required acid flow depends on the consumption of gas determined by FY 3130 and is calculated by ratio controller FY 3131. This calculation is adjusted by the pH-measurement AIC 3140 in the reactor outlet line in cascade control to guarantee a slightly acidic condition of the AN solution in the reaction loop. The nitric acid flow to reactor 31R001 is measured by FIC 3131 and controlled by control valve FV 3131.

The pressure of the incoming acid is monitored by PI 3102 at battery limits.

### 1.1.4 Reaction loop

The KRUPP UHDE forced circulation reaction loop consists of reactor 31R001, vapour separator 31D001 and circulation pump 31P001.

The feedstocks – ammonia gas and nitric acid – are fed into the reactor 31R001. In an ideal case the feedstocks should be brought to reaction in stoichiometric ratio. An exact operation at the equilibrium point, however, is not possible due to technical measuring and control reasons. In fact the pH-value would permanently oscillate between acidic and ammoniacal range. To prevent this the AN-reaction is operated in acidic condition at a pH-value of approx. 1.5. The acidic condition is chosen because otherwise the nitrogen losses would considerably increase due to free ammonia in the AN-solution and the process vapours.

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The quantity of acid needed to maintain the pH-value is determined by AIC 3140 in the reactor discharge line to vapour separator 31D001. This pH-measurement adjusts the flow controller FIC 3131 by a cascade control of the calculator FY 3131 (see 1.1.3).

The acid is distributed into circulating AN solution by four injection nozzles made from PTFE. The acid and the circulating stream of AN solution from vapour separator 31D001 are homogenised by the mixing elements of the static mixer just downstream of the injection nozzles. The ammonia sparger distributes the ammonia gas into the acidified solution through holes in the reactor tubes. Most of the spontaneous exothermic reaction takes place in the tubes and creates a moderate temperature increase.

The circulation pump 31P001 together with the flow orifice RO-3180 at the inlet nozzle of vapour separator 31D001 maintain a pressure higher than the solution vapour pressure. Therefore no flashing takes place in the reactor or in the line to the vapour separator. The heat of reaction is stored in the solution by increasing the temperature from approx. 114 °C to approx. 140 °C. In order to keep the temperature increase low a recirculation ratio of approx. 20:1 compared to the AN solution production is maintained.

At the inlet nozzle of vapour separator 31D001 the hot AN solution passes restriction orifice RO-3180. At this point the solution flashes into the vapour separator where the formed process vapours are separated from the AN solution. Most of the AN solution is circulated back to reactor 31R001 by circulation pump 31P001, the AN product stream is fed through an overflow into neutralizer II 31R002 by gravity flow.

Vapour separator 31D001 is working at a vacuum pressure of 0.45 bar abs. The water introduced to the process mainly via the aqueous nitric acid is partly vaporised by means of flash evaporation, resulting in a decrease in temperature to 114 °C and an increase in concentration to 90.5 %wt.


The well defined temperature of the flashed AN-solution and the high circulation rate results in a controlled reaction temperature and therefore avoids superheating of the ammonium nitrate.

### 1.1.5 Vapour scrubbing and condensate system

The process vapours leaving the vapour separator 31D001 are cleaned from entrained AN and HNO<sub>3</sub> by scrubbing in vapour scrubber 31C001. This scrubber consists of two parts:

The lower part (31C001 B), filled with pall rings, is used for neutralising the ammonia from a vapour stream fed in from battery limits. The scrubbing liquid is collected at the bottom of the scrubber and recirculated to the liquid distributor by scrubber pump 31P005. This flow is controlled by flow controller FIC 3135 and control valve FV 3135. The surplus of scrubbing liquid is sent to battery limits, adjusted via level controller LIC 3170 at the scrubber and control valve LV 3170.

In the upper part (31C001 A) both vapours (from BL and from 31D001) are washed with clean condensate on five Thorman type trays. These trays provide an excellent operating flexibility at a very

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good washing performance and a low pressure drop. The top tray is fed with clean condensate from the clean condensate tank/pump, adjusted by flow controller FIC 3134 and control valve FV 3134. The saturated vapour from the top tray passes a demister pad to catch entrained droplets before it is condensed.

Part of the vapours is used to evaporate and superheat the ammonia in ammonia vaporizer/superheater E-10. The rest of the vapour is condensed in condenser 31E001. From there it flows by gravity to the clean condensate tank.

