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FVO Nr. **3208**

Preliminary Process Design
Department for Chemical Process Technology

Subject

Purification of waste water with low concentration of ammonium nitrate using freeze concentration and eutectic freeze crystallization

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Waste water (purification), ammonium nitrate, freeze concentration, eutectic freeze crystallization

Date of Assignment : 2nd of October, 1997
Date of Report : 2nd of January, 1998

TUDelft
Technische Universiteit Delft

Faculteit der Scheikundige Technologie en der Materiaalkunde

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SUMMARY

During the production of ammonium nitrate at the industrial chemical company, Kemira Agro Rozenburg B.V., a waste stream of 130 000 ton p.a. of dilute (<0.5%) ammonium nitrate brine is produced. This project aims to design a preliminary process design for the upgrading of this waste stream into high purity water and concentrated ammonium nitrate.

With an operational cost analysis evaluation between the available technologies of reverse osmosis, freeze concentration and evaporation for concentrating brine, a choice was made for freeze concentration. The freeze concentration produces a more concentrated ammonium nitrate brine and ice. The concentrated ammonium nitrate brine is crystallised using eutectic freeze crystallisation to produce solid ammonium nitrate and ice. The ice is washed in wash columns and melted producing high purity water.

From the waste stream the process design produces 884 tons of highly concentrated (90%) ammonium nitrate and 129 000 m^3 of high purity water per annum. No by-products are produced. Process integration has produced benefits to the ammonium nitrate and nitric acid processes at Kemira. Economically the process requires a total investment of 7.4 M Nfl. (including subsidies 3.7 M Nfl), has a pay-out time of 60 years and a rate of return of -2.93 %

The process is technically feasible and the process design is considered quite sound. However, as such the project is not economically viable. In hindsight the high investment costs of freeze concentration probably make reverse osmosis the better alternative for the concentration step which would reduce the process costs considerably (approximately 25 %). Still, with the given basis of design the major bottleneck appears to be the low revenues from products. The revenues are insufficient for the investments required and thus the economic feasibility of upgrading the waste stream without subsidies.

At Kemira the combination of reverse osmosis with eutectic freeze crystallization appears to be a positive option for which it surely is worthwile examining further.

CONTENTS

Appendices

1 INTRODUCTION

The industrial chemical company, Kemira, situated in Rozenburg, The Netherlands, has industrial scale installations for the production of urea, ammonium nitrate and nitric acid. In the production process of ammonium nitrate, which is used in the production of fertilizers, a wastewater stream of approximately 130,000 ton/year, containing low concentrations of ammonium nitrate (<1%) is produced. Currently this wastewater is dumped via the sewer to the local environment. Kemira wishes to eliminate this wastewater stream in the near future.

Separating the wastewater stream has a number of benefits for Kemira:

1. Kemira cuts the costs of dumping the wastewater in the sewer.
2. Continuity of the process is improved in reducing environmental load.
3. A considerable amount of pure water is produced which is suitable to send through the ionexchanger. The recycling of water results in a decrease in the total water consumption of the plant.
4. A small amount of ammonium nitrate is retrieved which is high enough in concentration to fulfill product demands.

The aim of this project is to eliminate the wastewater stream by separating the stream into pure water (< 50 ppm AN) and highly concentrated ammonium nitrate (>90%). It is important that the treatment of the wastewater is integrated into the industrial processes on location to improve economic viability.

Several industrial technologies are available to separate the water and ammonium nitrate. These methods include reverse osmosis, evaporation and freeze concentration. The ammonium nitrate concentrating with reverse osmosis is limited by the maximum pressure difference across the membrane. In practice a maximal concentration of 12.5 w% of ammonium nitrate can be attained (see appendix IV h). This being the case a combination of technologies is necessary to achieve high (>90%w) ammonium nitrate concentrations. Two steps can be identified in the process, a concentrating (removal of large bulk of water) and a purification step (product >90%w ammonium nitrate). After comparing costs of different technologies for each step the optimal technologies can be chosen.

This project aims to utilize the new technology of eutectic freeze crystallization (EFC) to separate the wastewater stream. EFC is currently part of the research field of the Laboratory for Process Equipment at Delft University of Technology. EFC is becoming a more viable purification method in recent years as the specific implementation technology improves. With the background of EFC at the Laboratory for Process Equipment, EFC will be used as the purification step.

The quantity of ammonium nitrate produced is quite small in comparison to the production from the ammonium nitrate installation at Kemira. The quantity of ammonium nitrate produced can be added to the current production of ammonium nitrate without significant impact on the total quantity of ammonium nitrate produced and without affecting downline processing. More important are the costs saved by not dumping the ammonium nitrate to the sewer. Also, the high purity water produced is used in the nitric acid plant and reduces the total amount of water consumed by Kemira. Furthermore, the heating for the vaporization of ammonia is no longer required as the ammonia is vaporized in the purification process.

Upgrading a waste stream, which is currently released via the sewer is a benefit to the environment and as the government regulations regarding release of waste streams tighten the cost of releasing the waste stream may increase. In this perspective the process appears to have a positive future.

2 BASIS OF DESIGN

Kemira has specified a wastewater flow of 16,230 kg/h, with 1242 mg/kg of ammonium nitrate to be treated (for details of the basis of design see appendix I).

The wastewater treatment produces 129,080 ton/yr water and 884 ton/yr ammonium nitrate. The yearly operating hours are 8000 hrs. The plant life is estimated at 20 years.

2.1 Basis of Process

A flow scheme of the total treatment process is given in figure 2.1.

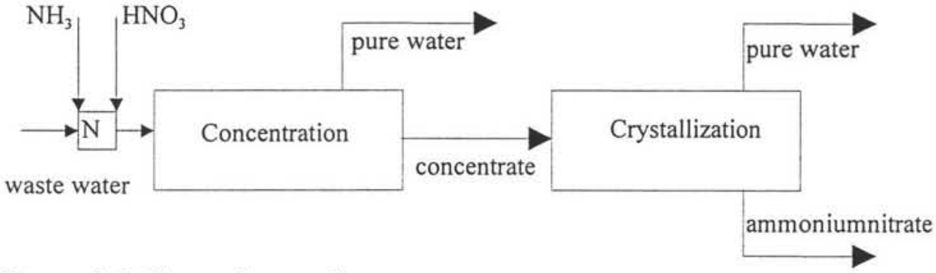


Figure 2.1 Flow scheme of treatment process.

The wastewater stream is continuous such that the treatment facility will be designed as a continuous process. A list of chemical properties is given in appendix II b.

During the purification the reactions taking place are:

$HNO_3(aq) + NH_3(aq) \rightarrow NH_4^+(aq) + NO_3^-(aq)$ ($\Delta H_{reaction}$= -340 kJ/mol)
$NH_4^+ (aq) + NO_3^-(aq) \rightarrow NH_4HNO_3 (s)$ ($\Delta H_{reaction}$= -321 kJ/mol)

Direct crystallisation is not practical because of a too large and too much diluted stream so concentrating of the waste stream is required. There are a variety of technologies available for the concentration step. These include:

a) reverse osmosis
b) freeze concentration
c) evaporation

a) Reverse osmosis:
Reverse osmosis can be used to reach concentrations which are more suitable for EFC, reducing the quantity of water needing to be cooled. Reverse osmosis allows concentrating of a dilute solution up to a limiting concentration which is determined by the maximum pressure difference possible across the membrane and water activity coefficient of the brine. Major costs are incurred in the regular replacing of membranes and this should be taken into account when comparing with freeze concentration and evaporation.
In preliminary cost calculations the costs of regularly having to replace the membranes has been taken into account of the operating costs by converting the costs of replacement into energy equivalents (kW/Nfl). In this manner a comparison can be made with the alternative technologies.

b) Freeze concentration:
Freeze concentration can be used to freeze out a quantity of water such that concentrations are reached that are more suitable for EFC. Freeze concentration need not operate at as low a temperature as EFC. However for higher concentrations a lower temperature required and the compression costs become higher.

c) Evaporation
To make a complete comparison of alternatives for concentration, evaporation is also taken into account.

The concentration step is followed by eutectic freeze crystallisation (EFC) or evaporation. Both EFC and evaporation have the advantage of separating the components water and ammonium nitrate in their pure form. The eutectic temperature of the ammonium nitrate-water system determines the operating temperature of the crystalliser.

2.2 Choice of Design

A choice of which combination is best suitable is made after a preliminary comparison of energy costs of different combinations (see table 2.1 and 2.2). Although total evaporation is not an economically favourable option, it is calculated to create a more complete view. Furthermore it has to be mentioned that the membrane (capital) investment costs have been converted to energy consumption to be able to make a comparison (thus not energy consumed by membrane).

Table 2.1: Comparison of energy consumption

Method	**Energy Consumption (kW)**				
	Concentration		**Crystallisation**		**Total**
	membrane	**Compressor**	**Compressor**	**Thermal ***	
RO+EFC	113	79	19		211
FC+EFC		187	14		201
RO+evap	113	79		260	279
2 stage evap				5965	1988
Evap+EFC			14	5705	1915

*Converted to electrical energy (electrical = 1/3x thermal)

Table 2.2 : Comparison of costs (in the case of Kemira)

Method	**Costs of energy consumption Nfl p.a.**				
	Concentration		**Crystallisation**		**Total**
	Membrane	**Compressor**	**Compressor**	**Thermal**	
RO+EFC	90 000	64 000	15 000		169 000
FC+EFC		150 000	11 000		161 000
RO+evap	90 000	64 000	0	69 000	223 000
2 stage evap			0	1591 000	1591 000
Evap+EFC			11 000	1521 000	1532 000

On the basis of this comparison a choice has been made to design a treatment installation for Kemira with a combination of freeze concentration and eutectic freeze crystallisation. Due to the low cooling costs at Kemira, freezing concentration is the most viable technology for the concentrating step.
At Kemira ammonia feed has to be evaporated for the ammonium nitrate plant. Utilising the cooling energy of this stream the cooling costs are very low in comparison to normal cooling. Realising that the cooling energy available at Kemira makes the freeze concentration the most economical, in cases where this cooling energy is not available, reverse osmosis is a more economical alternative.

2.3 Specification Feedstock and Products

In table 2.3 the specifications of the incoming and outcoming streams are given. For a complete overview of the 'basis of design' see appendix I. For an overview of chemical properties see appendix II b.

Table 2.3: specification incoming and outcoming streams

Incoming Stream	**Composition**	**Pressure (bara)**	**Temperature (°C)**	
NH_3 (l) HNO_3 Waste water	99.8 %, 0.02% water 57.5 %, 42.5% water	22.5 14 9	19 40 66	
Outcoming Stream	**Composition**	**Pressure (bara)**	**Temperature (°C)**	**Demands**
Ammonium Nitrate	90 %	2	95	
Highly pure water	100 % water	7	20	< 50 ppm AN < 10 ppm HNO_3
Waste water to HNO_3 plant	0.3 % AN, 99.7 % water	11	<40	
NH_3 (g) to AN plant	100 % NH_3	4.2	30-60	

2.4 Specification of Utilities

The utilities used in the process are limited to low-pressure steam and electricity.

Low-pressure steam, used in the dissolver, is specified by:
T = 190 °C
p = 3 bar
T_{cond} = 133,5 °C

Specifications of electricity:
Alternating current = 220 V
Three phase alternating current = 380 V
Three phase alternating current (larger power) = 10,000 V
The price of electricity at Kemira is 0.1 Nfl/kWh

2.5 Product Destination

The highly pure water is sent to the nitric acid plant and a demineralizing plant for use as demi water on the industrial site at Kemira. The ammonium nitrate (>90 w %) produced can easily be added to the ammonium nitrate stockpile from the ammonium nitrate plant. The majority of the ammonium nitrate produced at Kemira is used to increase the nitrogen content of artificial fertilisers.

2.6 Costs and Benefits

The following table gives an economic consideration of the costs and benefits of the process. Also a margin for the plant installation is calculated.

Table 2.5: cost and benefits

Costs	Nfl	Benefits	Nfl
Energy	126 000	AN	189 000
Ammonia	31 000	Water	453 000
		Sewer*	414 000
Subtotal	157 000		1 056 000
Margin	899 000		
Turnover	1 056 000		1 056 000

*Costs cut by not releasing wastewater to sewer

2.7 Plant Location

The ammonium nitrate plant of Kemira, Rozenburg, is situated on the same site as the ammonia, nitric acid and urea plants of Kemira. This enables a partial integration of the processes. Apart from the wastewater stream the main feedstocks are liquid ammonia and nitric acid. These are both produced on the neighbouring plants of Kemira and available in sufficient quantities.

The ammonium nitrate plant of Kemira is situated in Rozenburg, the Netherlands, which is part of the Rotterdam Industrial harbour network. The location is accessible for freight via rail, road and water transport. Safety and environmental considerations have to be made with regard to the location being part of an intensive industrial area and situated on the river Maas. Situated in an industrial area the population density surrounding the plant location is relatively low.

3 PROCESS STRUCTURE AND PROCESS FLOWSHEET

3.1 Background to Process Structure

The aim of this project is to produce highly pure water and highly concentrated ammonium nitrate from very diluted ammonium nitrate brine. From the preliminary cost analysis it has been determined that this is best achieved using freeze concentration followed by eutectic freeze crystallisation. Freeze concentration is possible up to the eutectic concentration of 42.3 w% ammonium nitrate in solution, further freeze concentrating leads to eutectic freeze crystallisation where solid ammonium nitrate is formed but the concentration of ammonium nitrate in solution remains the same. To achieve 90% or higher ammonium nitrate concentrations requires eutectic freeze crystallisation and it follows that the process consists of a freeze concentration step followed by eutectic freeze crystallisation.

3.1.1 Relationship between major Units

The freeze concentration (FC) and eutectic freeze crystallisation (EFC) units largely determine the structure of the process. The ice produced in the FC and EFC is separated from the ammonium nitrate brine in separate wash columns. The ice leaving the top of the wash columns is melted and is of high purity. The ammonium nitrate brine leaving the bottom of the wash columns is recycled back to the FC and EFC units. The EFC unit not only produces ice but solid ammonium nitrate as well, the ice being less dense than the brine leaves at the top of the EFC unit and the solid ammonium nitrate being more dense than the brine leaves at the bottom.

3.1.2 Restrictions and Requirements

To be able to transport the solids (ice and ammonium nitrate) without special pumps, slurries of no more than 20% solids are used. This means 80% brine is required to transport the ice produced in the FC and EFC, and the 80% brine to the wash columns has to be recycled back to the FC and EFC units. The ammonium nitrate brine transporting the solid ammonium nitrate is recovered in a filter and also recycled to the EFC unit.

Due to 80% brine that is required to transport the ice and the fixed operating ammonium nitrate concentration of the FC, the ice from the FC and EFC cannot be separated from the ammonium nitrate brine in the same wash column. Too much ammonium nitrate in solution is lost from the EFC in transporting the ice, such that the ammonium nitrate cannot accumulate and reach the eutectic concentration of 42.3 w% ammonium nitrate in the EFC without affecting the concentration in the FC. The concentration ammonium nitrate in the FC would have to be higher than 22 w% ammonium nitrate to use the same wash column for FC and EFC. A high ammonium nitrate concentration in the FC would require cooling to a much lower temperature in the FC unit for ice formation This would increase the cooling costs considerably. The degrees of freedom of the process when using one wash column are not sufficient to also specify the ammonium nitrate concentration in the FC unit.

The cooling costs are largely determined by the costs of recompressing the ammonia to 4.2 bara. Cooling to a lower temperature requires flashing of the ammonia to a lower pressure and more work for recompressing.

The FC and EFC units are operated at a pressure of 1 bara because at a higher pressure ice has a lower freezing point and so it is more difficult to produce ice. The operating pressure for the hydraulic wash columns is slightly higher than 1 bara.

Since a large liquid ammonia stream is available for cooling by evaporating, this ammonia stream is used for the cooling. The ammonia runs through a separate cooling process, which is integrated with the process only by means of heatexchanging. For the cooling of the FC the ammonia is flashed to 2.6 bara and for the EFC to 1.7 bara and respective temperatures of -13°C and –22°C.

The FC unit consists of a scraped surface heat exchanger (SSHE) and a recrystalliser in which the smaller ice crystals are converted to larger crystals. A recycle to the SSHE of brine is required to increase the amount of ice crystals in the recrystalliser. The SSHE makes it possible to transfer large amounts of cooling energy, which would be difficult in a tank because of ice formation at the cooling surface. In the SSHE the cooling surface is scraped and the solid ice formed on the cooling surface removed. Another advantage of this type of crystal growth and the reason why this process has been chosen is the production of relatively large ice crystals which can be washed efficiently in the wash column.

For the EFC unit a cooling disc crystalliser column (CDCC) is used because it is capable of large heat transfer in one crystalliser unit. The ice and solid ammonium nitrate can be separated in the EFC unit by withdrawal at the top and bottom respectively.

3.2 Thermodynamics

The reaction scheme is relatively simple with water and ammonium nitrate as the important components, however several ions are formed in solution. The reactions taking place in the process are listed below.

$NH_4NO_3(s) \Leftrightarrow NH_4^+(aq) + NO_3^-(aq)$ (1) Ionisation/formation of solid ammonium nitrate

$\Delta H_{cryst}=321 kJ/mol$

$NH_3(l) + H_2O(l) \Leftrightarrow NH_4^+(aq) + OH^-(aq)$ (2) Ionisation of ammonia

$HNO_3(l) \Leftrightarrow H^+(aq) + NO_3^-(aq)$ (3) Ionisation of nitric acid

$H_2O(l) \Leftrightarrow H^+(aq) + OH^-(aq)$ (4) Hydrate/hydroxide equilibrium

The formation of solid ammonium nitrate (right to left of equation (1)) only takes place when the ammonium nitrate brine is saturated. This occurs in the EFC unit, where at -16.9 °C the solution is saturated at 42.3 w% ammonium nitrate. See figure 3.1 for a phase diagram of ammonium nitrate solution and ice. This diagram shows that a ammonium nitrate solution of 90 % will be reached at 95 °C.

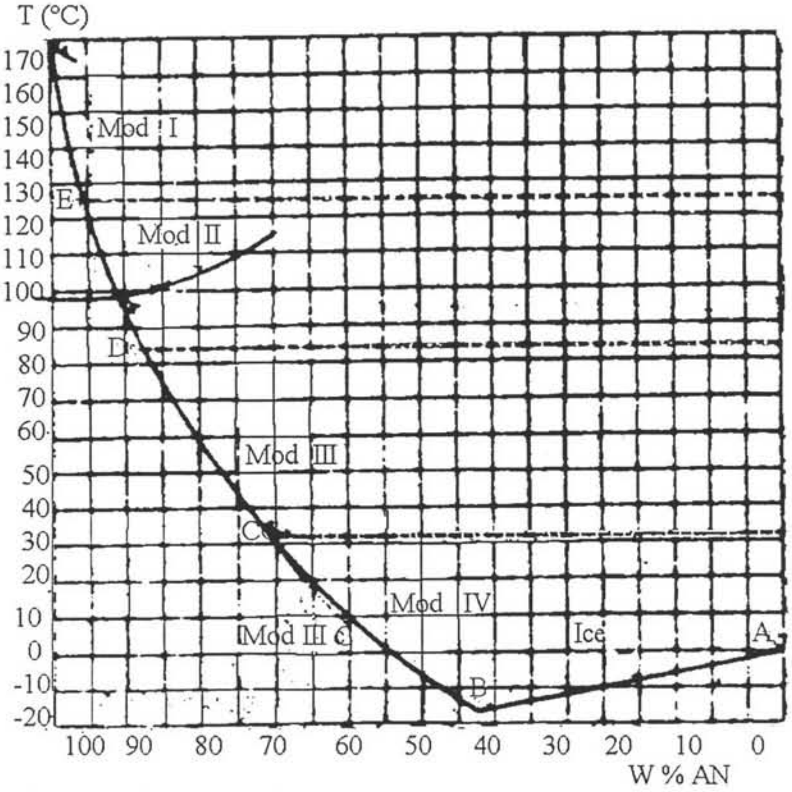


Figure 3.1: Eutectic diagram of water-ice-ammonium nitrate system

3.3 Motivation of Equipment selection

3.3.1 Optimisation of the combination FC and EFC

The freeze concentration unit (FC) and the eutectic freeze crystallisation (EFC) unit are interdependent. The ice produced in the FC unit, does not have to be produced in the EFC unit. A cost optimisation is necessary to obtain the best weight fraction of ammonium nitrate in solution that leaves the FC unit. In figure 3.2 the weight fraction ammonium nitrate is plotted against the water remaining in the brine. From this figure it is easy to see how much water has to be frozen in the FC and in the EFC unit when fixing the weight fraction ammonium nitrate coming from the FC unit.

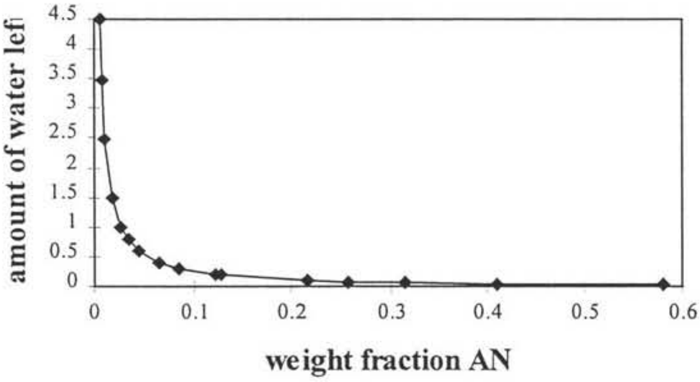


Figure 3.2: Remaining water in brine plotted against weight fraction ammonium nitrate

For the ammonium nitrate plant the ammonia is required at a pressure of 4.2 bara and a temperature of 60 °C. After evaporation of ammonia for cooling in the FC and EFC units, the ammonia has to be recompressed. The FC compression costs are based on the amount of ammonia that is needed to freeze the water and the temperature of ammonia. The ammonia temperature has to be about ten degrees lower than the freezing point of the solution leaving the FC unit. The higher the fraction ammonium nitrate that leaves the FC unit, the lower the freezing temperature because of 'depression of freezing point'. As a consequence the compression costs become higher. For EFC the temperature is fixed at the eutectic temperature and the compression costs are only determined by the amount of water that still has to be frozen out of the brine. In figure 3.3 the weight fraction ammonium nitrate leaving the FC is plotted against the compression costs.

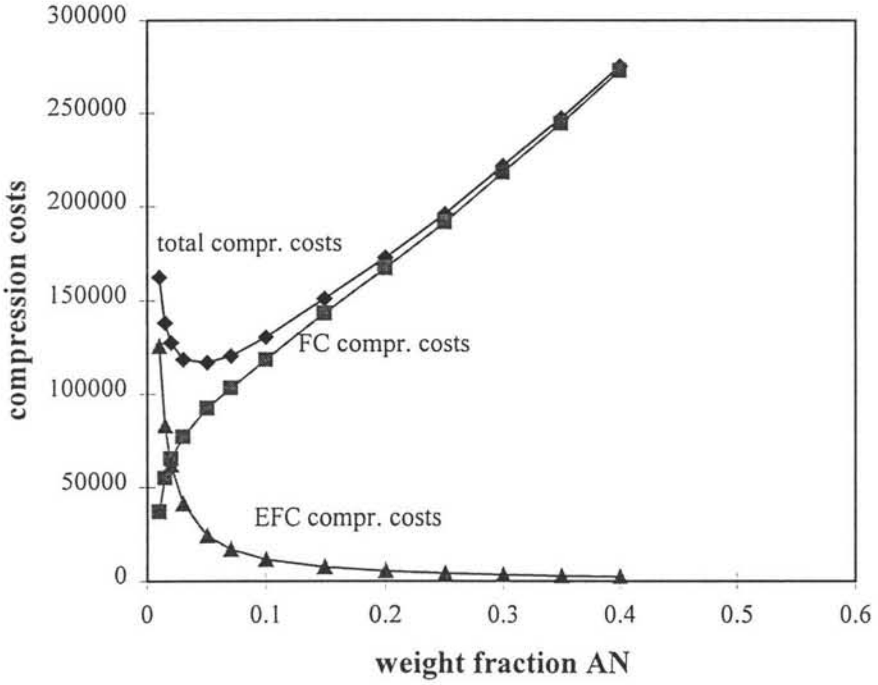


Figure 3.3: Compression costs optimisation

In calculating the compression costs the depression of freezing point is taken into account. Due to the depression of freezing point the compression work per kg ammonia and the heat of vaporation per kg ammonia changes. This can be seen in the Mollier diagram in appendix III. The values used in figure 3.3 can be found in appendix V a.

As can be seen in figure 3.2. there is minimum in costs at a weight fraction of 0.05 (5 w%). Although the minimum running cost has been determined at 5 w%, in the design 7 w% has been chosen because this reduces the size of the EFC unit considerably and only increases the FC marginally with respect to relative size. Also due to the added complexity in EFC the investment costs can be lowered by taking 7 w% as basis of design.

3.3.2 Freeze Concentration

Two types of freeze concentration units with indirect cooling are available for industrial scale. These are a standard crystal growth vessel and the combination of a scraped surface heat exchanger (SSHE) with a recrystalliser.

Crystal Growth Vessel

In the crystal growth vessel the cooling capacity is limited by the build-up of ice on the cooling surface. Ice build-up on the cooling surface reduces the heat transfer coefficient and thus the heat transferred. A scraping mechanism, scraping the cooling surface is installed to prevent the ice build up. However, scale up is limited due to the decrease in heat exchanging area to volume ratio as the tank size increases.

Scraped Surface Heat Exchanger (SSHE) and Recrystalliser

In the SSHE the brine is undercooled and in the recrystalliser most of the crystal growth takes place. To avoid ice build-up on cooling surfaces the FC unit consists of a scraped surface heat exchanger (SSHE) and a recrystalliser tank in which small ice crystals are converted to larger crystals. A recycle of brine from the recrystalliser to the SSHE is required to remove sufficient heat for the growth of the ice crystals in the recrystalliser. The SSHE makes it possible to transfer large amounts of cooling energy, which would be difficult in a tank because of ice formation in the unit and at the cooling surface.

The SSHE is in principle a heat exchanger with a scraping mechanism to keep the cooling surface free of ice. The scrapers in the SSHE and the high superficial velocity of the brine along the cooling interface in the SSHE prevent ice formation in the unit. See figure 3.4 for a diagram of the SSHE. Leaving the SSHE is an undercooled brine with small ice crystals (some nucleation does take place). In the recrystalliser the energy of the undercooling of the brine and the small ice crystals are converted into larger ice crystals which may leave as product. A recycle from the recrystalliser to the SSHE is fairly large to maintain high superficial velocities in the SSHE, the high superficial velocity of brine helps prevent ice build-up. The recycle is filtered from the recrystalliser such that the ice crystals remain behind.

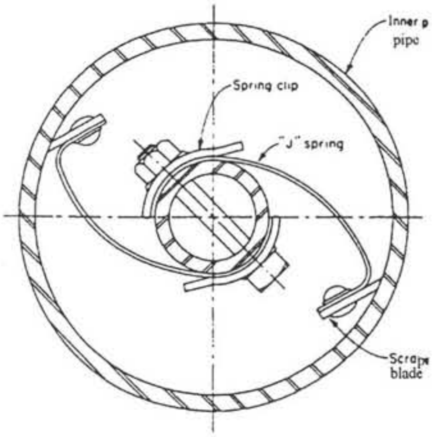


Figure 3.4: Cross sectional view of Scraped Surface Heat Exchanger

Another advantage of this type of crystal growth is the relatively large ice crystals, approximately 0.2 mm, which are produced. Larger sized ice crystals can be washed more efficiently in the wash column. The installation of SSHE units makes it possible to provide sufficient cooling without problems of ice build up on the cooling surface. The SSHE with recrystalliser has already been implemented on industrial scales of up to 35 ton/hr ice production (NIRO, 33). See figure 3.5 for build up of FC with SSHE and recrystalliser.

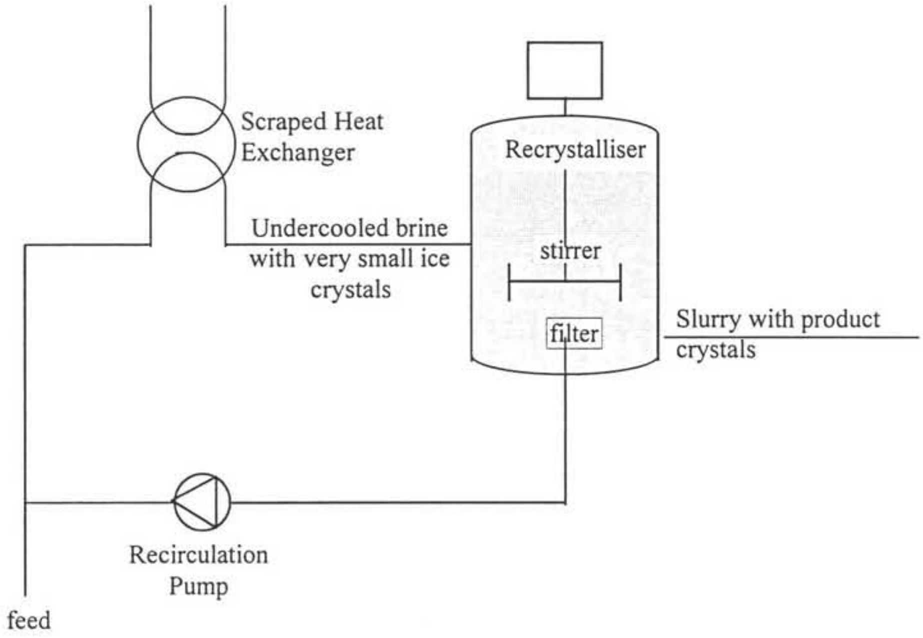


Figure 3.5: Schematic of freeze concentration with SSHE and recrystalliser

As previously mentioned a major advantage of the recrystalliser is the product crystal size. The difference in equilibrium temperature of small and large ice crystals in the recrystalliser provides the driving force for the melting of the smaller and the growth of larger ice crystals. See figure V.1 in appendix V b (Arkenbout, 16).

3.3.3 Eutectic Freeze Crystallisation

During the EFC process the following phase change and reaction take place:

water (l) → ice (s) $\Delta H = -335$ kJ/kg (1)
$NH_4^+ (aq) + NO_3^- (aq) \rightarrow NH_4NO_3 (s)$ $\Delta H = -321$ kJ/kg (2)

In the EFC slow 'suspension cooling' takes place. This means that nucleation and crystal growth of ice and ammonium nitrate are combined in one vessel. These operating conditions are possible in a Cooling Disc Column Crystalliser (CDCC) (van der Ham, 1). In the CDCC the eutectic temperature is maintained. Ice and solid ammonium nitrate crystals are produced. Due to difference in density, the ice crystals float to the top of the column and the ammonium nitrate crystals sink to the bottom. Figure 3.6 shows a schematic representation of as CDCC. In the CDCC the cooling discs are mounted with scrapers on both sides (in figure 3.6A only one scraper is shown).

In the discs ammonia evaporates at a temperature of 251 K to withdraw the heat from the brine. The scrapers prevent ice formation on the cooling discs (some nucleation does occur however) the suspension becomes slightly undercooled which causes the ice crystals to grow in the suspension. Orifices in the cooling discs allow the ice crystals to rise and the ammonium nitrate crystals to settle to the top and bottom of the column respectively.

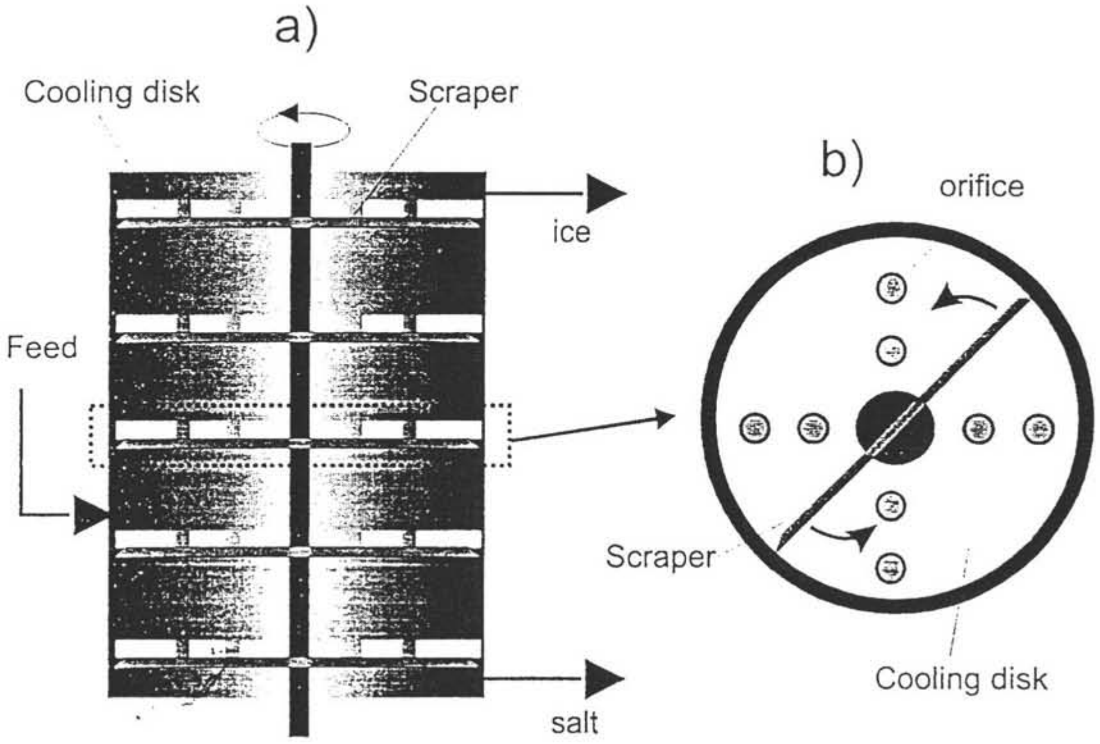


Figure 3.6: A) side view of a Cooling Disc Column Crystalliser B) top view of a disc in the CDCC.

