18 juni, 9.30

Spare

FVO Nr. 3170

Fabrieksvoorontwerp
Vakgroep Chemische Procestechnologie

Onderwerp

The production of 2,4 dihydroxybenzophenone with zeolite H-beta catalyst

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Keywords

2,4-dihydroxybenzophenone, benzoic acid, crystallization, esterification, Fries-arrangement, resorcinol, zeolite H-beta.

Datum opdracht : 13-02-1996
Datum verslag : 30-05-1996

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The Production of 2,4-Dihydroxybenzophenone with Zeolite H-beta as Catalyst

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Project : FVO no. 3170
Period : 13 April-28 May 1996

Summary

The preliminary design of a process for production of 2,4 dihydroxybenzophenone (DHBP) with zeolite H-beta as catalyst is described in this report. Recently, DHBP is produced via $FeCL_3$ catalyst which is not environmental friendly.

This plant is designed to produce 1.324 kton/year with a production time of 8000 hours per year. The DHBP has a purity about 98%, using resorcinol (961.44 ton/year), benzoic acid (1066.32 ton/year), n-butylbenzene (0.7312 ton/year) and zeolite H-beta (6.6561 ton/year) as catalyst.
The advantage of this new process in comparison to the old process, is environmental friendly (no chloor compounds emmited). Regenerable catalyst has been used in the process, this is also consider as another advantage of this new process.

The total estimated investment costs are 24.4M Hfl. If the DHBP price is taken as 227.2 kHfl/ton an annual profit of 342.24 MHfl can be achieved. Pay Out Time (POT) of the plant is 1.8 years. Over a period of 10 years the Return on Investment (ROI) is 10% and the Internal Rate of Return (IRR) is 50%.

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Appendices

1. Introduction

2,4-Dihydroxybenzophenone (DHBP) and its derivatives are used mainly as UV-light absorbent to prevent degradation and discolouring of synthetic materials. It is also applied in the cosmetic's industry.

Currently, DHBP has been produced from resorcinol, benzotrichloride and water with $FeCl_3$ as catalyst by Cytec, Rotterdam with production capacity of 1kton/year. Further, DHBP is used as an intermediate to produce 4-O-octyl-2-hydroxy-benzophenone, which is applied as an UV-absorbent. Because of aggressive catalysts used by this old process, care must be taken to prevent corrosion and therefore expensive apparatus material has to be used. This old process produces yearly high chloor contained waste.

By section *Organische Chemie and Katalyse* led by Prof. H van Bekkum has a new catalyst (*zeolite H-beta*) examined to eliminate the previous problems. Zeolite H-beta is an ion exchange catalyst from zeolite beta catalyst with Si/Al ratio of 14. The advantage of this catalyst that it can be regenerated and activated.

The reactants of this new process are resorcinol and benzoic acid and n-Butylbenzene used as solvent. This new synthesis is based on esterification reaction, followed by *Fries rearrangement*. Based on experimental data, it can be proven that zeolite H-beta is the best catalyst with only trace of byproducts detected.

The aim of this project is to design an industrial plant for the production of DHBP with a clean method with capacity of 1.324 kton/year and purity of approximately 98%. Reaction kinetics of this new reaction is not known yet, the unknown parameters are estimated based on experimental data. The thermodynamic information of compounds is estimated by UNIFAC group contribution method.

In chapter 3 of this report, the DHBP process structure is described and in chapter 4 the design and calculation of equipment with its parameters are given. Chapter 5 describes process control. Safety aspects are presented in chapter 6 while the process economics is given in chapter 7. The conclusions and recommendations can be found in chapter 8.

2. Starting Points

The designed process consists mainly of three sections (figure 2.1).

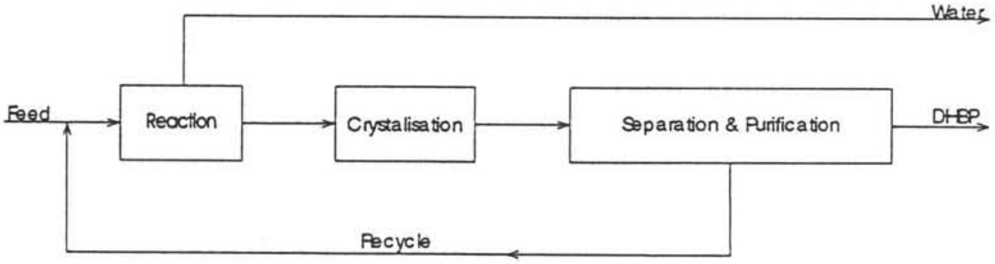


Figure 2.1: A schematic structure of the process

In the first section the reaction is carried out in packed bed reactor with a conversion of 57% of resorcinol after which DHBP is crystallized by cooling crystallization. In the third section the crystallized DHBP is separated from the reaction mixture and further purified.

In the first section following reaction takes place:

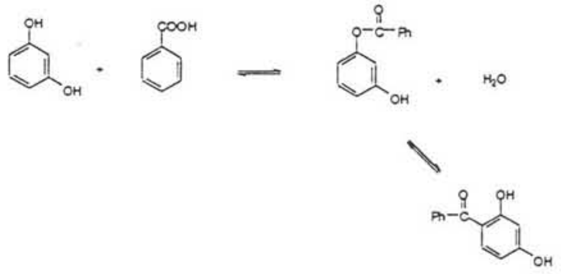


Figure 2.2: Reaction mechanism

The plant is located in Botlek, Rotterdam. The feed streams are commercial standard products, delivered at standard conditions (20° C and 1 bar) which are used without any purification. For crystallization section, sea water is assumed available as 15° C and can be emitted at 40° C. Cool water, used for other cooling purposes, is available at 20° C and also emitted at 40° C. Low pressure steam is available at 3 bar. The produced DHBP is delivered at 20° C, 1bar. Water which is formed during the reaction is cooled and emitted at 40° C.
The properties of the compounds which are present in the process can be found in appendix II.

3. Process Structure

3.1 Process description

The description of the process is based on the process flowsheet in appendix I. Temperature and process condition are also found in this scheme. The material properties used of this process are in appendix II. The component mass/energy balance and overall mass/energy balance are found in appendix III and appendix IV respectively. Equipment specifications are found in appendix V.

I. Reaction/crystallization section

The recycled stream (stream 4; 3601.47 kg/h) from purification section and fresh feed (120.18 kg/h resorcinol, 133.29 kg/h benzoic acid and 0.0914 kg/h n-butylbenzene) are introduced to a continous stirred mixer [M1] in order to dissolve resorcinol and bezoic acid which are in the solid form. Fresh feed containing benzoic acid are feeded to the mixer. The total massa is 3855 kg/h with almost 85 wt% of n-butylbenzene.

This mixer is warmed with steam up to 60° C to increase the solubility of the reactants. The outlet stream (stream 5) was pumped to the heat exchanger [H3] in order to reach the reaction temperature (160 °C). Stream 7 was introduced to the one of the packed bed reactor [R4/R5] which is equiped with heaters with temperature controller (TC) to maintain a constant temperature. The heaters are cilinder tube s with a diameter of 25 mm which are arrange across the cross sectional area of the reactor.

Water produced by this reaction is constantly removed via the top of the reactor (stream 8; 90.608 kg/h) and cooled to 40° C by a condensor placed at the top of the reactor [H6]. The residence time in each reactor is 3 hours.

The conversion of the reaction is assumed to be approximately 50-60 %. Outlet stream (stream 10) from the reactor which contains 223.3 kg/h DHBP liquid and 95.702 kg/h RMB is cooled [H8] to 60° C and feeded to the crystallizer [K9]. Supersaturation of the solution in the crytallizer is achieved by cooling. The cooling water enters the crystallizer at 15° C and leaves it at 40° C. To control the cooling water needed, a temperature and flow controller is coupled with the cool water valve.

II. Separation and purification section

After crystallization process, the suspension is introduced to the hydrocyclone [M11] to remove the particles from the mother liquoir. Stream 16 (contain almost 90 wt% n-butylbenzene) is mixed with stream 14 from the L/V separator and recycled to the mixer. Stream 17 (1317.56 kg/h) is feeded to the horizontal belt-filter [W12] and washed with water (stream 20, 666.534 kg/h) and pentane (stream 21, 379.305 kg/h). Water and pentane are delivered at 2 bar and sucked to the bottom of the filter and received in a vessel [V13/V14] before pumped to the L/V separator. Nitrogen is feeded to the belt filter in order to remove the vaporized pentane which is flammable.

The L/V separator is heated with steam in order to vaporize the liquid. Water is vaporized (stream 33) from L/V separator [M25] than cooled with heat exchanger [H17] to 25° C and received in a vessel tank. Pentane is also treated in the same way. The temperature in M20 and M25 is 40° C (above the boiling point of pentane) and 100° C (above the boiling point of water) respectively. The liquid from M20 and M25 are mixed and pumped back to the mixer (stream 14; total:1154.63 kg/h).

Wet product from horizontal belt filter (stream 26; 167.617 kg/h) is transported with screw conveyor [T27] to pneumatic dryer. Nitrogen of 1 bar (20° C) is compressed [C28] to 2.68 bar (142.803° C) and feeded to the pneumatic dryer to dry the product. The stack gas (stream 44) is recommended to be treated with active coal before leaves the stack (not handled in this project). The product (165.6 kg/h) which leaves the cyclone (stream 45) is cooled to 20° C with solid cooler and has a purity of approximately 98% .