The ammonia vaporizer/superheater E-10 (existing OSBL) is a vertical shell and tube heat exchanger with ammonia being evaporated and superheated on the shell side and the vapour condensing in the tubes. The condensate is collected and discharged into the clean condensate tank by gravity flow. The small amount of inerts in the process vapour is discharged to the acid heater by steam ejector 31J001.

The condenser 31E001 is a horizontal shell and tube heat exchanger. The cooling water flows on tube side, the cleaned process vapours on shell side. The vapour pressure (PIC 3103) in the vapour system consisting of 31D001 and 31C001 is determined by the condensation in the condenser. To control the condensation a small amount of air (PV 3103) is admixed to the vapour before entering the condenser. The inerts (from the feedstocks and the admixed air) are discharged to steam ejector 31J001.

The steam ejector is driven by fresh steam from battery limits. In order to use the energy of the driving steam the resulting mixture of vent gases and steam is mixed with fresh steam and sent to acid heater 31E002. The amount of fresh steam is determined by the temperature leaving the acid heater (TIC 3111) and adjusted by control valve TV 3111. The condensate from the acid heater is discharged to the clean condensate tank. As the acid heater serves as a deflector for the condensing vapour the inerts can now be discharged to atmosphere.

From the clean condensate tank the clean condensate is distributed to the top tray of vapour scrubber 31C001 A, to circulation pump 31P001, to the nitric acid plant (after mixing with the excess scrubbing liquid from 31C001 B and to battery limits.

In case of a plant trip process condensate will also be fed into the nitric acid line to reactor 31R001. Immediately after a general plant trip (31/1) check valve XV 3161 shall be opened by push button HS 3151 for about 60 seconds in order to flush the nitric acid lines. On high temperature trip (31/2) off-delay-timer XKC 3161 opens check valve XV 3161 for at least 2 minutes in order to cool down the reactor loop and dilute the circulated AN-solution. The flow is measured by magmeter FI 3137.

If the level in the clean condensate tank decreases the tank level controller LIC 3171 will close regulating valve LV 3171. Valve LV ~~3147~~ <sup>3171</sup> will also be closed on plant trip 31/1.

The flow to the nitric acid plant is measured by magmeter FIC 3136 and adjusted by control valve FV 3136. This valve is also closed on plant trip 31/1 in order to ensure that enough condensate for reactor flushing remains in the tank.

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### 1.1.6 Spillages

Drains from the reactor loop are fed into the AN tank. The remaining low drains (tanks and scrubber pump 31P005) will be discharged to the sewer system. Contaminated drains (e.g. from pump seals, reactor drip pan, etc.) are not allowed to be drained into the AN tank or the clean condensate tank.

### 1.1.7 Utilities

#### 1.1.7.1 Steam system

Low pressure saturated steam is provided at the battery limits with a temperature of approx. 160 °C and a pressure of 4.5 bar abs.

**3.5**

Low pressure steam is required for:

- steam injection into the reactor loop at shut-down and for start-up (to safety pot)
- steam ejector 31J001
- acid heater 31E002
- heating of AN tank
- Steam tracing and utility stations

*- Start up steam for NH<sub>3</sub> Vaporizer*

Particularly for start-up steam is further required to heat pipelines and equipment in order to prevent thermal shock and crystallisation.

#### 1.1.7.2 Cooling water system


Cooling water is provided at the battery limits at a temperature of 33 °C and a pressure of approx. 3.5 bar abs. The temperature of the cooling water leaving battery limits should not be higher than 43 °C.

**2.5**

Cooling water is required for the following equipment:

- Condenser 31E001
- Sample cooler 31Z003 (for pH-measurement AIC 3140)

Cooling water must be running before putting any energy/feedstocks into the plant in order to prevent damage to process equipment!

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### 1.1.7.3 Demineralized water

Demineralized water is provided at the battery limits at ambient temperature and a pressure of 2 bar g. This water is required for

- pH analysers AIC 3140 and AIC 3141
- start-up when no process condensate is available.

*Provide demin. water for start-up*

The demin. water for start-up is fed directly into the clean condensate tank, where the level is monitored by LIC 3171.