The CDCC is not yet commercially available but is still in development at the Laboratory for Process Equipment at Delft University of Technology. There are still some bottlenecks in this unit. There is no ripening tank as in the FC unit. This makes the crystal size control difficult. Also the CDCC is a crystalliser and a settler in one, this means the residence time must be large enough to ensure that the crystals reach the top or bottom of the column within the residence time.

Problems arise in calculating the settling and rise time with Stoke's law. The stirring of the scrapers and the baffling of the discs increase the settling and rise times. Despite these limitations Stoke's law is still used to make an estimate of the minimum rise and settling times.

3.3.4 Wash Column

From the FC and EFC slurries with 20 w% ice crystals and 80 w% brine are produced. The ice crystals have to be separated from the brine and the mother liquor adhered to the surface of the ice crystals removed. Separate wash columns are used for the ice slurries from the FC and EFC unit. These wash columns separate the ice crystals from the mother liquor and wash the ice crystals. The mother liquor is sent back to either the FC or the EFC, in accordance with where the slurry came from. At the top of the wash column the washed ice crystals are melted and high purity water is the product. A part of this melted ice is recycled to the wash column as wash water and reslurry-water.

There are two types of countercurrent wash columns: gravity transport columns and forced transport columns. The gravity transport columns are not practical in this process because ice has a lower density than water and floats. The gravity transport column is based on a crystal mass with a higher density than water. The forced transport columns are discussed further.

There are two main types of forced transport wash columns: mechanical and hydraulic.
In a 'mechanical' column the transport of the bed is effected by mechanical means such as a piston or a screw. In a 'hydraulic' column the driving force for crystal transport is exerted by mother liquor flowing through the crystal bed.
In appendix V c, the screw type, the piston columns are discussed in general. The hydraulic type wil be discussed in the following.

The principles in the various types of wash columns are the same: the slurry is fed to the column, where a packed ice bed forms. The mother liquor is separated from the ice crystals and the ice bed is washed countercurrently with recycled melt. At the top the ice bed is scraped off and melted.

The equilibrium temperature of the suspension feed is substantially lower than the equilibrium temperature in the reslurry section at the top of the column where the crystals are in contact with pure water. The heat required for raising the temperature of the ice crystals when passing through the washing zone is withdrawn from the crystallisation of the washing liquid in the wash zone. The crystallisation of the washing liquid results in a decrease in the porosity (volume fraction of liquid in the bed) of the packed bed. Consequently, very high temperature and concentration gradients occur at the wash-front.

The flow of wash liquid is controlled in such a way that the wash-front is located in the washing zone about halfway in between the filters and the reslurry section. The wash-front usually has a thickness of about 10-20 mm.

Wash Columns in this Process

In this process, a continuous feedstream of ice crystals has to be treated in a wash column. A hydraulic wash column has been chosen. This type of column does not exert large forces on the crystals and can handle small crystals better than the piston bed and screw type wash columns. The operation of the hydraulic wash column is explained in the following text, for more details on the operation of the piston bed and screw type refer to appendix V c.

Hydraulic Wash Column

As mentioned in the explanation in appendix V c, the screw conveyor in the screw type wash column exerts a large force on the crystal bed. The hydraulic wash column is developed to overcome this disadvantage (extra demands on design). An example of a hydraulic wash column is the TNO-Thijssen column, which is illustrated in figure 3.7 (Arkenbout, 16).

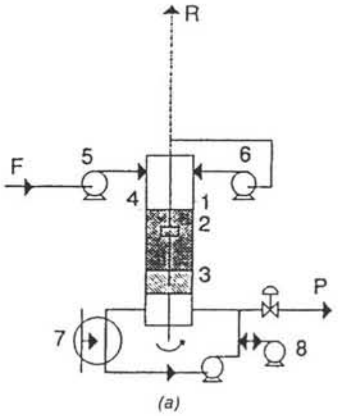


Figure 3.7: TNO-Thijssen Hydraulic Wash Column
1-wash-column; 2-tube(s) containing filter; 3-wash-front; 4-level crystal bed; 5-feed pump; 6-recirculation of filtrate for control purposes; 7-melting circuit; 8-liquid pulse device to reduce wall friction resistance.

This wash column operates continuously. The feed slurry enters at the top and is transported downward. In this column tubes with filters are installed to drain the mother liquor. The mother liquor flows through the filters and a packed crystal bed is formed. The crystal bed moves further downward to the scraper where the crystals are scraped off and sent to a melting circuit. Melted crystals are recycled to the column as wash liquid.

In the hydraulic wash column two zones in the crystal bed can be identified;

- between the feed side and the filters where the ice crystals and feed move cocurrently.
- between the reslurry section and the filters with the liquid moving upwards and the crystal bed downwards.

The hydraulic wash column in figure 3.7 is based on crystals with a density higher than the mother liquor. In this process the ice crystals have a density lower than the mother liquor (ammonium nitrate brine) and will rise.

The hydraulic ice wash column is the upside down version of the general hydraulic wash column. This implies that the feed will enter at the bottom and the scraper is installed at the top of the wash column, see figure 3.8.

The two zones in the hydraulic wash column are now;

- between the feed side and the filters with mother liquor and the ice bed moving upwards cocurrently.
- between the reslurry section and the filters with the wash water moving downwards and the ice bed upwards.

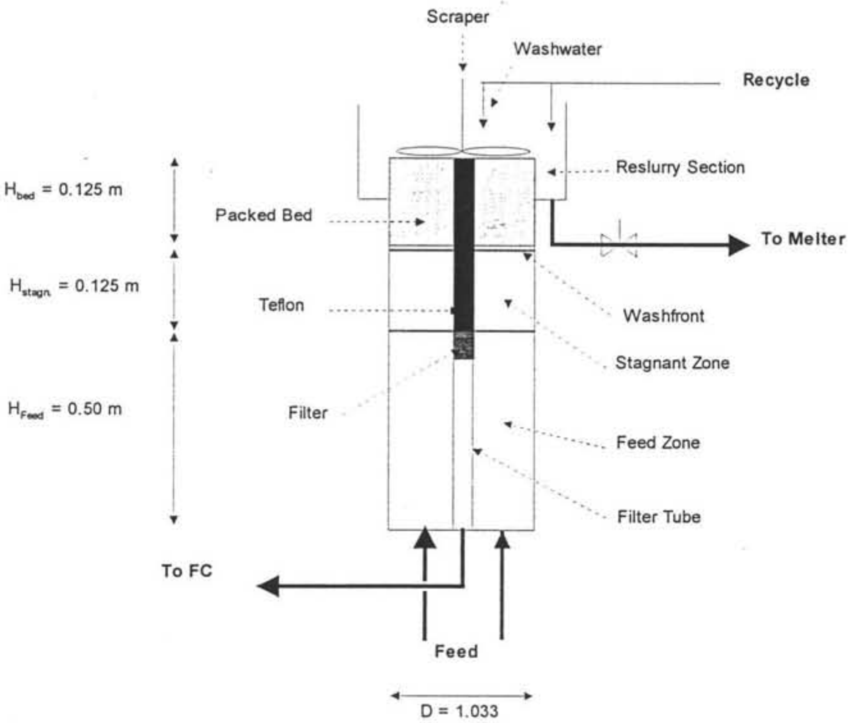


Figure 3.8: A Hydraulic Wash Column.

The feed slurry enters at the bottom and the slurry will move upward. The mother liquor flows through the filters and is recycled back to the FC or EFC. With the mother liquor drawn off the slurry, the volume fraction of the ice crystals increases considerably to a value of typically 55 %. The packed ice bed is forced upward by the feed pressure to the scraper. In the reslurry section the ice crystals are made transportable by adding recycled melt water. Subsequently the ice slurry is melted completely in a heatexchanger. During their passage from filter to reslurry section, the crystals are washed countercurrently by melt water flowing down the wash column to the wash-front.

3.3.5 Filter

A 90 % filter thickener has to be used at the end of the process to filter the ammonium nitrate solids slurry from the EFC.
There are several types of filters available (Perry, 17).

- gravitation filter
- cross flow filter
- rotary disc filter (thickener)

A gravitation filter is not suitable for the relative small amount of ammonium nitrate slurry to be filtered because of the large scale and filtering time required to reach a 90 w% slurry of ammonium nitrate.

Cross flow filtration (see fig. 3.9) has certain advantages over regular cake-filtration (Perry,17):

- filtration rate is not affected significantly by the particle-suspending-medium density difference.
- particle accumulation at the filter surface is minimised, so high filtration rates are obtained.

- feed additives such as flocculating agents and filter aid are not required.

A cross flow filter though is not favourable because of the following disadvantages:

- the energy required to move the slurry tangentially to the filter medium must be supplied in addition to the energy required to supply the pressure difference for filtration.
- an ultrafiltration membrane, which has high operating costs, has to be used.
- system is complex because it requires a recirculation pump, valves, and controls.

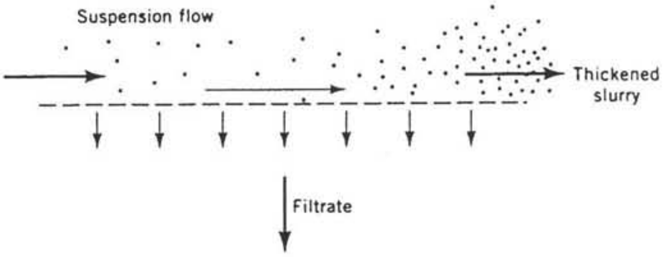


Figure 3.9: Principle of cross flow filtration.

So the rotary disc filter will be used in this process. It consists of a rotating disc filter with total submerging (180°), where cake is blown off into high solids product slurry (see figures 3.10 and 3.11). Disc filters are similar to rotary drum filters, but consists of several thin discs mounted on a shaft, over which a cloth is stretched, instead of the drum. This results in a larger effective filtering area and lower cost per unit area of filter. A scraper blade is used to discharge cakes thicker than 10 mm. Blowing back air is often used to assist the discharge. One disadvantage of the disc thickener is the difficulty of totally enclosing the filter for explosion hazardous materials. Since a wet ammonium nitrate cake is produced the explosion hazard (due to dry ammonium nitrate dust) is avoided.

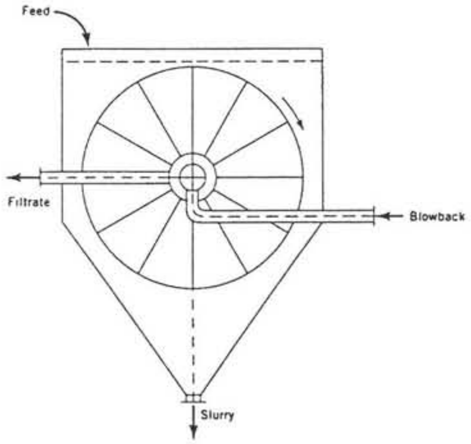


Figure 3.10: Rotary disc filter

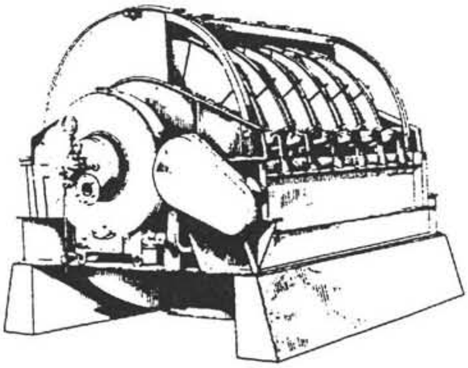


Figure 3.11: Figure of rotary disc filter

3.3.6 Centrifugal Pumps

The centrifugal pump is a dynamic pump, often used in the chemical and oil industries, and will be used in this process. It is able to pump liquids with very wide ranging properties and suspensions with a high solids content. The main advantages are (C&R, 18):

- simple and small of construction
- absence of valves
- operates at high speed
- steady delivery
- low maintenance costs

The main disadvantages are:

- single stage pump will not develop a high pressure
- limited operating range of conditions
- a no-return valve has to be incorporated to avoid back flow of liquid
- very viscous liquids cannot be handled efficiently

4 EQUIPMENT DESIGN

In this chapter the calculations for equipment design (appendix VI) are explained and the results are shown (see also specifications forms, appendix VII).

4.1 Freeze Concentrator

The design of freeze concentration step has to take into account that the brine becomes undercooled in the SSHE and most of the crystallisation takes place in the recrystalliser, see figure 4.1. The amount of undercooling is restricted to about 1 °C (Ref van Strate, Niro), cooling further results in increased possibility of a freeze up. The energy content of the cooling stored in the undercooling of the brine must be the same as the energy required for the formation of ice.

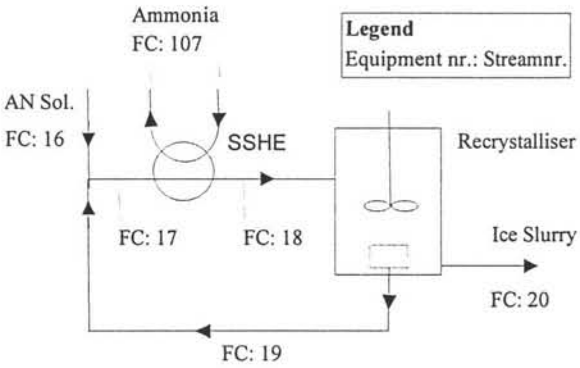


Figure 4.1: Freeze Concentration Unit

4.1.1 Scraped Surface Heat Exchanger (SSHE)

The restriction of maximum undercooling of 1°C determines the required flow through the SSHE. The SSHE can be specified by quantifying the total heat which has to be removed. The most important is the cooling required for the ice formation, the freezing energy.

$$Q_{ice} = F_{SSHE} \cdot c_{p,w} \cdot \Delta T = F_{SSHE} \cdot \Delta H_{s,w} \qquad [4\text{-}1]$$

where: Q_{ice} = cooling energy required for ice production (kW)
F_{SSHE} = total flow through SSHE (kg/s)
ΔT = temperature difference feed to and flow out SSHE (°C)
$\Delta H_{s,w}$ = freeze/melt enthalpy of water (kJ/kg)
$c_{p,w}$ = specific heat content of water (kJ/kg °C)

The fresh feed coming into the FC unit also needs to be cooled in the SSHE. Furthermore the heat generated by the recirculation pump and the agitator in the tank must not be neglected. The total cooling energy in the undercooling of the brine is calculated in equation 4-2.

$$Q_{tot} = Q_{ice} + (T_{in} - T_f) \cdot c_{p,w} \cdot F_{in} + Q_{pump} + Q_{agitator} \qquad [4\text{-}2]$$

where: Q_{tot} = total energy of undercooled brine required in FC unit (kW)
Q_{pump} = heat generated by the recirculation pump (kW)
$Q_{agitator}$ = heat generated by the agitator (kW)
T_{in} = temperature of feed in FC unit (K)
T_f = temperature of feed to SSHE (K)
F_{in} = fresh feed coming into FC unit (kg/s)

With a recycle of 344 kg/s and a total flow of 365 kg/s through the SSHE, the brine is undercooled by 1 °C in agreement with the former restriction imposed to avoid freeze up.

Calculating the logarithmic averaged temperature difference in the SSHE with equation 4-3 the required heat exchanging area can be calculated with equation 4-4.
To calculate the required heat exchanging area an estimate for the total heat transfer coefficient is necessary. For the heat transfer coefficient Niro Procestechnology B.V. (lit. 33) claim a value of 1500 W/m^2°C is possible whereas Perry claim a value between 170-850 W/m^2°C. As the heat transfer coefficient is affected by many factors a more conservative 1000 W/m^2°C is used.

$$\Delta T_{lm} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln \frac{(T_2 - t_2)}{(T_2 - t_1)}} \qquad [4\text{-}3]$$

where: ΔT_{lm} = logarithmic mean temperature difference (°C)
T_1 = inlet shell-side temperature (°C)
T_2 = outlet shell-side temperature (°C)
t_1 = inlet tube-side temperature (°C)
t_2 = outlet tube-side temperature (°C)

and: $$A_{SSHE} = \frac{Q_{tot}}{U.\Delta T_m} \qquad [4\text{-}4]$$

where: A_{SSHE} = required heat exchanging surface area for SSHE (m^2)
U = heat transfer coefficient (W/m^2 °C)
ΔT_m = mean temperature difference (in this case = ΔT_{lm}) (°C)

The required heat exchange area is 157 m^2 (see appendix VI a, Freeze concentrator). The SSHE prototypes available at Niro Procestechnology B.V. have 12 m^2 for each SSHE unit. A total of 14 units would have adequate heat exchange area to cool the brine sufficiently.

The superficial velocity of the brine in the SSHE is calculated from the flow and cross sectional area in the SSHE. The cross sectional area is calculated from the volume of the inner pipe and subtracting an estimate of the volume taken up by the axle and scrapers in the pipe and dividing by the length. The superficial velocity is approximately 0.73 m/s which ensures a turbulent flow in the SSHE which helps to prevent ice build up. A summary of the specifications of the SSHE is given in table 4.1. The numeric calculations can be found in appendix VI a.

Table 4.1 Design Specifications of SSHE units

Requirements		Units
Surface Area	157	m^2
Undercooling	1	°C
Total Cooling	1531	kW
Specifications SSHE		
Area	12	m^2
Inner Diameter	0.8	m
Height	4.8	m
Design		
Flow	0.365	m^3/s
Superficial Velocity	0.73	m/s
Number of SSHE	14	#

4.1.2 Recrystalliser

The recrystalliser is designed to convert the smaller crystals to crystals of size 0.2 mm. For the growth of the ice crystals a residence time of one hour is sufficient (NIRO, 33). With the flow and residence time the volume can be calculated.

$$V_{recryst} = F_{in} \cdot \rho_w \cdot \tau_{recryst} \qquad [4\text{-}5]$$

where: $V_{recryst}$ = volume of recrystalliser (m^3)

$\tau_{recryst}$ = residence time in recrystalliser (s)

The recrystalliser volume was calculated as 75 m^3, a small safety margin was added to result in a crystalliser of 4 m high and a diameter of 5 m resulting in a volume of 78.5 m^3. A summary of the dimensions of the tank are given in table 4.2

Table 4.2: Design Specifications of Recrystalliser

Requirements		
Residence Time	1	h
Specifications		
Flow	0.325	m^3/s
Design Recrystalliser		
Volume	78.5	m^3
Height	4	m
Diameter	5	m
Filter/Sieve Area	10	m^2
Filter/Sieve Cut	≈0.01	10^{-3}m

4.2 Eutectic Freeze Crystalliser – Cooling Disc Column Crystalliser

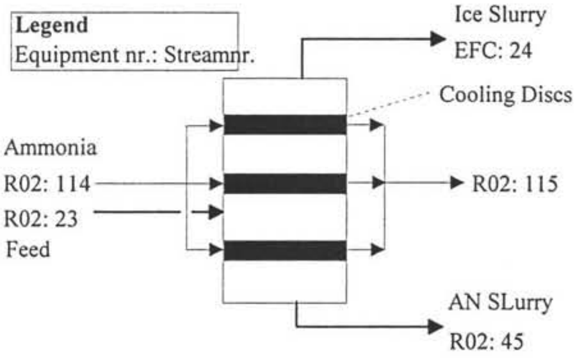


Figure 4.2: Cooling Disc Column Crystalliser

The design of the CDCC, see figure 4.2 is based on an estimation of dimensions and calculating whether they comply with requirements. The volume of the Cooling Disc Column Crystalliser (without discs) is calculated with equation 4-6.

$$\tau_{EFC} = \frac{V_{brine}}{F_{EFC}} \qquad [4\text{-}6]$$

where: τ_{EFC} = residence time in eutectic freeze crystalliser (s)
V_{brine} = volume CDCC (without discs) (m^3)
F_{EFC} = feed to EFC (m^3/s)

A residence time of one hour is sufficient for the crystals to grow to a mean size of 0.2 mm (Ref. NIRO). A residence time in the CDCC larger than the rise time and settling time of the ice and salt crystals respectively is required. The cooling discs also take up considerable volume and sufficient volume is required and must be placed and spaced adequately. Furthermore the baffling by the cooling discs and the mixing effect of the scrapers hinder the settling and rising of the crystals. The volume of the CDCC is then determined by a residence time of two hours and the extra volume required for the installing of the cooling discs. The settling and rising times and the spacing of the cooling discs must comply with previously mentioned guidelines.

To calculate the heat exchange area required for cooling equation 4-7 is used.

$$Q_{CDCC} = U_{CDCC} . A_{CDCC} . \Delta T \qquad [4\text{-}7]$$

where: Q_{CDCC} = cooling required for ice and AN formation (W)
U_{CDCC} = heat transfer coefficient ($W/m^2\,°C$)
A_{CDCC} = heat exchange area required (m^2)
ΔT = temperature difference between ammonia and brine (K)

A general estimate for the heat transfer coefficient in the CDCC is 300 W/m^2°C (van der Ham, 1). A temperature difference between coolant and brine of 5 K is sufficient to transport the heat. Bigger temperature differences would be better because it would reduce the heat exchange area and the number of discs which have to be installed. Higher temperature difference would require evaporating the ammonia at lower pressure and increase recompression costs of ammonia.

For the diameter of the column is taken 2.5 m. With the diameter of the column the cross sectional surface area of the column can be calculated determining the surface area of each cooling disc .

$$A_{disc} = \frac{1}{4}\pi . D_{column}^2 \qquad [4\text{-}8]$$

where: A_{disc} = surface area of the cooling disc (one side) (m^2)
D_{column} = diameter of the column (m)

Using both sides of the cooling discs, the number of discs required is calculated.

$$N = \frac{A_{CDCC}}{2A_{disc}} \qquad [4\text{-}9]$$

where: N = number of discs required (#)

The factor 2 represents use of both sides of the cooling disc.

The cooling discs have a thickness of approximately 0.2 m. Minimum spacing should be 0.2 m. With these specifications the total volume and height of the column can be calculated.

$$V_{discs} = A_{disc} h_{disc} N \qquad [4\text{-}10]$$

$$V_{total} = V_{discs} + V_{brine} \qquad [4\text{-}11]$$

$$H_{column} = \frac{V_{total}}{A_{column}} \qquad [4\text{-}12]$$

where: V_{discs} = total volume of cooling discs (m^3)
h_{disc} = thickness of cooling disc (m)
V_{total} = volume of brine and discs=volume column (m^3)
H_{column} = height of column (m)

The most important results of the dimension calculation are given in the following table.

Table 4.3: Dimensions of CDCC

Design		**Units**	**Value**
Residence time	τ_{CDCC}	s	7200
Volume of column	V_{column}	m^3	23.2
Diameter of column	D_{column}	m	2.5
Height of column	H_{column}	m	4.7
Number of discs	N_{disc}	-	11
Disc thickness	h_{disc}	m	0.2
Spacing of discs	h_{space}	m	0.2

By applying Stoke's law for laminar flow, the rise and sedimentation velocities can be estimated and the time ice and salt crystals need to rise and settle respectively. As previously mentioned, the assumed dimensions of the CDCC must result in a residence time larger than the rise and settling times of the crystals.

$$v_s = \frac{d_{crystal}^2 \cdot \Delta P_{c-b} \cdot g}{18\eta_w} \qquad [4\text{-}13]$$

$$t_s = \frac{H_{column}}{v_s} \qquad [4\text{-}14]$$

where: v_s	= rise or settling velocity	(m/s)
$d_{crystal}$	= diameter of crystal	(m)
$\Delta\rho_{c\text{-}b}$	= density difference between brine and crystal	(kg/m^3)
g	= acceleration due to gravity	(m/s^2)
η_w	= viscosity of water	(Pa.s)
t_s	= rise or sedimentation time	(s)

For correct calculation with this model the Reynolds number has to be smaller than 2000 with the calculated.

$$Re = \frac{\rho_f \cdot v_s \cdot d_p}{\eta_w} \qquad [4\text{-}15]$$

where: ρ_f	= density of fluid	(kg/m^3)
d_p	= particle diameter	(m)

The numeric calculations are given in appendix VI b

4.3 Wash Columns

For the quantity of ice crystals produced in the freeze concentration two washcolumns will be required to achieve sufficient capacity. See figure 4-3 for a schematic diagram of a hydraulic wash column. Arkenbout (16) states that a hydraulic wash column can be scaled up to a maximum capacity of about 10 tons/m^{2}h with a maximum column diameter of 1.2 m. This is confirmed by D. Verdoes of TNO-Apeldoorn. In the freeze concentrating unit 14.8 tons/h is produced, this means that two wash columns are required with a capacity of 7.4 tons/h each.

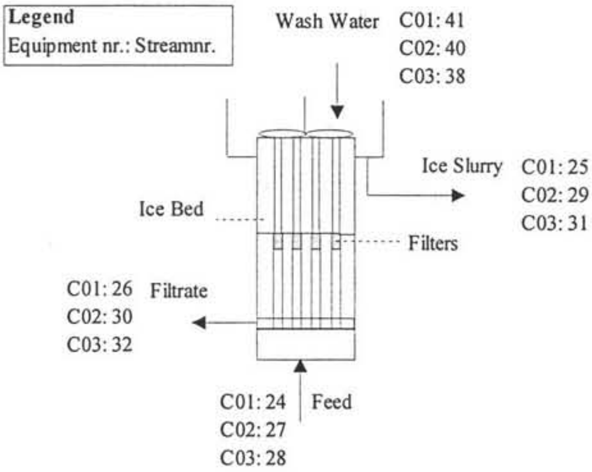


Figure 4.3: Wash Column

In the eutectic freeze crystalliser 0.36 kg/s ice is produced. This is about 1.3 ton/h and here one relatively small wash column will be adequate.

The following assumptions have been made for the wash columns:

- The porosity, ε, of the packed bed of ice crystals is constant, and has a value of $\varepsilon = 0.45$
- The particle diameter has a value $d_p = 200\ \mu m$

The cross sectional area of the wash column is related to the commercial wash column according to the capacity difference:

$$A_{wc} = A_{Comm} \cdot \frac{C_{wc}}{C_{Comm}} \qquad [4\text{-}16]$$

where: A_{WC} = cross sectional area of wash column (m^2)
A_{comm} = cross sectional area of commercial wash column (m^2)
C_{WC} = capacity of wash column (kg/m^2 h)
C_{comm} = capacity of commercial wash column (kg/m^2 h)

The column diameter can be calculated with equation 4-17, for numeric calculations see appendix VI c:

$$D_{wc} = \sqrt{\frac{4 \cdot A_{wc}}{\pi}} \qquad [4\text{-}17]$$

where: D_{WC} = diameter of wash column (m)

The wash water enters at the top of the wash column. From the flow of wash water the velocity is calculated by dividing the volume flow with the cross sectional area of the wash column. Similarly, from the flow of ice crystal slurry that enters at the bottom of the wash column the particle velocity can be calculated by dividing by the cross sectional area. The ice crystals are forced upward as they can only exit at the top. The wash water flows down the wash column to the wash-front. On the way down the wash water washes and warms up the ice bed. At the wash-front part of the wash water freezes again and moves up as ice in the ice bed. The downward flow of wash water to the wash-front displaces the mother liquor. The mother liquor passes through the filters leaving behind the ice. This washing action results in very pure ice and water leaving the top of the wash column. The numeric calculations are summarized in appendix VI c.

The pressure drop over the bed can be calculated with the Kozeny-Carman equation, equation 4-18.

$$\frac{\Delta P_{bed}}{H_{bed}} = \frac{v \cdot 180 \cdot (1-\varepsilon_{bed})^2 \cdot \eta}{\varepsilon_{bed}{}^3 \cdot d_p^2} \qquad [4\text{-}18]$$

Where: ΔP_{bed} = pressure drop over the bed (Pa)
H_{bed} = height of the bed (m)
v = superficial velocity (m/s)
ε_{bed} = porosity of the bed (-)
η = viscosity of the fluid (Pa.s)

Commercial wash columns have the following characteristics. Referring to figure 3.8

- The inlet pressure (P_{inlet}) is in the range of 1-2 barg,
- The wash pressure (P_{wash}) is about 0.75 times the inlet pressure,
- The feed zone has a height (H_{feed}) of about 0.50 m,
- The ice crystal filters are installed at the top of the feed zone,
- The total bed height ($H_{bed,total}$) is approximately 0.25 m, with the wash-front located halfway along the bed. This means that the packed bed height (H_{bed}) is about 0.125 m and the stagnant zone height ($H_{stag.}$) about 0.125 m
- The purity of the outcoming ammonium nitrate stream will be less than 1 ppm (ref. NIRO).

By controlling the wash pressure, the location of the wash-front can be controlled. If the wash zone is situated too low, the wash pressure is lowered and the wash-front will rise.
The difference between the pressure drop over the bed and the difference between the feed and wash pressure should be low so the wash column is controllable. The calculations are summarized in appendix VI c.

Near the wash-front, a temperature change can be observed. This temperature change is caused by the difference between the crystallization temperature and the melting temperature of the crystals.

The recrystallization of wash water reduces the bed porosity, this can be taken into account with the equation 4-19.

$$\varepsilon^* = \varepsilon_{bed} - (1-\varepsilon_{bed}) \cdot \frac{C_{p,s} \cdot \Delta T}{\Delta_{cryst} H} \qquad [4\text{-}19]$$

where: ε^* = bed porosity after recrystallisation (-)
ε_{bed} = bed porosity of stagnant zone (-)
$c_{P,S}$ = specific heat of ice crystals (J/kg °C)
$\Delta_{cryst}H$ = crystallisation enthalpy (J/kg)

The pressure drops over the washed bed and the stagnant zone can be calculated with the Kozeny-Carman equation. Summing the pressure drops gives the total pressure drop over the packed bed, see appendix VI c.

There are no specific guidelines available to determine how many filter tubes are needed. At TNO-Apeldoorn, two hydraulic wash columns are operational with:

1) D_{wc} = 0.15 m; 6 filter tubes,
2) D_{wc} = 0.08 m; 1 filter tube.

The filter tubes have a diameter (D_{filter}) of 0.02 m and a filter height (H_{filter}) of 0.03 m. With the cross sectional area of the desired wash column determined by the maximum diameter, the TNO wash columns can be used for an indication as to the number of tubes necessary. With equation 4-20 an estimate for the number of filter tubes is calculated.

$$N_{tube} = N_{TNO} \cdot \frac{A_{wc}}{A_{TNO}} \qquad [4\text{-}20]$$

where: N_{tube} = number of tubes in the wash column (#)
N_{TNO} = number of tubes in TNO-wash column (#)
A_{TNO} = cross area of the TNO-wash column (m^2)

The filter tubes have very little resistance such that the pressure drop over the filter is negligible.
The dimensions and specifications of the wash column from the calculations are summarized in table 4.4.

Table 4.4: Results of the Wash Column Design Calculations

Design		Units	C01	C02	C03
Ice washed	$\phi_{m,ice}$	kg/h	1312	7412	7412
Cross sectional area	A_{wc}	m^2	0.103	0.582	0.582
Wash column diameter	D_{wc}	m	0.362	0.861	0.861
Total bed height	$H_{bed,total}$	m	0.25	0.25	0.25
Height feed zone	H_{feed}	m	0.50	0.50	0.50
Height wash column	H_{wc}	m	0.75	0.75	0.75
Pressure drop over bed	$\Delta P_{bed,total}$	bar	0.45	0.44	0.44
Diameter filter tubes	$D_{filter\ tube}$	m	0.02	0.02	0.02
Height filter	H_{filter}	m	0.03	0.03	0.03
Number of tubes	N_{tube}	#	28	157	157

4.4 Cooling Process

The FC and EFC operate at low temperature. To reach this low temperatures liquid ammonia (p = 22.5 bara; T = 292 K), is flashed to a lower pressure. The liquid ammonia is flashed to different pressures for the FC and EFC because they operate at different temperatures.

The ammonia used for cooling does not come into contact with any other process streams. For the FC, in which the brine is cooled to 270 K (-3 °C), the liquid ammonia is flashed to 260 K (-13 °C) because a temperature difference of 10°C is required for effective heat exchange. To reach this temperature, the liquid ammonia is flashed to 2.6 bara, see Mollier diagram (appendix III).
In the EFC the brine is cooled to the eutectic temperature of 256 K (-17 °C) and 5°C is sufficient temperature difference for heat exchange. Thus, in the EFC liquid ammonia is flashed to 251 K (-22 °C). This temperature is reached by flashing the liquid ammonia to 1.7 bara, see appendix III (Mollier diagram).

Two separate flash vessels are installed to flash the ammonia to the required pressure and temperature. In the FC and EFC the ammonia evaporates cooling the brine. Not all the ammonia is evaporated in one pass so a knock-out drum is installed. The knock-out drum separates the liquid ammonia from the ammonia vapour. The liquid ammonia is sent back to the FC or EFC respectively as recycle.

Finally, two compressors are installed to compress the ammonia vapour to 4.2 bara to comply with downstream demands.

4.4.1 Flash Vessels

Flash vessels are required to flash the liquid ammonia (p = 22.5 bara and T = 292 K) to the required pressure and temperature for the FC and EFC. The pressure is relieved in the valve before the vessel. In the vessel the liquid and vapour separate. A demister is installed to prevent liquid ammonia droplets to go with the vapour (entrainment).

A flash vessel is shown in figure 4.4.