3.2 Thermodynamics

There is no thermodynamical data available in the literature. The reaction is endothermal (based on the information from lit. 12).
The reaction enthalpy is calculated by Chemcad.

The process is simulated with Chemcad. The compounds RMB and DHBP was not available in the compound list and therefore had to be self defined using Unifac groups.

Since there is no thermodynamical model parameters available of these compounds, the enthalpy and K-values are calculated with Unifac group contribution method.

4. Design/Calculation of the Equipments

4.1 Mixer

The mixer is needed to prepare solution of n-Butylbenzene with the reactants which is transported to the reactor. This liquid and solids mixing operation is carried out as a continuous process. The most suitable agitator for a particular application will depend on the type of mixing required, the capacity of the vessel, and the fluid properties, mainly the viscosity. The type of the agitator from the mixer M1 is a turbine impeller with 1150 rpm due to low viscosity of the liquid n-Butylbenzene i.e. 0.7917 10^{-3} Ns/m^2.

The volume of the tank can be estimated from figure 10.57 (lit. 6) which has a value of 9 m^3 and assuming that tank diameter (D_t)≈1/3 tank height (H) give D_t=1.56 m and H=4.68 m.
The power number (N_p) can be estimated from figure 10.58 from lit. 6 with Reynold number define as :

$$Re = \frac{D^2 N \rho}{\mu} \tag{4.1}$$

where:
ρ = fluid density [kg/m^3]
μ = fluid viscosity [Ns/m^2]
N = agitator speed [s^{-1} (rps)]
D = agitator diameter [m]

Take the ratio of the impeller diameter (D) and the tank diameter (D_t) as 1/3, give the power number (N_p) of 0.25. The agitator power consumption (P) can be calculated by:

$$P = N_p\, D^5\, N^3\, \rho \tag{4.2}$$

where:
P = power consumption [W]
N_p = power number [-]

Table A4 in appendix V give the details of the mixer apparatus.

4.2 Reactor

Introduction

To produce 1.325 kton each year of 2,4-dihydroxybenzophenone (DHBP) a packed bed reactor filled with catalyst zeolite H-beta has been designed. The reactor is equipped with a condenser at the top to remove water from the reactor. The catalyst is delivered as pellets form (d=1.6 mm) by PQ Zeolites B.V, Leiden, the Netherlands. After four residence times used, the catalyst must be regenerated. If the left column used for the reaction, the right column is being regenerated. The life time of the catalyst is estimated as four years.

This process is relatively new, therefore most data are taken from laboratory result (i.e.: conversion and reaction equilibria) and simulated with CHEMCAD program (i.e.: heat of reaction). Due to unknown information of catalyst regeneration, the regeneration process is not handled in this project. It can be recommended to regenerate with high temperature nitrogen gas.

Reaction mechanism and kinetics

DHBP was produced from reaction of resorcinol and benzoic acid with resorcinol monobenzoate (RMB) and water as intermediary product.
It can be assumed that there is no by product formed. The reaction occurred at 160° C.

Since there are not all parameters available of the reaction kinetic, reactor could not be simulated with equilibrium reactor and therefore, the simulation of the reactor is carried out with a stoichiometric reactor, with a conversion of 0.8. The following stoichiometric coefficients are assumed (lit. 12):

Resorcinol ---> -1
benzoic acid ---> -1
RMB ---> 0.3
Water ---> 1
DHBP ---> 0.7

The process is simulated approximately according to appendix I. The recycle streams are 'connected' and in order to achieve a convergence, a low accuracy of the iteration is chosen, namely 0.05. Therefore, the mass/heat balance was not correct. With a spreadsheet program the mass balance is improved..
The following equitions are used:

$$K_1 = \frac{[RMB][H_2O]}{[resorcinol][benzoic\ acid]} = 1.377$$

$$K_2 = \frac{[DHBP]}{[RMB]} = 2.333$$

These two equilibrium constants are calculated, based on the experimental data from lit. 12.
The mass balance calculated by spreadsheet program is used for this report, while heat balance is taken from Chemcad.

Reactor dimensions

Typical fixed bed reactor has porosity between 0.3 and 0.4. The chosen porosity is 0.4. With a feed mass debit of 3855 kg/h and assumed that almost n-Butylbenzene present in the feed stream (ρ_{nbb}=860 kg/m3), the volume debit will be 3855/860 = **4.483 m^3/h.** With reaction time of 3 hours , the volume of the fixed bed reactor filled with catalyst will be : 4.483*3 = **13.447 m^3**.
The volume of the catalyst needed will be 13.447*(1-ϵ)=13.447*0.6=**8.068 m^3**.

The ratio between diameter and height of the column has been chosen as $D \approx 1/3$ H. The height of the reactor filled with catalyst can be calculated by $13.447=1/4*\pi*(1/3\ H)^2*H$, give $H_{fixed\ bed,total}$ =5.4 m . The diameter of the reactor will be $H_{fixed\ bed,total}/3$= 5.4/3 → **D=1.8m** .

The height of the reactor filled with catalyst is divided in three section, 1.8 m for each section. The void space in order to place heaters is chosen as 0.5 m each. The void space between the in let/outlet and the catalyst is estimated as 0.2 m each. The total height of the reactor will be (1.8*3)+(0.5*2)+(0.2*2) → **H=6.8 m.**

Further detailed reactor design has been presented in table A5 in appendix V.

Pressure drop

The pressure drop has been calculated due to method describe by Coulson & Richardson (lit. 5, Figure 4.1, Carman's graph). Further detailed design and calculation can bee seen in table A6 (appendix V) and appendix VIII respectively.

Reactor heater

Amount heat added due to endothermic reaction has been calculated with CHEMCAD which has the value of 63.33 kW.
This amount of heat has been added by Cupro-nickel (10 per cent Ni) tube with 25 mm inside diameter in order to maintain a constant temperature of the reactor, i.e. 160 °C. One unit has to add about 63.33 kW/2=31.665 kW of heat. Due to unknown information about the reaction mechanism and the temperature profile, almost all values (i.e. overall heat transfer coefficient and average steam temperature) are estimated. Each heater was controlled by a temperature control (TC controller).
Total area needed of this heat exchanger has given by:

$$A = \frac{Q}{U(T_{s,av} - T_r)} \tag{4.3}$$

with:

A	= total heat transfer area	[m^2]
Q	= heat transferred per unit time	[W]
U	= the overall heat transfer coefficient	[W/ m^2 °C]
Tr	= isotherm reactor temperature	[160 °C]
$T_{s,av}$	= average steam temperature = $(T_{steam\ in} - T_{steam\ out})/2$ = (190+165)/2 =177.5° C	

The average steam temperature ($T_{s,av}$) has been defined as the average of the steam inlet temperature (190° C) and the outlet temperature (estimated as 165° C). The overall heat transfer coefficient (U) can be estimated from table 12.1 Coulson & Richardson (heaters: Steam , process fluid: organic solvent) and has a value of 750 W/m^2 ° C. Details calculation of this heater is found in appendix VIII.

The tubes in this heater are arranged across the cross sectional area of the reactor (5 tubes each section, total length :6.8 meter, see design scheme). Details results of each heater and design scheme are presented in table A7 in appendix V.

4.3 Crystallizer

In the crystallizer takes the separation of DHBP from the reaction mixture place as well as its simultaneous purification.

The advantage of crystallisation compared to other separation techniques like distillation, adsorption and extraction is that it can be applied at low temperatures and without an auxiliary solid (adsorbent) or liquid (extract) phase. Furthermore, during growth from solution one crystallization step often suffices to reach a high purity product since crystallisation is a selective technique with respect to the incorporation of foreign ions, atoms or molecules into the crystalline solid.

A drawback is the low rate in which the crystals increase their dimensions, implying a low yield rate. Growth rates of well-soluble compounds are typically between 10^{-8} and 10^{-7} m/s.

After crystallisation process, further downstream product handling is required to remove the mother liquor from the particles. Separation units can usually handle particles ranging in size from 200 to 1000μm. To reach these sizes generally a residence time of crystals in the crystallizer of 0.5 to 5 hours is needed. The crystal size distribution also affects the washability and drying of the crystals and also the storage (high density) and transport (non-caking) behaviour of the product.

Selection of crystallisation method:
For most organic substances, increasing in temperature leads to high solubilities so that supersaturation of the solution increase by decreasing the temperature.
Therefore, the cooling crystallisation method is chosen to separate DHBP from the reaction mixture. In particular a cooling disc crystallizer (figure 4.1) may be chosen for this purpose which can be considered as a cascade of cooling crystallizers. The cooling disc crystallizer consists of a trough containing several hallow discs, through which a cooling liquid is circulated. The disc build up a set of compartments containing part of suspension that enabled to flow from one compartment to the next through a segmental opening present in each disc. The suspension is countercurrent to the coolant. A gentle agitation is provided by slow rotation of the disc.