### 1.1.8 Analysers

The plant originally has two analysers for pH-measurement. The following is a list of the analysers and their function:

- pH control of the reactor loop: AT 3140
- pH control in the neutralizer II: AT 3141


Other analysers may be added later for convenience acc. to MCFI requirements (e.g. for pH control of the condensate to the nitric acid plant and battery limits or in the cooling water discharge line for monitoring possible leaks in the condenser tubes).



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## 1.2 AN Equipment Name Only List

31C002 A/B	Vapour Scrubber
31D001	Vapour Separator
31E001	Condenser
31E002	Acid Heater
E-10	NH <sub>3</sub> Vaporizer/Superheater (existing OSBL)
31J001	Steam Ejector
31P001 A/(B)	Circulation Pump
31P005 A/(B)	Scrubber Pump
31R001	AN-Neutralizer
31R002	Neutralizer II

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**1.3 Process diagrams**

**1.3.1 Process flow diagram and material balance**

PFD	Dwg. D80575-0	Process Flow Diagram Vacuum Neutralization
	Material Balance	(separate)

Stream No.	13	14	15	16	17	18	19	20
Runcase 01: incl. scrubbing unit	AN SOLUTION 31 R002 OUTLET	pH-LIQUID 31 R002 INLET	PROC. VAPOUR 31 D001 OUTLET	CLEAN VAPOUR 31 C002 AB OUTLET	PROC. VAPOUR NH3 VAPOURIZER INLET	CLEAN COND. NH3 VAPOURIZER OUTLET	PROC. VAPOUR 31 E001 INLET	PROC. COND. 31 C002 AB OUTLET
Component								
Ammonia	kg/h							
Nitric Acid	kg/h	- 0,0	0,0	3,9				85,4
Ammonium Nitrate	kg/h	9 375,0	30,6	2,4				116,3
Water	kg/h	1 041,7	71,3	4 818,4	5 566,0	1 069,4	1 065,7	4 496,6
Inerts (dry)	kg/h			5,0	5,0	1,1		3,9
Air (dry)	kg/h							
Steam	kg/h							
Steam Condensate	kg/h							
Cooling Water	kg/h							
Mass Flow total	kg/h	10 416,7	102,0	4 829,7	5 571,0	1 070,5	1 065,7	4 500,5
Volume Flow eff.	m <sup>3</sup> /h	7,4	0,1	19 073,3	22 380,1	4 300,5	1,1	18 079,6
Concentration	% wt.	90,0	30,0					1,4
Operating Temperature	°C	113,7	- 67,8	114,1	77,9	77,9	67,7	77,9
Operating Pressure	bar a	1,0	1,0	0,45	0,4	0,4	0,35	0,4
Density eff.	kg/m <sup>3</sup>	1 401,5	1 100,2	0,25	0,25	0,25	979,0	0,25
Viscosity	mPas	2,8	0,41	0,01	0,01	0,01	0,41	0,01
Enthalpy	kJ/kg	358,6	306,9	2 710,7	2 640,8	2 640,8	283,4	2 640,8

Neut II    Neut II    steam  
Separator

Condensate  
Preheater

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Stream No.	21	22	23	24	25	26	27	28
Runcase 01: incl. scrubbing unit	PROC. COND 31 C002 AB CIRCULATION	PROC. COND. TO NAPLANT	CLEAN COND. 31 E001 OUTLET	CLEAN COND. TANK OUTLET	CLEAN COND. 31 C002 AB REFLUX	CLEAN COND. EXPORT TO BL.	VAPOUR + INERTS 31 E001 OUTLET	VAPOUR + INERTS NH3 VAPOURIZER OUTLET
Component								
Ammonia kg/h								
Nitric Acid kg/h	82,6	2,8						
Ammonium Nitrate kg/h	112,4	3,9						
Water kg/h	13 500,0	464,0	4 483,5	5 970,2	650,0	5 320,2	13,1	3,7
Inerts (dry) kg/h							3,9	1,1
Air (dry) kg/h								
Steam kg/h								
Steam Condensate kg/h								
Cooling Water kg/h								
Mass Flow total kg/h	13 695,0	470,7	4 483,5	5 970,2	650,0	5 320,2	17,0	4,8
Volume Flow eff. m³/h	14,1	0,5	4,6	6,1	0,7	5,4	76,9	21,9
Concentration % wt.	1,4	1,4						
Operating Temperature °C	76,7	76,7	67,7	67,7	67,7	67,7	67,7	67,7
Operating Pressure bar a	2,1	2,8	0,35	4,0	2,1	4,0	0,35	0,35
Density eff. kg/m³	973,7	973,8	979,0	979,1	979,1	979,1	0,22	0,22
Viscosity mPas	0,37	0,37	0,41	0,41	0,41	0,41	0,01	0,01
Enthalpy kJ/kg	322,6	322,6	283,4	283,7	283,7	283,7		