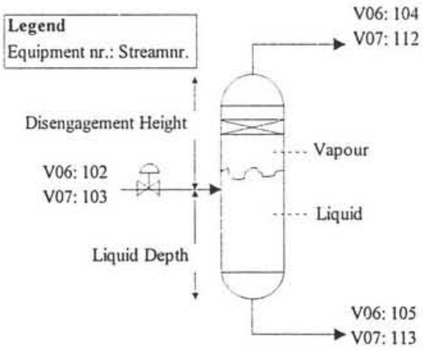


Figure 4.4: Flash Vessel

Equation 4-21 (C&R, 20) can be used to estimate the maximum design gas velocity for the sizing of a vertical separating vessel.

$$\hat{u}_V = 0.035 \cdot \sqrt{\frac{\rho_L}{\rho_V}} \qquad [4\text{-}21]$$

where $\hat{u}_V$ = maximum design vapour velocity (m/s)
ρ_V = vapour density (kg/m^3)
ρ_L = liquid density (kg/m^3)

Above the liquid level a disengagement height equal to the vessel diameter is necessary. The liquid level will depend on the hold-up time required for smooth operation and control. Here a hold-up time of 1 minute is used instead of a normal hold-up time of 10 minutes, as it is not necessary for the liquid to settle completely.

First the flow rates and densities of the incoming and outcoming streams are needed.

Using equation 4-22 to determine the maximum design vapour velocity,

$$\phi_{v,V} = \frac{\phi_{m,V}}{\rho_V} \qquad [4\text{-}22]$$

where: $\phi_{v,V}$ = vapour volumetric flow rate (m^3/s)
$\phi_{m,V}$ = vapour mass flowrate (kg/s)

The cross sectional area and diameter of the flash vessel can be determined using equations 4-23 and 4-24.

$$A_{vessel} = \frac{\phi_{v,V}}{\hat{u}_V} \qquad [4\text{-}23]$$

$$D_{vessel} = \sqrt{\frac{4 \cdot A_{vessel}}{\pi}} \qquad [4\text{-}24]$$

where: A_{vessel} = cross sectional area of flash vessel (m^2)
D_{vessel} = diameter of flash vessel (m)

The disengagement height (H_d) equals the vessel diameter.

Similarly the liquid volume flow rate can be calculated with equation 4-25.

$$\phi_{v,L} = \frac{\phi_{m,L}}{\rho_L} \qquad [4\text{-}25]$$

where: $\phi_{v,L}$ = liquid volumetric flow rate (m^3/s)
$\phi_{m,L}$ = liquid mass flow rate (kg/s)

The required liquid volume for a 1 minute hold-up time is then calculated with equation 4-26.

$$V_L = \phi_{v,L} \cdot t_{hold-up} \qquad [4\text{-}26]$$

where: V_L = required liquid volume (m^3)
$t_{hold\text{-}up}$ = hold up time (s)

With the vessel cross sectional area enables the liquid depth (H_L) to be calculated with equation 4-27.

$$H_L = \frac{V_L}{A_{vessel}} \tag{4-27}$$

$$H_{vessel} = H_d + H_L \tag{4-28}$$

$$V_{vessel} = H_{vessel} \cdot A_{vessel} \tag{4-29}$$

The calculations have been carried out for both flash vessels. For the numeric calculations see appendix VI d, the results are given in table 4.5.

Table 4.5: Results of flash vessel design

Specifications		**Units**	**V06**	**V07**
Liquid flow	$\phi_{m,L}$	kg/s	1.0647	0.1007
Vapour flow	$\phi_{m,V}$	kg/s	0.1137	0.0158
Liquid density	ρ_L	kg/m^3	770	770
Vapour density	ρ_V	kg/m^3	2.38	1.25
Design		**Units**	**V06**	**V07**
Diameter	D_{vessel}	m	0.311	0.136
Disengagement height	H_d	m	0.311	0.136
Liquid level	H_L	m	1.093	0.539
Vessel height	H_{vessel}	m	1.404	0.675
Vessel volume	V_{vessel}	m^3	0.107	$9.83\ 10^{-3}$

4.4.2 Knock-Out Drums

The knock-out drums have the same function as the flash vessels, see figure 4-5. The ammonia is not evaporated completely in the FC and EFC so a knock-out drum is required to separate the liquid ammonia from the vapour. The vapour is sent to the compressors and the liquid is recycled to the FC or EFC.

An amount of liquid ammonia is sent through the units such that 80% of liquid ammonia evaporates and is sent to the compressor, the remaining liquid ammonia is collected at the base of the knock-out drum and is recycled. First 10% of liquid ammonia was recycled but this was insufficient to control the liquid level in the knock-out drum. A hold-up time of 15 minutes is required for so that the liquid level is high enough for good control.

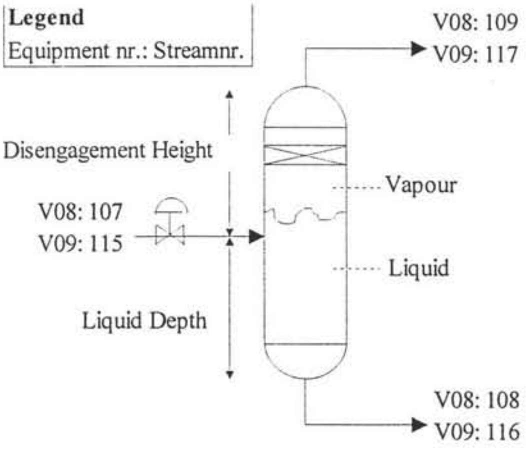


Figure 4.5: Knock-Out Drum

The calculations are the same as for the flash vessels and can be found in appendix VI d. The results are summarized in table 4.6.

Table 4.6: Results of Knock-Out Drum design

Specification		Unit	V08	V09
Liquid flow	$\phi_{m,L}$	kg/s	0.2662	0.0252
Vapour flow	$\phi_{m,V}$	kg/s	1.0647	0.1007
Liquid density	ρ_L	kg/m^3	770	770
Vapour density	ρ_V	kg/m^3	2.38	1.25
Design		**Unit**	**V08**	**V09**
Diameter	D_{vessel}	m	0.951	0.344
Disengagement height	H_d	m	0.951	0.344
Liquid level	H_L	m	0.438	0.317
Vessel height	H_{vessel}	m	1.389	0.661
Vessel volume	V_{vessel}	m^3	0.987	0.061

4.4.3 Compressors

The ammonia vapour from cooling the FC and EFC units is compressed to 4.2 bara to comply with the demands as feed to the ammonium nitrate plant. Centrifugal type compressors are used.

If the pressure ratio P_{out}/P_{in} is higher than 3 (de Graauw, 21) multistage compression is usually implemented to reduce the overall work required. The pressure ratios for compressors K01 and K02 are 1.62 and 2.47 respectively, hence multistage compression is not needed. Equation 4-30 is used to determine the isentropic power.

$$P_{S,isen} = \phi_m \cdot \frac{P_{in}}{\rho_{in}} \cdot \frac{\kappa}{\kappa - 1} \cdot \left\{ \left(\frac{P_{uit}}{P_{in}} \right)^{\frac{\kappa-1}{\kappa}} - 1 \right\} \qquad [4\text{-}30]$$

with $\kappa = \frac{C_p}{C_v}$

where:			
	$P_{S,isen}$	= isentropic power	(kW)
	ϕ_m	= mass flow rate of ammonia vapour	(kg/s)
	P_{in}	= pressure in of ammonia vapour	(Pa)
	ρ_{in}	= density of ammonia vapour in	(kg/m^3)
	P_{uit}	= pressure out	(Pa)

Equation 4-30 is not applicable for multistage compression.

To calculate the real power, the isentropic power is divided by the compressor efficiency (equation 4-31), which is the product of the mechanical and hydraulic efficiency. Normally, this efficiency lies between 70 en 80%. A compressor efficiency of 70% is used.

$$P_{real} = \frac{P_{S,isen}}{\eta} \qquad [4\text{-}31]$$

where: P_{real} = real power (kW)
η_c = compressor efficiency (-)

The specifications for compressors K01 and K02 are summarized in table 4.7.

Table 4.7: Specifications of Compressors

Design		**Units**	**K01**	**K02**
Vapour mass flow rate	$\phi_{m,V}$	kg/s	1.1784	0.1165
Pressure in	P_{in}	Pa	$2.6\ 10^5$	$1.7\ 10^5$
Pressure out	P_{out}	Pa	$4.2\ 10^5$	$4.2\ 10^5$
Isentropic power	$P_{S,\ isen}$	kW	65.41	16.01
Total efficiency	η	-	0.70	0.70
Real power	P_{real}	kW	93.4	22.9

4.5 Neutraliser

The neutraliser is a well stirred mixing vessel. A simple cylindrical tank will fulfill the demands with a stirring mechanism adequately. The volume of the neutraliser is calculated with equation 4-32.

$$V_n = \phi_{v,n} \cdot \tau_n \qquad [4\text{-}32]$$

where: V_n = volume neutraliser (m^3)
$\phi_{v,n}$ = volumetric flow through neutraliser (m^3/s)
τ_n = residence time in neutraliser (s)

The mixing process is very fast. A residence time of 3 minutes is sufficient for complete mixing to take place. For the diameter of the tank 1 m is assumed. The tank height can then be calculated with equation 4-33.

$$H_n = \frac{V_n}{\frac{1}{4} \cdot \pi \cdot D_n^2} \qquad [4\text{-}33]$$

where: H_n = tank height (m)

The dimensions of the neutraliser are given in table 4.8.

Table 4.8: Dimensions of neutraliser

Design		Units	Value
Residence time	τ_n	s	780
Neutraliser volume	V_n	m^3	3.5
Diameter	D_n	m	1
Tank height	H_n	m	4.5

4.6 Design of Filter

When the mass flow is known, the total filter area can be calculated from equation 4-34.

$$A_F = \frac{\varphi_{v,F}}{N_r \cdot Q_f} \qquad [4\text{-}34]$$

where: A_F = filter area (m^2)

$\varphi_{v,F}$ = volumetric flow through filter (m^3/s)

Q_F = total volume of filtrate per unit area (m^3/m^2)

N_r = rotation frequency ($1/s$)

Q is determined using the Ruth equation, see appendix VI j.
To determine the number of discs needed in the filter, the total filter area must be divided by the filter area per disc (see table 4.8).

Table 4.8: Specification of rotary disc filter

Design	Units	Value
A_{tot}	m^2	1.33
A_{disc}	m^2	0.14
N_{discs}	-	9

4.7 Dissolver

The dissolver has to heat the ammonium nitrate slurry that comes out of the filter. The slurry will dissolve and reach the desired concentration of 90 w% at a temperature of 95 °C. A continuously stirred vessel with an internal coil is required. Low pressure steam of 3 bar and 190 °C is condensed in the heating coil of the dissolver. This is a simple and economical manner to transfer heat in a vessel. To calculate the dimensions of the vessel and the coil the following equations are used.

$$V_v = \phi_v \cdot \tau \qquad [4\text{-}35]$$

where: V_v = vessel volume (m^3)
ϕ_v = volume flow (m^3/s)
τ = residence time (s)

The residence time is set at 30 minutes so the slurry has enough time to dissolve.
The diameter of the vessel is set at 0.3 m. The diameter of the coil can be calculated by using equation 4-36 (C&R, 20).

$$D_c = \frac{D_v}{30} \qquad [4\text{-}36]$$

where: D_c = coil diameter (m)
D_v = vessel diameter (m)

The surface area required for heat exchange can be calculated by using equation 4-37

$$A_c = \frac{Q}{U \cdot \Delta T} \qquad [4\text{-}37]$$

where: A_c = heat exchanging area (m)
U = heat transfer coefficient (W/m^2 °C)
Q = heat to be transferred (W)
ΔT = temp. diff. between steam and brine in dissolver (K)

A value of 1207 W/m^{2}K (lit. Perry) is used for the heat transfer coefficient.
The length of the coil can be calculated with equation 4-38.

$$L_c = \frac{A_c}{\pi \cdot D_{coil}} \qquad [4\text{-}38]$$

where: L_c = coil length (m)
D_{coil} = thickness of the coil (m)

The total volume is determined by the volume of the vessel and the volume of the coil.

$$V_c = L_c \cdot \frac{1}{4} \cdot \pi \cdot D_c^{\,2} \qquad [4\text{-}39]$$

$$V_{total} = V_c + V_v \qquad [4\text{-}40]$$

where: V_c = volume of the coil (m^3)
V_{total} = total volume (m^3)

Dividing this volume by the surface area of the cylindrical vessel the height can be calculated.

$$H = \frac{V_{total}}{\frac{1}{4} \cdot \pi \cdot D_v^{\ 2}} \qquad [4\text{-}41]$$

where: H = height of dissolver vessel (m)

The most important dimensions of the dissolver are given in the next table.

Table 4.9: dimensions of the dissolver

Design		Units	Value
Residence time	τ	s	1800
Vessel diameter	D_v	m	0.3
Diameter coil	D_c	m	0.01
Area coil	A_c	m^2	0.43
Length coil	L_c	m	13.4
Total volume	Vt	m^3	0.04
Vessel height	H	m	0.57

4.8 Design of Heat Exchangers

In the process three heat exchangers of the inline tube and shell type are used.
The main equation in the design of this type of heat exchangers according to *Coulson and Richardson* is used to calculate the heat transfer area. It includes the heat transfer per time Q, the overall heat transfer coefficient U and the mean temperature difference, the driving force for heat transfer, ΔT_m.

$$A_{HE} = \frac{Q}{U * \Delta T_m} \tag{4-42}$$

$$\Delta T_m = F_t * \Delta T_{lm} \tag{4-43}$$

where: A_{HE} = required heat exchanging surface area (m^2)

The correction factor F_t is a function of the shell and tube fluid temperatures and the number of tube and shell passes. The manner of calculating the correction factor F_t is shown in appendix VI j.

As for the SSHE the logarithmic mean temperature difference, ΔT_{lm} is given by equation 4-3.

The overall heat transfer coefficient is first estimated. After calculation of the individual coefficients resulting from the heat-transfer area, the overall heat transfer coefficient U_o is calculated and compared with the trial value.

$$\frac{1}{U_o} = \frac{1}{h_s} + \frac{1}{h_{od}} + \frac{d_o \ln(d_o / d_i)}{2 * k_w} + \frac{d_o}{d_i} * \frac{1}{h_{id}} + \frac{d_o}{d_i} * \frac{1}{h_i} \tag{4-44}$$

where			
	h_s	= outside fluid film coefficient	(W/m^2 °C)
	h_i	= inside fluid film coefficient	(W/m^2 °C)
	h_{od}	= outside dirt coefficient (fouling factor)	(W/m^2 °C)
	h_{id}	= inside dirt coefficient	(W/m^2 °C)
	k_w	= thermal conductivity of the tube wall material	(W/m °C)
	d_i	= tube inside diameter	(m)
	d_o	= tube outside diameter	(m)

Shell side

At the shell side the bundle diameter and the shell diameter can be calculated as follows

$$D_b = d_o * \left(\frac{N_T}{k_1}\right)^{\frac{1}{n_1}} \tag{4-45}$$

$$D_s = D_b + C \tag{4-46}$$

where N_T = number of tubes
K_1, n_1 = constants, dependent on number of tube passes and triangular pitch (obtained from table 12.4 C&R (20))
C = clearance, obtained from figure 12.10 C&R (20) (split-ring floating head)

Calculation of the shell-side coefficient h_s:

$$h_s = \left(\frac{k_f}{d_e}\right) * j_h * \text{Re} * \text{Pr}^{0.33} * \left(\frac{\mu}{\mu_w}\right)^{0.14} \qquad [4\text{-}47]$$

$$\text{Re} = \frac{\rho * u * d_e}{\mu} = \frac{G_s * d_e}{\mu} \qquad [4\text{-}48]$$

The mass velocity G_s equals the mass flow rate divided by the total shell cross flow area A_s.

$$G_s = \frac{F}{A_s} \qquad [4\text{-}49]$$

$$A_s = \frac{p_t - d_o}{p_t} * L_b * D_s \qquad [4\text{-}50]$$

$$d_e = \frac{1.10}{d_o}\left(p_t^2 - 0.917 d_o^2\right) \quad \textit{in case of a triangular pitch} \qquad [4\text{-}51]$$

where: A_s	= shell cross flow area	m^2
p_t	= pitch	m
L_b	= baffle spacing	m

Calculation of the shell-side pressure drop ΔP_s:

$$\Delta P_s = 8 j_f \left(\frac{D_s}{d_e}\right)\left(\frac{L}{L_B}\right)\frac{\rho u_s^2}{2} \qquad [4\text{-}52]$$

where: j_f = shell side friction factor (figure 12.20 C&R (20)) -

Tube side

Calculation of tube-side coefficient h_i:

$$h_i = Nu * \frac{k_f}{d_i} \tag{4-53}$$

$$Nu = j_h * Re * Pr^{0.33} * \left(\frac{\mu}{\mu_w}\right)^{0.14} \tag{4-54}$$

$$Re = \frac{\rho * u * d_e}{\mu} = \frac{G_t * d_e}{\mu} \tag{4- 55}$$

$$Pr = \frac{C_p * \mu}{k_f} \tag{4-56}$$

where			
	k_f	= fluid thermal conductivity	(W/m °C)
	j_h	= heat-transfer factor (fig. 12.23/12.29 C&R)	(-)
	μ	= fluid viscosity (at the bulk fluid temperature)	(Pa.s)
	μ_w	= fluid viscosity at the wall	(Pa.s)
	d_e	= equivalent diameter (equals d_i for tubes)	(m)
	G_t	= mass velocity	(kg/m^2 s)
	Pr	= Prandtl Number	(-)
	Re	= Reynolds Number (inertia forces/viscous forces)	(-)
	Nu	= Nusselt Number (total heat transfer/conductive heat tr.)	(-)

The ratio of the viscosities in the Prandtl number is approximately 1 with a low viscous fluid. The mass velocity G_t equals the mass flow rate divided by the total tube cross flow area A_t.

Calculation of the tube-side pressure drop ΔP_t:

$$\Delta P_t = N_p * [8 j_f * \left(\frac{L}{d_i}\right) + 2.5] * \frac{\rho u_t^2}{2} \tag{4-57}$$

where:			
	j_f	= shell side friction factor (figure 12.20 C&R (20))	-
	N_p	= number of tube side passes	-

The results for heat exhangers E01, E02, E03 are summarized in table 4.10. For the numeric calculations see appendix VI h.

Table 4.10: Pressure Drops Heat Exchangers

Heat Exchanger	**E01**	**E02**	**E03**
ΔP_s (bara)	0.18	0.07	0.11
ΔP_t (bara)	0.31	0.2	0.13

4.9 Design of Centrifugal Pumps

The isentropic power needed has been calculated with equation 4-58.

$$P_{p,isen} = -W_{p,isen} = \phi_m * \frac{\Delta P}{\rho} = \phi_v * \Delta P \qquad [4\text{-}58]$$

The pump shaft power, the power required for pumping an incompressible fluid is given by equation [4-59]. The total pump efficiency is the product of the mechanical and a hydraulic efficiencies.

$$P_p = \frac{\phi_v * \Delta P}{\eta_p} \qquad [4\text{-}59]$$

where: P_p = pump shaft power (kW)
η_p = overall pump efficiency (-)

The overall pump efficiency depends on the capacity of the pump and can be estimated from figure 10.62 (C&R, vol. 6).

$$_p = \eta_m * \eta_h \qquad [4\text{-}60]$$

where: η_m = mechanical efficiency (-)
η_h = hydraulic efficiency (-)

The head is calculated with equation 4-61.

$$h_{man} = \frac{\Delta P}{\rho \cdot g} \qquad [4\text{-}61]$$

The Net Positive Suction Head (NPSH) is a measure for the minimal suction pressure required to avoid cavitation. To avoid cavitation it is important that the pump suction pressure does not drop below the vapour pressure of the liquid being pumped. The margin between those two pressures is called the available NPSH and is dependent of the type of pump and not of the system.

$$NPSH_{avail} = \frac{P_{in} - P_v}{\rho * g} \qquad [4\text{-}61]$$

where: P_{in} = suction pressure (Pa)
P_v = vapour pressure (Pa)

The results of the pumpdesign calculations are summarized in appendix VI i.

5 MASS AND HEAT BALANCES

5.1 Heat Integration

In the next table an summary is given of all the heat exchanging streams. In this table Q represents the quantity of heat to be exchanged.

Table 5.1 Summary of heat to be exchanged in different equipment (stream numbers are in brackets)

Equipment	Heating of Cold Stream	M (kg/s)	**Q (kW)**	Cooling of Hot Stream	M (kg/s)	**Q (kW)**
E01	ice (34)	3.456	**1156.2**	feed (14)	4.513	**1156.2**
E02	ice (35)	1.142	**381.9**	to HNO3 plant (9)	4.722	**381.9**
E03	productstream (42)	4.482	**376.5**	to HNO3 plant (10)	4.722	**376.5**
R01 (FC)	ammonia (evap) (102)	1.314	**1524.0**	feed (16)	4.513	**146.5**
				water (16)	4.118	**1377.5**
	Total		**1524.0**	Total		**1524.0**
R02 (EFC)	ammonia (evap) (103)	0.135	**153.5**	feed for EFC (23)	0.395	**23.4**
				water (23)	0.364	**121.9**
				AN (cryst) (23)	0.025	**8.2**
	Total		**153.5**	Total		**153.5**
C01	ice (24)	0.364	**13.5**	ice (cryst) (41)	0.040	**13.5**
C02	ice (27)	2.059	**12.5**	ice (cryst) (40)	0.038	**12.5**
C03	ice (28)	2.059	**12.5**	ice (cryst) (38)	0.038	**12.5**
V05	90 w% AN (46)	0.031	**14.4**	steam (cond)	0.094	**20.4**
	Solution of AN	0.025	**8.2**	steam	0.094	**2.2**
	Total		**22.6**	Total		**22.6**
E405a	ammonia	1.875	**2625.3**	air	38.333	**2625.3**
	Total		**6278.5**	Total		**6278.5**

In this balance the energy required for phase transitions is calculated with equation 5-1.

$$Q = M \cdot \Delta H_{phase\ transition} \qquad [5\text{-}1]$$

where: Q = energy per second (kW)
M = mass flow (kg/s)
$\Delta H_{phase\ transition}$ = enthalpy of phase transition (kJ/kg)

The energy required for temperature changes is calculated with equation 5-2.

$$Q = M \cdot c_p \cdot \Delta T \qquad [5\text{-}2]$$

There are three heat exchangers required to reach the goals mentioned in chapter 2. The heat exchangers are already present on the Kemira Rozenburg plant site, they are described below.

1. In the first the air can be cooled by the ammonia that is not used in this process. This is equipment E405a (see table 5.1).
2. After cooling of the air, the 0.091 kg/s remaining ammonia is evaporated in the second heat exchanger.
3. The last heat exchanger is required to heat the ammonia after compression of the ammonia gas. The ammonia is heated to a temperature of 60°C for the ammonium nitrate process.

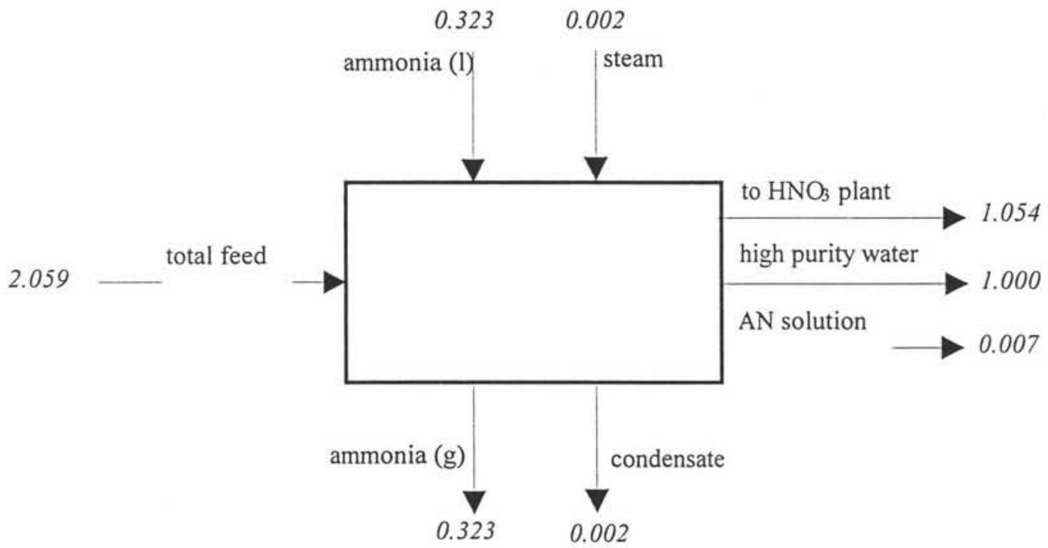
5.2 Mass, Heat and Component Balances

In appendix II c the overall mass and heat balance is shown. This balance is split up in two parts. The cooling part with the ammonia streams and the rest of the plant. The mass balance is made with help of the component balance in appendix II d, which is also split up in two parts. In this component balance the stream heat content Q is calculated using liquid water at 273.15 K as a reference. The stream heat content in the ammonia component balance is interpolated from the Mollier diagrams for ammonia in appendix III.

In the plant energy balance the entropy change in concentrating the solution (35.1 kW) is taken into account. In the ammonia energy balance the power that is put in the compressors is also taken into account, because this has influence on the temperature of the ammonia.

In figure 5.1 the total input/output diagram is given. The main product is high purity water and this stream is set to unity.

Figure 5.1: Input/output diagram



6. PROCESS CONTROL

In this chapter the control of process units during operation is discussed. Some specifics with regard to process control during start-up are also discussed.

6.1 Flow and Pressure Control

The flow of liquid streams are monitored with flow meters and regulated with simple valves. All incoming streams are at sufficient pressure to control the flow with the valves.

The pressures of the liquid streams are mostly determined by the installation of pumps, however in specific recycle streams the pressure is let off with a simple valve and pressure monitor to the desired level.

6.2 Neutraliser

In the neutraliser the control objective is to add ammonia or nitric acid to the feed to achieve a 1:1 molar ratio of nitric acid and ammonia. This ensures that in the process all the waste water can be converted to just water and ammonium nitrate.

Ammonia and nitric acid feeds are available to regulate the pH, they are at pressures of 22 and 14 bara respectively. These pressures are sufficient to regulate the flow with a regulating valve.

Normal disturbances in the feed of nitrate and ammonia concentrations and acidity are relatively small compared to the disturbance when parts of the ammonium nitrate plant is shutdown. Partial operation of the ammonium nitrate plant influences the feed composition considerably and this has been taken into account for the pH control.

To achieve this stoichiometric range the pH in the neutraliser is measured. Measuring the pH of the feed for feed forward control is not considered worthwhile as the dead time resulting from measuring the pH in the neutraliser is not very large. Also considering the large mixing time in the FC unit a lag in control is smoothed out considerably.

The nitric acid and ammonia are added continuously. When making a small step change in the addition of ammonia or nitric acid, the change in pH is registered. At the point of inflection the change in pH is minimal and the molar ratio of nitric acid and ammonia is also 1:1.. The controller must make step changes in the flows of ammonia and nitric acid in the direction that minimises the response in pH.

Working with the pH change is necessary as the pH is also dependent on the total concentration of the ammonia and nitric acid even when in the correct molar ratio. The pH should be within the range of ammonium nitrate salts in solution. This is the range of pH of of the process ammonium nitrate concentrations (stoichiometric ammonia and nitric acid) in the neutraliser. For good control a minimum flow for ammonia and nitric acid is maintained. The minimum flow is taken as 10% of the maximum required flow.

The controller should minimise the flow of either the ammonia or nitric acid to 10% and increase the addition of the other flow to regulate the pH.

6.3 Flash vessels and knock-out drums

The flash vessels and knock-out drums are both controlled with the same principles. The pressure is controlled with a relief valve. At pressures above the setpoint the opening of the relief valve releases the pressure. When the pressure is too low, closing the gas outlet valve allows the evaporisation of ammonia to increas the pressure.

In the flash vessels the liquid level is controlled with a valve on the inlet. When the liquid level is low, opening of the inlet valve increases the inlet flow so the liquid level rises and vice versa.

With the knock-out drums the liquid level is controlled with a valve on the liquid outlet. When the liquid level is low the closing the outlet valve decreases the liquid flow out and so the liquid level rises and vice versa.

6.4 FC and EFC

The control objective in the FC is to maintain an as high as possible cooling rate for the ice production in the FC. Under normal operating conditions this will be close to the maximum cooling capacity. The temperature of the stream leaving the SSHE should be controlled by increasing the ammonia flow until the leaving stream is 1°C undercooled. If further undercooling occurs the temperature controller should reduce the ammonia flow. As the residence times of the ammonia and brine in the SSHE is very short, a feed back controller will function well.

When the valve position for the liquid flow out of the knock-out drum approaches full open, the fresh ammonia feed into the FC should not be allowed to increase further. If the liquid flow out of the knock-out drum with the valve at full open is calibrated at 20% of the total ammonia flow through the FC this would ensure no excessive ammonia recycle takes place. A higher level controller can enforce this.

The EFC is controlled in an identical manner to the FC.

6.5 Wash Columns

During the start-up procedure the washed ice slurry outlet valve at the top of the wash column is closed. The only way out is then through the filters in the filter tubes. The ammonium nitrate brine passes through the filters while the ice crystals that remain form a crystal bed around the filters. Due to the feed pressure, a pressure difference over the filter is established for flow through the filter.

When the ice bed has formed, the ice slurry outlet valve at the top of the wash column is opened. By regulating with the washed ice slurry outlet valve the outlet flow can be balanced against the ice crystal transport rate and the ice crystal feed. This can be monitored accurately by detecting the level of the wash-front in the wash column. During operation the wash column is regulated by maintaining a constant level of the wash-front.

The pressure difference between the feed and outlet provides part of the driving force for the transport of the ice bed upwards. The ice bed already has a tendency to float upwards.

The wash-front is easily detected by the high temperature gradient over a 10-20 mm zone.

6.6 Compressors

Both the compressors are designed to increase the pressure to 4.2 bara. Pressure control is achieved with a recycle valve.

6.7 Dissolver

The dissolver has to be heated to 95 °C. A temperature controller is used regulating the flow of the steam. For temperatures below 95 °C the flow of steam is increased and vice versa.

6.8 Heat exchangers

Only heat exchanger E02 is regulated, the other heat exchangers operate at maximum capacity and cannot be regulated as both incoming streams are fixed. These heat exchangers operate at design capacity. Some control mechanisms will be required for start-up procedures.

Heat exchanger E02 is regulated with a temperature sensor on the melt water coming out of the exchanger (str. nr. 36). Feed back operation wih a simple by-pass allows the flow to be regulated such that the melt water is not heated beyond 273K.

7 PROCESS SAFETY

The process safety can be determined with the chemicals in the process and the *Dow Fire & Explosion Index*. The *Dow Fire & Explosion Index* will be calculated for the units where the most dangerous chemicals of the process are present. Also a general HAZOP analysis of the process is given in appendix VIII.

7.1 Toxicity of the Chemicals

To determine the toxicity of the chemicals the MAC (maximum acceptable concentrations) and the LD_{50} (lethal dose for 50% population) values are used as an indication, see table 7.1

Table 7.1: MAC and LD_{50} values

	MAC value		LD_{50}
	ppm	mg/m^3	mg/kg (oral)
Ammonia	25	18	350
Water			$1.9\ 10^5$
Nitric Acid	2	5	2345
Ammonium Nitrate			?

It can be seen from table 7.1 that ammonia and nitric acid are the most toxic substances in this process.

7.2 Dow Fire & Explosion Index

The dangerous units in this process are the neutraliser (V04) and the dissolver (V05), because in the neutraliser the neutralisation reaction is exothermic and the dissolver works at a high temperature.
The risks of the units are ranked according to the *Dow Fire & Explosion Index*, see table 7.2.

Table 7.2: Dow Fire & Explosion Index

Dow Fire & Explosion Index	Risk
1-60	*light*
61-96	*moderate*
97-127	*intermediate*
128-158	*heavy*
≥159	*severe*

Both indexes for the neutraliser and the dissolver are in the light risk range.

7.3 HAZOP

From the HAZOP study (see appendix VIII) it can be concluded that the most important hazard in the process is freezing of solution which can cause blockage in or damage to equipment. In the process the pumps and heat exchangers are most vulnerable to break down due to freeze up.

Care has to be taken with ammonium nitrate on the filter due to the explosion hazard.. The ammonium nitrate filtrate must not dry on the filter because dry ammonium nitrate dust is highly explosive. Ammonium nitrate is unlikely to dry in the process and ammonium nitrate requires a very large activation energy for explosion. For these reasons this hazard and the risk is relatively small. The ammonium nitrate stores have to monitored to prevent even small quantities of ammonium nitrate to be stolen. Because of ammonium nitrate's explosive power and untracability it is often used in terrorist bombs.

8 ECONOMY

8.1 Investment Costs

The total investments of a chemical plant design can be divided into fixed capital costs and working capital costs (C&R, chapter 6):

$$C_T = C_F + C_W \qquad [8\text{-}1]$$

Where C_T = total investment costs (Nfl)
C_F = fixed capital costs (Nfl)
C_W = working capital costs (Nfl)

8.1.1 Fixed Capital Costs

The fixed capital costs consist of direct costs for equipment and installation, buildings, utilities and indirect costs for design, engineering and contigencies (unforeseen circumstances).
To determie an estimate for the fixed captal costs the factorial method (Lang's Method) is used.

$$C_F = L \cdot C_E \qquad [8\text{-}2]$$

$$L = F_1 \cdot F_2 \qquad [8\text{-}3]$$

$$C_e = C \cdot S^n \qquad [8\text{-}4]$$

Where: L = the Lang factor, which depends on the process
F_1 = factor based on direct costs ($1 + f_1 \ldots f_9$)
F_2 = factor based on indirect costs ($1 + f_{10} .. f_{12}$)
C_e = purchased (major) equipment costs (Nfl)
C = cost constant (given in table 6.2 C&R)
S = characteristic size parameter (given in table 6.2 C&R)
n = index for type of equipment (given in table 6.2 C&R)

When calculating the equipment costs two correctionfactors must be taken into account: the currencyfactor and the inflationfactor (with the help of indices).