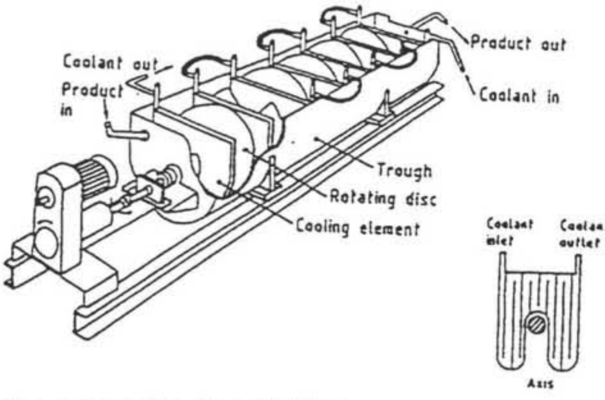


Figure 4.1: Cooling disc crystallizer

Calculation of material and energy flows:

The crystallizer could not be simulated by ChemCad. Since there is no solubility data available of DHBP the material balance over the crystallizer is based on the experimental results (lit.12) in which is given that 72.72% of DHBP is crystallized by cooling to 20° C. Assuming that a small amount of RMB (3.5%) also crystallize, following results are calculated based on 3764.342 kg/h feed:

Table 4.1: Material balance over the crystallizer

Stream	DHBP (l)	DHBP(s)	RMB (l)	RMB(s)	Rest	Total
Feed [kg/h]	223.3	0	95.702	0	3445.34	3764.342
Product [kg/h]	60.901	162.4	92.35	3.3496	3445.34	3764.342

The energy withdrawn per unit mass of crystals, Q_{cool}, given by:

$$-Q_{cool} = \frac{\rho_o}{c}\, C_p\, \Delta T + \Delta H_{crys} \qquad (4.4)$$

where:

ρ_o = outlet density of the slurry [$890.64 \mathrm{kg_{slurry}/m^3_{slurry}}$]
c = solute concentration of the solution in the crystallizer [$62.907\ \mathrm{kg_{solute}/m^3_{slurry}}$]
C_p = specific heat of the mother liquor [$1.8463\ \mathrm{kJ/kg_{slurry}K}$]
ΔT = temperature difference between inlet feed and slurry [40 K]
ΔH_{crys} = enthalpy of crystallisation [$60.454 \mathrm{kJ/kg_{crys}}$]

Approximate values for the specific heat capacity of the components is calculated using Chueh-Swenson method (lit. 6) at 20° C. Since liquid specific heats do not vary much with temperature (at temperatures below the critical temperatures), the specific heat of the liquid mixture at 60° C is calculated by taking heat capacities of the components as additive.

The enthalpy of crystallisation of DHBP has been taken approximately as the enthalpy of fusion, estimated by (lit. 7):

$$\Delta H_f = \frac{R}{2}\, T_m^{4/3} \qquad (4.5)$$

where R = gas constant [8.314 J/molK]
T_m = melting point of DHBP [417.15K

Consequently, total energy withdrawn per unit mass crystals:
$Q_{cool} = 1106.0519 \mathrm{kJ/kg_{crys}} \times 190.5034/3600\ \mathrm{kg_{crys}/s} = 58.5296$ kW.

The heat transfer area can be calculated with:
$Q_{cool} = U \cdot A \cdot \Delta T_{lm}$

where: U = overall heat transfer coefficient [$0.5\ \mathrm{kW/m^2}$]
A = heat transfer area [$\mathrm{m^2}$]
ΔT_{lm} = logarithmic mean temperature difference [K]

Assuming countercurrent flow, $\Delta T_1 = 333 - 313 = 20$ K

$\Delta T_2 = 293 - 288 = 5\ K$

and logarithmic mean, $\Delta T_{lm} = (20-5) / \ln(20/5) = 10.82\ K$.

The heat transfer area required is then:
$A = Q_{cool} / U\ \Delta T_{lm} = 58.5296 / (0.5\times 10.82) = 10.82\ m^2$.

The maximum cooling energy available per unit heat transfer area:
$P_{cool,max} = Q_{cool} / A = 5.41\ kW/m^2$

For the cooling disc crystallizer $P_{cool,max} = 0.5 - 1.5\ kW/m^2$. Therefore, the required heat transfer area must be minimum $39\ m^2$.

Crystallizer volume
The surface area and the volume of the cooling disc crystallizer is:

$A = 2 \cdot N \cdot \pi/4 \cdot D^2$ and $V = \pi/4 \cdot D^2 \cdot \Delta L \cdot N$
where in general, $A/V = 2 / \Delta L \approx 4\ m^{-1}$

with D = diameter of crystallizer [m]
N = number of discs [-]
ΔL = distance between two discs [-]

The volume of the crystallizer is: $39 / 4 = 9.75\ m^3$.

The average residence time:

The average residence time necessary to produce the desired CSD is determined by:

$$\Gamma = \frac{L_{av}}{G} \tag{4.5}$$

where:
L_{av} = average particle diameter [m]
G = growth rate [m/s]

To produce crystals of an average particle diameter of $500\mu m$ with a growth rate of 10^{-8} m/s the average residence time will be 5000 seconde or 1.389 hour.

4.4 Hydrocylone

Hydrocylones are used for solid-liquid separation with particle diameter range from 4 to 500 µm. Design method of hydrocylone are estimated by nomogram given from Zanker (lit. 22).

The value of d_{50} was assumed approximately 30% lower from the particle size of each hydrocylone. Table A8 in appendix V give the design parameter of such hydrocylone.

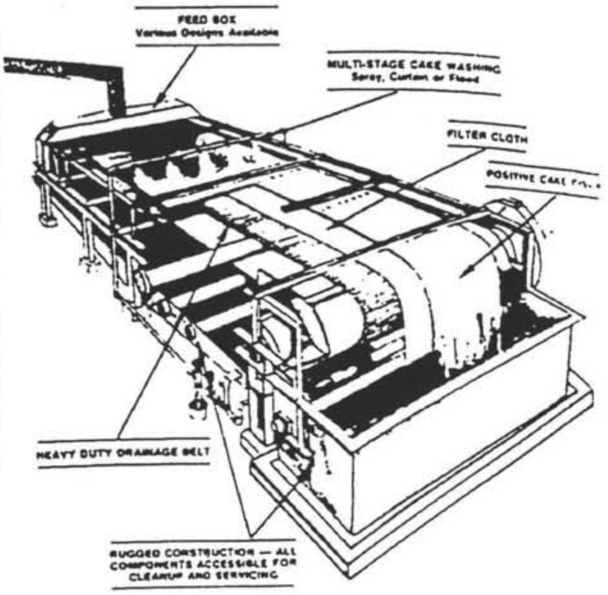
4.5 Washer and filter

Horizontal-belt filter was used to wash the product. Water and pentane are used for this washing process and sprayed on the top of the belt filter. These components were sucked to the bottom of this filter.

The residence time was estimated about 15 minute for each washing section. The total area used for this washing process is 8 m^2 (2m by 2m for each washing section, see figure 4.2). Typical horizontal-belt filter is produced by *Eimco Process Eqiupment & co.*

The belt velocity will be 2m/15 minute = **0.13 m/minute.** Area use for this process : 0.13 m/minute × 2 m = **0.26 m^2/minute** The slurry feed is **2.553.10^{-2} m^3/min** and introduced at the beginning of the filter belt. The cake thickness will be 2.553.10^{-2} m^3/min /0.26 m^2/minute = **9.82 mm.**

It is difficult to calculate pressure drop over the filter without experiment data. The theory to calculate the pressure drop across the filter is taken from 'Particle Technology I' lecture from TU Delft (Scarlett et. all). By used of Ruth equation :



$$\frac{\eta\alpha f}{\Delta P}\frac{Q^2}{2}+\frac{\eta R}{\Delta P}Q=t \qquad (4.6)$$

Figure 4.2: Horizontal belt filter

with:

$$\alpha = \frac{1}{K\ (1-\varepsilon)\ \rho_s} \qquad (4.7)$$

and

$$f=\frac{\rho_1}{(1-Ss)/Ss-(1-Sc)/Sc} \qquad (4.8)$$

which:

α = specific cake resistance for unit area of filter per unit weight of cake
K = permeability
ϵ = voidage [-]
ρ_s = solid density [kg/m^3]
ρ_l = liquid density [kg/m^3]
R = filter medium resistance
Sc = mass fraction of solids in cake [-]
Ss = mass fraction of solids in suspension [-]
m = mass solid per unit area [kg/m^2]

Plotting t/Q as function of Q give $0.5 * \eta \alpha f/\Delta P$ en $\eta R/\Delta P$ as slope and intercept respectively. By knowing the α,f and the viscosity of the filtrate, the pressure drop can be determined.

4.6 Dryer

Pneumatic dryer, which has almost the same principal as fluidized bed, has been used to dry the product. The solid is introduced into the dryer by some form of mechanical feeder (screw conveyor).

Typical installation is illustrated in figure 4.3. The wet feed (debit=167.62 kg/h) is delivered in a double paddle mixer and mixed with the previously dried material. It than passes to a cage mill where it comes into contact with hot nitrogen gas (temperature=142.803 °C and debit=1500 kg/h) from the compressor. Moisture components are immediately evaporated.

The stream of particles leaves the dryer through a cyclone separator and the hot gasses pass out of the stream. In this unit the time of contact of particles with the hot gas is small (typically 5 seconds).