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Stream No.	29	30	31	32	33	40	51	52
Runcase 01: incl. scrubbing unit	LP STEAM 31 J001 INLET	VENT GAS TO 31 E002	LP STEAM to 31 E002	STEAM / VAPOUR 31 E002 INLET	PROC. COND. 31 E002 OUTLET	PROC. VAPOUR FROM EVAPORATION	COOLING WATER 31 E001 INLET	COOLING WATER 31 E001 OUTLET
Component								
Ammonia kg/h						0,3		
Nitric Acid kg/h								
Ammonium Nitrate kg/h						0,2		
Water kg/h		116,8		421,0	407,9	548,5		
Inerts (dry) kg/h		5,0		5,0				
Air (dry) kg/h								
Steam kg/h	- 100,0		321,0					
Steam Condensate kg/h								
Cooling Water kg/h							250 500,0	250 500,0
Mass Flow total kg/h	100,0	121,8	321,0	426,0	407,9	548,9	250 500,0	250 500,0
Volume Flow eff. m³/h	42,8	212,5	137,4	749,2	0,4	2 303,3	251,8	252,7
Concentration % wt.						0,1		
Operating Temperature °C	160,0	146,7	160,0	150,0	95,0	137,8	33,0	43,0
Operating Pressure bar a	4,5	1,1	4,5	1,1	1,05	0,45	2,0	1,4
Density eff. kg/m³	2,34	0,57	2,34	0,57	961,7	0,24	994,8	991,1
Viscosity mPas	0,01	0,01	0,01	0,01	0,29	0,01	0,75	0,62
Enthalpy kJ/kg	2 770,3	2 769,0	2 770,3	2 775,6	398,0	2 756,7	138,4	180,1

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Stream No.	01	02	03	04	05	10	11	12
Runcase 02: without scrubbing unit	NITRIC ACID PLANT INLET	NITRIC ACID 31 R001 Inlet	AMMONIA GAS NH3 VAPOURIZER OUTLET	AMMONIA GAS BYPASS	AMMONIA GAS 31 R002 INLET	AN SOLUTION 31 R001 OUTLET	AN SOLUTION 31 P001 INLET	AN SOLUTION 31 D001 OUTLET
Component								
Ammonia kg/h			1 993,5	0,0	2,0			
Nitric Acid kg/h	7 381,5	7 381,5				144,9	137,4	7,4
Ammonium Nitrate kg/h						181 108,2	171 733,2	9 288,4
Water kg/h	5 799,7	5 799,7	3,0	0,0	0,0	23 753,2	17 954,7	970,3
Inerts (dry) kg/h								
Air (dry) kg/h								
Steam kg/h								
Steam Condensate kg/h								
Cooling Water kg/h								
Mass Flow total kg/h	13 181,2	13 181,2	1 996,5		2,0	204 861,5	189 687,9	10 266,1
Volume Flow eff. m³/h	10,0	10,3	453,2		0,8	148,2	135,0	7,3
Concentration % wt.	56,0	56,0	99,9	99,9	99,9	88,4	90,5	90,5
Operating Temperature °C	40,0	66,0	60,0	60,0	60,0	140,0	114,1	114,1
Operating Pressure bar a	6,0	3,5	7,0	7,0	4,0	1,4	1,15	0,45
Density eff. kg/m³	1 319,5	1 285,4	4,41	4,41	2,46	1 382,4	1 405,0	1 405,0
Viscosity mPas	1,3	0,88	0,01	0,01	0,01	2,2	2,9	2,9
Enthalpy kJ/kg	1 551,4	1 625,0	1 745,0	1 768,0	1 768,0	413,3	358,1	358,1