8.1.2 Working Capital Costs

Working capital contains additional investments for start-up which can be recovered at end of plant-life. It can be estimated as 5-30 % of the fixed capital costs.

The investments cost will be reduced by taken into account a subsidy of the Economy, Ecology and Technology arrangement of the Dutch government.

8.2 Operating/Production Costs

The operating costs consists of fixed costs and variable costs.
Fixed costs:

- maintenance (labour and materials, 5-10% of fixed capital)
- operating labour (maximum of 15 % of total operating costs)
- laboratory costs (process monitoring/quality control, 20 % of operating labour costs, 2-4% of total operating costs)
- supervision (management, 20 % of operating labour costs)
- plant overheads (general costs, 50 % of operating labour costs)
- capital charges (10-20 % of fixed capital)
- rates (and any other local taxes, 1-2% of fixed capital)
- insurance (1-2% of fixed capital)
- licence fees and royalty payments (1 % of fixed capital, will add 1-5 % to sales price)

Variable costs:

- raw materials
- miscellaneous operating materials (e.g. safety clothes, instrument charts, gaskets, 10 % of maintenance costs)
- utilities (services)
- shipping and packaging (usually negligble)

Because the design will be incorporated in the existing plant, it will have to share in the company's general operating expenses such as:

- general overheads
- research and development costs
- sales expenses
- reserves

This will add an amount of 20-30 % of the direct production costs to the total production costs.

8.3 Sales Income

The incomes in this process come mainly from the savings that are made compared to the present process situation. In the present process the waste stream is sent to the sewer, which costs a certain amount of money per year, because of the nitrate contents in the waste stream. In the present process, liquid ammonia is evaporated and used in the ammonium nitrate plant. Because the liquid ammonia in this design will be used in the cooling process and will be delivered at the required pressure, it will not be necessary to evaporate this ammonia. This is also considered as income for the designed process.

Additional benefits come from the value of ammonium nitrate and highly pure water. In table 8.1 an overview is given of the costs and benefits of the design, see also appendix IX a,b.

Table 8.1: Overview costs and benefits

Costs	**M Nfl**		**M Nfl**
Total Investment Costs	7.4	Fixed Capital Costs	7.05
		Working Capital	0.35
Total Investment less subsidies	3.7		
Operating Costs (annual)	1.79	Variable Costs	0.22
		Fixed Costs	1.27
		Additional Costs	0.3
Benefits	**M Nfl**		**M Nfl**
Total Sales Income	2.26	Ammonium Nitrate	0.21
		Highly Pure Water	0.45
		Waste Water	0.41
		NH_3 evaporation	0.62

8.4 Economical Evaluation

The following criteria will be calculated to be able to compare the economic performance of the project.

- Cash-flow diagram including pay-back time
- Rate of return
- Discounted cash-flow rate of return (DCFRR)
- Sensitivity analysis

8.4.1 Cash-Flow Diagram

The net cash-flow, also called net future worth (NFW), at any time is the difference between the earnings and expenditure (investment costs and operating costs, excluding tax, depreciation, capital grants). The pay back time is the period of time needed to pay off the initial investment from income (the time taken to reach the break even point).

Figure 8.1 shows the cash-flow diagram of this project. From this diagram can be seen that the pay back time is about 60 years.

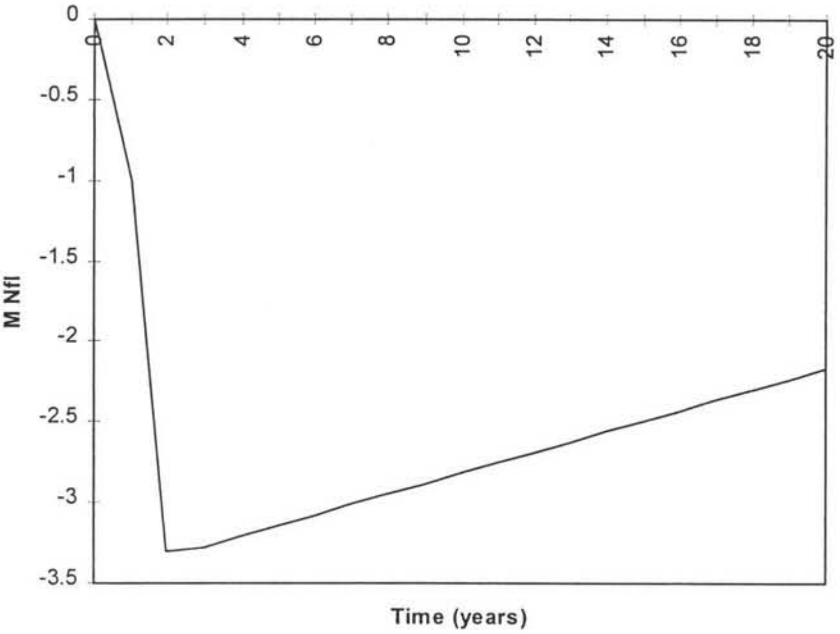


Figure 8.1: Cash flow diagram of project

8.4.2 Rate of Return (ROR)

The rate of return is a simple index of performance of the capital invested.

$$\mathrm{ROR} = \frac{\text{Cumulative Net Cash flow at end of project}}{\text{Life of project} * \text{Original investment}} * 100\% \tag{8-5}$$

The cumulative net cash flow at the end of the project is also called the total net future worth of project (Project NFW).

8.4.3 Discounted Cash-Flow Rate of Return (DCFRR)

The net cash flow in each year of the project is brought to its 'present' worth at the start of the project by discounting it at some chosen compound interest rate: net present worth (NPW). The cumulative net cash flow at the end of the project is then called the total net present worth of project (Project NPW), see equation [8-3].

$$\mathrm{NPW} = \frac{\text{Estimated net cash flow in year n (NFW)}}{(1+r)^n} \tag{8-6}$$

$$\mathrm{Pr\,oject\ NPW} = \sum_{n=1}^{n=t} \frac{\mathrm{NFW}}{(1+r)^n} \tag{8-7}$$

The discounted cash-flow rate of return is the interest rate at which the cumulative net present worth at the end of the project is zero, see equation 8-8. So it is the maximum rate the project could pay and still break even by the end of the project life. This value is found by trial-and-error calculations. The more profitable the project, the higher the DCFRR that it can afford to pay.

$$\sum_{n=1}^{n=t} \frac{\text{NFW}}{(1+r')^n} = 0 \qquad [8\text{-}8]$$

Where r' is the discounted cash-flow rate of return (%) and t the last year of the plant life.

In Table 8.2 the results of these criteria are presented.

Table 8.2: economic criteria

Criteria	**Value**	**Unit**
time of project t	20	yr
discount rate r	10	%
Project NFW	-2.17	M Nfl
Project NPW	-2.40	M Nfl
Rate of Return	-2.93	%
DCFRR	NA	%

Obviously the project is not economically viable under the present circumstances. The DCFRR cannot be calculated because the Project NPW is negative.

8.4.4 Sensitivity Analysis

In table 8.3 a number of parameters are varied by 10 % to calculate the effect on the cash-flow.

Table 8.3: Sensitivity analysis

Parameter	Design	Change	New Value	Effect on cash-flow
English Pound	3.2 Nfl/£	-10%	2.88	+33.7 %
		+10%	3.52	-33.5 %
Ammonium	236 Nfl/ton	-10%	212.4	-4.5 %
Nitrate		+10%	259.6	+4.5%
Water	3.5 Nfl/ton	-10%	3.15	-9.7%
		+10%	3.85	+9.7%
Electricity	0.1 Nfl/kW-h	-10%	0.09	+3.8%
		+10%	0.11	-3.8%
Nitric Acid	191 Nfl/ton	-10%	171.9	+0.2%
		+10%	210.1	-0.2%
Ammonia	335 Nfl/ton	-10%	301.5	+0.7%
		+10%	368.5	-0.7%
Steam	30 Nfl/ton	-10%	27	-25.5%
		+10%	33	+25.5%

8.5 Conclusions

The rate of return is negative, indicating the process is not economically productive. This is also why other criteria have negative values or cannot be calculated. An economic problem of the process is the low sales income. This is due to treating of a waste stream which has by definition a low economic value. Also it appears the largest equipment costs are the freeze concentration and freeze concentration seems the economic bottleneck.

Considering the high purity of the water the price given by Kemira seems below its real value. The investment costs are reduced by subsisidies. For further design stage, more subsidies may be possible.

In appendix IX f a (last minute) fax from SETEC is included. In this fax a cost estimation for reverse osmosis is made. A reduction in costs for the concentration step by using reversis osmosis instead of freeze concentration is estimated at 25 %. This would result in a pay-out time between 5 and 10 years.

9 CONCLUSIONS AND RECOMMENDATIONS

The process design for treatment of very diluted ammonium nitrate brine to produce highly concentrated ammonium nitrate and high purity water has been completed successfully. Unfortunately on the present basis of design the process does not appear economically viable without considerable subsidies.

From the waste stream the process design produces 884 tons of highly concentrated (90%) ammonium nitrate and 129 000 m^3 of high purity water (<1 ppm ammonium nitrate) per year. No by-products are produced. Furthermore the stream to the nitric acid plant (str. nr. 12) has been cooled from 66°C to 28°C. Approximately half of the ammonia feed to the ammonium nitrate plant has been vaporised and delivered at 4.2 bara and 32 °C as specified by Kemira. Enough liquid ammonia remains for the cooling of air Kemira require. Process integration has produced the above benefits to the ammonium nitrate and nitric acid processes at Kemira.

Economically the process requires a total investment of 7.4 M Nfl (including subsidies, 3.7 M Nfl) and has a pay-out time of approximately 60 years. A number of factors which affect the economic viability of the process design should be considered carefully.

Regarding the chosen process design, a choice between different concentration technologies had to be made on the basis of comparing operational costs. It was beyond the scope of this project to complete a full cost analysis including investment costs and detailed specifics of both reverse osmosis and freeze concentration. In the cost evaluation between concentration technologies the costs of regularly replacing the membranes for reverse osmosis were regarded as operation costs. In hindsight the costs of membrane replacement compare quite favourably to the surprisingly large investment necessary for the scraped surface heat exchangers in the freeze concentration and also the extra wash columns needed. As the difference between the operational costs between reverse osmosis (including regular replacing of membranes) and freeze concentration is relatively small, the total costs of concentration with reverse osmosis would most likely cost less. Considering the concentration step is by far the largest investment, reductions in costs could have a reasonable influence.

Regarding the situation at Kemira, a very diluted ammonium nitrate waste stream is treated and in general it is difficult to make upgrading of diluted waste streams profitable. Kemira has already installed a demister in one of their ammonium nitrate reactors and have plans for a demister in their second ammonium nitrate reactor. The demisters reduce the amount of entrained ammonium nitrate solution and the concentration of ammonium nitrate in the waste stream. The investments made in installing the demisters and also their effect on the total ammonium nitrate produced in the designed process are worthwhile considering. A more concentrated waste stream would produce considerably more revenues by producing much more ammonium nitrate.

Eutectic freeze crystallization appears to be positive in the process design. The concentrated ammonium nitrate brine is efficiently separated with the cooling disc crystalliser with wash column and filter into ammonium nitrate and high purity water.

Furthermore, the process design has been established on a sure but perhaps conservative basis. Some cost reduction is expected in refining, improvements and decreasing over-design margins.

A final recommendation is the investigation into injecting liquid carbon dioxide into the crystalliser and making use of clathrate structures.

In summary, the project is technically feasible and the process design is considered quite sound. However as such the project is not economically viable. In hindsight the high investment costs of freeze concentration probably make reverse osmosis the better alternative for the concentration step which would reduce the process costs considerably (approximately 25%). Still, with the given basis of design the major bottleneck appears to be the low revenues from products and the high equipment costs of the freeze concentration. The revenues are insufficient for the investments required and thus the economic feasibility of upgrading the waste stream without subsidies.

At Kemira the combination of reverse osmosis with eutectic freeze crystallization appears to be a positive option for which it surely is worthwile examining further.

10 LIST OF SYMBOLS AND MEANING

10.1 Greek Symbols

α	specific cake resistance	m^2/kg
$\Delta H_{s,w}$	freeze/melt enthalpy of water	kJ/kg
$\Delta H_{phase\ tr.}$	Enthalpy required for phase transition	kJ/kg
$\Delta_{cryst}H$	crystallisation enthalpy	J/kg
ΔP_{bed}	Pressure drop over the bed	Pa
ΔP_f	pressure drop over filter	Pa
ΔP_s	shell side pressure drop	Pa
ΔP_t	tube side pressure drop	Pa
$\Delta \rho_{c\text{-}b}$	density difference between brine and crystal	kg/m^3
ΔT	temperature difference	K
ΔT_{lm}	logarithmic mean temperature difference	°C
ΔT_m	mean temperature difference	°C
ε_{bed}	bed porosity	-
ε^*	bed porosity after recrystallisation	-
η_m	mechanical efficiency	-
η_h	hydraulic efficiency	-
η_c	compressor efficiency	-
η_p	overall pump efficiency	-
η_w	viscosity of water	Pa.s
μ	viscosity	Pa.s
$\phi_{m,L}$	liquid mass flow rate	kg/s
$\phi_{m,V}$	vapour mass flowrate	kg/s
$\phi_{v,L}$	liquid volumetric flow rate	m^3/s
$\phi_{v,V}$	vapour volumetric flow rate	m^3/s
$\phi_{v,F}$	volumetric flow through filter	m^3/s
$\phi_{v,n}$	volumetric flow through neutraliser	m^3/s
φ	angle of submergence	rad
ρ	density	kg/m^3
ρ_f	density of fluid	kg/m^3
ρ_L	liquid density	kg/m^3
ρ_V	vapor density	kg/m^3
τ_n	residence time in neutraliser	s
$\tau_{recryst}$	residence time in recrystalliser	s
τ_{EFC}	residence time in eutectic freeze crystallizer	s

10.2 Common Symbols

A_{HE}	heat transfer area	m^2
A_{CDCC}	heat exchange area required in CDCC	m^2
A_{comm}	cross sectional area of commercial wash column	m^2
A_{disc}	surface area of the cooling disc (one side)	m^2
A_F	filter area	m^2
A_s	shell cross flow area	m^2
A_t	tube cross flow area	m^2
A_{TNO}	cross area of the TNO-wash column	m^2
A_{vessel}	cross sectional area of flash vessel	m^2
A_{wc}	cross sectional area of wash column	m^2
$c_{p,w}$	specific heat content of water	J/(kg °C)
$c_{P,S}$	specific heat of ice crystals	J/kg °C
C_{Comm}	capacity of commercial wash column	kg/(m^2 h)
C_e	purchased (major) equipment costs	Nfl
C_F	fixed capital costs	Nfl
C_T	total investment costs	Nfl
C_W	working capital costs	Nfl
C_{wc}	capacity of wash column	kg/(m^2 h)
d	diameter	m
$d_{crystal}$	diameter of crystal	m
d_p	diameter of particle	m
D_c	diameter of coil	m
D_{coil}	thickness of the coil	m
D_{column}	diameter of column	m
D_v	diameter of vessel	m
D_{vessel}	diameter of flash vessel	m
D_{wc}	diameter of wash column	m
F_{in}	fresh feed coming into FC unit	kg/s
F_{EFC}	feed to EFC	m^3/s
F_{SSHE}	total flow through SSHE	kg/s
g	acceleration due to gravity	m/s^2
G_s	mass velocity (mass flow per tube unit area)	kg/m^2 s
G_t	mass velocity (mass flow per shell unit area)	kg/m^2 s
h_{disc}	thickness of cooling disc	m
h_i	inside fluid film coefficient	W/(m^2 °C)
h_{id}	inside dirt coefficient	W/(m^2 °C)
h_o	outside fluid film coefficient	W/(m^2 °C)
h_{od}	outside dirt coefficient (fouling factor)	W/(m^2 °C)
H	height of dissolver vessel	m
H_{bed}	height of the bed	m
H_{column}	height of column	m
H_n	height of neutraliser tank	m
j_h	heat-transfer factor	-
k_f	fluid thermal conductivity	W/(m °C)
k_w	thermal conductivity of the tube wall material	W/(m °C)
M	mass flow	kg/s

N	number of discs required	#
N_r	rotation frequency	1/s
N_{tube}	number of tubes in the wash column	#
N_{TNO}	number of tubes in TNO-wash column	#
P	pressure	Pa
P_{in}	suction pressure	Pa
P_p	pump shaft power	kW
$P_{S,isen}$	isentropic power	kW
P_{real}	real power	kW
P_v	vapour pressure	Pa
Q	heat duty	W
$Q_{agitator}$	heat generated by the agitator	kW
Q_F	total volume of filtrate per unit area	m^3/m^2
Q_{ice}	cooling energy required for ice production	W
Q_{pump}	heat generated by the recirculation pump	W
Q_{to_t}	total energy of undercooled brine required in FC unit	W
Q_{CDCC}	cooling required for ice and AN formation	W
R	filter medium resistance	-
S_s	solid fraction in slurry	kg/kg
S_c	solid fraction in cake	kg/kg
$t_{hold\text{-}up}$	hold up time	s
t_s	rise or sedimentation time	s
T	temperature	°C
T_{in}	temperature of feed in FC unit	K
T_f	temperature of feed to SSHE	K
$\hat{u}_V$	maximum design vapor velocity	m/s
U	heat transfer coefficient	W/(m^2 °C)
U_{CDCC}	heat transfer coefficient	W/(m^2 °C)
v	Superficial velocity	m/s
v_s	rise or settling velocity	m/s
V_{brine}	volume CDCC (without discs)	m^3
V_c	volume of the coil	m^3
V_{discs}	volume of cooling discs	m^3
V_L	required liquid volume	m^3
V_n	volume neutraliser	m^3
$V_{recryst}$	volume of recrystalliser	m^3
V_{total}	volume of brine and discs (volume column)	m^3
V_v	volume vessel	m^3

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APPENDIX I BASIS OF DESIGN

Incoming Feed Streams

Components	Stream	E401	E421	E402	E403	Total Feed	Boundary limits
NH_3 (aq)	(mg/l)			4000	1500	326	<400
HNO_3 (l)	(mg/l)	4260	3700			3694	<3694
AN (s)	(mg/l)	1000	1000	4000	2000	1242	
Flow	(kg/h)	21000	9000	2400	830	33230	
Pressure	(bara)	0.3	0.3	0.3	0.3	9	
Temperature	(°C)	66	66	66	66	66	

Conditions other incoming streams

		NH_3 storage	NH_3 process	HNO_3	air
Flow	(ton/day)		295		
Flow	(m3/hr)				115000
Density	(kg.m3)				1.2
gew %		99.8	99.8	57.5	
Pressure	(bara)	1	22.5	14	1
Temperature	(°C)	-33	19	40	-5 to 25
State		liquid	liquid	liquid	gas

Conditions Outcoming Streams

	Units	NH_3 to AN plant	Feed to HNO_3 plant	Air to CAN plant	Water
Mass flow	(ton/hr)	295 /day	17 /hr	13.8 /hr	
Pressure	(barg)	3.2	10	0.038	6
Pressure	(bara)	4.2	11	1.038	7
Temperature	(°C)	60	<40	1 to 4	max 60

Demands Product Concentrations

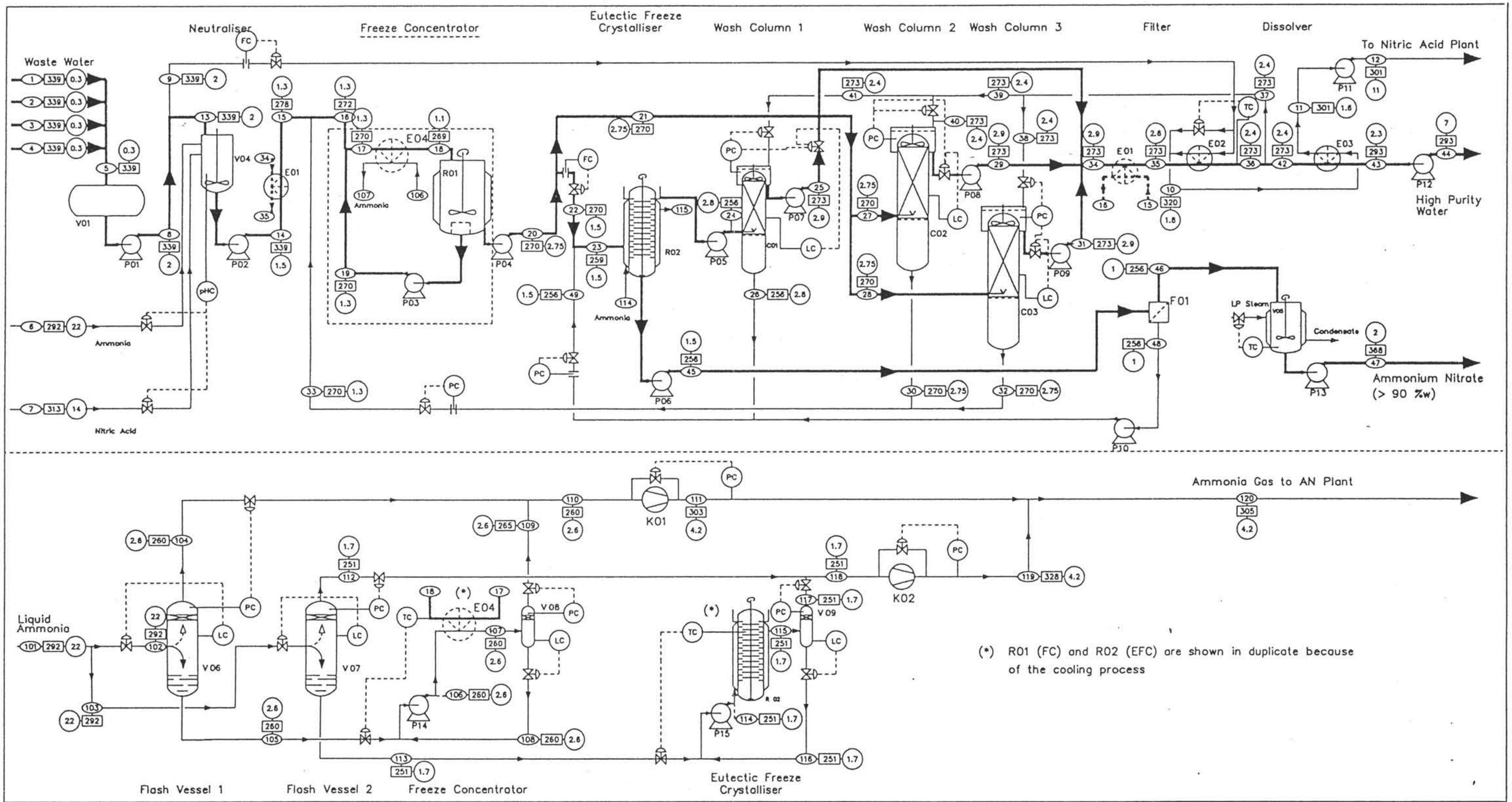
Water	<50ppm AN
	<10ppm HNO_3
AN	>90 gew %

Prices Feedstocks and Utilities (Source = Kemira)

NH_3	335	Nfl/ton
HNO_3	191	Nfl/ton
Electricity	0.1	Nfl/kwh
Water	3.5	Nfl/m^3
AN	236	Nfl/ton
IE	65.2	Nfl/IE

APPENDIX II PROCESSFLOW INFORMATION

Appendix II a Process Flow Diagram



(*) R01 (FC) and R02 (EFC) are shown in duplicate because of the cooling process

R 1	FREEZE CONCENTRATOR
R 2	EUTECTIC FREEZE CRYSTALLISER
E 1	HEAT EXCHANGER
E 2	HEAT EXCHANGER
E 3	HEAT EXCHANGER
E 4	SURFACE SCRAPED HEAT EXCHANGER
C 1	WASH COLUMN
C 2	WASH COLUMN
C 3	WASH COLUMN
F 1	FILTER
V 1	TANK 401
V 4	NEUTRALISER
V 5	DISSOLVER
V 6	FLASH VESSEL
V 7	FLASH VESSEL
V 8	KNOCKOUT DRUM
V 9	KNOCKOUT DRUM
K 1	COMPRESSOR
K 2	COMPRESSOR
P 1	PUMP
P 2	PUMP
P 3	PUMP
P 4	PUMP
P 5	PUMP
P 6	PUMP
P 7	PUMP
P 8	PUMP
P 9	PUMP
P 10	PUMP
P 11	PUMP
P 12	PUMP
P 13	PUMP
P 14	PUMP
P 15	PUMP

Ammonium Nitrate Waste Water Purification Proces

Dennis Sterzenbach
Josefine Treijtel
Martin Vervoorn
Reinout von Zevenbergen

FVO 3208
Date: 24/12/1997 .
TU Delft

⬭	Stroom Nr.
▭	Temperature (K)
○	Pressure (bara)

II b Properties of Chemicals

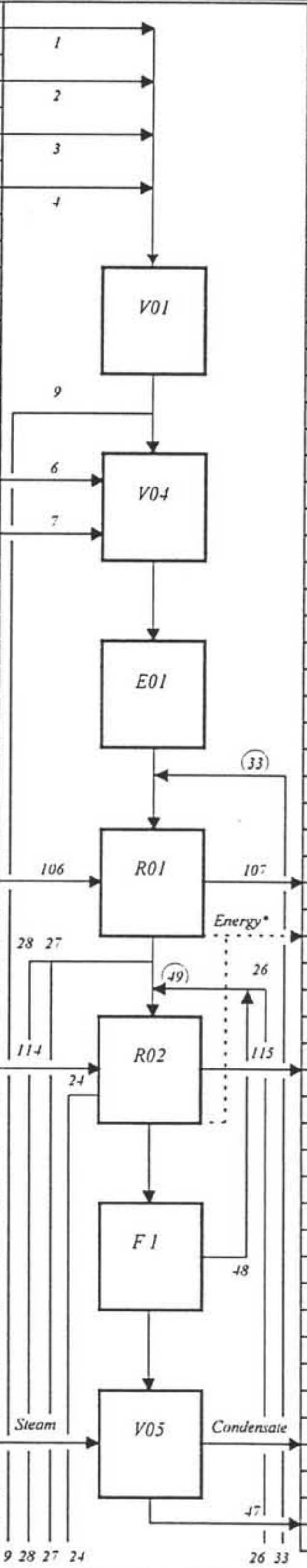
Name	Formula	Mole mass	Boiling point	Melting point	Solubility	Density
		g/mol	°C	°C	g/100 ml 20 °C	10^3 kg/m^3
Ammonia	NH_3	17.03	-33	-78	53	0.77
Water	H_2O	18.02	100	0	-	0.998 (20°C)/0.917 (0°C)
Air	80% N_2,20% O_2 0,03% CO2	29	-193	-213	-	$1.293*10^{-3}$
Nitric acid	HNO_3	63.01	83	-42	completely	1.5027
Ammonium nitrate	NH_4NO_3	80.04	210	170	1910	1.725

Name	ΔH melt/ΔH evap.*	ΔH cryst	Cp	Explosion limit	MAC	LD_{50}
	kJ/kg	kJ/kg	kJ/kgK	vol % in air	ppm / mg/m^3	mg/kg (oral)
Ammonia	1370*	-	2.06	15-29	25 / 18	350
Water	-334 / 2260*	-	4.20	-	-	190,000
Air	210*	-	1.00	-	-	-
Nitric acid	-	-528.2	2.70	-	2 / 5	2345
Ammonium nitrate	-	321.0	1.74	-	-	-

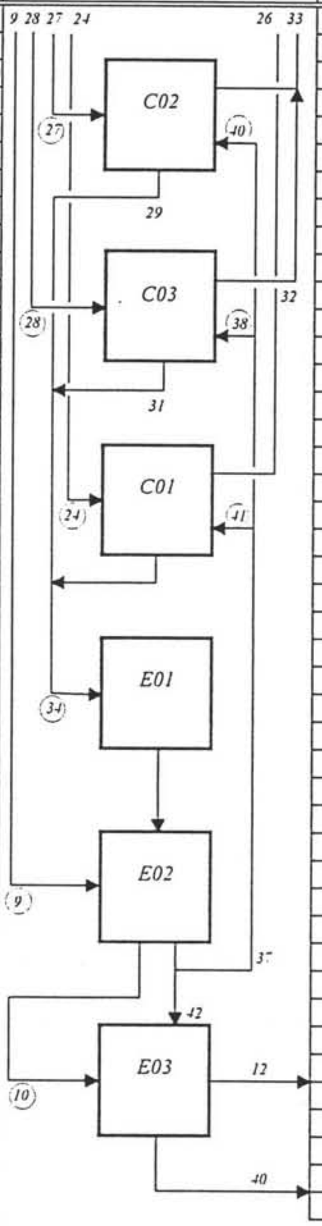
II c Mass and Heat Balances

Balance without ammonia cooling cycle

IN		forwards	retour	OUT	
M (kg/s)	Q (kW)	M Q	M Q	M (kg/s)	Q (kW)
5.8333	1617.0				
2.5000	693.0				
0.6667	184.8				
0.2306	63.9				
0.0032	0.3				
0.0012	0.2				
			16.4705 -191.6		
1.4666	-1182.0			1.4666	342.0
					-35.1
			1.5538 -110.3		
0.1449	-122.7			0.1449	30.8
0.0094	27.9			0.0094	5.3
				0.0307	12.3



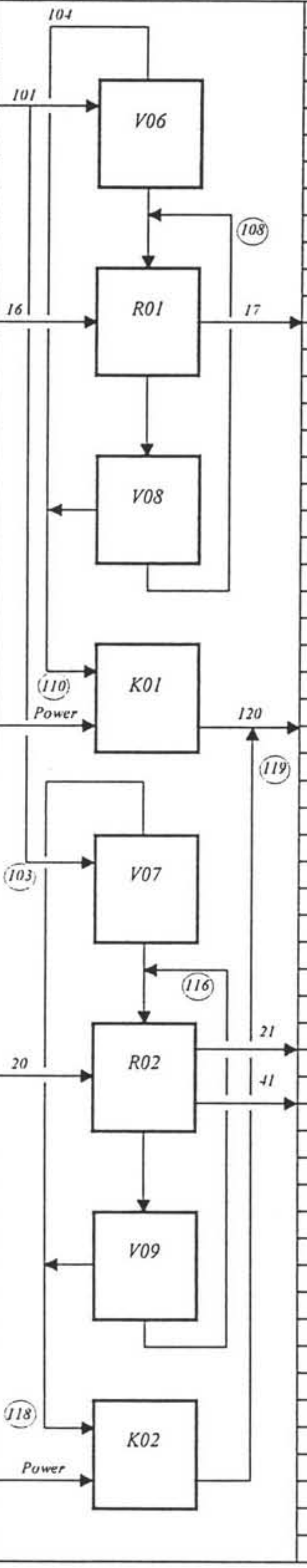
*Energy lost due to concnetrating ammonium nitrate



IN M (kg/s)	IN Q (kW)	forwards M	forwards Q	retour M	retour Q	OUT M (kg/s)	OUT Q (kW)
		10.2940	-808.5				
				8.4227	0.0		
		10.2940	-808.5				
				8.4227	0.0		
		1.8218	-251.2				
				1.6599	0.0		
		22.9874	-1538.1				
		4.7222	1309.0				
		4.7222	927.1			4.7222	550.6
						4.4820	376.5
10.8559	1282.3	← Total		→		10.8559	1282.3

II c Mass and Heat Balances

Ammonia Balance



IN M (kg/s)	IN Q (kW)	forwards M Q		retour M Q	OUT M (kg/s)	OUT Q (kW)
1.4480	-965.8		101			
			108	0.2933 -236.4		
20.9832	-97.6		16 / 17		20.9832	-1621.7
		1.3138 647.7	110			
	133.1		Power / 120		1.4480	868.7
			119	0.1342 87.8		
		0.1342 -876.3	103			
			116	0.0290 -24.5		
1.9489	-114.9		20 / 21		1.8218	-251.2
			41		0.1270	-17.2
		0.1342 64.0	118			
	23.8		Power			
24.3800	-1021.4	◄	Total	►	24.3800	-1021.4

II d Process Streams and Component Balance

Stream number		1 (E401)	2 (E421)	3 (E402)	4 (E403)	5	6	6 design	7
Component	mollmass	M	M	M	M	M	M	M	M
NH_3 (aq)	17.03			0.0027	0.0003				
NH_3 (l)	17.03						0.0032	0.0032	
NH_3 (g)	17.03								
AN (s)	80.04								
NH_4^+	18.03	0.0013	0.0006	0.0006	0.0001	0.0058			
NO_3^-	62.01	0.0290	0.0111	0.0021	0.0004	0.0425			0.0007
H^+	1.00	0.0004	0.0001			0.0004			0.0000
Water (l)	18.02	5.8026	2.4882	0.6613	0.2297	9.1819	0.0000		0.0005
Water (s)	18.02								
Total		5.8333	2.5000	0.6667	0.2306	9.2306	0.0032	0.0032	0.0012
weight fraction AN		0.0010	0.0010	0.0040	0.0020	0.0028	0.0000	0.0000	0.0000
P (bara)		0.3	0.3	0.3	0.3	0.3	22.0	22.0	14.0
T (K)		339	339	339	339	339	292	292	313
Q (kW)		1617.0	693.0	184.8	63.9	2558.7	0.3	0.3	0.2