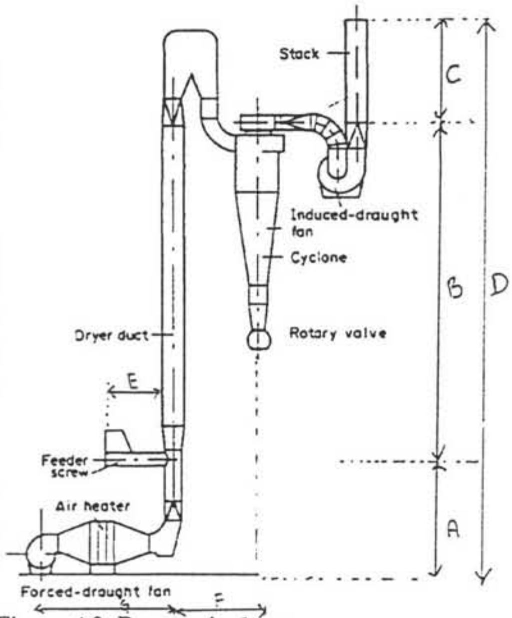


Figure 4.3: Pneumatic dryer

Pneumatic dryer specification needed for DHBP production can be found in *Perry Chemical Engineering Handbook* (lit. 17). Typical apparatus with capacity up to 2 tons/hour is delivered by Combustion Engineering Inc. Details data and design specification will be presented in table 4.2.

Table 4.2: Pneumatic dryer summaries

Evaporation capacity [kg/h]	A [m]	B [m]	C [m]	D [m]	E [m]	F [m]	G [m]	Price [$]
2000	3.353	7.315	3.139	13.807	4.053	6.400	4.815	190

4.7 L/V Separator

The L/V separator M20 and M25 are used to separate the solvents from liquid phase. The heat of separation is calculated using CHEMCAD. The separation has been done by increasing temperature of mixtures from 25 °C to 40 °C and 100° C for pentane and water respectively. In case of pentane, vapour phase will leave at the top of separator M20 and liquid phase is recycled via the bottom of the vessel. The same way has been done by separator M25 . The recovery of pentane and water are 99.39wt% and 99.65wt% respectively. 99 wt% of other compounds can be recycled.
The sizing of separating vessels can be estimated with the following equation:

$$\hat{u}_v = 0.035\sqrt{\frac{\rho_L}{\rho_v}} \tag{4.9}$$

where: $\hat{u}_v$	= maximum design vapour velocity	[m/s]
ρ_L	= liquid density	[kg/m^3]
ρ_v	= vapour density	[kg/m^3]

The disengagement height equal to the vessel diameter should be provided above the liquid level. The liquid level depends on the hold-up time. A typical value for the hold-up time is 10 minutes. The parameters of the vessels are given in table A9 (appendix V).

4.8 Heat transfer equipments

Heat transfer is to predict the energy transfer between materials of different temperatures. A heater is designed and built based on heat transfer principles.

The function of heater H3 is to heat the feed stream before into the reactor from 60 °C (T_1) to 160 °C (T_2). The incoming medium (tube side) is LP steam of 3 bar at 190 °C (t_1) and the output steam has a temperature of 160 °C (t_2). The unit H3 consists one shell side pass and two tube side passes. The basic heat-transfer equation can be written as :

$$Q = U * A * \Delta T_m \tag{4.10}$$

and

$$\Delta T_m = F_t * \Delta T_{lm} \tag{4.11}$$

(for heat exchangers and condensers)

where	Q	= the heat transferred per unit time	[W]
	U	= the overall heat transfer coefficient	[W/m^{2}°C]
	A	= heat transfer area	[m^2]
	ΔT_m	= the mean temperature difference and is expressed in terms of the counter flow heat exchanger	
	F_t	= the temperature correction factor	[-]
	ΔT_{lm}	= logarithmic mean temperature difference	[°C]

The logarithmic mean temperature is given by:

$$\Delta T_{lm} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln \frac{(T_1 - t_2)}{(T_2 - t_1)}} \tag{4.12}$$

where T_1 = inlet shellside fluid temperature

T_2 = outlet shellside fluid temperature

t_1 = inlet tubeside fluid temperature

t_2 = outlet tubeside fluid temperature

The overall heat transfer coefficient can be estimate as :

$$\frac{1}{U} = \frac{1}{h_{od}} + \frac{d_o}{d_i*h_{id}} + \frac{d_o*\ln\frac{d_o}{d_i}}{2*k_w} + \frac{1}{h_o} + \frac{d_o}{d_i*h_i} \qquad (4.13)$$

with: h_{od} = outside dirt coefficient [W/m^2 °C]
h_o = outside fluid film coefficient [W/m^2 °C]
h_i = inside fluid film coefficient [W/m^2 °C]
h_{id} = inside dirt coefficient [W/m^2 °C]
d_i = inside diameter [mm]
d_o = outside diameter [mm]
k_w = thermal conductivity of the tube wall material=45 [W/m^2 °C]

From the equation (4.10) the heat transfer area is calculated. This value divided by the area of one tube (tubelength is 1 and diameter d_o are chosen) is the total number of tubes N_t. The tubes in this heat exchanger are arranged in an triangle and square pattern. The bundle diameter D_b of the tube sheet is given as the following equation:

$$D_b = d_o\left(\frac{N_t}{K_1}\right)^{\frac{1}{n_1}} \qquad (4.14)$$

where D_b = bundle diameter [mm]
d_o = tube outside diameter [mm]
N_t = number of tubes [-]
K_1, n_1 = constants, and depend on the pitch and the number of the tube passes (see table 12.4, lit. 6).

The estimated U value is then recalculated with the theory, described in Chemical Engineering, vol.6, Coulson & Richardson (lit. 6).

4.9 Pumps, screw conveyor and compressors

4.9.1 Pumps

In the production of DHBP centrifugal pumps are used for pumping process fluids. The power required for pumping and incompressible fluid is given by:

$$P_p = \frac{\Delta P * Q_p}{\eta_p} \times 100 \qquad (4.15)$$

where P = power required [W]
ΔP = pressure differential across the pump [N/m^2]
Q_p = flow rate [m^3/s]
η_p = pump efficiency [%]

The efficiency of centrifugal pumps depends on the flow rate. The values from Fig. 10.62 of Coulson & Richardson can hereby be used. Resume of the calculated power are presented in table A10 (appendix V).

4.9.2 Screw conveyor

Transportation of solids is usually more expensive than the movement of liquids and gases, which can be easily pumped down a pipeline. In case of the DHBP processing, screw conveyor was used. This consists helical screw rotating in a U-shaped trough. They can horizontally or, with some loss of capacity, at an incline to lift materials. Due to friction between the solids ,the flight of the screw and the trough, screw conveyors are less effective than belt conveyors , but are cheaper and easier to maintain. The capacities of this type conveyor are generally limited to around 4.72 m^3/min .

Screw conveyor specification needed for DHBP production can be found in *Perry Chemical Engineering Handbook.* Typical screw conveyor with capacity up to 5 tons/hour is delivered by Fairfield Engineering and Co. Details data and specification will be presented in table 4.3

Table 4.3: Screw conveyor summaries

Capacity [ton/h]	Diameter of the pipe [m]	Approx. area occupied by material	Speed [rpm]	Max length [m]	Power [kW]
5	0.3	31%	40	4.5	0.32

4.9.3 Compressors

The work produced (or required) by polytrophic compression W_{pol} of is given by:

$$-W_{pol} = Z \frac{R\,T_i}{M} \frac{n}{n-1} \left[\left(\frac{P_2}{P_1} \right)^{\frac{n-1}{n}} - 1 \right] \qquad (4.16)$$

with

$$n = \frac{1}{1-m}$$

$$m = \frac{\left(\frac{C_p}{C_v}\right)_{id.gas} - 1}{\left(\frac{C_p}{C_v}\right)_{id.gas} * \eta_{pol}}$$

where Z = compressibility factor (1 for an ideal gas) [-]
R = universal gas constant=8.314 [J/mol/K]
T_i = inlet temperature [K]
P1 &P2 = resp. the inlet and the outlet pressure [bar]
Mw = molecular mass (weight) of gas [g/mol]
$(C_p/C_v)_{id.gas}$= specific heat at constant pressure/ specific heat at constant volume=1.4,
η_{pol} = polytrophic efficiency. [-]

The polytrophic efficiency depends on the volumetric flow rate. The values in fig. 3.6. of Coulson & Richardson vol. 6 can hereby be used. The polytrophic work divided by the polytrophic efficiency gives the actual work that is required. The summaries of the designed pumps and compressors are given in table 4.4

$$W_{actual} = \frac{W_{pol}}{\eta_{pol}} \qquad (4.17)$$

Table 4.4: Compressor summaries

Φm [kg/s]	Φv [m^3/s]	η_{pol} [-]	m [-]	n [-]	Ti [K]	P1 [Bar]	P2 [Bar]	Mw [g/mol]	W_{pol} [kW]	W_{act} [kW]
0.417	0.362	0.65	0.439	0.784	298	1	2.68	28	152.54	234.61

4.10 Liquid storage tanks

Liquids are stored in bulk horizontal cylindrical steel tanks and rectangular tanks, usually for relatively small quantities. There are two kinds of tanks used namely, fixed and floating-roof tanks. Floating-roof tanks are used to eliminate evaporation losses and, for flammable liquids, to obviate the need for inert gas blanketing to prevent an explosive mixture forming above the liquid, as would be the situation with a fixed-roof tanks.

There are four liquid storage tanks which are two for water (V13/V23) and the others for pentane (V14/V18). Water and pentane have a function as liquid washer. The storage of pentane have a special attention because of high flammability of this compound.