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Stream No.	13	14	15	16	17	18	19	20
Runcase 02: without scrubbing unit	AN SOLUTION 31 R002 OUTLET	pH-LIQUID 31 R002 INLET	PROC. VAPOUR 31 D001 OUTLET	PROC. VAPOUR	PROC. VAPOUR NH3 VAPOURIZER INLET	PROC. COND. NH3 VAPOURIZER OUTLET	PROC. VAPOUR 31 E001 INLET	
Component								
Ammonia kg/h								
Nitric Acid kg/h	- 0,0	0,0	3,9	3,9	0,8	0,8	3,0	
Ammonium Nitrate kg/h	9 375,0	30,6	2,4	2,4	0,5	0,5	1,9	
Water kg/h	1 041,7	71,3	4 818,4	4 818,4	1 041,8	1 038,2	3 776,6	
Inerts (dry) kg/h			5,0	5,0	1,1		3,9	
Air (dry) kg/h								
Steam kg/h								
Steam Condensate kg/h								
Cooling Water kg/h								
Mass Flow total kg/h	10 416,7	102,0	4 829,7	4 829,7	1 044,2	1 039,5	3 785,4	
Volume Flow eff. m³/h	7,4	0,1	19 073,3	19 073,3	4 123,9	1,1	14 949,4	
Concentration % wt.	90,0	30,0			0,1	0,1	0,1	
Operating Temperature °C	113,7	- 67,8	114,1	114,1	114,1	70,9	114,1	
Operating Pressure bar a	1,0	1,0	0,45	0,45	0,45	0,4	0,45	
Density eff. kg/m³	1 401,5	1 100,2	0,25	0,25	0,25	977,2	0,25	
Viscosity mPas	2,8	0,41	0,01	0,01	0,01	0,40	0,01	
Enthalpy kJ/kg	358,6	306,9	2 710,7	2 710,7	2 710,7	296,7	2 710,7	

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Stream No.	21	22	23	24	25	26	27	28
Runcase 02: without scrubbing unit			PROC. COND. 31 E001 OUTLET	PROC. COND. TANK OUTLET		PROC. COND. EXPORT TO B.L.	VAPOUR + INERTS 31 E001 OUTLET	VAPOUR + INERTS NH3 VAPOURIZER OUTLET
Component								
Ammonia kg/h								
Nitric Acid kg/h			3,0	3,9		3,9		
Ammonium Nitrate kg/h			1,9	2,4		2,4		
Water kg/h			3 763,5	5 209,6		5 209,6	13,1	3,6
Inerts (dry) kg/h							3,9	1,1
Air (dry) kg/h								
Steam kg/h								
Steam Condensate kg/h								
Cooling Water kg/h								
Mass Flow total kg/h			3 768,4	5 215,9		5 215,9	17,0	4,7
Volume Flow eff. m³/h			3,9	5,3		5,3	68,0	18,7
Concentration % wt.			0,1	0,1		0,1		
Operating Temperature °C			70,9	70,9		70,9	70,9	70,9
Operating Pressure bar a			0,4	4,0		4,0	0,4	0,4
Density eff. kg/m³			977,2	977,3		977,3	0,25	0,25
Viscosity mPas			0,40	0,40		0,40	0,01	0,01
Enthalpy kJ/kg			296,7	297,0		297,0		



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Runcase 02: without scrubbing unit	LP STEAM 31 J001 INLET	VENT GAS TO 31 E002	LP STEAM to 31 E002	STEAM / VAPOUR 31 E002 INLET	PROC. COND. 31 E002 OUTLET		COOLING WATER 31 E001 INLET	COOLING WATER 31 E001 OUTLET
Component								
Ammonia	kg/h							
Nitric Acid	kg/h							
Ammonium Nitrate	kg/h							
Water	kg/h	116,7		421,0	407,9			
Inerts (dry)	kg/h	5,0		5,0				
Air (dry)	kg/h							
Steam	kg/h	- 100,0	321,0					
Steam Condensate	kg/h							
Cooling Water	kg/h						243 500,0	243 500,0
Mass Flow total	kg/h	100,0	121,7	321,0	426,0	407,9	243 500,0	243 500,0
Volume Flow eff.	m³/h	42,8	212,6	137,4	749,2	0,4	244,8	245,7
Concentration	% wt.							
Operating Temperature	°C	160,0	147,2	160,0	150,0	95,0	33,0	43,0
Operating Pressure	bar a	4,5	1,1	4,5	1,1	1,05	2,0	1,4
Density eff.	kg/m³	2,34	0,57	2,34	0,57	961,7	994,8	991,1
Viscosity	mPas	0,01	0,01	0,01	0,01	0,29	0,75	0,62
Enthalpy	kJ/kg	2 770,3	2 770,0	2 770,3	2 775,6	398,0	138,4	180,1