Stream number	8	9	10	11	12	13	14	15	16
Component	M	M	M	M	M	M	M	M	M
NH_3 (aq)									
NH_3 (l)									
NH_3 (g)									
AN (s)									
NH_4^+	0.0058	0.0030	0.0030	0.0030	0.0030	0.0028	0.0062	0.0062	0.2659
NO_3^-	0.0425	0.0218	0.0218	0.0218	0.0218	0.0208	0.0214	0.0214	0.9146
H^+	0.0004	0.0002	0.0002	0.0002	0.0002	0.0002			
Water (l)	9.1819	4.6973	4.6973	4.6973	4.6973	4.4846	4.4851	4.4851	19.8026
Water (s)									
Total	9.2306	4.7222	4.7222	4.7222	4.7222	4.5083	4.5127	4.5127	20.9832
weight fraction AN	0.0028	0.0028	0.0028	0.0028	0.0028	0.0028	0.0061	0.0061	0.0563
P (bara)	2.0	2.0	2.0	2.0	11.0	2.0	1.5	1.3	1.3
T (K)	339	339	320	301	301	339	339	278	272
Q (kW)	2558.7	1309.0	927.1	550.6	550.6	1249.7	1250.2	94.0	-97.6

Stream number	17	18	19	20	21	22	23	24	25
Component				M	M	M	M	M	M
NH_3 (aq)									
NH_3 (l)									
NH_3 (g)									
AN (s)									
NH_4^+	5.6905	5.6905	5.4246	0.2659	0.2597	0.0062	0.1543	0.1389	
NO_3^-	19.5712	19.5712	18.6566	0.9146	0.8932	0.0214	0.5306	0.4776	
H^+									
Water (l)	339.7382	339.7382	319.9356	15.6850	15.3175	0.3674	1.2640	0.8410	1.6195
Water (s)				4.1176	4.1176			0.3644	0.4049
Total	365.0000	365.0000	344.0168	20.9832	20.5881	0.3951	1.9489	1.8218	2.0243
weight fraction AN	0.0692	0.0692	0.0700	0.0700	0.0700	0.0700	0.3514	0.4230	0.0000
P (bara)	1.3	1.1	1.3	2.8	2.8	1.5	1.5	2.8	2.9
T (K)	270	269	270	270	270	270	259	256	273
Q (kW)	-4246.4	-5779.4	-4002.3	-1621.7	-1617.1	-4.6	-114.9	-251.2	-135.4

Stream number	26	27	28	29	30	31	32	33	34
Component	M	M	M	M	M	M	M	M	M
NH_3 (aq)									
NH_3 (l)									
NH_3 (g)									
AN (s)									
NH_4^+	0.1389	0.1299	0.1299		0.1299		0.1299	0.2597	
NO_3^-	0.4776	0.4466	0.4466		0.4466		0.4466	0.8932	
H^+									
Water (l)	0.8410	7.6588	7.6588	8.3852	7.6588	8.3852	7.6588	15.3175	18.3899
Water (s)		2.0588	2.0588	2.0963		2.0963			4.5975
Total	1.4575	10.2940	10.2940	10.4815	8.2352	10.4815	8.2352	16.4705	22.9874
weight fraction AN	0.4230	0.0700	0.0700	0.0000	0.0700	0.0000	0.0700	0.0700	0.0000
P (bara)	2.8	2.8	2.8	2.9	2.8	2.9	2.8	1.3	2.9
T (K)	256	270	270	273	270	273	270	270	273
Q (kW)	-103.5	-808.5	-808.5	-701.3	-95.8	-701.3	-95.8	-191.6	-1538.1

Stream number	35	36	37	38	39	40	41	42	43
Component	M	M	M	M	M	M	M	M	M
NH_3 (aq)									
NH_3 (l)									
NH_3 (g)									
AN (s)									
NH_4^+									
NO_3^-									
H^+									
Water (l)	21.8458	22.9874	18.5054	8.4227	10.0827	8.4227	1.6599	4.4820	4.4820
Water (s)	1.1416								
Total	22.9874	22.9874	18.5054	8.4227	10.0827	8.4227	1.6599	4.4820	4.4820
weight fraction AN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
P (bara)	2.6	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.3
T (K)	273	273	273	273	273	273	273	273	293
Q (kW)	-381.9	0.0	0.0	0.0	0.0	0.0	0.0	0.0	376.5

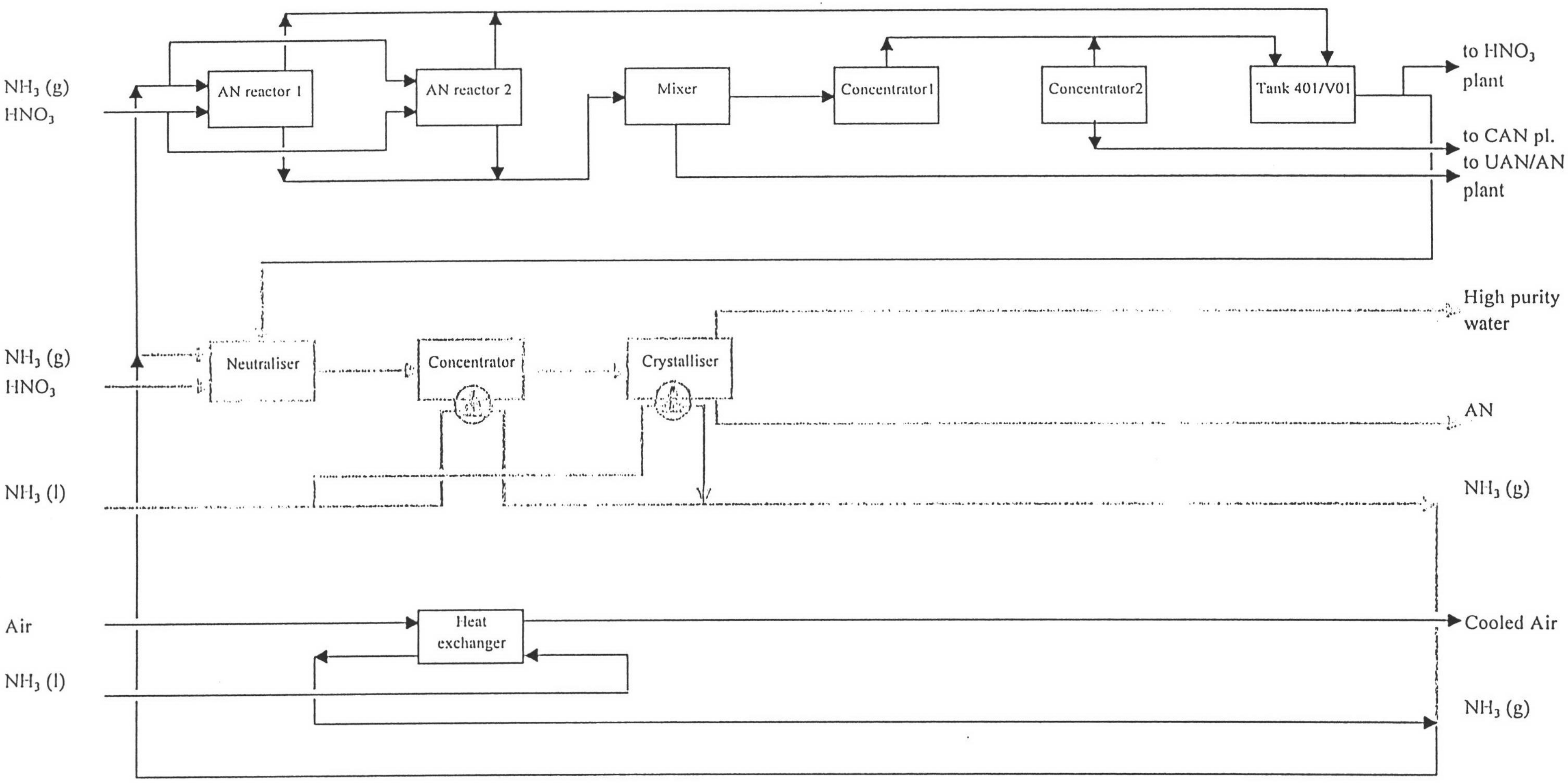
Stream number	44	45	46	47	48	49
Component		M	M	M	M	M
NH_3 (aq)						
NH_3 (l)						
NH_3 (g)						
AN (s)		0.0254	0.0254			
NH_4^+		0.0097	0.0005	0.0062	0.0092	0.1481
NO_3^-		0.0333	0.0017	0.0214	0.0316	0.5092
H^+						
Water (l)	4.4820	0.0586	0.0031	0.0031	0.0556	0.8965
Water (s)						
Total	4.4820	0.1270	0.0307	0.0307	0.0963	1.5538
weight fraction AN	0.0000	0.4230	0.4230	0.9000	0.4230	0.4230
P (bara)	7.0	1.5	1.0	2.0	1.0	1.5
T (K)	293	256	256	368	256	256
Q (kW)	376.5	-17.2	-10.3	12.3	-6.8	-110.3

stream number		101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120
component	molmass	M	M	M	M	M				M	M	M	M	M	M	M	M	M	M	M	M
NH_3 (l)	17.03	1.4480	1.3138	0.1342		1.1732	1.4666	0.2933	0.2933					0.1159	0.1449	0.0290	0.0290				
NH_3 (g)	17.03				0.1406			1.1732		1.1732	1.3138	1.3138	0.0182			0.1159		0.1159	0.1342	0.1342	1.4480
Total		1.4480	1.3138	0.1342	0.1406	1.1732	1.4666	1.4666	0.2933	1.1732	1.3138	1.3138	0.0182	0.1159	0.1449	0.1449	0.0290	0.1159	0.1342	0.1342	1.4480
P (bara)		22	22	22	2.6	2.6	2.6	2.6	2.6	2.6	2.6	4.2	1.7	1.7	1.7	1.7	1.7	1.7	1.7	4.2	4.2
T (K)		292	292	292	260	260	260	260	260	260	260	303	251	251	251	251	251	251	251	328	305
Q (kW)		-965.8	-876.3	-89.5	69.3	-945.6	-1182.0	342.0	-236.4	578.4	647.7	780.9	8.7	-98.2	-122.7	30.8	-24.5	55.3	64.0	87.8	868.7

IIe

Process integration scheme

Designed process integrated in the existing ammonium nitrate plant



——— = already present at the Kemira ammonium nitrate plant

- - - - = designed

APPENDIX III MOLLIER DIAGRAMS AMMONIA

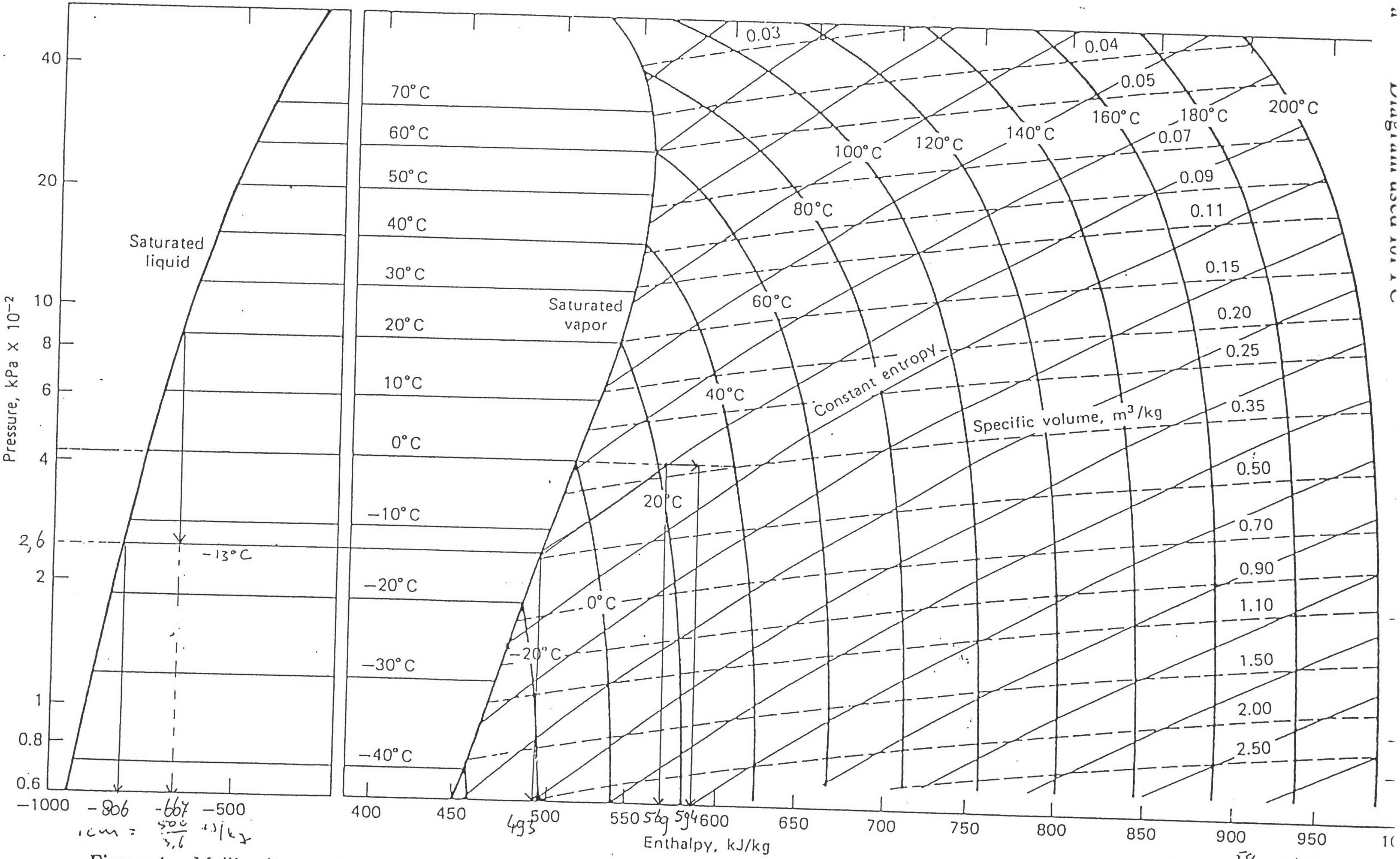


Figure 1. Mollier diagram for ammonia. Courtesy of Elliot Co. To convert kPa to atm divide by 101.3. To convert kJ to kcal divi

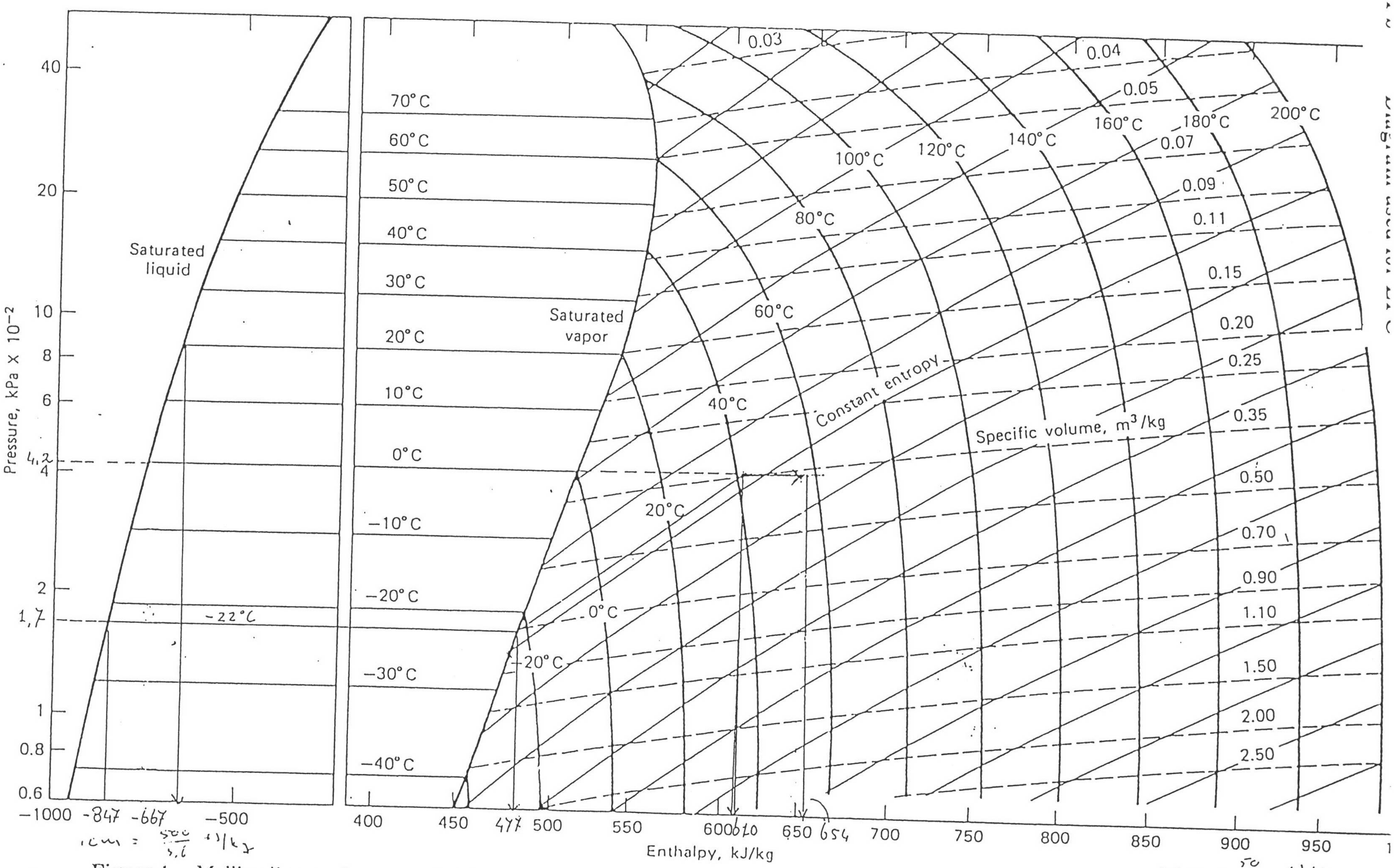


Figure 1. Mollier diagram for ammonia. Courtesy of Elliot Co. To convert kPa to atm divide by 101.3. To convert kJ to kcal divide by 4.184.

APPENDIX IV ENERGY CALCULATIONS FOR PROCESS-COMPARISON

IV a Summary

Method	Energy Consumption (kW)				
	preconcentration		crystallisation		total*
	membrane	compressor	compressor	thermal	
RO+EFC	112.69	79.41	18.92	NA	211.02
FC+EFC	NA	186.89	14.25	NA	201.14
RO+evap	112.69	79.41	0	260.45	278.91
evaporation	NA	0	0	5964.57	1988.19

*Converted to electrical energy (electrical = 1/3x thermal)

Costs per year

These are costs calcutated for Kemira's situation (in presence of fluid ammonia) For an honest comparison between the four possibilities we should take the ammonia cooling cycle into account.

Method	Costs of energy consumption per year				
	Membrane	preconcentration	crystallisation		total
		compressor	compressor	thermal	
RO+EFC	90151	63528	15139	0	168817
FC+EFC	0	149513	11401	0	160914
RO+evap	90151	63528	0	69452	223131
evaporation	0	0	0	1590553	1590553

1 kWh =	0.10	Nfl.
total plant hours	8000	hours

IV b Calculation of energy use of concentration of AN solution with RO

Permeability factor	K	1.00E-12	m^3/m^2Pas	
Max. operating pressure	DPmax	100	Bar	
Min. pressure difference across membrane	DPmax-DPmax	30	Bar	
Max. osmotic pressure	DPmax	70	Bar	
Water density at 70 bar, 298 K	rw	999.57	kg/m^3	
Molecular weight water	Mw water	18.02	g/mol	
Molecular weight ammoniumnitrate	Mw NH4NO3	80.04	g/mol	
Universal gas constant	R	8.314	J/molK	
Temperature	T	298	K	
Molar volume of water	v	1.80E-05	m^3/mol	
Activity coefficient of water at 70 bar, 300K	aw	0.9503	-	
Activity coefficient of permeate	ap	1.0000	-	
Maximum activity coefficient of retained solution	as	0.9503	-	
Concentration of ammoniumnitrate for which		14.400	w%	*See Mercury calculation
activity coefficient = 0.9503 (from Mercury calc.)	c	12.587	w%	*See Mercury calculation
Total Flow	Ftotal	4.5114	kg/s	
Concentration of ammoniumnitrate flow in	c NH4NO3	0.5973	w%(g/g)	
Ammoniumnitrate flow	F NH4NO3	0.0269	kgAN/s	
Mass balance RO unit: Retained flow	Fretained	0.2141	kg/s	
Permeate flow	Fpermeate	4.2973	kg/s	
Volumetric flow permeate	Fvol permeate	0.0043	m^3/s	
Maximum waterflux through membrane	J	3.00E-06	m^3/m^2s	J=K(DP-DP)
Required membrane surfacearea	Atot	1435.30	m^2	Atot=Fvol permeate/J
Volumetric flow feed	Fvol	0.00452	m^3/s	
Pump efficiency	h	0.75	-	
Electric Power compression pump	W Pcomp	60272	W	W Pcomp=(F/hr)DP
Electric Power circulation pump	W Pcirc	19137	W	W Pcirc=10Atot/h
Total Electric Power consumption	Wtot	79.410	kW	
Electricity costs	E.u.	0.10	Nfl/kWh	
Electricity costs p.a.	e	67022	Nfl/year	e=(W Pcomp+ W Pcirc)xE.u.
Compression pump cost	p1	32512	Nfl	p1=10^(3.4+0.4log(Power/100))
Circulation pump cost	p2	20547	Nfl	p2=10^(3.4+0.4log(Power/100))
Membrane costs	m	90151	Nfl/year	m=10^(3.1+0.86log(Atot/10))
Membrane costs in kW equivalents		112.7	kW	
Total Costs	TC	167785	Nfl./year	TC=e+m+(p1+p2)*0.2

IV c Calculation of energy use of concentration of AN solution with FC

Feed to FC	Ftotal	4.511 kg/s
	fraction AN	0.005973
feed temperature	Tfeed	298.150 K
freezing point	Tfreeze	268.150 K (-5°C)
	ΔT	10.000 K
	Tammonia	258.150 K
specific heat	Cp (water)	4.180 kJ/kgK
heat of melting of ice	ΔHmelt	334.548 kJ/kg
stream water out	Fwater	4.297 kg/s
	Q cooling	565.726 kW
	Q ice formation	1437.652 kW
	Qtotal	2003.377 kW
evaporation of ammonia (at 10 bar and 25°C see Mollier diagram)	ΔHevap	1172 kJ/kg
	Fammonia	1.7094 kg/s
	Fammonia	147.689 ton/day
compressing NH3	ΔHcomp	109.333 kJ/kg
electrical power consumption	Wcomp	186.891 kW
Coefficient of performence	COP	10.720

IV d Calculation of energy use of EFC after RO

feed to EFC	Ftotal	0.214	kg/s
	fraction AN	0.12587	
feed temperature	Tfeed	298.150	K
eutectic point	Teut	256.250	K (-16.9°C)
temperature gradient	ΔT	10.000	K
specific heat	Cp (water)	4.180	kJ/kgK
heat of melting of ice	ΔHmelt	334.548	kJ/kg
heat of solution of AN	ΔHsol	320.961	kJ/kg
stream water out	Fwater	0.187	kg/s
stream AN out	F AN	0.027	kg/s
energy required	Q cooling	37.493	kW
	Q ice formation	62.603	kW
	Q salt formation	8.649	kW
	Q total	108.745	kW
evaporation of ammonia (at 10 bar and 25°C see Mollier diagram)	ΔHevap	1157	kJ/kg
	Fammonia	0.094	kg/s
	Fammonia	8.121	ton/day
compressing NH3	ΔHcomp	201.333	kJ/kg
electrical power consumption	Wcomp	18.923	kW
coefficient of performence	COP	5.747	

IV e Calculation of energy use of EFC after FC

feed to EFC	Ftotal	0.214 kg/s
	fraction AN	0.12587
feed temperature	Tfeed	268.150 K
eutectic point	Teut	256.250 K (-16.9°C)
temperature gradient	ΔT	10.000 K
specific heat	Cp (water)	4.180 kJ/kgK
heat of melting of ice	ΔHmelt	334.548 kJ/kg
heat of solution of AN	ΔHsol	320.961 kJ/kg
stream water out	Fwater	0.187 kg/s
stream AN out	F AN	0.027 kg/s
energy required	Q cooling	10.648 kW
	Q ice formation	62.603 kW
	Q salt formation	8.649 kW
	Q total	81.900 kW
evaporation of ammonia (at 10 bar and 25°C see Mollier diagram)	ΔHevap	1157 kJ/kg
	Fammonia	0.0708 kg/s
	Fammonia	6.116 ton/day
compressing NH3	ΔHcomp	201.333 kJ/kg
electrical power consumption	Wcomp	14.252 kW
coefficient of performence	COP	5.747

IV f Calculation of energy use of evaporation after RO

feed to evaporator	Ftotal	0.214	kg/s
	fraction AN	0.126	
	Fwater	0.187	kg/s
	FAN	0.027	kg/s
pressure	P	1.000	bar
temperature	Tfeed	339.150	K (66°C)
	Tvap step1	373.928	K (101°C)
	Tvap step2	373.150	K (102°C)
	ΔT	5.000	K
	Tsteam step 1	378.150	K
	Tsteam step 2	373.150	K
specific heat	Cp (water)	4.180	kJ/kgK
heat of evaporation of water	ΔHvap	2260.000	kJ/kg
heat of solution of AN	ΔHsol	320.961	kJ/kg
stream water out	Fwater	0.187	kg/s
stream AN out	F AN	0.027	kg/s
enthalpy (from steam tables)	Hwater (373.15 K)	416.064	kJ/kg
	Hsol 1	419.316	kJ/kg
	Hsol 2	416.064	kJ/kg
	Hsteam1 (395 K)	2686.080	kJ/kg
	Hsteam2 (390 K)	2676.000	kJ/kg
	Hfeed	277.196	kJ/kg
step 1	Fin	0.214	kg/s
	Fsteam out	0.094	kg/s
	Fsol out	0.121	kg/s
	%w	0.224	
	Qfeed	59.341	kW
	Qsteam	251.320	kW
	Qsolution	50.532	kW
	Qtotal 1	242.512	kW
step 2	F in	0.121	kg/s
	condensor duty	0.95	
	ΔHcondensor	2156.515	kg/s
	Qin	252.304	kW
	Fsteam	0.089456	kg/s
	Qvap	9.285	
	Qcrystallisation	8.649	kW
	Qtotal 2	17.934	kW
	Qtotal	260.445	kW

IV g Calculation of energy use of evaporation of total waste stream

feed to evaporator	Ftotal	4.511	kg/s
	fraction AN	0.006	
	Fwater	4.484	kg/s
	FAN	0.027	kg/s
pressure	P	1.000	bar
temperature	Tfeed	339.150	K (66°C)
	Tvap step1	373.928	K (101°C)
	Tvap step2	374.665	K (102°C)
	ΔT	5.000	K
	Tsteam step 1	379.665	K
	Tsteam step 2	374.665	K
specific heat	Cp (water)	4.180	kJ/kgK
heat of evaporation of water	ΔHvap	2260.000	kJ/kg
heat of solution of AN	ΔHsol	320.961	kJ/kg
stream water out	Fwater	4.484	kg/s
stream AN out	F AN	0.027	kg/s
enthalpy (from steam tables)	Hwater (373.15 K)	416.064	kJ/kg
	Hsol 1	419.316	kJ/kg
	Hsol 2	422.395	kJ/kg
	Hsteam1 (395 K)	2689.134	kJ/kg
	Hsteam2 (390 K)	2679.054	kJ/kg
	Hfeed	277.196	kJ/kg
step 1	Fin	4.511	kg/s
	Fsteam out	2.242	kg/s
	Fsol out	2.269	kg/s
	%w	0.012	
	Qfeed	1250.534	kW
	Qsteam	6029.606	kW
	Qsolution	951.494	kW
	Qtotal 1	5730.567	kW
step 2	F in	2.269	
	condensor duty	0.95	
	ΔHcondensor	2159.416	kg/s
	Qin	5793.362	kW
	Fsteam	2.142496	kg/s
	Qvap	225.358	
	Qcrystallisation	8.649	kW
	Qtotal 2	234.007	kW
	Qtotal	5964.574	

IV h Equations used in energy calculations

EFC and FC

$$Q_{cooling\ required} = (T_{feed} - T_{eut}) * C_p * F_{solution\ to\ EFC} \tag{1}$$

$$Q_{ice\ formation} = \Delta H_{melt} * F_{water\ out} \tag{2}$$

$$Q_{salt\ formation} = \Delta H_{solution} * F_{AN} \tag{3}$$

$$Q_{total} = Q_{ice\ formation} + Q_{salt\ formation} \tag{4}$$

$$F_{ammonia} = \frac{Q_{tot}}{\Delta H_{flash}} \tag{5}$$

$$W_{compressor} = F_{ammonia} * \Delta H_{compressor} \tag{6}$$

Evaporation of water in two steps

step 1

$$Q_{feed} = H_{feed} * F_{in} \tag{7}$$

$$Q_{steam} = H_{steam} * F_{steam\ out} \tag{8}$$

$$Q_{solution} = H_{solution} * F_{solution\ out} \tag{9}$$

$$Q_{total\ 1} = Q_{steam} + Q_{solution} - Q_{feed} \tag{10}$$

step 2

$$\Delta H_{condensor} = (H_{steam} - H_{water}) * 0.95 \tag{11}$$

$$Q_{in} = Q_{solution} + \Delta H_{condensor} * F_{steam\ out} \tag{12}$$

F_{steam} can be calculated from the energy balance:

$$Q_{in} = F_{steam} * (H_{steam} - H_{solution}) + F_{in} * H_{solution} \tag{13}$$

$$Q_{vap} = F_{water\ left} * \Delta H_{vap} \tag{14}$$

$$Q_{cryst} = F_{AN} * \Delta H_{sol} \tag{15}$$

Equations for measurement maximal concentration for reverse osmosis.

```
{from calc. as=aw=0.9503 } {aw=activity coefficient of water at osmotic Pressure}
Omega=55.51                {mol/kg; Moles of solvent in a kg}
zM=1                       {cation charge}
zX=1                       {anion charge}
mM=1.8
mX=1.8
m=1.8                      {molality of NH4NO3}
vX=1                       {stoech. coefficient}
vM=1                       {stoech. coefficient}
v=vX+vM

RhoP=999.57                {Density of water at 70 bar}
P=70E5                     {Osmotic Pressure}
Vw=0.01802/RhoP            {Molar volume of water at 70 bar}
Temp=298                   {Temperature}
R=8.314
ln(aw)=-P*Vw/(R*Temp)

{Universele constanten}
alpha=2                    {(kg/mol)^0.5, pg 87}
b=1.2                      {(kg/mol)^0.5, pg 87}

{NH4NO3 specifieke constanten uit tabellen}
Aphi=0.3915                {25 graden Celcius, (kg/mol)^0.5, tabel 1, pg 99}
Beta0MX= -0.0154           {tabel 2, pg 101}
Beta1MX= 0.1120            {tabel 2, pg 101}
CphiMX= -0.00003           {tabel 2, pg 101}

Phi=-(Omega/(mM+mX))*ln(aw)      {equation 28, pg 85}

Phi-1=(zM*zX)*fphi + m*(2*vM*vX/v)*BphiMX +m^2*(2((vM*vX)^1.5)/v)*CphiMX
                                 {equation 40, pg 87}
fphi=(-Aphi*I^0.5)/(1+b*I^0.5)   {equation 41, pg 87}

BphiMX=Beta0MX+Beta1MX*exp(-alpha*I^0.5)
                                 {equation 42, pg 87}
I=0.5*((mM*zM^2)+(mX*zX^2))      {Ionic strength, equation 24, pg 84}
c=m*80.04*0.998                  {g/l}
gew=(c/998)*100                  {kg/kg (gew%)}
```

{} References made to Pitzer

The activity co-efficient (aw) of water at 70 bar has been calculated. By iteration the concentration of salt which results in the same activity coefficient (as) as water at 70 bar has been determined.