The minimum wall thickness required to resist the hydrostatic pressure can be calculated from the equation for the membrane stresses in thin cylinders:

$$e_s = \frac{\rho_L * H_L * g}{2 * f_t * J} * \frac{D_t}{10^3} \qquad (4.18)$$

where e_s = tank thickness required at depth H_L [mm]
H_L = liquid dept [m]
ρ_L = liquid density [kg/m^3]
J = joint factor [-]
g = gravitational acceleration=9.81 [m/s^2]
f_t = design stress for tank material [N/mm^2]
D_t = tank diameter [m]

Thin-walled vessels under internal pressure

For a cylindrical shell the minimum thickness to resist internal pressure can be determined from the following equation:

$$e = \frac{P_i * D_i}{2 * f P_i} \qquad (4.19)$$

The equation for a spherical shell is given as:

$$e = \frac{P_i * D_i}{4*f - 1.2*P_i} \qquad (4.20)$$

where e = the minimum thickness required [mm]
P_i = internal pressure [N/mm^2]
D_i = internal diameter [m]
f = design stress [N/mm^2]

Minimum practical wall thickness

There will be a minimum wall thickness required to ensure that any vessel is sufficiently rigid to withstand its own weight, and any incidental loads. As a general guide the wall thickness of any vessel should not be less than the values given below; the values include a corrosion allowance of 2 mm:

Table 4.5: Values of wall thickness

Vessel diameter [m]	Minimum thickness [mm]
1	5
1 to 2	7
2 to 2.5	9
2.5 to 3.0	10
3.0 to 3.5	12

Summaries of tanks parameters can be found in table A11 (appendix V).

5. Process control

Mixer

Temperature control is used to maintain the mixer temperature at 60° C. It is coupled with steam inlet. Level controller is connected to the pump to avoid pumping problems.

Recycle

The flow control for the recycle is used to maintain sufficient solvent quantity in the mixer. This is needed to dissolve resorcinol and benzoic acid (fresh feed).

Reactor

Due to endothermic reaction, intern heaters are placed inside the reactors. Temperature control provides a constant reactor temperature at 160° C.

Crystallizer

Flow controller is used to prevent fluctuations in the cooling water stream. A slow cooling is crucial to produce large crystals, because increasing in the cooling rate cause rapid nucleation and small size of crystals. Liquid controller is also placed to control the liquid level of the crystallizer and the pump outlet.

Vessels

Vessels V13, V14, V17 and V23 provide for intermediate storage of the liquid. The level controller avoids dry up or overflow problems of the vessels.

Evaporators

Evaporator M20 and M25 provide the evaporation of pentane and water respectively. Temperature control is used to maintain an accurate evaporation temperature. A constant liquid level in the evaporator is controlled by level controller.

6. Process Safety

The potential fire and explosion hazards of the process are evaluated by calculating the Dow Fire and Explosion Index for the main process units. The index is based on the nature of the process and the properties of the materials.

1. Hazard and Operability Study (HAZOP)

The HAZOP is a qualitative procedure to systematically check the effects of deviations from intended operating conditions (Appendix VI).

Table 6.1: MAC-values and explosion limits (lit. 4).

Compound	MAC-value [ppm, mg/m^2]	Explosion limit [vol. % in air]
Resorcinol	10, 45	1.4-?
Benzoic acid	-	-
Resorcinolmonobenzoate	-	-
2,4-Dihydroxybenzophenone	-	-
n-Butylbenzene	10, 30	0.8 - 5.8
Pentane	600, 1800	1.4 - 8

It is recommended to place everywhere on the plant terrain special sensors to ensure that any leakage in the apparatures or piping can be detected directly. Also a extensive sprinkler installation and good fire air should be available because of the low explosion limit of all compounds.

2. Safety, Health and Environment

The safety of a plant is not only dependence on the apparatus but also the properties of compounds . In the production of DHBP, there are flammable and combustible material used, namely resorcinol, benzoic acid, n-butylbenzene, and pentane. Based on this information, hazardious properties can be determined.

A hazard identification method has been developed by The American National Fire Protection Agency (NFPA). This method gives an evaluating of potential risk from a process, and assessing the potential loss. The NFPA method assigns five "rating" (numbers) to materials. These ratings, which range from 0 (no hazard) to 4 (maximum hazard), are based on Flammability, Reactivity (related to explosiveness), and Health (related to toxicity). The calculation of "Fire and Excplosion Index" (F&EI) is based on the nature of the process and the properties of the materials used. Application of this method for the reactants and products gives the following results.

Table 6.2: NFPA ratings for the reactants and products.

Compound	Flammability (N_f)	Reactivity (Nr)	Health (N_h)
Resorcinol	1	0	0
Benzoic acid	1	0	2
Resorcinolmonobenzoate	1	-	-
2,4-Dihydroxybenzophenone	-	-	-
n-Butylbenzene	1	-	-
Nitrogen	0	0	1
Pentane	4	0	1
Water	0	0	0

The reactants and wash solutions are flammable, so the use of open fire and smoking in the plant are strictly forbidden. To ensure safe operation if flammability limits and ignition temperatures are simultaneously reached, standard procedures should additionally used.

7. Process Economics

All calculations are listed in Appendix VII.

7.1 Production costs

The production costs consist of direct and indirect costs. The direct costs are the sum of production dependant costs, semi-variable costs and investments costs. Indirect costs consist of overhead, terrain, safety etc. The model for the calculation of the production costs is:

$$K_T = a\ K_p + d\ K_L + f\ K_I \qquad (7.1)$$

in which:

K_T = total costs [MHfl/yr]
K_P = production volume dependent costs [MHfl/yr]
K_L = semi-variable costs [MHfl/yr]
K_I = investment dependent costs [MHfl/yr]

In this model, a is a factor to rectify for the production dependent costs that have not been taken into consideration. The factor d can be considered to be a constant as it varies with every venture and product. The factor f is called capital charge and its magnitude depends on depreciation and maintenance.
The values of a, d and f are given by "beste model" in table II-12 [Montfoort, 1991].

$$K_T = 1.13\ K_P + 2.6\ K_L + 0.13\ K_I \qquad (7.2)$$

The value for f (0.13) does not take into account the interest and depreciation over the years. Therefore a factor, called as capital charge, has to be added, obtained from table IV-9 [Montfoort, 1991]. A project duration of 10 years and a rent of 8% gives capital charge as 14.9% and the factor f becomes 0.13+0.117 = 0.279.
Consequently, equation (7.2) becomes:

$$K_T = 1.13\ K_P + 2.6\ K_L + 0.279\ K_I \qquad (7.3)$$

7.1.1 Production volume dependent costs

The production volume dependent costs can be calculated as follows:

$$K_P = P \sum_{1}^{N} v_i\ q_i \qquad (7.4)$$

in which:

P = production capacity of the plant [ton/yr]
v_i = cost of base chemical or utility i per ton [MHfl/ton]
qi = amount of i needed per ton product [ton/ton]

The costs of base chemicals and utilities have been calculated in Appendix VII. The total production cost (K_P) of the process is MHfl 14.894 per year.

7.1.2 Labour costs

For a continuous process the labour costs can be calculated from Wessel model:

$$K_L = 32\ N\ C^{0.24} \tag{7.5}$$

in which

N = number of steps [-]

C = capacity of the plant [kton/yr]

The process consists of 6 steps: mixing, reaction, crystallization, product separation, wash & filter and drying. The capacity of the plant is 1.3248 kton/year. So the calculated K_L is:
$K_L = 32 \times 6 \times 1.3248^{0.24} = 205.41$ kHfl/year.

The calculated value of K_L has to be corrected to include the amount of function sites. The Wessel model is based on a standard function site cost in 1986 of 350 kHfl/function site. The number of function sites is equal to 205.41 / 350 = 0.587 and it is rounded up to 1. The corrected value of K_L becomes 350 kHfl/year.

7.1.3 Investment dependent costs

The total investments of a plant can be divided into four groups:

$$K_I = I_B + I_H + I_L + I_w \tag{7.6}$$

I_B	= investments in process units [MHfl]	(64% of total)
I_H	= investments in side apparatures [MHfl]	(16%of total)
I_L	= investments in overhead [MHfl]	(14% of total)
I_W	= investments in work capital [MHfl]	(6% of total)

There are several models to estimate the investments based on I_B. The methods of Zevnik-Buchanan-Jansen, Taylor and Wilson are used.

Zevnik- Buchanan- Jansen method

In this method the investments depend on the capacity and complexity of the different process units.

$$I_B = 0.1\ C_I \sum_{i=1}^{N} C_{fi}\ P_i^{m} \tag{7.7}$$

in which

C_I = C.E. plant costs index (= 380.08, Chemical Engineering, April,1995)

N = number of functional units [-]

C_{fi} = complexity factor of the functional unit [-]

P_i = unit capacity [kton/yr]

m = degression exponent (= 0.6)

The complexity factor can be found from:

$$C_f = 2 \times 10^{(F_t + F_p + F_m)} \tag{7.8}$$

Values for the temperature factor (F_t), the pressure factor (F_P) and the material factor (F_m) are found in Montfoort, 1991. The investment costs according to Zevnik-Buchanan-Jansen is calculated as k$ 314,5 (for results refer to AppendixVII, table A16).