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Stream No.	01	02	03	04	05	10	11	12
Runcase 01: incl. scrubbing unit	NITRIC ACID PLANT INLET	NITRIC ACID 31 R001 Inlet	AMMONIA GAS NH3 VAPOURIZER OUTLET	AMMONIA GAS BYPASS	AMMONIA GAS 31 R002 INLET	AN SOLUTION 31 R001 OUTLET	AN SOLUTION 31 P001 INLET	AN SOLUTION 31 D001 OUTLET
Component								
Ammonia	kg/h		1 993,5	0,0	2,0			
Nitric Acid	kg/h	7 381,5	7 381,5			144,9	137,4	7,4
Ammonium Nitrate	kg/h					181 108,2	171 733,2	9 288,4
Water	kg/h	5 799,7	5 799,7	3,0	0,0	23 753,2	17 954,7	970,3
Inerts (dry)	kg/h							
Air (dry)	kg/h							
Steam	kg/h							
Steam Condensate	kg/h							
Cooling Water	kg/h							
Mass Flow total	kg/h	13 181,2	13 181,2	1 996,5	2,0	204 861,5	189 687,9	10 266,1
Volume Flow eff.	m³/h	10,0	10,3	453,2	0,8	148,2	135,0	7,3
Concentration	% wt.	56,0	56,0	99,9	99,9	99,9	88,4	90,5
Operating Temperature	°C	40,0	66,0	60,0	60,0	60,0	140,0	114,1
Operating Pressure	bar a	<b>5</b> 6,0	<b>2.5</b> 3,5	<b>6</b> 7,0	<b>6</b> 7,0	<b>3</b> 4,0	1,4	1,15
Density eff.	kg/m³	1 319,5	1 285,4	4,41	4,41	2,46	1 382,4	1 405,0
Viscosity	mPas	1,3	0,88	0,01	0,01	0,01	2,2	2,9
Enthalpy	kJ/kg			1 745,0	1 768,0	1 768,0	413,3	358,1

Reactor


Next IT  
Reactor

Reactor

AN PUMP


Steam  
separator



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**1.3.2 P&I Diagram**

PID #301      Dwg. D80749-0      Vacuum Neutralization

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### 1.3.3 Interlocking system and diagrams

#### 1.3.3.1 General

The alarm and interlocking system is designed to protect plant and personnel from damages due to dangerous operating conditions.

If conditions arise which will lead to a dangerous operating condition, the plant will be shut down automatically and, at the same time, the necessary safety devices will be switched into operation.

Deviations from normal operating parameters will be signalled by alarms, to allow the operating personnel to take action for the required corrections.

#### 1.3.3.2 Operating

The interlocking system is a fail-safe type and operates by the closed-circuit-current principle, i.e. during normal operation all contacts are closed, all signal units and solenoids are energised. Voltage failure, cable break, intentional or unintentional interruption of a cable connection as well as opening of an interlocking contact will cause a plant shut-down.

#### 1.3.3.3 Structure

The interlocking system itself is a unit completely independent from the control system.

#### 1.3.3.4 Function of Alarms and Interlocks

Solid state type alarm systems operating on the closed-circuit (fail-safe) principle. Alarm signals are given with last-failure operating sequence as per ISA-1 and for interlocking signals with first-out sequence as per ISA-4 A.

The interlocking system is built up of electronic logic elements and operates on 24 V DC.

The first out-alarms can be acknowledged only by key-operated switches (or similar devices) to ensure that the originating alarms are reliably recognised.

For the interlocking system, provision is made for a battery-stabilised power supply device in order to compensate any transient voltage drops in the plant power mains. In the event of a total power failure, this device will ensure the power supply to instruments which are important for a smooth plant shutdown.

Remark: Alarm indication and acknowledgement can be transferred to the distributed control system (DCS) if no separate indication table for the interlock alarms is foreseen. But for safe operation of the interlocking system it is absolutely necessary that all inputs and outputs

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with interlocking functions (process values, field contacts, shut-off switches, overriding switches, solenoid valves, etc.) are directly connected to the interlocking System. Any malfunction of DCS should not effect the interlocking functions.