IV i Calculation of energy use of FC under process conditions

Feed to FC	Ftotal	4.513	kg/s
	fraction AN	0.006129	
feed temperature	Tfeed	278.110	K
freezing point	Tfreeze	270.380	K (-3°C)
	DT	10.000	K
	Tammonia	260.380	K
specific heat	Cp (water)	4.200	kJ/kgK
heat of melting of ice	DHmelt	334.548	kJ/kg
stream water out	Fwater	4.118	kg/s
	Q cooling	146.506	kW
	Q ice formation	1377.539	kW
	Qtotal	1524.045	kW
evaporation of ammonia (at 10 bar and 25°C see Mollier diagram)	DHevap	1160	kJ/kg
	Fammonia	1.3138	kg/s
	Fammonia	113.515	ton/day
compressing NH3	DHcomp	101.333	kJ/kg
electrical power consumption	Wcomp	133.135	kW
Coefficient of performence	COP	11.447	
fraction liquid after flash		0.893	

IV j Calculation of energy use of EFC under process conditions

feed to EFC	Ftotal	0.395	kg/s
	fraction AN	0.070	
feed temperature	Tfeed	270.380	K
eutectic point	Teut	256.250	K (-16.9°C)
temperature gradient	ΔT	5.000	K
specific heat	Cp (water)	4.200	kJ/kgK
heat of melting of ice	ΔHmelt	334.548	kJ/kg
heat of solution of AN	ΔHsol	320.961	kJ/kg
stream water out	Fwater	0.364	kg/s
stream AN out	F AN	0.025	kg/s
energy required	Q cooling	23.448	kW
	Q ice formation	121.899	kW
	Q salt formation	8.154	kW
	Q total	153.500	kW
evaporation of ammonia (at 10 bar and 25°C see Mollier diagram)	ΔHevap	1144	kJ/kg
	Fammonia	0.1342	kg/s
	Fammonia	11.593	ton/day
compressing NH3	ΔHcomp	177.333	kJ/kg
electrical power consumption	Wcomp	23.794	kW
coefficient of performence	COP	6.451	
fraction liquid after flash		0.864	

APPENDIX V ADDITIONAL INFORMATION ON PROCESS EQUIPMENT

V a Compression costs optimalisation FC-EFC

weight fraction AN	remaining water in brine	t_{freeze}	t_{NH3}	compression work	heat of evaporation	costs of fc	costs of efc	total compression costs
	(kg/s)	(°C)	(°C)	(kJ/kg NH_3)	(kJ/kg NH_3)	(Nfl.)	(Nfl.)	(Nfl.)
0,01	2,768	-0,4	-10,4	79	1164	36904	125545	162448
0,02	1,846	-0,6	-10,6	80	1163	55004	83121	138125
0,02	1,384	-0,8	-10,8	82	1163	65292	62171	127463
0,03	0,923	-1,2	-11,2	85	1162	77209	41221	118430
0,05	0,554	-2,0	-12,0	92	1161	92426	24460	116887
0,07	0,395	-2,8	-12,8	98	1160	103142	17277	120419
0,10	0,277	-4,0	-14,0	108	1159	118424	11890	130315
0,15	0,185	-5,9	-15,9	125	1156	143219	7709	150928
0,20	0,138	-7,9	-17,9	141	1154	167061	5612	172673
0,25	0,111	-9,9	-19,9	157	1151	191669	4353	196023
0,30	0,092	-11,9	-21,9	174	1148	218345	3514	221859
0,35	0,079	-13,9	-23,9	190	1146	244474	2915	247390
0,40	0,069	-15,8	-25,8	207	1143	272768	2469	275238

V b Melt temperature as function of ice crystal diameter

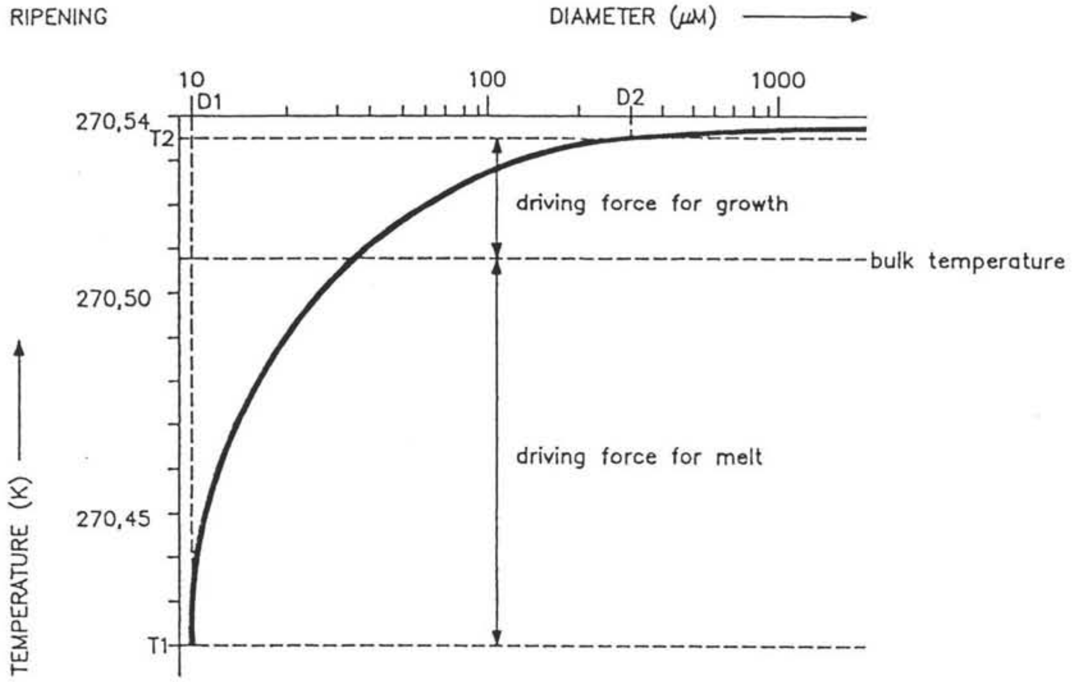


Figure V.1: Specific procedure to make an ice suspension (by courtesy of Niro Process Technology B. V.,'s-Hertogenbosch, the Netherlands). (a) Aqueous solution recirculating through a filter from the ripening tank passes through a scraped heat exchanger to generate small crystals. In the ripening tank the small crystals will melt, resulting in a further growth of the larger crystals being present in this tank.

V c Wash Columns

Screw Type Wash Column

In a screw type wash column, the suspension is fed at the bottom and the mother liquor taken off through a scraped filter. The remaining crystals are picked up by the screw conveyor and transported to the upper side of the wash column and transferred to the melting circuit. This means that this wash column doesn't work completely continuous. The flow of molten crystals as wash-liquid countercurrent to the crystals is adjusted by controlling the product withdrawal rate and, by this, the liquid pressure in the reslurry section. This mechanical column type is scaled up to a diameter of 1.2 m and is widely used for freeze concentration.

In a screw type wash column, relatively large forces are exerted on a crystal bed when using a screw conveyor. Hydraulic type columns have been developed to overcome this disadvantage.

Piston Bed Wash Column

In this type of wash column, see figure V-2 (a and b), the packed bed of ice crystals is moved upward by means of a piston, with a porous head. This type of wash column does not work completely continuous, because the wash column has to be filled first and then the crystal will be separated and washed. The slurry from the crystalliser section enters at the bottom of the wash column through a central inlet in the porous head. The liquid concentrate can leave through the head while the ice crystals remain in the column to form a packed bed of crystals. When the column is filled the feed valve closes and the piston moves upward and thus moving the crystal bed upward into the scraper. Ice is scraped off and enters the melting circuit, where it is circulated through and melted in a heat exchanger. Controlling the pressure in the melting circuit allows the possibility to force the melted water back through the ice bed, which washes the incoming ice crystals.

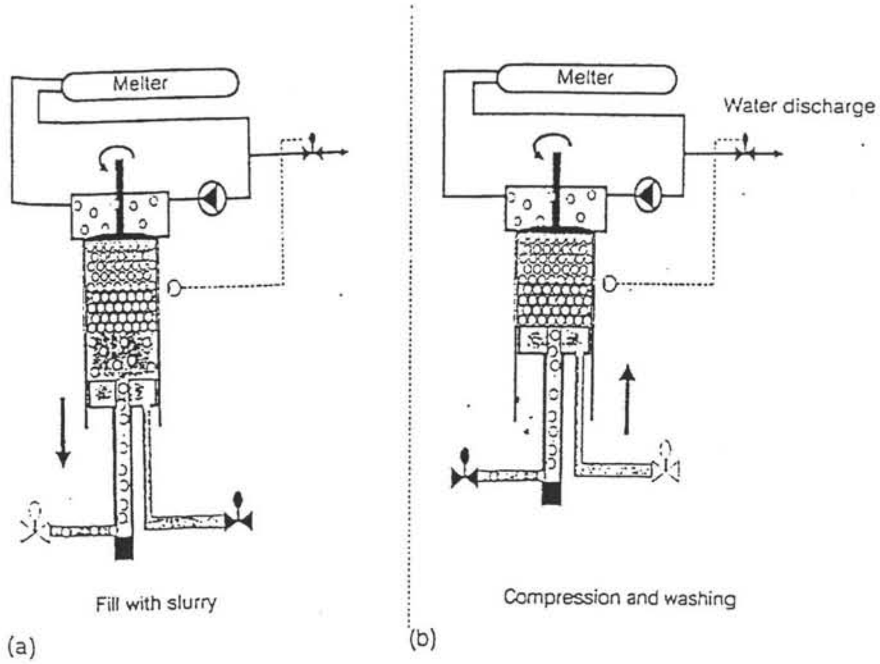


Figure V-2: A Piston Type Wash Column
Where: (a) The column is filled with slurry; (b) The piston compresses the slurry.

APPENDIX VI CALCULATIONS EQUIPMENT

VI a Freeze Concentrator

Specification/Design		Value	Unit	Source
Total Fresh Feed	Ft	4.5127	kg/s	Str. Nr. 15
Ice production	P	4.118	kg/s	Process
Melt Enthalpy	DH_m	334.548	kJ/kg	Lit.
Heat Capacity	c_p	4.22	kJ/kg	Lit.
Freezing of water	Q_{ice}	1377669	W	Equation [4-1]
Heat Produced by agitator	$Q_{agitator}$	1250	W	25% of total power (=5000W)
Heat Produced by recirc. Pump	Q_{pump}	3200	W	25% of total power (=12800W)
Cooling of Feed	Q_c	148500	W	Heat Balance
Total Cooling	Q	1530619	W	Equation [4-2]
Heat Transfer Coefficient	U	1500	W/m^2°C	Niro
Heat Transfer Coefficient	U	170-850	W/m^2°C	Perry
Heat Transfer Coefficient	U	1000	W/m^2°C	used
Temp. Dif.	DT	15-20	°C	Niro
Temp. Dif.	DT	17	°C	Perry
Temp. Dif.	DT	10	°C	used in process
Temp. Ammonia (l) in	T_{in}	-13	°C	used
Temp. Ammonia (g) out	T_{out}	-13	°C	used
Temp. brine in	t_{in}	-2.77	°C	
Temp. brine out	t_{out}	-3.76	°C	
Log mean Temp. Dif.	DT	-9.72	°C	Equation [4-3]
Exchange Area	A	157.4	m^2	Equation [4-4]
Exch. Area Pipe	A_p	12	m^2	Niro prototype
Length of Pipes	l_p	4.8	m	Calculated (estimate)
Diameter inner pipe	d_i	0.80	m	Niro
Number of Pipes	N_p	13.12		Calculated
Number of Pipes	N_p	14		
FC Feed	F	20.98	kg/s	Str.Nr. 16
Recycle	R	344.02	kg/s	Str. Nr. 19
Flow through SSHE	F_{SSHE}	365	kg/s	Niro Maximum 20000kg/s
Freeze Temperature	T_f	-2.77	°C	
Maximum undercooling	T_{SSHE}	1	°C	Undercooling used by Niro
Calculated undercooling	T_{SSHE}	0.99	°C	Undercooling attained
Superficial velocity in Pipes		0.73	m/s	Pipes in series
Working height		11.55	m	For instance for replacing scrapers
Reynolds		323358		Turbulent flow

Design Crystalliser Tank		Value	Unit	Source
Residence time	t	3600	s	Time required to grow to 0.2mm
Flow in-out FC	F	20.9847	kg/s	
Volume of Tank	V	75.54	m^3	Equation [4-5]
Height	H	4	m	estimated
Diameter	D	5	m	estimated
Actual Volume of Tank	V_a	78.54	m^3	

Design Filter in Tank		Value	Unit	Source
Area		10	m^2	
Cut		0.01	mm	

Design Recirculation Pump		Value	Unit	Source
Capacity		0.365	m^3/s	
Power Requirement		12.8	kW	

VI b Eutectic Freeze Crystalliser

Design		Value	Units	
residence time in CDCC	τ	7200	s	
volume flow	Φv	0.002	m^3/s	
volume in brine	V_{brine}	12.445		Equation [4-6]
heat to be exchanged	Q	153.500	kJ/kg	
heat transfer coefficient	U	300.000	W/m^2K	
temperature difference	ΔT	5.000	K	
total disc surface area	A_{discs}	102.334	m^2	Equation [4-7]
surface area CDCC	A_{column}	4.909	m^2	Equation [4-8]
amount of discs needed	N	10.424		Equation [4-9]
diameter of CDCC	D_{column}	2.500	m	
thickness of one disc	D_{disc}	0.200	m	
total volume of discs	V_{discs}	10.799	m^3	Equation [4-10]
height of CDCC without discs	H	2.535	m	
height of CDCC with discs	H_{column}	4.735	m	Equation [4-12]
space between two discs	H_{space}	0.211	m	
volume of CDCC	V_{column}	23.244	m^3	Equation [4-11]

Calculation of settling and rising time of one particle that has to go through the whole column

1. Ice				
diameter ice particle	$d_{ice\ particle}$	2.00E-04	m	
density difference between particle and brine	$\Delta\rho$	283	kg/m^3	
viscosity	n	1.79E-03	Pa*s	
gravitational acceleration	g	9.8	m/s^2	
velocity of particle	v_s	0.0034392	m/s	Equation [4-13]
floating time	t	737.15759	s	Equation [4-14]
floating time	t	12.28596	min	
Reynolds number	Re	0.460612		Equation [4-15]

2. Ammonium nitrate				
diameter salt particle	$d_{ice\ particle}$	2.00E-04	m	
density difference between particle and brine	$\Delta\rho$	525	kg/m^3	
viscosity	n	1.79E-03	Pa*s	
gravitational acceleration	g	9.8	m/s^2	
velocity of particle	v_s	0.0063802	m/s	Equation [4-13]
sinking time	t	397.36304	s	Equation [4-14]
sinking time	t	6.6227174	min	

VI c Wash Columns: C01

	Feed kg/s	To FC kg/s	To Melter kg/s	Washwater kg/s	Reslurry water kg/s	Recrystallizatio Water kg/s	Total Recycle kg/s
NH4+	0.1389	0.1389					
NO3-	0.4776	0.4776					
water (l)	0.8410	0.8410	1.6196	0.3313	1.2883	0.0405	
water (s)	0.3644		0.4049				
total	1.8218473	1.45747787	2.0245	0.3313	1.2883	0.0405	1.6601

Design		Value	Unit	Source
Ice Flowrate	$F_{m,s}$	0.3644	kg/s	
Ice Flowrate	$F_{m,s}$	1311.73008	kg/h	
Crystal Diameter	dp	2.00E-04	m	
Bed Porosity	e (bed)	0.45		
Stagnant Zone Porosity	e (stag)	0.461		Equation [4-19]
A wash column with 10 ton/h m^2 has a diameter:				
Diameter	D_{10}	1	m	
Cross Area	A_{10}	0.7854	m^2	
A wash column with 7.412 ton/h m^2 has an cross area:				
Wash Column Cross Area	A_{WC}	0.1030	m^2	Equation [4-16]
And thus is the diameter of the wash column:				
Wash Column Diameter	D_{WC}	0.3622	m	Equation [4-17]
Wash water enters at the top:				
Washwater Flowrate	$F_{m,wash}$	0.3718	kg/s	
Washwater Flowrate	$F_{v,wash}$	3.726E-04	m^3/s	
Particles enter at the bottom:				
Ice Flowrate	$F_{m,s}$	0.3644	kg/s	
Ice Flowrate	$F_{v,s}$	3.973E-04	m^3/s	
Superficial Velocity	v_{wash}	3.616E-03	m/s	
Superficial Velocity	v_s	3.857E-03	m/s	
Relative Velocity	v_{rel}	7.473E-03	m/s	

Pressure Drop over the Packed Bed				
		Packed bed		
Kozeny-Carman	dP/H	-199828.421	Pa/m	Equation [4-18]
Bed Height	H	0.125	m	
Pressure Drop	dP	-24978.5526	Pa	
Pressure Drop	dP	-0.24978553	bar	
		Stagnant zone		
Kozeny-Carman	dP/H	-178502.447	Pa/m	Equation [4-18]
Bed Height	H	0.125	m	
Pressure Drop	dP	-22312.8058	Pa	
Pressure Drop	dP	-0.22312806	bar	
Total Pressure Drop	dP_2	-0.47291358	bar	
Pressure Drop over the Wash Column				
Inlet Pressure	P_{inlet}	1.800	barg	TNO
Wash Pressure	P_{wash}	1.350	barg	TNO
Inlet Pressure	P_{inlet}	2.800	bara	TNO
Wash Pressure	P_{wash}	2.350	bara	TNO
Total Pressure Drop	dP_1	0.450	bara	
Pressure Drop Difference	dP_1-dP_2	-0.023	bara	
Number of Filtertubes in the Wash Column:				
Two laboratory scale hydraulic wash column:				
		1)		
Wash Column Diameter	D_{TNO}	0.15	m	TNO
Wash Column Cross Area	A_{TNO}	1.767E-02	m^2	TNO
Number of Filtertubes	N_{TNO}	6	#	TNO
Number of Filtertubes	N_{tube}	34.9795	#	Equation [4-20]
		2)		
Wash Column Diameter	D_{TNO}	0.08	m	TNO
Wash Column Cross Area	A_{TNO}	5.027E-03	m^2	TNO
Number of Filtertubes	N_{TNO}	1	#	TNO
Number of Filtertubes	N_{tube}	20.4958	#	Equation [4-20]

Wash Columns: C02 and C03

	Feed kg/s	To FC kg/s	To Melter kg/s	Washwater kg/s	Reslurry water kg/s	Recrystallization Water kg/s	Total Recycle kg/s
NH_4^+	0.1299	0.1299					
NO_3^-	0.4466	0.4466					
water (l)	7.6588	7.6588	8.3852	1.7152	6.6700	0.0375	
water (s)	2.0588		2.0963				
total	10.294034	8.23522689		1.7152	6.6700	0.037493278	8.4227

Design		Value	Unit	Source
Ice Flowrate	$F_{m,s}$	2.0588	kg/s	
Ice Flowrate	$F_{m,s}$	7411.68	kg/h	
Crystal Diameter	dp	2.00E-04	m	
Bed Porosity	e (bed)	0.45		
Stagnant Zone Porosity	e (stag)	0.461		Equation [4-19]
A wash column with 10 ton/h m^2 has a diameter:				
Diameter	D_{10}	1	m	
Cross Area	A_{10}	0.7854	m^2	
A wash column with 7.412 ton/h m^2 has an cross area:				
Wash Column Cross Area	A_{WC}	0.5821	m^2	Equation [4-16]
And thus is the diameter of the wash column:				
Wash Column Diameter	D_{WC}	0.8609	m	Equation [4-17]
Wash water enters at the top:				
Washwater Flowrate	$F_{m,wash}$	1.7526	kg/s	
Wash water Flowrate	$F_{v,wash}$	1.756E-03	m^3/s	
Particles enter at the bottom:				
Ice Flowrate	$F_{m,s}$	2.0588	kg/s	
Ice Flowrate	$F_{v,s}$	2.245E-03	m^3/s	
Superficial Velocity	v_{wash}	3.017E-03	m/s	
Superficial Velocity	v_s	3.857E-03	m/s	
Relative Velocity	v_{rel}	6.874E-03	m/s	

Pressure Drop over the Packed Bed				
		Packed bed		
Kozeny-Carman	dP/H	-183801.71	Pa/m	Equation [4-18]
Bed Height	H	0.125	m	
Pressure Drop	dP	-22975.214	Pa	
Pressure Drop	dP	-0.2297521	bar	
		Stagnant zone		
Kozeny-Carman	dP/H	-164186.13	Pa/m	Equation [4-18]
Bed Height	H	0.125	m	
Pressure Drop	dP	-20523.266	Pa	
Pressure Drop	dP	-0.2052327	bar	
Total Pressure Drop	dP_2	-0.4349848	bar	
Pressure Drop over the Wash Column				
Inlet Pressure	P_{inlet}	1.750	barg	TNO
Wash Pressure	P_{wash}	1.313	barg	TNO
Inlet Pressure	P_{inlet}	2.750	bara	TNO
Wash Pressure	P_{wash}	2.313	bara	TNO
Total Pressure Drop	dP_1	0.438	bara	
Pressure Drop Difference	dP_1-dP_2	0.003	bara	
Number of Filtertubes in the Wash Column:				
Two laboratory scale hydraulic wash column:				
		1)		
Wash Column Diameter	D_{TNO}	0.15	m	TNO
Wash Column Cross Area	A_{TNO}	1.767E-02	m^2	TNO
Number of Filtertubes	N_{TNO}	6	#	TNO
Number of Filtertubes	N_{tube}	197.6448	#	Equation [4-20]
		2)		
Wash Column Diameter	D_{TNO}	0.08	m	TNO
Wash Column Cross Area	A_{TNO}	5.027E-03	m^2	TNO
Number of Filtertubes	N_{TNO}	1	#	TNO
Number of Filtertubes	N_{tube}	115.8075	#	Equation [4-20]
Wash Column Filtertubes	N_{tube}	157	#	Average

VI d Cooling Process

Flash Vessels

Design		Value	Value	Unit	Source
		V06	V07		
Flash Pressure	P_{flash}	2.6	1.7	bara	Process
Flash Temperature	T_{flash}	260	251	K	Process
Feed Flowrate	$F_{m,F}$	1.1784	0.1165	kg/s	Process
Liquid Flowrate	$F_{m,L}$	1.0647	0.1007	kg/s	Process
Vapour Flowrate	$F_{m,V}$	0.1137	0.0158	kg/s	Process
Rho liquid		770	770	kg/m^3	Appendix II b
Rho vapor		2.38	1.25	kg/m^3	Appendix III
u		0.6294	0.8687	m/s	Equation [4-21]
Vapour Volume Flowrat	$F_{v,V}$	0.0478	0.0126	m^3/s	Equation [4-22]
Vessel Area	A_{vessel}	0.0759	0.0146	m^2	Equation [4-23]
Vessel Diameter	D_{vessel}	0.3108	0.1361	m	Equation [4-24]
Disengagement space	H_d	0.3108	0.1361	m	
Liquid Volume Flowrate	$F_{v,L}$	1.383E-03	1.308E-04	m^3/s	Equation [4-25]
Volume 1 min hold-up	V_{HU}	0.0830	0.0078	m^3	Equation [4-26]
Liquid Depth	H_L	1.0935	0.5393	m	Equation [4-27]
Height Vessel	H_{vessel}	1.4043	0.6754	m	Equation [4-28]
Volume Vessel	V_{vessel}	0.1065	9.827E-03	m^3	Equation [4-29]

Knock-Out Drums

Design		Value	Value	Unit	Source
		V08	**V09**		
Inlet Pressure	P_{flash}	2.6	1.7	bara	Process
Inlet Temperature	T_{flash}	260	251	K	Process
Feed Flowrate	$F_{m,F}$	1.3309	0.1259	kg/s	Process
Liquid Flowrate	$F_{m,L}$	0.2662	0.0252	kg/s	Process
Vapour Flowrate	$F_{m,V}$	1.0647	0.1007	kg/s	Process
Rho liquid		770	770	kg/m^3	Appendix II b
Rho vapor		2.38	1.25	kg/m^3	Appendix III
u		0.6294	0.8687	m/s	Equation [4-21]
Vapour Volume Flowrat	$F_{v,V}$	0.4472	0.0806	m^3/s	Equation [4-22]
Vessel Area	A_{vessel}	0.7105	0.0927	m^2	Equation [4-23]
Vessel Diameter	D_{vessel}	0.9511	0.3436	m	Equation [4-24]
Disengagement space	H_d	0.9511	0.3436	m	
Liquid Volume Flowrate	$F_{v,L}$	3.457E-04	3.269E-05	m^3/s	Equation [4-25]
Volume 15 min hold-up	V_{HU}	0.3111	0.0294	m^3	Equation [4-26]
Liquid Depth	H_L	0.4379	0.3173	m	Equation [4-27]
Height Vessel	H_{vessel}	1.3890	0.6609	m	Equation [4-28]
Volume Vessel	V_{vessel}	0.9868	0.0613	m^3	Equation [4-29]

Compressors

Design		Value	Value	Unit	Source
		K01	**K02**		
Feed Flowrate	$F_{m.F}$	1.1784	0.1165	kg/s	Process
Inlet Pressure	P_{in}	2.60E+05	1.70E+05	Pa	Process
Inlet Temperature	T_{in}	260	251	K	Process
Outlet Pressure	P_{out}	4.20E+05	4.20E+05	Pa	Process
Rho V		2.38	1.25	kg/m^3	Appendix III
Specific Heat Ratio	K	1.317	1.317	-	Lit ##
Gasconstant	R	8.3144	8.3144	J/mol K	Lit ##
Isentropic Work	P_{isen}	65415	16010	J/s	Equation [4-30]
Isentropic Work	P_{isen}	65.41	16.01	kW	
Efficiency		0.7	0.7	-	
P_{real}		93.4	22.9	kW	Equation [4-31]

VI e Neutraliser

Design		Value	Units	
Mass flow	M	4.5127129	kg/s	
Density of water	r	998	kg/m3	
Volume flow	Fv	0.0045218	m3/s	
residence time	t	780	s	
volume	V	3.52697	m3	Equation [4-32]
diameter dissolver	D	1	m	
surface area dissolver	A	0.7853982	m2	
height dissolver	H	4.4906776	m	Equation [4-33]

VI f Filter

Design	Value	Unit	Source
Mass flow	0.127	kg/s	
Viscosity	1.80E-03	Pa.s	
Liquid density	1300	kg/m^3	
Specific cake resistance	1E+11	-	
Solid fraction in slurry	0.2	kg/kg	
Solid fraction in cake	0.82	kg/kg	Design factor
Pressure drop	1.00E+04	Pa	
phi	3.141593	rad	
N	0.016667	rps	
f	343.871	kg/m^3	Equation [VI j-2]
t	30	s	Equation [VI j-3]
Volume flow	9.77E-05	m^3/s	
Q	0.004403	m^3/m^2	
A	1.331236	m^2	Equation [4-34]
d	0.3	m	
Area per disc side	0.070686	m^2	
Number of disc sides	19		
Number of discs	9		

VI g Dissolver

Design		Value	Units	
Mass flow	M	0.03073	kg/s	
Density of 90 w% water	ρ	1408	kg/m^3	
Volume flow	Φv	2.183E-05	m3/s	
residence time	τ	1800	s	
volume dissolver	V	0.0392855	m^3	Equation [4-35]
diameter dissolver	D	0.3	m	
surface area dissolver	A	0.0706858	m^2	
diameter coil tube	Dc	0.01	m	Equation [4-36]
heat flow	Q	20.3904	kW	
temperature difference	ΔT	40	K	
temperature suspension in	T1	256	K	
temperature suspension out	T2	368	K	
temperature steam in	t1	408	K	
temperature steam out	t2	408	K	
heat trasfer coefficient	U	212.500	Btu/h ft^2 °F	(Perry)
heat trasfer coefficient	U	1207.000	W/m^{2}K	
coil surface area	A_c	0.4223364	m^2	Equation [4-37]
length of coil	L_c	13.443384	m	Equation [4-38]
volume of coil	Vc	0.0010558	m^3	Equation [4-39]
volume of tank with coil	Vt	0.0403413	m^3	Equation [4-40]
height dissolver	H	0.5707126	m	Equation [4-41]

VI h Heat Exchangers

Heat exchanger	E1	E2	E3	
shell side stream	**AN-solution**	**HNO_3-solution**	**HNO_3-solution**	
mass flow rate (kg/s)	4.5127	4.7222	4.7222	
T1 (°C)	66	66	47	
T2 (°C)	5	47	28	
T mean (°C)	35.5	56.5	37.5	
number of passes	1	1	1	
triangular pitch (*d0)	1.25	1.25	1.25	
K1	0.249	0.249	0.249	from table 12.4,
n1	2.207	2.207	2.207	dep of no of passes
density (kg/m^3)	995	985	995	
viscosity (10^{-6} Ns/m^2.s)	715	600	690	
heat capacity (kJ/kg °C)	4.2	4.2	4.2	
thermal conductivity (10^{-3} W/m °C)	625	640	630	
tube side	**ice-slurry**	**ice-slurry**	**water**	
mass flow rate (kg/s)	22.6769	22.6769	4.4820	
t1 (°C)	0	0	0	
t2 (°C)	0	0	20	
t mean (°C)	0	0	10	
number of passes	2	2	2	
density (kg/m^3)	983	995	999	
viscosity (10^{-6} Ns/m^2.s)	1435	1700	1307	
heat capacity (kJ/kg °C)	3.8	4.1	4.2	
thermal conductivity (10^{-3} W/m °C)	870	640	580	
tube inside diameter (m)	0.016	0.016	0.016	
tube outside diameter (m)	0.02	0.02	0.02	
L tube (m)	5	1	5	
Area one tube (m^2)	0.314	0.063	0.314	
Delta T lm (°C)	23.64	55.96	27.50	Equation [4-3]
R	-	-	0.9500	Appendix VI j, Equation [VI j-5]
S	0.0000	0.0000	0.4255	Appendix VI j, Equation [VI j-6]
F	1.0000	1.0000	0.9097	1 shell pass, 2 tube passes, Appendix VI j
Delta T m (°C)	23.64	55.96	25.01	otherwise from fig. 12.19/12.20
				Equation [4-43]
Heat duty Q (kW)	1156	382	377	
U estimate (W/m^2 °C)	1000	1000	1000	
Heat transfer Area (m^2)	48.90	6.83	15.07	Equation [4-42]
Number of tubes	156	109	48	
Tubes per pass	78	54	24	

Calculation hs (Kern)				
D_b Bundle diameter (m)	0.370	0.314	0.217	Equation [4-45]
Clearance (m)	0.055	0.053	0.050	read from fig. 12.10, split-ring float. head
D_s Shell diameter (m)	0.425	0.367	0.267	otherwise read from 12.19/12.20
L_B Baffle spacing (m)	0.170	0.147	0.107	L_b is 0.4 D_s
pitch (m)	0.025	0.025	0.025	
A_s Shell cross flow area (m^2)	0.0144	0.0108	0.0057	Equation [4-50]
G_s Mass flow velocity (kg/s m^2)	312.703	437.916	828.563	Equation [4-49]
u_s (m/s)	0.314	0.445	0.833	
d_e Equivalent diameter (m)	0.014	0.014	0.014	Equation [4-51]
Re	6210.761	10364.734	17052.791	$d=d_e$, Equation [4-48]
Pr	4.805	3.938	4.600	Equation [4-56]
j_h	0.0073	0.0058	0.0047	Read from figure 12.29, 25 % baffle cut
h_s (W/m^2 C)	3349.513	4258.631	5883.355	Equation [4-47]

Calculation hi				
Tube cross area (m^2)	0.0002	0.0002	0.0002	
Tube cross area per pass (m^2)	0.0156	0.0109	0.0048	
Volumetric flow (m^3/s)	0.0231	0.0228	0.0045	
u_t (water velocity) (m/s)	1.4743	2.0868	0.9303	
A_t Tube cross flow area (m^2)	0.0156	0.0109	0.0048	Equation [4-50]
G_t Mass flow velocity (kg/s m^2)	1449.2707	2076.3713	929.3453	Equation [4-49]
Re	16159.12	19542.32	11376.84	$d=d_i$, Equation [4-55]
Pr	6.2678	10.8906	9.4645	Equation [4-56]
L/di	312.5	62.5	312.5	
j_h	4.00E-03	3.80E-03	4.00E-03	Read from figure 12.23
Nu	118.45	163.30	95.54	Equation [4-54]
h_i (W/m^2 °C)	6440.58	6532.01	3463.39	Equation [4-53]

Calculation U				
fouling factors h_o en h_{od} (W/m^2 °C)	6000	6000	6000	From table12.2
k_w thermal conduct. tube wall mat	16	16	16	From table 12.6, material is stainless steel
1/U calculated	0.00101	0.00094	0.00105	
U calculated (W/m^2 °C)	992.95	1063.10	956.61	

Calculation Pressure drops				
j_f (shell side friction factor)	5.20E-02	5.00E-02	4.30E-03	Read from uit figure 12.24
Pressure drop over shell side (Pa)	17992.65	6854.82	10445.95	Equation [4-52]
Pressure drop over shell side (bar)	0.1799	0.0685	0.1045	
j_f (tube side friction factor)	4.70E-03	4.00E-03	4.90E-03	Read from figure 12.30 (25% baffle cut)
Pressure drop over tube side (Pa)	30448.11	19498.42	12752.07	Equation [4-57]
Pressure drop over tube side (bar)	0.3045	0.1950	0.1275	

VI i Calculation of Pump Characteristics

Pump No.	P01	P02	P03	P04	P05	P06	P07	P08	
Mass flow (kg/s)	9.2306	4.5127	375	20.5881	1.8218	0.127	1.9183	10.3793	
Density (kg/m^3)	980	980	1030	985	985	1300	985	985	
Volume flow (m^3/s)	0.009419	0.0046	0.364078	0.0209	0.00185	9.8E-05	0.00195	0.01054	
Capacity (m^3/h)	33.90833	16.5773	1310.68	75.2458	6.65836	0.35169	7.01105	37.9345	
P_{in} (Pa)	100000	100000	100000	100000	100000	100000	235000	230000	
P_{uit} (Pa)	200000	150000	130000	275000	280000	150000	290000	290000	
Pressure Difference (Pa)	100000	50000	30000	175000	180000	50000	55000	60000	
Head h_{man} (m)	10.40	5.20	2.97	18.11	18.63	3.92	5.69	6.21	Equation [4-61]
Isentropic power (W)	941.90	230.24	10922.33	3657.78	332.92	4.88	107.11	632.24	Equation [4-58]
Pump efficiency	0.71	0.65	0.85	0.77	0.55	0.30	0.56	0.71	Fig. 10.62, lit 20
Pump shaft power (W)	1326.62	354.22	12849.80	4750.37	605.31	16.28	191.27	890.48	Equation [4-59]
Temperature (°C)	66	66	-3	-3	-17	-17	0	0	
P_{vap} (Pa)	26163	26613	476	476	137	137	611	611	
$NPSH_{avail}$ (m)	7.680314	7.63351	9.849668	10.2997	10.3347	7.83055	24.2567	23.7393	Equation [4-62]

Pump No.	P09	P10	P11	P12	P13	P14	P15
Mass flow (kg/s)	10.3793	0.0963	4.7222	4.482	0.0307	1.4666	0.1449
Density (kg/m^3)	985	1300	996	998	1652.3	770	770
Volume flow (m^3/s)	0.010537	7.4E-05	0.004741	0.00449	1.9E-05	0.0019	0.00019
Capacity (m^3/h)	37.9345	0.26668	17.06819	16.1675	0.06689	6.85683	0.67745
P_{in} (Pa)	230000	140000	160000	230000	100000	250000	160000
P_{uit} (Pa)	290000	280000	1100000	700000	200000	260000	170000
Pressure Difference (Pa)	60000	140000	940000	470000	100000	10000	10000
Head h_{man} (m)	6.21	10.98	96.21	48.01	6.17	1.32	1.32
Isentropic power (W)	632.24	10.37	4456.69	2110.76	1.86	19.05	1.88
Pump efficiency	0.71	0.30	0.65	0.65	0.1	0.54	0.3
Pump shaft power (W)	890.48	34.57	6856.45	3247.33	18.58	35.27	6.27
Temperature (°C)	0	-17	28	20	95	-13	-22
P_{vap} (Pa)	611	137	378	234	20000	2767	2213
$NPSH_{avail}$ (m)	23.73927	10.9671	16.3367	23.4685	4.93551	32.7301	20.8887

VI j Additional Equations used for design filter and heat exchanger

Filter

Equation 4-34, the Ruth equation can be derived from Darcy's law.

$$\frac{\eta\cdot\alpha\cdot f}{2\Delta P_f}*\frac{Q^2}{2}+\frac{\eta * R}{\Delta P_f}Q=t \qquad \text{[VI-1]}$$

where: α = specific cake resistance (m^2/kg)
f = function of slurry composition (eq. VI-2) (kg/m^3)
R = filter medium resistance (-)
t = residence time of element of disc in suspension (s)
ΔP_f = pressure drop over filter (Pa)

$$with:\ f=\frac{\rho_l}{\dfrac{1-S_s}{S_s}-\dfrac{1-S_c}{S_c}} \qquad \text{[VI-2]}$$

$$and:\ t=\frac{\varphi}{2\pi}*\frac{1}{N} \qquad \text{[VI-3]}$$

where: S_s = solid fraction in slurry kg/kg
S_c = solid fraction in cake kg/kg
φ = angle of submergence rad
N = rotations per second 1/s

Through substitution of equation VI-2 and VI-3 in equation VI-1, Q can be calculated.