Taylor's method

The Taylor's method is based on process steps, not on the number of the apparatures. The investments costs are determined by

$$I_B = 93\ f\,P^{\,0.39}\ \frac{C_{I,T}}{300} \tag{7.9}$$

where:

$$f = \sum_{1}^{N} (1.3)^{S_i} \tag{710}$$

in which:

f = costliness index [-]
N = number of process steps [-]
S_i = complexity score for step i [-]
P = plant capacity [kton/yr]
$C_{I,T}$ = EPE cost index [-]

The costliness index (f) gives the complexity of the process steps. It includes the influences of throughput, construction material, residence time, pressure and temperature. Values for S_i are found from Montfoort,1991 and are listed in Appendix VII, table A17. The investment costs according to Taylor have been calculated as k$ 7,985.

Wilson method

This method is on the number of all apparatures, excluded the pumps.

$$I_B = f\ N\ (AUC)\ F_p\ F_t\ F_m\ C_{I,T} / 115 \tag{7.11}$$

where:

$$AUC = 21 \times P^{0.675} \tag{7.12}$$

in which:

f = investment factor (= 1.7)
N = number of apparatures without pumps (= 21)
AUC = average unit cost (= 2690 £)
P = plant capacity (= 1324.8 ton/yr)
F_p = pressure factor (1)
F_m = material factor (1.01)
F_t = temperature factor (1)
$C_{I,T}$ = EPE cost index (873)

Values f, F_p, F_m, F_t are found in Montfoort, 1991. The investment costs according to Wilson calculated as k£ 736.3. Taking the rate of exchange as 2.475 (= £/Hfl, 24 may 1996), investment costs become MHfl 1.82.

For the three calculated values of I_B the exchange rates of 1.66 Hfl/$ and 2.475 Hfl/£ is taken (May, 1996). In table 7.1, the results of the different I_B's and the average value of I_B are listed.

Table 7.1: Values for the three different methods

Method	I_B	I_B **(MHfl)**
Zevnik-Buchanan-Jansen	k$ 314.5	0.52
Taylor	k$ 7,985	13.25
Wilson	k£ 736.3	1.82
Average		15.59

Using the average I_B the total investment costs become:
$K_I = 15.59 \times 100/64 = 24.4$ MHfl.

The total production costs is given by equation (7.3) and equal to MHfl 24.54 per year.

In table 7.2 income is given.

Table 7.2: Income

Product	Production rate (ton/yr)	Price* (kHfl/ton)	Income (MHfl/yr)
DHBP	1324.8	277.2	367.24

The profits can be calculated with: (Incomes - K_T) = 342.7 MHfl/yr. This value strongly depends on the costs of the DHBP, of which the commercial price is unknown. A value of 277.2 Hfl/kg is taken from FLUKA.

7.2 Economic Criteria

The purpose of economic criteria is to give a quic overview in order to decide whether to start a project or not. There are several ways to calculate the rendability in which different parameters are counting. Most usual is the ratio between the exploitation and the investments. Examples are the Return On Investments (ROI), Pay Out Time (POT) and Internal Rate of Return (IRR). Important is how to define exploitation. When is taken into account the method is dynamical, like the Internel Rate of Return (IRR), otherwise the method is static. The project time does not play a role within the static method, it does within the dynamical method.

7.2.1 Return on Investments (ROI)

This method is well suited for comparing process alternatives with equal life time and production patterns. Hence, this method is often used in the design stage.The return on investment is the ratio of the annual profit and the total investment [Montfoord, 1991]:

$$ROI = \frac{\overline{W}}{I_F + I_W} \qquad (7.13)$$

where W = annual profits = Incomes - K_T
K_T = total production costs (= MHfl 24.54 per year)
I_F = fixed capital investments
I_W = work-costs investments.

$I_F + I_W = 0.86\ K_I$ = MHfl 20.95.

For a plant with a working period of 10 years, the ROI should be minimum 10% [Montfoord, 1991]. The incomes is MHfl 26.635 per year. For 1324.8 ton production per year, the minimum product price of DHBP becomes Hfl 20,105 per ton.

7.2.2 Pay Out Time (POT)

The POT is defined as the number of years needed to pay back the original investments. The original investments are the depreciable capital investments. Interest and taxes are not considered; this makes the POT more a risk ratio, which gives information about how long the investments are risky and make it less a rentability ratio. The POT is expressed as follows:

$$POT = \frac{I_F}{exploitation\ surplus} \qquad (7.14)$$

I_F = $I_B + I_H = 0.8\ K_I$ (= MHfl 19.52).
exploitation surplus = incomes - K_P - 2.6 K_L (= 26.635 -14.894 -2.6×0.35 = MHfl 10.831 per year)

Avalue of 1.8 years is calculated. According to the rentability criterium, the lifetime of the project has to be at least tree times the POT, that is 5.4 years. With a lifetime of 10 years, this criterium is fulfilled.

7.2.3 Internal Rate of Return (IRR)

The IRR is a dynamic method in which the cash flows, including the investments, are evaluated over the lifetime of the project with a return percantage r resulting from the sum of the discounting cashflows to be zero. The basic idea is the investigation of the annual cash flow in projects with the same value of r. A value of 50% is calculated (Appendix VII).

8. Conclusions and Recommendations

The DHBP process is designed to produce 1.324 kton/year with a production time of 8000 hours per year. The DHBP has a purity of about 98%, produced by resorcinol (961.44 ton/year), benzoic acid (1066.32 ton/year), n-butylbenzene (0.7312 ton/year) and zeolite H-beta (6.6561 ton/year).
The advantage of this process in comparison to the old process, is environmental acceptance (no chloor compounds emitted). Regenerable catalyst has been used in the process. This is also considered as another advantage of this new process.

The calculated investment costs are MHfl 24.4. If the DHBP price is taken as 227.2 kHfl/ton an annual profit of MHfl 342.24 can be achieved. This value strongly depends on the costs of the DHBP, of which the commercial price is unknown. Pay Out Time (POT) of the plant is 1.8 years. Over a period of 10 years the Return on Investment (ROI) is 10% and the Internal Rate of Return (IRR) is 50%.

It is very difficult to design a process with unknown information about the reaction. All information needed was estimated or assumed from laboratory results. The authors have recommended the following points due to the further optimalisation of the process:

1. More laboratory research about esterification and Fries rearrangement, with H-beta as catalyst, to determine the reaction rate constant and order of the reaction
2. Looking for regenerating methods and more information about the catalyst (i.e. life time, activity, selectivity)
3. Choosing a better kind of reactor in which the reaction and regenerating of the catalyst can be carried out at the same apparatus.

9. List of symbols

A	= column cross sectional area	m^2
A	= heat transfer area	m^2
AUC	= average unit cost	£
c	= solute concentration of the solution in the crystallizer	kg_{solute}/m^3_{slurry}
C_p	= specific heat of the mother liquor	$kJ/kg_{slurry}K$
C	= capacity of the plant	(kton/yr)
C_I	= C.E. plant costs index	-
$C_{I,T}$	= EPE cost index	-
C_{fi}	= complexity factor of the functional unit	-
d_{50}	= the particle diameter for which the cyclone is 50% efficient	μm
d_p	= particle diameter	μm
D	= column diameter	m
D	= diameter of crystallizer	m
F_p	= pressure factor	-
F_m	= material factor	-
F_t	= temperature factor	-
f	= costliness index	-
f	= investment factor	-
G	= growth rate	m/s
H	= column height	m
I_B	= investments in process units	MHfl
I_H	= investments in side apparatures	MHfl
I_L	= investments in overhead	MHfl
I_W	= investments in work capital	MHfl
I_F	= fixed capital investments	MHfl
I_W	= work-costs investments.	MHfl
K	= permeability	-
K_T	= total costs	MHfl/yr
K_P	= production volume dependent costs	MHfl/yr
K_L	= semi-variable costs	MHfl/yr
K_I	= investment dependent costs	MHfl/yr
l	= bed height	m
L	= feed flow rate	kg/h
L_{av}	= average particle diameter	m
m	= mass solid per unit area	kg/m^2
m	= degression exponent	-
N	= number of discs	-
N	= number of process steps	-
N	= number of functional units	-
N	= number of apparatures without pumps	-
$P_{cool,max}$	= maximum cooling energy available per unit heat transfer area	kW/m^2
P	= plant capacity	kton/yr
P_i	= unit capacity	kton/yr
qi	= amount of i needed per ton product	ton/ton

Q_{cool}	= cooling energy	$kJ/kg_{crystall}$
R	= filter medium resistance	
R	= gas constant=8.3144	J/molK
S	= specific surface area	m^2/m^3
Sc	= mass fraction of solids in cake	-
S_i	= complexity score for step i	-
Ss	= mass fraction of solids in suspension	-
T_m	= melting point	K
u_c	= superficial velocity	m/s
U	= overall heat transfer coefficient	kW/m^2
V	= column volume	m^3
v_i	= cost of base chemical or utility i per ton	MHfl/ton