### 1.3.3.5 Trip System

The alarm system with switching function is subdivided into the following groups:

1. ammonium nitrate trips (IS 31/1 and IS 31/2)
2. individual interlocks (IS 31/3)

#### 1.3.3.5.1 Ammonium Nitrate Trip IS 31/1:

31/1

Reactor feedstocks

The following alarm units with switching function will actuate the ammonium nitrate trip:

- |   |   |   |
|---|---|---|
| 1 | • Emergency 'off                                | main control panel<br>HS 3152 A   |
| 2 | • Emergency 'off                                | local panel (AN-circulating pump)<br>HS 3152 B                                    |
| 3 | • Ammonia gas feed<br>pressure low              | PSL 3101  |
| 4 | • Ammonia gas feed<br>flow low                  | FSL 3130                      15 Vol. %<br>(overridable for start-up by FHS 3130) |
| 5 | • Nitric acid feed<br>flow low                  | FSL 3131                      15 Vol. %<br>(overridable for start-up by FHS 3131) |
| 6 | • AN-solution temperature high                  | TSH 3112 <i>after reactor</i>   |
| 7 | • AN-solution circulating<br>pump motor failure | ISL 3190  |
| 8 | • Instrument air                                | by MCFI   |
| 9 | <i>High Level AN Storage Tank</i>               |   |

If one of the above listed alarm units sets off an alarm the following valves will be switched to their safe position:

- ammonia flow control valve FV 3130                      close (overridable by FHS 3130)\*
- nitric acid flow control valve                      FV 3131                      close (overridable by FHS 3131)\*
- nitic acid shut down valve                      XV 3160                      close (overridable by FHS 3131)\*

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- heating steam control valve      TV 3111      close
- clean condensate control valve    LV 3171      close
- clean condensate control valve    FV 3136      close

The above mentioned valves can be restarted by means of a reset button (control room) which energises the solenoid valves.

Remark: { \*) In addition FV 3130 and FV 3131 can be operated only with time delay of <sup>15 minutes</sup> ~~9 sec~~. In case that FV 3130 or FV 3131 opens within the ~~3 sec~~, the plant will be tripped again by position switch ZSL 3130 or ZSL 3131. The controllers should be switched to manual with 0 percent output signal.

1.3.3.5.2 Ammonium Nitrate Trip IS 31/2:      **31/2**      High temperature emergency

The following alarm unit with switching function will actuate the high temperature emergency trip:

- AN-solution temperature high high      TSH 3112

If the above listed alarm unit sets off an alarm the following actions will be taken:


- ammonium nitrate trip 31/1      initiated again
- reactor flush valve      XV 3161      open (via off delay timer for 5 minutes)

1.3.3.5.3 Individual Interlocks

Remark: These interlocks can be realised as part of the fail safe interlocking system or as part of DCS.

Last alarms with switching functions

IS 31/3      Vapour Scrubber 31 C002 B  
 LSL 3170 will stop pump 31 P005 in order to prevent pump damage.  
 min. level: 20%

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Following OSBL switches are proposed by Krupp Uhde, but are in MCFI responsibility:

- IS 31/4      AN-Tank  
 Low level switch will stop AN transfer pump  
 min. level: approx. 10%
- IS 31/5      Clean Condensate Tank  
 Low level switch will stop clean condensate pump in order to prevent pump damage.  
 min. level: approx. 10%
- IS 31/6      AN-Tank  
 high temperature switch will close steam supply for tank heating.  
 max. temperature: 125 °C
- IS 31/7      Ammonia Vaporizer  
 high level switch will close NH<sub>3</sub> liquid valve PV 3100  
 max. level: approx. 85%




Trip INITIATOR	CONTROL POINT	PRE ALAR.	Trip	Trip LINK UP	Trip ACTION	PROCESS	REMARK
	INST.-NO.	INST.-NO.	INST.-NO.				