Heat Exchangers

The correction factor F_t is a function of the shell and tube fluid temperatures and the number of tube and shell passes.

$$F_t = \frac{\sqrt{(R^2+1)}\ln[(1-S)/(1-RS)]}{(R-1)\ln\left[\dfrac{2-S[R+1-\sqrt{(R^2+1)})]}{2-S[R+1+\sqrt{R^2+1}]}\right]} \qquad \text{[VI-4]}$$

After calculation of the two dimensionless temperature ratios R and S, this factor can be obtained from either equation VI-4 (in the case of one shell pass and two tube passes) or from figure 12.19 and 12.20 (C&R).

$$R = \frac{T_1 - T_2}{t_2 - t_1} \qquad \text{[VI-5]}$$

$$S = \frac{t_2 - t_1}{T_1 - t_1} \qquad \text{[VI-6]}$$

APPENDIX VII LIST OF EQUIPMENT AND SPECIFICATION FORMS

VII a List of Wash Columns

UNIT NUMBER	C01	C02	C03
Name Type	Wash Column Hydraulic	Wash Column Hydraulic	Wash Column Hydraulic
Feed Stream	Ice slurry	Ice slurry	Ice slurry
Ice Capacity [kg/s]	0.36	2.06	2.06
Diameter [m]	0.36	0.58	0.58
Height [m]	0.75	0.75	0.75
Wash water flow [kg/s]	0.33	1.72	1.72
Inlet/Wash Pressure (abs) [bar]	2.8/2.35	2.75/2.31	2.75/2.31
Diameter Tube [m]	0.02	0.02	0.02
Number of Tubes [#]	28	157	157

VII b List of Flash Vessels and Knock-out Drums

UNIT NUMBER	V06	V07	V08	V09
Name	Flash Vessel	Flash Vessel	Knock-out Drum	Knock-out Drum
Feed Stream	NH_3	NH_3	NH_3	NH_3
Flow rate [kg/s]	1.1784	0.1165	1.3309	0.1259
Liquid flow [kg/s]	1.0647	0.1007	0.2662	0.0252
Vapour flow [kg/s]	0.1137	0.0158	1.0647	0.1007
Temperature (°C)	-13	-22	-13	-22
Pressure (bara)	2.6	1.7	2.6	1.7
Diameter [m]	0.31	0.14	0.95	0.34
Disengagement space H_d [m]	0.31	0.14	0.95	0.34
Liquid depth H_l [m]	1.09	0.54	0.44	0.32
Vessel Height [m]	1.4	0.68	1.39	0.66

VII c List of Heat Exchangers

UNIT NUMBER	E01	E02	E03
Name	Heat Exchanger	Heat Exchanger	Heat Exchanger
Type	Floating head	Floating head	Floating head
Medium			
- tubes	ice slurry	ice slurry	water
- shell	AN solution (water)	HNO_3 solution (water)	HNO_3 solution (water)
Capacity Heat Exchanged [kW]	1156	382	377
Heat Exchanging Area [m^2]	48.90	6.83	15.07
Number of units			
- serial	1	1	1
- parallel			
Abolute Pressure [bar]			
- tubes	2.9	2.6	2.4
- shell	1.3	2	1.8
Temperature in/out [°C]			
- tubes	0/0	0/0	0/20
- shell	66/5	66/47	47/28
Special construction materials	stainless steel	stainless steel	stainless steel
Miscellaneous	1 shell pass 2 tube passes	1 shell pass 2 tube passes	1 shell pass 2 tube passes

VII d List of pumps and compressors

UNIT NUMBER	P01	P02	P03	P04
Name Type	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal
Fluid being pumped	AN solution	AN solution	AN solution	Ice slurry
Capacity [kg/s]	9.23	4.51	375	20.59
Density [kg/m^3]	980	980	1030	985
Suction/Discharge Pressure (abs) [bar]	1/2	1/1.5	1/1.3	1/2.75
Temp. In/Out [°C]	66/66	66/66	-3/-3	-3/-3
Power [kW] - theoretical - practical	 0.94 1.33	 0.23 0.35	 10.92 12.85	 3.66 4.75
Number of units	1	1	1	1
Special construction materials	S.S.	S.S.	S.S.	S.S.

UNIT NUMBER	P05	P06	P07	P08
Name Type	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal
Fluid being pumped	Ice slurry	AN slurry	Ice slurry	Ice slurry
Capacity [kg/s]	1.28	0.13	1.92	10.38
Density [kg/m^3]	985	1300	985	985
Suction/Discharge Pressure (abs) [bar]	1/2.8	1/1.5	2.35/2.9	2.3/2.9
Temp. In/Out [°C]	-17/-17	-17/-17	0/0	0/0
Power [kW] - theoretical - practical	 0.33 0.61	 4.88 16.28	 0.11 0.19	 0.63 0.89
Number of units	1	1	1	1
Special construction materials	S.S.	S.S.	S.S.	S.S.

UNIT NUMBER	P09	P10	P11	P12
Name Type	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal
Fluid being pumped	Ice slurry	AN solution	AN solution	H.P.Water
Capacity [kg/s]	10.38	0.10	4.72	4.48
Density [kg/m^3]	985	1300	996	998
Suction/Discharge Pressure (abs) [bar]	2.3/2.9	1.4/2.8	1.6/11	2.3/7
Temp. In/Out [°C]	0/0	-17/-17	28/28	20/20
Power [kW] - theoretical - practical	 0.63 0.89	 0.010 0.035	 4.46 6.86	 2.11 3.25
Number of units	1	1	1	1
Special construction materials	S.S.	S.S.	S.S.	S.S.

UNIT NUMBER	P13	P14	P15
Name Type	Pump Centrifugal	Pump Centrifugal	Pump Centrifugal
Fluid being pumped	AN solution	NH_3 (l)	NH_3 (l)
Capacity [kg/s]	0.031	1.47	0.15
Density [kg/m^3]	1652	770	770
Suction/Discharge Pressure (abs) [bar]	1/2	2.5/2.6	1.6/1.7
Temp. In/Out [°C]	95/95	-13/-13	-22/-22
Power [kW] - theoretical - practical	 0.0019 0.019	 0.019 0.035	 0.0019 0.0063
Number of units	1	1	1
Special construction materials	S.S.	S.S.	S.S.

UNIT NUMBER	K01	K02
Name Type	Compressor- Centrifugal	Compressor Centrifugal
Gas being compressed	Ammonia	Ammonia
Capacity [kg/s]	1.18	0.12
Density [kg/m^3]	2.38	1.25
Suction/Discharge Pressure (abs) [bar]	2.6/4.2	1.7/4.2
Temp. In/Out [°C]	-8/30	-22/55
Power [kW] - theoretical - practical	 65.41 93.40	 16.01 22.90
Number of units	1	1
Special construction materials	S.S.	S.S.

VII e List of utilities

UNIT NUMBER	Electricity (kW)	Low Pres. Steam (kg/s)
P01	1.33	
P02	0.35	
P03	12.85	
P04	4.75	
P05	0.61	
P06	0.02	
P07	0.19	
P08	0.89	
P09	0.89	
P10	0.04	
P11	6.86	
P12	3.25	
P13	0.02	
P14	0.04	
P15	0.01	
FC (scraper+agitator)	10	
Wash Column (scraper)	15	
Neutraliser	5	
K01	93.4	
K02	22.9	
Dissolver	1	0.0094
Total	179.4	0.0094

VII f SPECIFICATION FORM FREEZE CONCENTRATOR

SPECIFICATION FORM SSHE

UNIT NUMBER: E04
No. Serial: 14
No. Parallel: -

Type: Scraped Surface Heat Exchanger, Niro Prototype

Design: Concentric Tubes, Internal scraping mechanism inner tube.

Position: Vertical

Total Heat Capacity	: 1531	[kW]
Heat Capacity per Unit	: 109	[kW]
Total Heat Exchanging Area	: 168	[m^2]
Heat Exchange Area per unit	: 12	[m^2]
Overall Heat Transfer Coefficient	: 1000	[$W/m^2°C$]
Logarithmic Mean Temp. Diff.	: 9.72	[°C]
No. of passes (inner tube)	: 1	
No. of passes (outer tube)	: 1	
Correction Factor LMTD	: 1	
True Mean Temperature Diff.	: 9.72	[°C]

Operating Conditions

	Innertube	**Outertube**
Stream	Ammonium nitrate brine	Ammonia
Total Mass Flow per unit [kg/s]	365	1.02-1.11
-to evaporate	-	0.093
Specific Heat Capacity [kJ/kg°C]	4.2	2.06
Heat of Evaporation [kJ/kg]		1176
Temperature IN [°C]	-2.77	-13
Pressure IN [bara]	1.3	2.6
Temperature OUT [°C]	-2.77	-13
Pressure OUT [bara]	1	2.6
Material	Stainless Steel	Stainless Steel

SPECIFICATION FORM RECRYSTALLISER

UNIT NUMBER: R01
No. Serial: 1
No. Parallel: -

Type: Recrystalliser Tank

Design: Cylindrical, with agitator and filter installed.

Position: Vertical

Absolute Pressure	: 1	[bara]
Temperature	: -3.8	[°C]
Total Volume	: 78.5	[m^3]
-Diameter	: 5	[m]
-Height	: 4	[m]
Material	: Stainless Steel	

Specifics

Agitator installed (5 kW)
Filter installed (10 m^2)
Insulation required

VII g SPECIFICATIONFORM EFC COLUMN

UNIT NUMBER : RO2

General properties

Function = Freezing water and crystallising ammonium nitrate
Column type = Cooling Disc Column Crystalliser

Type of discs = Cooling Disc with four orifices
Number of discs
- **theoretical** = 10.4
- **practical** = 11
- **Feed disc (pract)** = 5

Disc distance = 0.21 [m] ----- **Material of discs :** Stainless steel
Diameter column = 2.5 [m] ----- **Column height** : 4.7 [m]
Column material : Stainless steel

Conditions

	Feed (23)	Top (24)	Bottom (45)
Temp. [°C]	-14	-17	-17
Pressure [bar]	1.5	1	1
Density [kg/m^3]	998	1143	1305
Massflow [kg/s]	1.9489	1.8218	0.1270
	wt%	wt%	wt%
Composition			
Ammonium nitrate			0.2000
NH4+	0.0792	0.0762	0.0762
NO3-	0.2723	0.2622	0.2622
Water	0.6486	0.4616	0.4616
Ice		0.2000	

Design

Number of orifices : 44

Surface area of discs : 108 [m^2]

VII h SPECIFICATION FORM WASH COLUMN

UNIT NUMBER C02			
Type:	Hydraulic Wash Column		
Operating Conditions			
Capacity	: 2.06	[kg/s]	
Feed Pressure	: 2.75	[bara]	
Wash Pressure	: 2.13	[bara]	
Temperature	: -3	[°C]	
Vessel Data			
Diameter	: 23.5	[m]	
Cross Sectional Area	: 0.582	[m^2]	
Height	: 0.75	[m]	
Bed height	: 0.125	[m]	
Stagnant zone height	: 0.125	[m]	
Feed zone height	: 0.50	[m]	
Filter			
Diameter tubes	: 0.02	[m]	
Height	: 0.03	[m]	
Number of tubes	: 157		
Material			
Vessel	: Stainless Steel	Tubes	: Stainless Steel

VII i SPECIFICATION FORM HEAT EXCHANGER

UNIT NUMBER: E01
No. Serial: 1
No. Parallel : -

Common Properties

Type : heat exchanger

Design : floating head

Position : horizontal

Heat Capacity : 1156 [kW] (Calculated)
Heat Exchanging Area : 48.90 [m^2] (Calculated)
Overall heat transfer coefficient: 1000 [$W/m^2.K$]
Logaritmic mean temp. diff. (LMTD) : 23.64 [°C]

No. of passes tubeside : 2
No. of passes shell side : 1

Correctionfactor LMTD : 1
True mean temp. diff. : 23.64 [°C]

Operation Conditions

	Shell-side	Tube-side
Liquid	AN solution (water)	ice slurry
Mass flow [kg/s]	4.5	22.7
Mass flow to		
- evaporate [kg/s]	-	-
- condense [kg/s]	-	-
Average specific heat [kJ/kg.°C]	4.2	3.8
Heat of evaporation [kJ/kg]	-	-
Temperature IN [°C]	66	0
Temperature OUT [°C]	5	0
Pressure [bar]	1.3	2.9
Material	stainless steel	stainless steel

VII j SPECIFICATION FORM CENTRIFUGAL PUMP

UNIT NUMBER P12			
Duty: 3.2 [kW] **Type:** Centrifugal Pump			
	Physical properties of pumpfluid		
Fluid	: Water		
Temperature (T)	: 20	[°C]	
Density (ρ)	: 996	$[kg/m^3]$	
Viscosity (η)	: $1.0 * 10^{-3}$	$[N.s/m^2]$	
Vapour pressure (p_d)	: 378	[Pa]	**at temperature (T) :** 20 [°C]
		Power	
Capacity (Φ_v)	: $4.5 * 10^{-3}$	$[m^3/s]$	
Suction Pressure (p_z)	: 2.3	[bar]	
Discharge Pressure (p_p)	: 7	[bar]	
Theoretical Power	: 2.1	[kW]	
Efficiency	: 0.65	[-]	
Pump Shaft Power	: 3.2	[kW]	
		Construction Data	
h_{man}	: 48	[m]	
N.P.S.H.$_{avail.}$	: 23.5	[m]	
		Material	
Pump-housing	: Stainless Steel		
Impeller	: Stainless Steel		
As	: Stainless Steel		

APPENDIX VIII HAZOP

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Pump P01 Duty: Pumping feed from vessel V01 to neutraliser V04 Stream: 8, feed to neutraliser V04				
NO	Flow	1. Pump fails (motor fault). 2. Vessel V01 empty because ammonium nitrate plant shut down.	Vessel V01 overfills. No feed for the plant.	a. Repair pump. b. Install low level alarm on vessel V04. c. Install low level alarm on V01. d. Install one way valve befor pump.
MORE	Flow	Ammonium nitrate plant produces more wastewater.	Neutraliser overfills	a. Catch wastewater in storage tank. b. Dump wastewater in the sewer.
Equipment: Neutraliser V04 Duty: Makes the feed a stoicionetric molar ratio of nitric acid and ammonia Streams: 13, 6 and 7 in; 14 out				
NO/LESS	1. NH_3 flow	Flow controller failure or valve fail-closed.	Accumulation of HNO_3 in the process.	a. Shut valve on stream 7 b. Release streams with HNO_3 in a vessel or the sewer
	2. HNO_3 flow	Flow controller failure or valve fail-closed.	1. Under normal operating conditions the total feed is acidic, the pH can still be control-led with stream 6. 2. If not normal operating conditions, the total feed is basic then: accumulation of NH_3 in the process.	Release streams with NH_3 in a storage vessel. Streams with too much NH_3 cannot be released in the sewer.
MORE	1. NH_3 flow	Flow controller failure or valve fail-open.	Accumulation of NH_3 in the process	Release streams with NH_3 in a storage vessel. Streams with too much NH_3 cannot be released in the sewer.
	2. HNO_3 flow	a. Flow controller failure or valve open. b. Feed stream is too acidic.	Accumulation of HNO_3 in the process.	Release streams with HNO_3 in a vessel or the sewer

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Heat exchanger E01 Duty: Cooling feed and melting ice product Streams: 14 (feed) and 34 (ice slurry) in; 15 and 35 out				
MORE	Flow	1. More flow in stream 14.	Temperature in 15 is higher.	Freeze concentrator must be cooled more; increase of ammonia flow through SSHE required.
		2. More flow in stream 34.	a. Less ice will melt and heat exchanger E03 can be blocked with ice. b. The temperature of stream 44 (product; water) will be lower than 20°C.	Leading more flow to nitric acid plant or the sewer untill the ice in E03 has melted. No action required.
MORE	Ice	More ice is produced in washcolumns	Pipes and equipment will freeze.	Shutting down the plant and waiting (in summer) or heating (in winter) till the ice has melted.
NO/LESS	Flow	1. Less flow in stream 14.	Temperature in 15 is lower.	Reduce ammonia flow through SSHE.
		2. Less flow in stream 34.	Temperature of stream 44 will be higher than 20°C.	Flow controller on stream 10.
		3. Heat exchanger is blocked with ice.	Tubes of heat exchanger can burst.	Shutting down the plant and waiting (in summer) or heating (in winter) till the ice has melted.
MORE	Temperature	Stream 14 is warmer.	1. Stream 15 is warmer. 2. More ice will melt. The temperature of stream 44 (product; water) will be higher than 20°C.	Increase ammonia flow through SSHE. Flow controller on stream 10.
LESS	Temperature	Stream 14 is colder	1. Stream 15 is colder. 2. Less ice will melt and heat exchanger E03 can be frozen up with ice.	Reduce ammonia flow through SSHE. Leading more flow to nitric acid or the sewer plant untill the ice in E03 has melted.
MORE	Pressure	1. P07, P08 and/or P09 are working too fast	Ice melts at lower temperature.	No action required
		2. Tubes of heat exchanger are blocked with ice.	Pipes can burst or plant flow stops.	Shutting down the plant and waiting (in summer) or heating (in winter) till the ice has melted.

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Freeze concentrator R01 Duty: Concentrating the slurry by freezing the water Streams: 16 in; 20 out				
MORE	Flow	1. Flow of stream 15 is higher. 2. Flow of recycle stream 33 is higher.	Not enough ice will be produced to reach 7 w% AN. Residence time will be shorter.	More ammonia has to be evaporated in SSHE. No action required.
NO/LESS	Flow	1. Ice filter in R01 broke down and ice froze up in SSHE. 2. No/less recycle stream 33.	SSHE breaks down. Ice cannot be transported because there is too much ice in the slurry. Pump P04 breaks down.	Stop NH_3 flow and wait untill the ice has melted. Repair SSHE. Stop evaporating ammonia and wait untill the ice has melted. Repair P04.
MORE	NH_3 flow	Temprature controller failure.	Brine in reactor becomes undercooled. Ice is formed later in pipes and pumps.	Close ammonia valve in stream 105. Wait untill the ice has melted. Repair controller.
LESS	NH_3 flow	1. Temprature controller failure. 2. Vessel V06 is empty.	Less ice formation.	Repair controller. Check level of vessel and refill when required.
MORE	Temperature	Ammonia flow controller failure or valve fail-closed.	No ice and no ammonia nitrate is being produced.	Shut down plant and repair flow controller.
MORE	Pressure	Too much ice is produced.	Pipes and pumps will freeze up with ice.	Stop ammonia flow and wait untill the ice has melted.
LESS	Temperature	Ammonia flow controller failure or valve fail-open.	Too much ice will be produced and pump P04 will break down.	Shut down the plant and repair or replace pump P04 and repair ammonia flow controller.

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: R02 Cooling Disc Column Crystalliser Duty: Freezing ice and crystallising ammonium nitrate Streams: 23 in; 24 (ice slurry) and 45 (ammonium nitrate) out				
MORE	Flow	1. Flow of steam 22 is higher. 2. Flow of recycle stream is higher.	Not enough ice will be produced to reach the eutecticum. Residence time will be shorter.	More ammonia has to be evaporated in the discs. No action required.
NO/LESS	Flow	1. Flow controller failure. 2. No/less recycle stream 49.	Production will be lower. Recycle 33 will accumulate. Ice cannot be transported because there is too much ice in the slurry. Pump P05 breaks down.	Repair flow control. Stop evaporating ammonia and wait untill the ice has melted. Repair P05.
MORE	NH_3 flow	Temprature controller failure.	Brine in reactor gets undercooled. Ice is formed later in pipes and pumps.	Close ammonia valve in stream 113. Wait untill the ice has melted. Repair controller.
LESS	NH_3 flow	1. Temprature controller failure. 2. Vessel V07 is empty.	No ice and ammonium nitrate produced.	Repair controller. Refill vessel.
MORE	Temperature	Ammonia flow controller failure.	No ice and no ammonia nitrate produced.	Shut down plant and repair flow controller.
MORE	Pressure	Too much ice is produced.	Pipes and pumps will be freeze up with ice.	Stop ammonia flow and wait untill the ice has melted.
LESS	Temperature	Ammonia flow controller failure.	Too much ice will be produced and pump P05 will break down.	Shut down the plant and repair or replace pump P05 and repair ammonia flow controller.
AS WELL AS	Ammonium nitrate crystals are present in stream 24	There is too much turbulence in CDCC (R02).	a. Pump P05 can break down. b. Product is unpure.	Check flow in CDCC.

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Washcolumns C01 (C02 or C03) Duty: Washing of ice slurry that comes from the CDCC (or R01) Streams: 24 (ice slurry) and 41 (water) in; 25 (pure ice slurry) and 26 (recycle) out				
MORE	Flow	1. More ice slurry (stream 24) is coming in C01.	a. More ice produced.	Increase recycle 41.
			b. Scraper can break.	Repair scraper.
		2. More water (stream 41) is coming in C01 because of flow controller failure or valve fail-open.	Too much washwater comes in the bed and stream 26 is diluted.	Repair flow controller and restart plant.
NO/LESS	Flow	1. Flow controller failure in stream 41or valve fail-closed.	Ice bed is too cold. Impure water will also freeze and the product will be impure.	Repair flow controller.
		2. Pump P05 is blocked with ice.	Pump P05 will break down.	Shut down the plant and wait untill the ice has melted. Repair pump P05.
MORE	Temperature	Flow controller failure of stream 41or valve fail-closed.	Less ice freezes up on the bed and the temperature on top of the bed increases.	Install temperature controller which controls the amount of water to be frozen on the bed (stream 41).
LESS	Temperature	Slurry coming out R02 is undercooled.	Washcolumn will be blocked with ice.	Remove ice block from C01 and restart process. When only C02 or C03 has a problem, plant can operate at half capacity.
MORE	Pressure	Pump P07 is blocked with ice.	Pump P07 will break down.	Shut down the plant and wait untill the ice has melted. Repair pump P07.
AS WELL AS	Ammonium nitrate present in ice high purity ice slurry (25)	Less washwater is added to C01 due to flow controller failure on stream 41.	Product of high purity water not pure enough.	Repair flow controller.

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Filter F01 Duty: Filtration of ammonium nitrate slurry Streams: 45 in; 46 (filtrate) and 48 (retentate) out				
MORE	Flow	Pump P06 runs too fast	a. Residence time in filter is shorter so solution of AN is < 90 w% AN. b. Recycle 48 is too small. Pumps can be choked up with ice.	Install flow controller on stream 45. Shut down the plant.
NO/LESS	Flow	1. Pump P06 is blocked with ice. 2. Pump P06 runs too slow.	Pump can break down. Residence time in the filter is longer so solution of AN is > 90 w% AN.	Shut plant and wait untill the ice has melted. Install flow controller on stream 45.
AS WELL AS	Ice is present in ammonium nitrate slurry.	Ice is sucked in stream 45 by pump P06 due to turbulence in the R02.	Ice will melt in the dissolver and so the solution of AN is < 90 w% AN.	Check flow in CDCC.
MORE	Temperature	Isolation round the pipes is broken.	Solid ammonium nitrate dissolves. Concentration is higher. Less product is produced and a larger warmer recycle stream of too high concentration.	Repair isolation round the pipes.
LESS	Temperature	Solution is undercooled due to ammonia flow controller failure.	More ice and ammonium nitrate will be produced in pipes. Pipes and pumps will be blocked with solids.	Shut down the plant, repair flow controller and wait untill the ice melts.
NO	Water on filter	1. Filter runs too fast. 2. Plant shut and filter cake dries.	Nothing happens in dissolver. Explosion hazard.	Stop filter rotations. Add water. Install an humidity meter and alarm system
Equipment: Dissolver V05 Duty: Heating and dissolving the concentrated ammonium nitrate slurry Streams: 46 in; 47 out				
MORE	Flow	Build up of filter cake.	Solution doesn't reach the requirement of 90 w% AN. No recycle stream to R02.	Clean filter.
NO/LESS	Flow	Filter runs too slow.	Less product is produced	Check rotating speed of filter and filter pressure.
MORE	Temperature	Steam controller failure.	Product is warmer than 95°C. Steam costs are higher than necessary.	Repair steam controller.
LESS	Temperature	Steam controller failure.	Ammonium nitrate won't dissolve completely.	Repair steam controller.

Guide Word	Deviation	Possible Causes	Consequences	Action Required
Equipment: Flash vessel V06 Duty: Flashing ammonia Streams: 102 in; 104 (gas) and 105 (liquid) out				
MORE	Flow	Level controller failure or valve fail-open.	Flash vessel overfills.	Shut valve on ammmonia stream and repair level controller.
NO/LESS	Flow	Level controller failure or valve fail-closed.	Vessel empties and only vapour flows through reactors. No heat transport in the reactors.	Repair level controller.
MORE	Pressure	Pressure controller failure or valve fail-closed.	Temperature of liquid ammonia is too high. Driving force for heat transport in the reactors is too small.	Open valve in stream 104 and repair pressure controller.
LESS	Pressure	Pressure controller failure.	Temperature of liquid ammonia is very low. Compression costs to bring ammonia to 4.2 bar are much higher.	Shut valve in stream 104 and repair controller.
Equipment: Compressor K01 Duty: Compressing ammonia to 4.2 bara Streams: 110 in; 111 out				
MORE	Temperature	1. No isolation round pipes. 2. Compressor is running inefficiently.	Less compression work is required. More work is required.	No action required. Check efficiency of compressor and repair if required.
LESS	Temperature	Pressure controller failure on stream 104.	More compression work is required.	Shut valve in stream 104 and repair controller.
MORE	Pressure	1. Pressure controller failure in stream 111. 2. Compressor is running too hard.	Stream 111 flows to compressor K02.	Put one way valve in stream 119. Repair pressure controller.
LESS	Pressure	1. Pressure controller failure. 2. Compressor is not running efficiently.	Ammonia stream is not on the required pressure of 4.2 bar. Stream 119 will flow to compressor K01.	Put one way valve in stream 111 and repair pressure controller.
AS WELL AS	Liquid present	1. There is too much liquid present in the vessel due to level controller failure. 2. Demister has broken down.	Ammonia product stream doesn't fit the demands of being a gas.	Repair level controller. Repair demister.

APPENDIX IX ECONOMY

IX a Investment Costs Calculations

Total Purchase Cost Equipment (PCE)	2240928
f1: Equipment erection	0.45
f2: Piping	0.45
f3: Instrumentation	0.15
f4: Electrical	0.10
f5: Buildings, Process	0.10
Total factor F1	2.25
Total Physical Plant Cost (PPC)	5042089

f10: Design and Engineering	0.25
f11: Contractor's fee	0.05
f12: Contingency	0.10
Total factor F2	1.40
Fixed Capital (FC)	7058925

Working Capital (5% FC)	352946
Total Investment (FC+WC)	**7411871**

Subsidies (NOVEM, 25 %)	1852968
Investment less subsidies	**5558903**

Calculation Total Purchase Cost Equipment

Inflation Factor 92-97	1.35
Curr. Factor Pound-Nfl (92)	3.2

Eutectic Freeze Crystallizer

Part of Equipment (D(m))	Material	Height (m)	Mat.Fact.	Press.Fact	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
Vessel (2.5)	S.S.	4.63	2	1	12000	24000	32400	103680
	Material	Amount	Mat.Fact.		B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
Plates (2.5)	S.S.	11	1.7		500	9350	12623	40392
						Total Costs EFC		144072

Wash Columns

Vessel (D)	Material	Height (m)	Mat. Factor	Press. Fact.	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
C01 (0.36)	S.S.	0.75	2	1	1800	3600	4860	15552
C02 (0.86)	S.S.	0.75	2	1	2100	4200	5670	18144
C03 (0.86)	S.S.	0.75	2	1	2100	4200	5670	18144
	Material	Diameter (m	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
Tubes (L=0.75)	S.S.	0.02	1	31	0.6	2	3	10
Total Tubes Costs (342)						760	1027	3285
	Material	Power (kW)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
Scraper	S.S.	5	2	2000	0.6	10506	14183	45386
Total Scraper Costs (3)						31518	42550	136159
						Total Costs Wash Columns		191284

Heat Exchangers	Material	Area (m2)	Type Fact.	Press. Fact.	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
E01	S.S	48.9	1	1	30000	30000	40500	129600
E02	S.S	6.8	1	1	5500	5500	7425	23760
E03	S.S	15.1	1	1	12000	12000	16200	51840
						Total Costs HE		205200

Freeze Concentrator

Heat Exchanger	Material	Area (m2)	Mat. Fact.	Press. Fact.	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	12	2	1	4000	8000	10800	34560
Scraper	Material	Power (kW)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	5	2	2000	0.6	10506	14183	45386
						Total Cost 1 HE		79946
						Total Cost SSHE (14 tubes)		1119249.6
Crystalliser Tank	Material	Volume (m3	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
Tank (hor.)	S.S.	80	2	1500	0.6	41589	56145	179663
Insulation (5%)								8983
Filter	Material	Area (m2)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S	10	2	2300	0.6	18313	24722	79112
Agitator (propeller)	Material	Power (kW)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	5	2	1000	0.5	4472	6037	19320
						Total Ripening Tank		287078
						Total Freeze Concentrator		1406327

Neutraliser

Tank	Material	Volume (m3	Mat. Factor	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	3.53	2	1250	0.6	509	688	2200
Agitator (propellor)	Material	Power (kW)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	5	2	1000	0.5	4472	6037	19320
						Total Costs Neutraliser		21520

Cooling Process

Flash Vessels (D(m))	Material	Height (m)	Mat. Factor	Press. Fact.	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
V06 (0.31)	S.S.	1.40	2	1	2000	4000	5400	17280
V07(0.14)	S.S.	0.68	2	1	1600	3200	4320	13824
Knock-out Drums (D(m))	Material	Height (m)	Mat. Factor	Press. Fact.	B. Cost	P. Cost (92)	P. Cost (97)	Cost (Nfl)
V08 (0.95)	S.S.	1.39	2	1	2000	4000	5400	17280
V09 (0.34)	S.S.	0.66	2	1	1600	3200	4320	13824
Compressors	Material	Power (kW)		Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
K01	S.S.	93.4		500	0.8	18847	25444	81420
K02	S.S.	22.9		500	0.8	6121	8264	26444
						Total Costs Cooling Process		170072

Filter	Material	Area (m2)	Mat. Factor	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	1.33	2	9000	0.6	21359	28835	92271

Dissolver

Tank	Material	Volume (m3	Mat.Factor	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	0.039	2	1250	0.6	357	482	1542
Agitator (propellor)	Material	Power (kW)	Mat. Fact.	Constant	Index	P. Cost (92)	P. Cost (97)	Cost (Nfl)
	S.S.	1	2	1000	0.5	2000	2700	8640
						Total Dissolver Costs		10182

Total Purchase Equipment Costs	2240928

IX b Operating Costs Calculations

Inflation Factor 92-97	1.35
Curr. Factor Pound-Nfl (92)	3.2
Annual Operating Time (h)	8000

	Variable Costs			
			M (ton/a)	Nfl.
1	Raw Materials	Nitric Acid	34.56	6601
		Ammonia	92.16	30874
2	Miscellaneous Materials	10% Maintenance		35295
3	Utilities	Steam	271.872	8156
		Electricity		143500
4	Shipping and Packaging	Negligable		0
		Total Variable Costs		219296

	Fixed Costs		
5	Maintenance	5% of Fixed Capital	352946
6	Operating Labour	1/2 man = 10000 Pounds (92)	43200
7	Laboratory Costs	20% of Operating	8640
8	Supervision		0
9	Plant Overheads	50% of Labour Costs	21600
10	Capital Charges	10% of Fixed Capital	705892
11	Rates (and local taxes)	1% of Fixed Capital	70589
12	Insurance	1% of Fixed Capital	70589
13	Licence fees and royalty payments	-	0
		Total Fixed Costs	1273457

Direct Production Costs	1532549

14	Sales Expense		
15	General Overheads		306510
16	Research & Development		
17	Reserves		

Annual Production Costs	1839059

IX c Sales Income

		kg/s	ton/a				NFl
Ammonium Nitrate		0.0307	884.16				208662
Water		4.482	129082				645408
Waste Water		4.5083	129839				414600
		kg/s	kW	kg/s steam	ton/a steam	NFl	NFl
Evaporation and heating NH_3 in cold period		1.448	2079.21	1.37514	39604	1188121	594061
Evaporation and heating NH3 in rest of year		1.448	80.54	0.053266	1534	46022	23011
Sales Income							1862730

IX d Cash-Flow Data

End of Year	Sales	Sales Less Operating	Investment Costs (less subsidies)	Net Cash Flow (NFW)	Cum. Cash Flow (Project NFW)	Disc. Cash Flow at 10% (NPW)	Cum. DCF (Project NPW)
	M Nfl	M Nfl	M Nfl	M Nfl	M Nfl	M Nfl	M Nfl
1	0	0	1.00	-1.00	-1.00	-0.91	-0.91
2	0	0	2.30	-2.30	-3.30	-1.90	-2.81
3	1.86	0.065	0.40	0.025	-3.28	0.02	-2.79
4	1.86	0.065	0	0.065	-3.21	0.04	-2.75
5	1.86	0.065	0	0.065	-3.15	0.04	-2.71
6	1.86	0.065	0	0.065	-3.08	0.04	-2.67
7	1.86	0.065	0	0.065	-3.02	0.03	-2.64
8	1.86	0.065	0	0.065	-2.95	0.03	-2.61
9	1.86	0.065	0	0.065	-2.89	0.03	-2.58
10	1.86	0.065	0	0.065	-2.82	0.03	-2.55
11	1.86	0.065	0	0.065	-2.76	0.02	-2.53
12	1.86	0.065	0	0.065	-2.69	0.02	-2.51
13	1.86	0.065	0	0.065	-2.63	0.02	-2.49
14	1.86	0.065	0	0.065	-2.56	0.02	-2.47
15	1.86	0.065	0	0.065	-2.50	0.02	-2.46
16	1.86	0.065	0	0.065	-2.43	0.01	-2.44
17	1.86	0.065	0	0.065	-2.37	0.01	-2.43
18	1.86	0.065	0	0.065	-2.30	0.01	-2.42
19	1.86	0.065	0	0.065	-2.24	0.01	-2.41
20	1.86	0.065	0	0.065	-2.17	0.01	-2.40

ROR -2.93

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EET Subsidieregeling Economie Ecologie en Technologie

OMSCHRIJVING

Doel van deze regeling is een meerjarige ondersteuning van duurzame technologische ontwikkelingen.
In dit kader vindt steunverlening plaats voor fundamenteel onderzoek, industrieel onderzoek, pre-concurrentiële ontwikkeling of een combinatie van deze activiteiten.