α	= specific cake resistance for unit area of filter per unit weight of cake	m/kg
Γ	= residence time	s
ΔH_{crys}	= enthalpy of crystallisation	kJ/kg_{crys}
ΔL	= distance between two discs	-
ΔP	= pressure drop	bar
ΔT	= temperature difference between inlet feed and slurry	K
ΔT_{lm}	= logarithmic mean temperature difference	K
ϵ	= bed porosity	-
ϵ	= voidage	-
η	= liquid viscosity	Ns/m^2
ρ	= density	kg/m^3
ρ_l	= liquid density	kg/m^3
ρ_o	= outlet density of the slurry	kg_{slurry}/m^3_{slurry}
ρ_s	= solid density	kg/m^3
τ	= residence time	h
$\Phi_{v,in}$	= feed volume debiet	m^3/h

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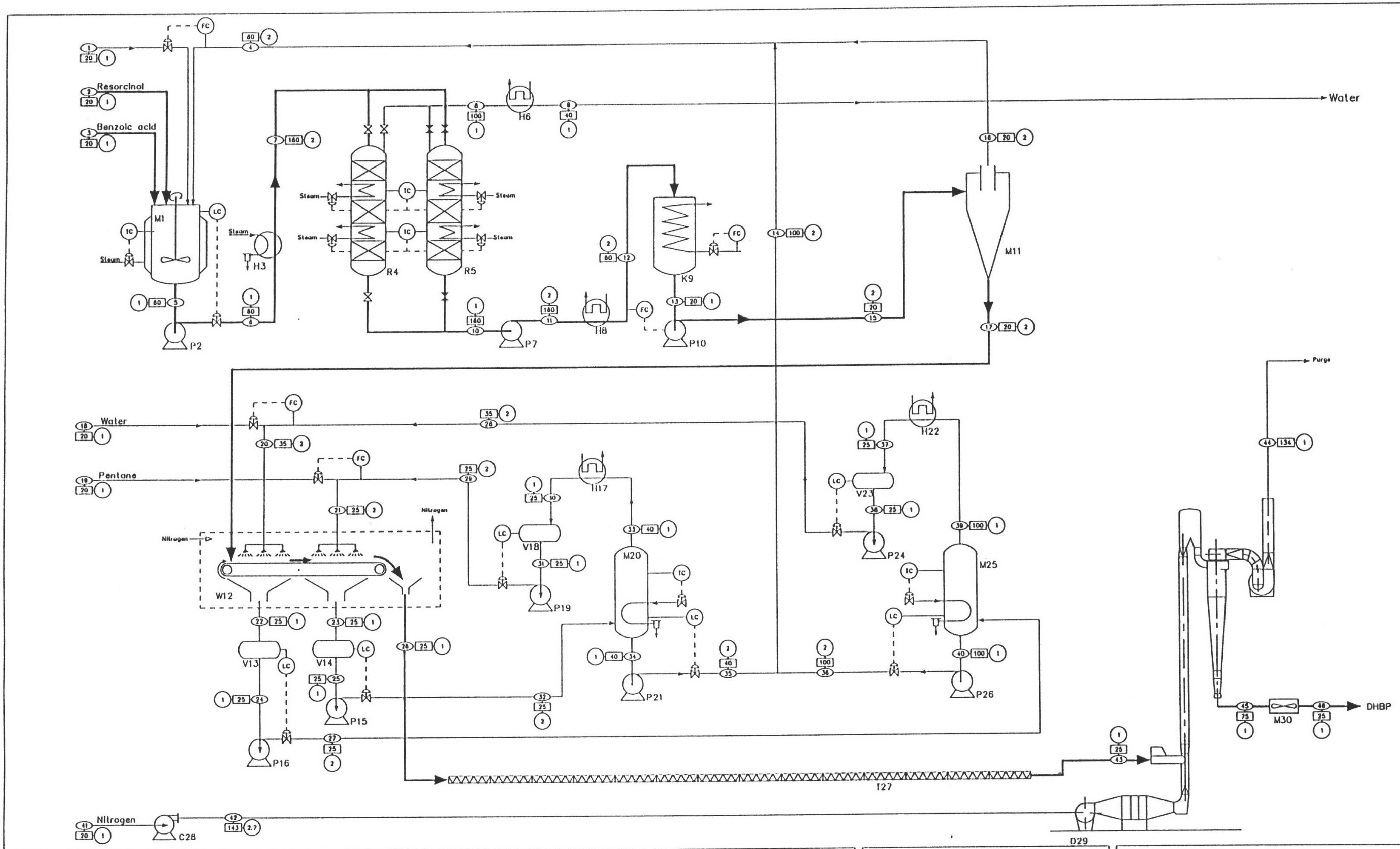
Appendix I: Process flowsheet



M1	MIXER
P2	PUMP
H3	HEATER
R4	PACKED BED REACTOR
R5	PACKED BED REACTOR
H6	CONDENSOR
P7	PUMP
H8	COOLER
K9	CRYSTALIZATOR
P10	PUMP
M11	HYDROCYCLON
W12	SOLID WASHER
V13	VESSEL
V14	VESSEL
P15	PUMP
P16	PUMP
H17	CONDENSOR
V18	PENTANE STORAGE TANK
P19	PUMP
M20	LV SEPARATOR
P21	PUMP
H22	CONDENSOR
V23	WATER STORAGE TANK
P24	PUMP
M25	LV SEPARATOR
P26	PUMP
T27	SCREW CONVEYOR
C28	COMPRESSOR
D29	PNEUMATIC DRYER
M30	SOLID COOLER

Production of
2,4-dihydroxybenzophenone

I.B. Janto Saputro
G. Kartal
C. Setijabudiharsa
Y.W. Wong

FVO: 3170
May 1996

○	Streamnr.
☐	Temp. in °C
○	Pressure in bar

Appendix II

Tabel A1: Physical properties of compounds

Name	Formula	Mol weight [kg/kmol]	Boiling point [°C]	Melting point [°C]	Liquid density [kg/m^3]	Specific heat capacity [kJ/kgK]
1,3-Benzenediol (resorcinol)	$C_6H_6O_2$	110.11	280.5	110	1176.9 (110°C)	1.8307
Benzoic acid	$C_7H_6O_2$	122.12	249.8	122.4	1086 (122.4°C)	1.6532
n-Butylbenzene	$C_{10}H_{14}$	134.22	183.2	135-136	-	1.5826
DHBP	$C_{13}H_{10}O_3$	214.24	194	144	694.9 (144°C)	1.7112
Nitrogen	N_2	28.01	-195.8	-	805	-
RMB	$C_{13}H_{10}O_3$	214.24	432.43	135-136	-	1.5847
Pentane	C_5H_{12}	72.15	36	-129.8	626 (20°C)	2.2841
Water	H_2O	18.01	100	0	998 (20°C)	4.18
Zeolite H-beta	NU-1	-	-	-		-

Appendix III: Component mass and energy balance

Table A2: Component mass and energy balance

Stream no	1	2	3	4	5	6	7	8	9	10
Temperature(°C)	20	20	20	59.9919	60	60.0004	160	100	40	160
Pressure(bar)	1	1	1	1.9972	1	2	2	1	1	1
Heat(kW)	-0.0122	-126.94	-126.06	-373.38	-837.60	-837.46	-614.55	-108.64	-110.4	-551.21
Component	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)
Nitrogen	0	0	0	0	0	0	0	0	0	0
N-pentane	0	0	0	31.056	31.056	31.056	31.056	0	0	31.056
N-butylbezene	0.0914	0	0	3252.5	3252.59	3252.59	3252.59	0	0	3252.6
1,3-benzenediol	0	120.18	0	34.971	155.151	155.151	155.151	0	0	66.817
Benzoid acid	0	0	133.29	49.912	183.202	183.202	183.202	0	0	94.867
Water	0	0	0	2.272	2.272	2.272	2.272	90.608	90.608	0
RMB(liq)	0	0	0	154.78	154.78	154.78	154.78	0	0	95.702
DHBP(liq)	0	0	0	75.894	75.894	75.894	75.894	0	0	223.3
DHBP(solid)	0	0	0	0.0893	0.0893	0.0893	0.0893	0	0	0
RMB(solid)	0	0	0	0	0	0	0	0	0	0
Total	0.0914	120.18	133.29	3601.47	3855.0	3855.0	3855.0	90.608	90.608	3764.34

Stream no	11	12	13	14	15	16	17	18	19	20
Temperature(°C)	170.712	60	20	99.9879	20.0763	20.073	20.0763	20	20	34.6591
Pressure(bar)	2	2	1	2	2	1.9944	1.9944	1	1	2
Heat(kW)	-346.54	-675.94	-722.94	-158.76	-722.83	-455.27	-267.53	-10.219	-1.5726	-1460.8
Component	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)
Nitrogen	0	0	0	0	0	0	0	0	0	0
N-pentane	31.056	31.056	31.056	10.742	31.056	20.982	10.073	0	2.3168	162.45
N-butylbezene	3252.6	3252.6	3252.6	1054.8	3252.6	2197.6	1055	0	0	194.53
1,3-benzenediol	66.817	66.817	66.817	21.669	66.817	45.144	21.673	0	0	0.3447
Benzoid acid	94.867	94.867	94.867	30.767	94.867	64.095	30.771	0	0	0.5499
Water	0	0	0	2.2126	0	0	0	2.3444	0	296.72
RMB(liq)	95.702	95.702	92.353	14.687	92.353	77.937	14.689	0	0	0
DHBP(liq)	223.3	223.3	60.901	19.749	60.901	41.147	19.754	0	0	11.939
DHBP(solid)	0	0	162.4	0	162.4	0.076	162.25	0	0	0
RMB(solid)	0	0	3.3496	0	3.3496	0	3.3496	0	0	0
Total	3764.34	3764.34	3764.34	1154.63	3764.34	2446.98	1317.56	2.3444	2.3168	666.534