Emergency off (control room)	HS 3152 A			→ 301 →			
Emergency off (local rack @ AN circulating pump)	HS 3152 B			→ 301 →			
					→ 301 →	HV-3150 permissive	Ammonia gas bypass to Reactor
Manual trip reset				--- 301 ---			
Low pressure of ammonia gas	PIC 3101	PAL 3101	PSL 3101	→ 301 →	→ 301 →	FV-3130 closed	Ammonia gas to Reactor
Manual trip reset				--- 301 ---			
Start up override	FHS 3130			--- 301 ---			
Low flow of ammonia gas	FIC 3130	FAL 3130	FSL 3130	→ 302 →	→ 301 →	FV-3131 closed	Nitric acid to Reactor
Manual trip reset				--- 301 ---			
Start up override	FHS 3131			--- 301 ---			
Low flow of nitric acid	FIC 3131	FAL 3131	FSL 3131	→ 301 →	→ 301 →	XV-3160 closed	Nitric acid to Reactor
Manual trip reset				--- 301 ---			
High temperature of AN solution	TI 3112 A/B	TAH 3112 A/B	TSH 3112	→ 301 →	→ 301 →	FV-3136 closed	Clean cond. balance water
Manual trip reset				--- 301 ---			
Pump 31 P001 failure		IAL 3190	ISL 3190	→ x →	→ 301 →	LV-3171 closed	Clean cond. export to B.L.

					Description: Trip 31.1			AN Neutralisation MCFI	
					Reactor Feedstocks				
					Trip and Interlock Schedule				
00	24.06.97	Eb			Basic Trip Schedule	UAN 01-6207-600	Drawn: 24.06.96	Appr.:	
Rev.	Date	Name	Checked	Approved	Description	Unit 38	Checked:	Microcopied:	31/1

Trip INITIATOR	CONTROL POINT	PRE ALAR	Trip	Trip LINK UP	Trip ACTION	PROCESS	REMARK
	INST.-NO.	INST.-NO.	INST.-NO.				

Manual trip reset				----- 301 -----			
High temperature emergency trip 31/2				----- 301 ----->	TV-3111 closed	LP steam to Acid Preheater	

 <b>KRUPP UHDE</b>					<b>Description: Trip 31.1</b> Reactor Feedstocks		<b>AN Neutralisation</b> <b>MCFI</b> <b>Trip and Interlock Schedule</b>	
00	24.06.97	Eb			Basic Trip Schedule	UAN 01-6207-600	Drawn: 24.06.96	Appr.:
Rev.	Date	Name	Checked	Approved	Description	Unit 38	Checked:	Microcopied: 31/2

Trip INITIATOR	CONTROL POINT	PRE-ALARM	Trip	Trip LINK UP	Trip ACTION	PROCESS	REMARK
	INST.-NO.	INST.-NO.	INST.-NO.				

High high temperature of AN solution	TI 3112 A/B	TAH 3112 A/B	TSHH 3112		Quick closing valve XV 6113 opened	Condensate to 31R001	to delay timer XKC 3161
					Initiation of trip 31/1		

					Description: Trip 31.2		<b>AN Neutralisation MCFI</b> <b>Trip and Interlock Schedule</b>	
					High Temperature Emergency			
00	24.06.97	Eb			Basic Trip Schedule	UAN 01-6207-600	Drawn: 24.06.97	Appr.:
Rev.	Date	Name	Checked	Approved	Description	Unit 38	Checked:	Microcopied:
								31/3

Trip INITIATOR	CONTROL POINT	PRE ALAR.	Trip	Trip LINK UP	Trip ACTION	PROCESS	REMARK
	INST.-NO.	INST.-NO.	INST.-NO.				
Low level in vapour scrubber 31C002 B	LIC 3170	LAL 3170	LSL 3170		Pump 31P005 stopped		
Low level in AN tank	LIC 31xx	LAL 31xx	LSL 31xx		AN transfer pump stopped	AN solution to NPK plant	(proposed)
Low level in celan cond. tank	LIC 3171	LAL 3171	LSL 3171		Clean cond. pump stopped		(proposed)
High temperature in AN tank	TIC 31xx	TAH 31xx	TSH 31xx		Heating steam to AN tank stopped		(proposed)
High level in Ammonia Vaporizer	LI 31xx	LAH 31xx	LSH 31xx				(proposed)

					<b>Description:</b> Trip 31.3 / 31.4 31.5 / 31.6 31.7		<b>AN Neutralisation</b> <b>MCFI</b> <b>Trip and Interlock Schedule</b>	
					<b>Miscellaneous</b>			
00	24.06.97	Eb			Basic Trip Schedule	UAN 01-6207-600	Drawn: 24.06.97	Appr.:
Rev.	Date	Name	Checked	Approved	Description	Unit 38	Checked:	Microcopied:
								31/4