Projecten moeten betrekking hebben op één van de volgende thema's:
1. de vermindering van de milieubelasting door proceswater, inclusief koelwater, in de Nederlandse industrie (bijvoorbeeld waterzuivering, recirculatie, een beter watermanagement);
2. de vermindering van het afvalprobleem van de Nederlandse industrie;
3. de integratie van het milieu in het productontwikkelingsproces (bijvoorbeeld een milieuvriendelijk kantoor of een keuken als geïntegreerd energiesysteem);
4. het beperken van emissies en energiegebruik in de sector 'verkeer en vervoer';
5. het gebruik van hernieuwbare grondstoffen;
6. het gebruik van duurzame energiebronnen.

DOELGROEP

Een bijdrage is beschikbaar voor een:
- onderneming:
- alle ondernemingen;
- non-profitinstelling:
- alle non-profitinstellingen;
- publiekrechtelijke instelling:
- semi-publiekrechtelijk:
- overige publiekrechtelijke instellingen.

Hieronder worden verstaan: samenwerkingsverbanden van bedrijven en kennisinstellingen.

REGIO

Activiteiten moeten plaatsvinden in:
- Nederland.

VOORWAARDEN

Bij de aanvraag gelden de volgende voorwaarden:
- het project moet een substantiële bijdrage leveren aan de doelstellingen van het programma;
- het moet gaan om een grootschalig project (circa f.1 miljoen per jaar en een looptijd van meerdere jaren);
- het moet gaan om vernieuwende technologische activiteiten (doorbraken);
- het project moet worden uitgevoerd door een samenwerkingsverband

dat bestaat uit ten minste 2 niet verbonden natuurlijke (rechts)personen, onder wie ten minste één ondernemer;
- het project moet op een termijn van 5 tot 20 jaar leiden tot belangrijke resultaten op het gebied van milieu en economie
- overlegging van een projectplan dat moet bevatten:
a. een omschrijving van de activiteit en een begroting met toelichting;
b. de aard en tijdsduur van de activiteit.

BIJDRAGE

Steunverlening vindt plaats tot maximaal 62,5% van de projectkosten, afhankelijk van de fase waarin het project zich bevindt, tot een maximum van f.10 miljoen.

Een bijdrage is mogelijk voor onder andere:
1. loonkosten, waarbij samenwerkingsverbanden een beroep kunnen doen op de Wet Bevordering Speur- en Ontwikkelingswerk (WBSO);
2. verbruikte materialen en hulpmiddelen;
3. aangeschafte machines en apparatuur;
4. reis- en verblijfkosten;
5. kosten gemaakt door derden.
Iedere partner in het samenwerkingsverband moet minimaal 25% van de eigen projectkosten bijdragen.

AANVRAAG

De aanvraag moet worden ingediend bij:
Programmabureau E.E.T.
Postbus 8242
3503 RE Utrecht.
Een aanvraagformulier is verkrijgbaar bij het aanvraagadres.
Inlichtingen zijn verkrijgbaar bij:
Mw. S. Smulders
Tel: (030) 239 34 36
Fax: (030) 231 64 91.

Aanvragen kunnen worden ingediend na een openbare uitnodiging.
De indiening van de voorstellen verloopt in 2 stappen:
- eerst moet er een beknopt projectvoorstel op hoofdlijnen worden ingediend.
- daarna kan de officiële aanvraag, voorzien van een pré-advies, worden ingediend.
In 1997 zijn er twee oproeprondes. Het indienen van aanvragen voor de eerste ronde is mogelijk:
a. van 22-01-1997 tot 71-03-1997 (de adviesronde);
b. van 22-04-1997 tot 30-06-1997 (definitieve voorstellen);
Het indienen van aanvragen voor de tweede ronde is mogelijk van 08-09-1997 tot 10-11-1997 (adviesronde).

Op de aanvraag beslist de Minister binnen 4 maanden na sluitingsdatum.
De behandeling van de aanvragen vindt plaats na prioriteitstelling. Hierbij zijn de volgende criteria van belang:
a. het realiseren van de ecologische doelstellingen;
b. het realiseren van een duurzame groei;
c. het bijdragen aan het vergroten van de technische innovativiteit van het bedrijfsleven en van de kennisinfrastructuur in Nederland;
d. het structureel verbeteren van de samenwerking tussen ondernemers onderling en tussen ondernemers en de kennisinfrastructuur.

NADERE VOORSCHRIFTEN

Bij steunverlening gelden de volgende voorschriften:
- overlegging van een accountantsverklaring;
- overlegging van een voortgangsrapportage;
- overlegging van een financieel en inhoudelijk verslag na beëindiging van het project, waarmee om definitieve vaststelling van het steunverleningsbedrag wordt gevraagd.

AANVULLENDE INFORMATIE

De EET-regeling zal in 1997 worden voortgezet. De nieuwe regeling is gepubliceerd als Besluit Subsidies Economie, Ecologie en Technologie. Begin 1997 gaat een nieuwe oproepronde van start. Bron: Stb.1997, nr.13.

In bepaalde gevallen kan een project gedeeltelijk in het buitenland worden uitgevoerd, als dat zorgt voor een aantoonbare meerwaarde.

BRON

De regeling is gepubliceerd in de Staatscourant 1996, nr.91.
De regeling is afkomstig van het ministerie van Economische Zaken en van Onderwijs, Cultuur en Wetenschappen.
Er is een brochure beschikbaar over deze regeling getiteld: Economie, Ecologie en Technologie.
Deze is verkrijgbaar bij het aanvraagadres.

RECHTSWERKING

Het programma is in werking getreden per 15-05-1996 en heeft een looptijd tot 01-01-1997. Na deze datum zal verlenging plaats vinden.
Er is voor de tender van 1996 f.20 miljoen beschikbaar.
Uiteindelijk zal er voor het E.E.T.-programma jaarlijks f.45 miljoen beschikbaar zijn.
Op de regeling is de Kaderwet financiële verstrekking EZ van toepassing, Staatsblad 1996, 180.

MUTATIES

IX-9

jan 97 De EET-regeling zal in 1997 worden voortgezet. De nieuwe regeling is gepubliceerd als Besluit Subsidies Economie, Ecologie en Technologie. Binnenkort gaat de eerste oproepronde van start. Het indienen van voorlopige voorstellen is mogelijk van 22-01-1997 tot 17-03-1997 (de adviesronde). De definitieve voorstellen kunnen worden ingediend van 22-04-1997 tot 30-06-1997. Later dit jaar zal nog een tweede oproep verschijnen. De adviesronde daarvoor loopt van 08-09-1997 tot 10-11-1997.
Bron: Stb.1997, nr.13.
sep 96 Dit is een nieuwe regeling.
nov 96 De eerste tender van de EET-regeling heeft geleid tot een groot
aantal kwalitatief goede aanvragen. Mogelijk zal de regering besluiten tot een verhoging van het budget. Meer duidelijkheid hierover wordt verwacht als alle definitieve voorstellen binnen zijn (28-10-1996). Overigens moet de EET-regeling bij een budgetverhoging worden gepubliceerd als AMvB. Inhoudelijke wijzigingen worden niet verwacht.
Bron: Kamerstukken 1995-1996, 24.400 XIII, nr.55.

DOEL

milieu: energie, bodem, water, lucht, overige milieu; bedrijfsvoering: investeren, overige bedrijfsvoering; research: fundamenteel/wetenschappelijk, haalbaarheidsstudie, ontwikkeling/demonstratie; technologie: materiaaltechnologie, productietechnologie, milieutechnologie.

NUMMER

01300

TREFWOORDEN

afvalverwerking, alternatieve energie, biomassa, brandstof, ecosysteem, elektriciteit, emissie, energiebesparing, isolatie, kernenergie, kooldioxide, verontreiniging, verwarming, windenergie, zonne energie, waterzuivering, kwaliteitszorg, milieuzorg, productvernieuwing, productieproces, recycling, kennisoverdracht.

IX-10

SETEC

Tel : +31 - 71 - 561 49 40
Fax : +31 - 71 - 561 81 80

FAXBERICHT

FAX : 31.12.97 / 1255
AAN : TU Delft, Lab. API - Delft
TAV : Hr. F. van der Ham
BETR : Concentreren van NH4NO3
UW : Project KEMIRA
ONS : Project 971252
REF : Uw fax van 11.12.97

BLZ. : 1 VAN 10
FAXNR : 015 - 2786975

Geachte heer van der Ham,

Het opconcentreren van een zuivere NH_4NO_3 stroom met een zoutgehalte van 6.129 mg/l tot een concentratie van 12 w% is mogelijk d.m.v. Omgekeerde Osmose.

SETEC heeft in 1996 het opconcentreren van een 12 - 16 w% tot 17 w% NaCl-oplossing uitgebreid getest. Doel van de proeven was het constant houden van de pekelconcentratie in pekelbaden voor de kaas- en vleesverwerkende industrie. De daartoe benodigde systeemdrukken varieerden van 140 - 180 bar. Uit het onderzoek is gebleken dat systeemdrukken tot ca. 140 bar een stabiele bedrijfsvoering gaven. Bij drukken boven de 140 bar trad compactie van de membranen op. Deze toepassing van RO is vanwege de hoge osmotische druk van 17 w% NaCl voor deze industrie thans nog niet mogelijk.

Vanwege de lagere osmotische druk van NH_4NO_3 en de gewenste lagere eindconcentratie van 12 w% is RO een aantrekkelijke mogelijkheid om NH_4NO_3 op te concentreren.

Voor een dergelijke toepassing de volgende informatie:

1. Doel is het opconcentreren van een zuivere NH_4NO_3 oplossing van 6.129 mg/l tot 12 w%. Dit is een 20-voudige indikking.
2. Aangenomen wordt dat de voeding zeer zuiver is en dat geen membraanvervuiling optreedt vanwege deze hoge indikkingsfactor.
3. Uit uw opgave van een membraanoppervlak van 1.431 m2 wordt afgeleid dat de voedingstroom ca. 37 m3/h bedraagt. Bij RO ligt de waterflux op ca. 25 l/h/m2.
4. De voedingwatertemperatuur is op 25 °C gesteld.
5. Bijgaand is een ontwerp van een dergelijke installatie gegeven. De installatie bestaat uit 2 achterelkaar geschakelde RO units.
   De eerste installatie, RO-1, Model SEARO8608, produceert een permeaatstroom van 35 m3/h uitgaande van een voedingstroom van 41 m3/h met een zoutgehalte van 6.129 mg/l NH_4NO_3. Het permeaatzoutgehalte bedraagt 340 mg/l NH_4NO_3. Deze RO heeft een drukvatschakeling in 4 banken (array) van 4+2+1+1. Vanwege de sterke stijging in osmotische druk dient het concentraat van elke bank in druk te worden verhoogd, de "boost pressure". Deze installatie is voorzien van standaard zeewater membranen met verhoogde zoutretentie.
   De tweede installatie, RO-2, Model SEARO4604, concentreert het concentraat van RO-1 op van 39.600 mg/l tot 120.000mg/l NH_4NO_3. Deze installatie is voorzien van membra-

nen met een maximale bedrijfsdruk van 120 bar. Deze membranen zijn niet in een computer programma beschikbaar. Het in het ontwerp van RO-2 membraantype, HR30-4040 kan deze druk niet weerstaan, maar geeft een indruk van RO-2. Het beschikbare hoge druk membraan heeft echter een hogere zoutpassage dan opgegeven in het ontwerp van RO-2. In de praktijk zal het zoutgehalte van het RO-2 permeaat ca. 5.000 mg/l NH4NO3 bedragen. Deze permeaatstroom kan dan aan de voeding van RO-1 worden toegevoegd. De netto permeaat stroom is die van RO-1; t.w. 35 m3/h.

6. In de bijgevoegde kostprijs berekening per m3 permeaat zijn de investering van een dergelijke RO en de membraanvervangingskosten van resp. NLG 750.000,– en NLG 250.000,– genoemd. Bij een verwachte levensduur van de membranen van 4 jaar is de kostprijs per m3 permeaat NLG 0,79, ofwel NLG 27,65 per uur. Dit bedrag is inclusief afschrijving. Voor het verdampen van 35 m3/h water is ca. 7 ton stoom nodig. Met een kostprijs van NLG 20,– per ton stoom zijn de stoomkosten alleen reeds NLG 140,– per uur. Toepassing van RO is derhalve zeer aantrekkelijk.
7. Het permeaat van RO-1 heeft een NH4NO3 gehalte van 340 mg/l. Indien dit NH4NO3 verlies niet acceptabel is of wanneer een lager NH4NO3 gehalte hergebruik van deze stroom mogelijk maakt kan door middel van een RO op het permeaat van RO-1 het NH4NO3 gehalte worden gereduceerd tot 20 mg/l. Het concentraat van deze RO kan aan de voeding van RO-1 worden toegevoerd. De investering in deze RO bedraagt ca. NLG 300.000,–.

Vertouwend dat deze informatie voldoende is om het fabrieksvoorontwerp af te ronden en daarmee de studie, wens ik u een voorspoedig 1998.

Met vriendelijke groet,

SETEC

George Schröder

Bijlagen: Ontwerp van RO-1
Ontwerp van RO-2
Kostprijsberekening per m3 permeaat

```
FilmTec Reverse Osmosis System Analysis, March 97 Version 4.00
Prepared For: TU DELFT - KEMIRA - 971252
Analysis by: GS
Date: 12-31-1997

Feed:                  41,13 M3/H,    6129 MG/L,    25,0 Deg C
Recovery:              85,3 Percent

Array:                  1           2            3            4
No.of PV:               4           2            1            1
Element:          SWHR-380    SWHR-380     SWHR-380     SWHR-380
No.El/PV:               6           6            6            6
El.Total:              24          12            6            6
Boost (BAR):          0,0        10,0          5,0         20,0

Fouling Factor:      0,85

                               FEED     REJECT    AVERAGE

Pressure(BAR)                  21,1      43,6       25,6
Osmotic Pressure(BAR)           3,5      23,3        7,9
NDP(Mean)=    17,9 BAR
Average Permeate Flux=   20,7 L/M2/H, Permeate Flow=   35,07 M3/H

                  Recovery      Permeate       Feed   Feed    Feed
Array El.No.   (Perm/Feed)    M3/D  MG/L       M3/H   MG/L  PRESS(BAR)

 1      1          ,070       17,37  139       10,3   6129    20,7
        2          ,072       16,60  160        9,6   6583    20,3
        3          ,075       15,86  185        8,9   7084    19,9
        4          ,077       15,12  216        8,2   7639    19,6
        5          ,079       14,38  253        7,6   8256    19,3
        6          ,081       13,64  299        7,0   8944    19,1

 2      1          ,073       22,31  210       12,8   9711    28,5
        2          ,074       21,14  247       11,9  10454    27,9
        3          ,076       19,99  286       11,0  11271    27,3
        4          ,077       18,85  327       10,2  12170    26,9
        5          ,079       17,72  376        9,4  13161    26,4
        6          ,080       16,59  434        8,6  14253    26,1

 3      1          ,052       19,86  385       15,9  15454    30,4
        2          ,051       18,46  434       15,1  16280    29,5
        3          ,050       17,13  490       14,3  17131    28,7
        4          ,049       15,88  552       13,6  18005    27,9
        5          ,047       14,69  623       12,9  18897    27,2
        6          ,046       13,57  703       12,3  19805    26,6

 4      1          ,103       29,03  380       11,8  20723    45,7
        2          ,105       26,52  464       10,6  23054    45,2
        3          ,106       24,05  568        9,4  25696    44,7
        4          ,106       21,52  705        8,4  28677    44,4
        5          ,105       19,10  875        7,6  32000    44,1
        6          ,102       16,60 1109        6,8  35667    43,8
```

RO-1
Model SEARO8608

```
FilmTec Reverse Osmosis System Analysis, March 97 Version 4.00
Prepared For: TU DELFT - KEMIRA - 971252
Analysis by: GS
Date: 12-31-1997

Array:           Total      Array 1    Array 2    Array 3    Array 4

Reject (M3/H):                 25,6       15,9       11,8        6,1
Reject (MG/L):                 9711      15454      20723      39610
Perm (M3/D):       842          372        233        100        137
Perm (MG/L):     340           204        306        517        638


Permeate, (MG/L as Ion)
Array:           Total     Array 1    Array 2    Array 3    Array 4

NH4               76,5       46,0       68,8      116,4      143,6
K                  0,0        0,0        0,0        0,0        0,0
Na                 0,0        0,0        0,0        0,0        0,0
Mg                 0,0        0,0        0,0        0,0        0,0
Ca                 0,0        0,0        0,0        0,0        0,0
Sr                 0,0        0,0        0,0        0,0        0,0
Ba                 0,0        0,0        0,0        0,0        0,0
HCO3               0,0        0,0        0,0        0,0        0,0
NO3              263,5      158,3      237,1      400,9      494,5
Cl                 0,0        0,0        0,0        0,0        0,0
F                  0,0        0,0        0,0        0,0        0,0
SO4                0,0        0,0        0,0        0,0        0,0
SiO2               0,0        0,0        0,0        0,0        0,0


Feed/Reject, (MG/L as Ion)
                  Feed   Reject 1   Reject 2   Reject 3   Reject 4

NH4             1379,0     2184,9     3477,0     4662,5     8912,1
K                  0,0        0,0        0,0        0,0        0,0
Na                 0,0        0,1        0,1        0,1        0,3
Mg                 0,0        0,0        0,0        0,0        0,0
Ca                 0,0        0,0        0,0        0,0        0,0
Sr                 0,0        0,0        0,0        0,0        0,0
Ba                 0,0        0,0        0,0        0,0        0,0
HCO3               0,0        0,0        0,0        0,0        0,0
NO3             4750,0     7526,1    11976,6    16060,0    30698,1
Cl                 0,0        0,0        0,0        0,0        0,0
F                  0,0        0,0        0,0        0,0        0,0
SO4                0,0        0,0        0,0        0,0        0,0
SiO2               0,0        0,0        0,0        0,0        0,0

To Balance 0,0 MG/L Sodium and 0,0 MG/L Chloride added to feed.
```

FilmTec Reverse Osmosis System Analysis, March 97 Version 4.00
Prepared For: TU DELFT - KEMIRA - 971252
Analysis by: GS
Date: 12-31-1997

Feed water is Well or Softened Water (BW) SDI < 3

```
FilmTec Reverse Osmosis System Analysis, March 97 Version 4.00
Prepared For: TU DELFT - KEMIRA - 971252
Analysis by: GS
Date: 12-31-1997

Feed:                    5,88 M3/H,  39610 MG/L,   25,0 Deg C
Recovery:               68,0 Percent

Array:                  1            2
No.of PV:               2            2
Element:       HR30-4040   HR30-4040
No.El/PV:               6            6
El.Total:              12           12
Boost (BAR):          0,0          0,0

Fouling Factor:       0,85

                                FEED      REJECT     AVERAGE
                                ----      ------     -------
Pressure(BAR)                  100,4       96,0        21,8
Osmotic Pressure(BAR)           22,8       76,4        17,3
NDP(Mean)=      5,7 BAR
Average Permeate Flux=   25,6 L/M2/H, Permeate Flow=      4,00 M3/H

                      Recovery      Permeate       Feed  Feed    Feed
Array El.No.         (Perm/Feed)   M3/D MG/L       M3/H   MG/L PRESS(BAR)
----- ------         -----------   ---- ----       ----   ---- ----------

  1      1               ,121      8,57  495       2,9  39610   100,1
         2               ,122      7,56  634       2,6  45019    99,3
         3               ,120      6,55  822       2,3  51182    98,8
         4               ,116      5,57 1080       2,0  58070    98,3
         5               ,110      4,64 1436       1,8  65563    97,9
         6               ,101      3,80 1921       1,6  73452    97,6

  2      1               ,090      3,05 2588       1,4  81468    97,0
         2               ,079      2,45 3454       1,3  89272    96,8
         3               ,069      1,95 4576       1,2  96661    96,6
         4               ,059      1,57 5928       1,1  03451    96,4
         5               ,051      1,26 7607       1,0  09607    96,3
         6               ,044      1,03 9574       1,0  15071    96,2

Array:            Total        Array 1    Array 2
                  -----        -------    -------

Reject (M3/H):                    2,8        1,9
Reject (MG/L):                  81468     119878
Perm (M3/D):         96            73         23
Perm (MG/L):       1842           937       4778
```

RO-2
Model SEPRO4604

```
FilmTec Reverse Osmosis System Analysis, March 97 Version 4.00
Prepared For: TU DELFT - KEMIRA - 971252
Analysis by: GS
Date: 12-31-1997

Permeate, (MG/L as Ion)
Array:         Total    Array 1    Array 2
               -----    -------    -------

NH4            414,5      210,9     1075,1
K                0,0        0,0        0,0
Na               0,0        0,0        0,0
Mg               0,0        0,0        0,0
Ca               0,0        0,0        0,0
Sr               0,0        0,0        0,0
Ba               0,0        0,0        0,0
HCO3             0,0        0,0        0,0
NO3           1427,9      726,4     3703,3
Cl               0,0        0,0        0,0
F                0,0        0,0        0,0
SO4              0,0        0,0        0,0
SiO2             0,0        0,0        0,0

Feed/Reject, (MG/L as Ion)
                Feed   Reject 1   Reject 2
                ----   --------   --------

NH4           8912,0    18329,5    26971,5
K                0,0        0,0        0,0
Na               0,4        0,8        1,2
Mg               0,0        0,0        0,0
Ca               0,0        0,0        0,0
Sr               0,0        0,0        0,0
Ba               0,0        0,0        0,0
HCO3             0,0        0,0        0,0
NO3          30698,0    63137,3    92905,1
Cl               0,0        0,0        0,0
F                0,0        0,0        0,0
SO4              0,0        0,0        0,0
SiO2             0,0        0,0        0,0

To Balance 0,4 MG/L Sodium and 0,0 MG/L Chloride added to feed.

Feed water is Well or Softened Water (BW) SDI < 3

WARNING ! MAXIMUM RECOMMENDED PRESSURE HAS BEEN EXCEEDED
WARNING ! MAXIMUM RECOMMENDED PERMEATE FLUX HAS BEEN EXCEEDED

DISCLAIMER:  NO WARRANTY, EXPRESSED OR IMPLIED, AND NO WARRANTY OF
MERCHANTABILITY OR FITNESS, IS GIVEN.  Neither FilmTec Corporation nor
The Dow Chemical Company assume liability for results obtained or damage
incurred from the application of this information.  Any final design
should be reviewed by the appropriate applications engineering personnel
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SETEC COSTPRICE per m3 of PERMEATE p.1 of 3

Separation Technologies BV - P.O.Box 90 - 2250 AB Voorschoten - the Netherlands - Tel: +31-71-5614940 - Fax: +31-71-5618180

CLIENT	TU DELFT
PROJECT	KEMIRA
PLANT MODEL	SEARO8608+4604
PROJECT NR	971252
DATE	31.12.97

1 BASIS FOR COST PRICE CALCULATION

Plant capacity	39,0	m3/h
R.O. Recovery	95	%
Differential pressure HP Pump	50,0	bar
HP Pump efficiency	65	%
Plantfactor	90	%
96% H2SO4 consumption	0	gr/m3 feed
Sequestrant consumption	0	gr/m3 feed
Hypochlorite consumption	0,00	gr/m3 permeate
Lime consumption	0	gr/m3 permeate
Hardware lifetime	10	years
Membrane lifetime	4,0	years
Operator attendance per day	0,1	h/day
Spare parts as % of equipment	1	%/year
Total Plant price	750.000	NLG
Cost of membranes	250.000	NLG
Interest rate	7,0	%
Cost of energy	0,15	NLG/kWh
Cost of 96% H2SO4	0,30	NLG/kg
Cost of sequestrant	12,00	NLG/kg
Cost of hypochlorite	6,00	NLG/kg
Cost of Lime	0,30	NLG/kg
Cost per m3 feedwater	0,00	NLG
Cost per m3 effluent	0,00	NLG
Cost of cleaning chemicals	500,00	NLG/year
Cost of manpower	40,00	NLG/h
Yearly production	307.476	m3/year
Consumed electrical load	86,1	kW

2 SUMMARY COST ITEMS per m3 PERMEATE

AMORTISATION	see item 3	0,232	NLG/m3
FEEDWATER and EFFLUENT	see item 4	0,000	NLG/m3
ENERGY	see item 5	0,331	NLG/m3
MEMBRANE REPLACEMENT	see item 6	0,203	NLG/m3
SPARE PARTS	see item 7	0,016	NLG/m3
CHEMICALS	see item 8	0,002	NLG/m3
MAINTANANCE	see item 9	0,005	NLG/m3
TOTAL COST PRICE PER m3 OF PERMEATE		0,789	NLG/m3

SETEC COSTPRICE per m3 of PERMEATE p.2 of 3

3 AMORTISATION

Total plantprice	750.000	NLG
Cost of membranes = operating cost, see item 6	250.000	NLG
Cost of equipment to be amortized	500.000	NLG
Hardware life time	10	years
Interst rate	7,0	%
Yearly cost	71.189	NLG
Yearly production	307.476	m3
COST OF AMORTISATION PER m3 OF PERMEATE	**0,232**	**NLG**

4 FEEDWATER and EFFLUENT

Plant capacity	39,0	m3/h
R.O. recovery	95	%
Feed flow rate	41,1	m3/h
Feed quantity per m3 permeate	1,1	m3/m3
Cost per m3 feed water	0,00	NLG
FEED COST PER m3 OF PERMEATE	0,000	NLG
Concentrate flow rate	2,1	m3/h
Concentrate quantity per m3 permeate	0,1	m3/m3
Cost per m3 effluent	0,00	NLG
CONCENTRATE COST PER m3 OF PERMEATE	0,000	NLG
COST OF FEED/CONCENTRATE PER m3 OF PERMEATE	**0,000**	**NLG**

5 ENERGY COST

Plant capacity	39,0	m3/h
R.O. recovery	95	%
Feed water flowrate	41,1	m3/h
System pressure	50	bar
Pump efficiency	65	%
Consumed electrical load HP Pump	86,1	kW
Energy consumption per m3 permeate	2,2	kWh/m3
Energy price per kWh	0,15	NLG
COST OF ENERGY PER m3 OF PERMEATE	**0,331**	**NLG**

6 MEMBRANE REPLACEMENT

Cost of membranes	250.000	NLG
Membrane lifetime	4	years
Yearly cost of membranes	62.500	NLG/year
Yearly production	307.476	m3
COST OF MEMBRANE REPLACEMENT PER m3 OF PER	**0,203**	**NLG**

7 SPARE PARTS

Cost of equipment,excl. membranes	500.000	NLG
Consumption as % of equipment	1	%
Yearly cost of spare parts	5.000	NLG/year
Yearly production	307.476	m3
COST OF SPARE PARTS PER m3 OF PERMEATE	**0,016**	**NLG**

SETEC COSTPRICE per m3 of PERMEATE

8 CHEMICALS

8.1 96% H2SO4

96% H2SO4 consumption	0	gr/m3 feed
Feed flow rate	41	m3/h
96% H2SO4 consumption	0,00	kg/h
Cost of 96% H2SO4	0,30	NLG/kg
Cost of 96% H2SO4	0,00	NLG/h
Plant capaciy	39,0	m3/h
COST OF 96% H2SO4	0,000	NLG/m3 permeate

8.2 SEQUESTRANT

Sequestrant consumption	0	gr/m3 feed
Feed flow rate	41	m3/h
Sequestrant consumption	0,00	kg/h
Cost of sequestrant	12,00	NLG/kg
Cost of sequestrant	0,00	NLG/h
Plant capaciy	39,0	m3/h
COST OF SEQUESTRANT	0,000	NLG/m3 permeate

8.3 HYPOCHLORITE

Hypochlorite consumption	0,0	gr/m3 permeate
Permeate flow rate	39,0	m3/h
Hypochlorite consumption	0,000	ltr/h
Cost of hypochlorite	6,00	NLG/ltr
Cost of hypochlorite	0,000	NLG/h
COST OF HYPOCHLORITE	0,000	NLG/m3 permeate

8.4 LIME

Lime consumption	0	gr/m3 permeate
Permeate flow rate	39,0	m3/h
Lime consumption	0,000	kg/h
Cost of Lime	0,30	NLG/kg
Cost of Lime	0,00	NLG/h
COST OF LIME	0,000	NLG/m3 permeate

8.5 CLEANING CHEMICALS

Cost cleaning chemicals	500	NLG/year
Yearly production	307.476	m3
COST OF CLEANING CHEMICALS	0,002	NLG/m3 permeate

TOTAL CHEMICAL COST PER m3 OF PERMEATE	**0,002**	**NLG**

9 MAINTENANCE

Operator attendance	0,1	h/day
Operator cost	40,00	NLG/day
Daily operator cost	4,00	NLG/day
Plant capacity	39,0	m3/h
Plant factor	90	%
Daily production at plantfactor	842	m3
COST PER m3 OF PERMEATE	**0,005**	**NLG**

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APPENDIX X PROJECT DESCRIPTION AND TIME SCHEDULE

Project Description (as in lit. 28)

The Preliminary Process Design ('Fabrieks Voor Ontwerp') is part of the curriculum of the 4th year Chemical Engineering at the Delft University of Technology. This design offers the possibility for the students to increase their skill in a characteristic aspect of the study: the design of an industrial process. It covers the design of a coherent system to produce desired chemicals with so called unit operations. The design results in a Process Flow Sheet, a mass balance, a heat balance and a cost and benefit overview.
The basis for this design lays in the knowledge of a number of relevant courses from the preceding years. The student uses the design to use this collected knowledge in an integrated, practical shape.

The design will be carried out in a team of 4 students, which is an excellent training for working as a team.

During the process of designing the students will be confronted with the following subjects:
Study of Literature: searching for the best process by making a correct combination of feedstocks and procucts with kinetics, reactor and processmethods to match. Also taking the market for raw materials and products into account.
Process Diagram: to design a logical combination of feed, reactor and separation section with the extent of process streams and conditions. To balance alternative combinations and make reasonable choices.
Balances and Process Flow Sheet calculations: mass and heat balances, process flow diagram
Equipment Calculations: including heat integration.
Process Evaluation: evaluation of safety, environment, economy.

The application of gradual method to work out the design is recommended. Start simple with a black-box presentation. The three pillars of the design are the process flow sheet, the mass and heat balances and the process streams overview. The number of detailed calculations is a compromise between a complete design and the available time.

The preliminary process design report has to be ready in three months after distribution of the assignment.

Time Schedule

Kick-off	Thursday,	October 2nd 1997	15:30
Informative meeting at Kemira	Friday,	October 3rd 1997	10:00
Presentation of review at Kemira	Thursday,	October 30th 1997	10:30
Progress meeting at Kemira	Monday,	December 1st 1997	13:30
Deadline report	Friday,	January 2nd 1998	17:00
Presentation of final report	Friday,	January 16th 1998	10:00
Review of final report (at univ.)	Tuesday,	February 17th 1998	9:30

APPENDIX XI JUSTIFICATION OF FIGURES

3.1	figure 13, p. 114, Gmelin (31)
3.4	figure 11.34, p11-32, Perry (17)
3.5	as figure 15.3, p 337, Arkenbout (16)
3.6	figure 4, p 10, van der Ham (1)
3.7	figure 5.3, p 109, Arkenbout (16)
3.9	figure 3, p 790, Kirk Othmer (29)
3.10	figure 13, p 815, Kirk Othmer (29)
3.11	figure 19-103, p 19-81, Perry (17)
4.1	as figure 15.3, p 337, Arkenbout (16)