Stream no	21	22	23	24	25	26	27	28	29	30
Temperature(°C)	24.9816	25.014	24.9814	25.014	24.9814	24.9816	25.014	34.8252	25.0788	25
Pressure(bar)	2	1	1	1	1	1	2	2	2	1
Heat(kW)	-315.94	-1670.8	-315.5	-1670.8	-315.5	-71.661	-1670.8	-1450.5	-314.36	-314.36
Component	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)
Nitrogen	0	0	0	0	0	0	0	0	0	0
N-pentane	358.54	171.83	357.59	171.83	357.59	1.6479	171.83	162.45	356.23	356.23
N-butylbezene	0.9975	1248.2	2.1776	1248.2	2.1776	0.2206	1248.2	194.53	0.9975	0.9975
1,3-benzenediol	0.0002	21.992	0.0218	21.992	0.0218	0.004	21.992	0.3447	0.0002	0.0002
Benzoid acid	0.0011	31.286	0.0319	31.286	0.0319	0.0055	31.286	0.5499	0.0011	0.0011
Water	19.744	296.58	19.755	296.58	19.755	0.1317	296.58	294.38	19.744	19.744
RMB(liq)	0	14.672	0.0144	14.672	0.0144	0.0026	14.672	0	0	0
DHBP(liq)	0.0217	31.659	0.0507	31.659	0.0507	0.0053	31.659	11.939	0.0217	0.0217
DHBP(solid)	0	0	0	0	0	162.25	0	0	0	0
RMB(solid)	0	0	0	0	0	3.3496	0	0	0	0
Total	379.305	1816.22	379.641	1816.22	379.641	167.617	1816.22	664.194	376.995	376.995

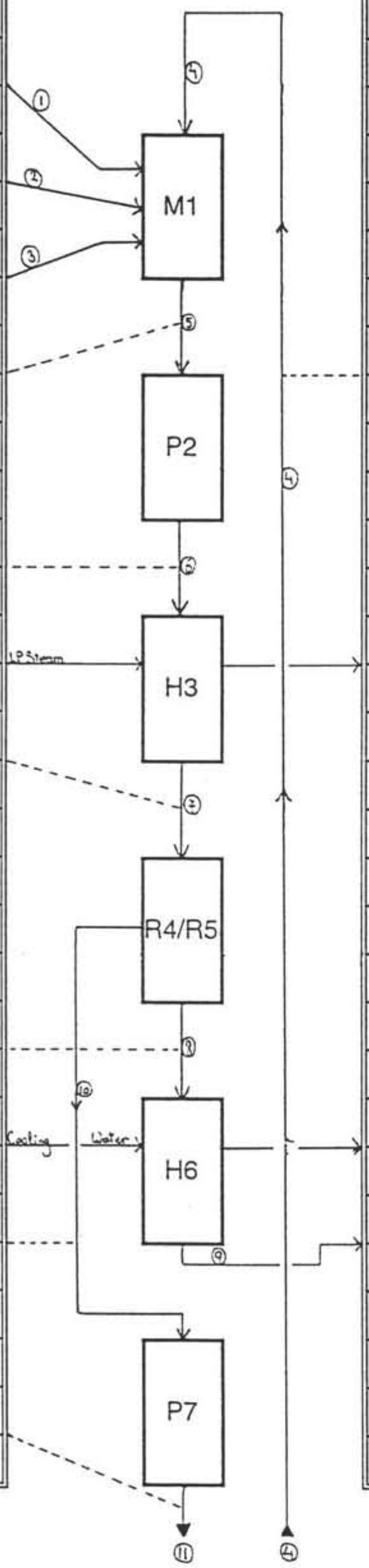
Stream no	31	32	33	34	35	36	37	38	39	40
Temperature(°C)	25	24.9814	40	40	40.006	100.07	25	25	100	100
Pressure(bar)	1	2	1	1	2	2	1	1	1	1
Heat(kW)	-314.36	-315.5	-263.58	-1.0954	-1.0953	-157.71	-1450.6	-1450.6	-1200.6	-157.76
Component	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)
Nitrogen	0	0	0	0	0	0	0	0	0	0
N-pentane	356.23	357.59	356.23	1.3615	1.3615	9.3807	162.45	162.45	162.45	9.3807
N-butylbezene	0.9975	2.1776	0.9975	1.1802	1.1802	1053.6	194.53	194.53	194.53	1053.6
1,3-benzenediol	0.0002	0.0218	0.0002	0.0216	0.0216	21.648	0.3447	0.3447	0.3447	21.648
Benzoid acid	0.0011	0.0319	0.0011	0.0308	0.0308	30.736	0.5499	0.5499	0.5499	30.736
Water	19.744	19.755	19.744	0.0108	0.0108	2.2018	294.38	294.38	294.38	2.2018
RMB(liq)	0	0.0144	0	0.0144	0.0144	14.672	0	0	0	14.672
DHBP(liq)	0.0217	0.0507	0.0217	0.029	0.029	19.72	11.939	11.939	11.939	19.72
DHBP(solid)	0	0	0	0	0	0	0	0	0	0
RMB(solid)	0	0	0	0	0	0	0	0	0	0
Total	376.995	379.641	376.995	2.6483	2.6483	1151.96	664.194	664.194	664.194	1151.96

Stream no	41	42	43	44	45	46				
Temperature(°C)	25	142.803	24.9816	134.367	75	25				
Pressure(bar)	1	2.68	1	1	1	1				
Heat(kW)	0.01822	49.4222	-71.661	44.3722	-66.611	-69.983				
Component	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)	F(kg/h)				
Nitrogen	1500	1500	0	1500	0	0				
N-pentane	0	0	1.6479	1.6479	0	0				
N-butylbezene	0	0	0.2206	0.2206	0	0				
1,3-benzenediol	0	0	0.004	0.004	0	0				
Benzoid acid	0	0	0.0055	0.0055	0	0				
Water	0	0	0.1317	0.1317	0	0				
RMB(liq)	0	0	0.0026	0.0026	0	0				
DHBP(liq)	0	0	0.0053	0.0053	0	0				
DHBP(solid)	0	0	162.25	0	162.25	162.25				
RMB(solid)	0	0	3.3496	0	3.3496	3.3496				
Total	1500	1500	167.617	1502.02	165.6	165.6				

Appendix IV: Overall mass and energy balance

Mass and heat balance

In M	In Q	Forward M Q	Back M Q	OUT M	OUT Q
0.0914	-0.0122				
120.18	-126.94				
133.29	-126.06				
		3855 -837.60	3601.47 -373.38		
		3855 -837.46			
6368.94	5069.5			6368.94	4846.59
		3855 -614.547			
		90.608 -108.64			
75.43	1.7571			75.43	3.5171
		3764.34 -506.86		90.608	-110.40
		3764.34 -346.54			



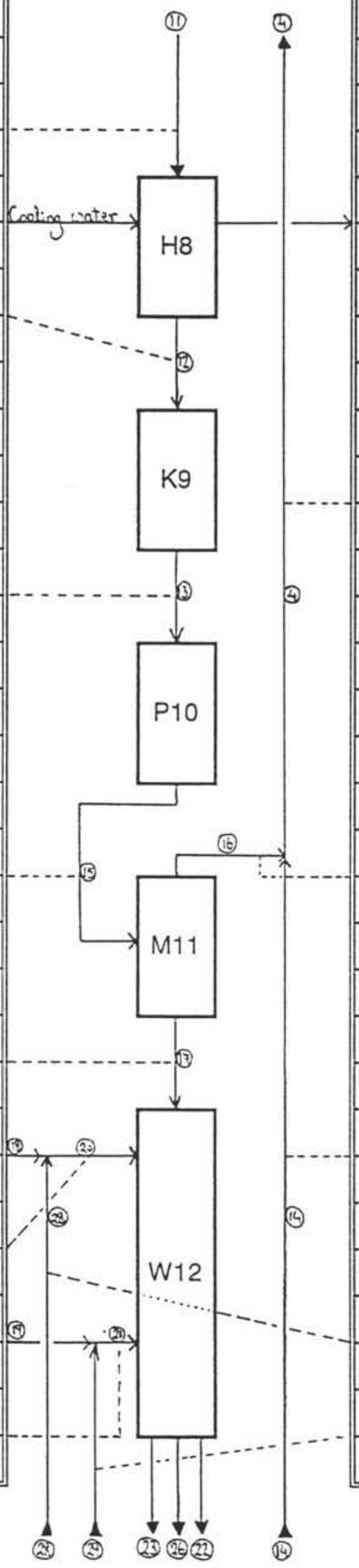
Mass in kg/hr

Heat in kW

FVO No. : 3170

Mass and heat balance

In		Forward
M	Q	M Q
		3764.34 -346.54
2550.52	59.41	
		3764.34 -675.94
		3764.34 -722.94
		3764.34 -722.83
		1317.56 -267.53
2.3444	-10.2194	
		666.534 -1460.75
2.3168	-1.5726	
		379.305 -315.94



Back	OUT	
M Q	M	Q
	2550.52	388.81
3601.47 -329.02	·	
2446.98 -455.27		
1154.63 -158.76		
664.194 -1450.53		
376.995 -314.36		

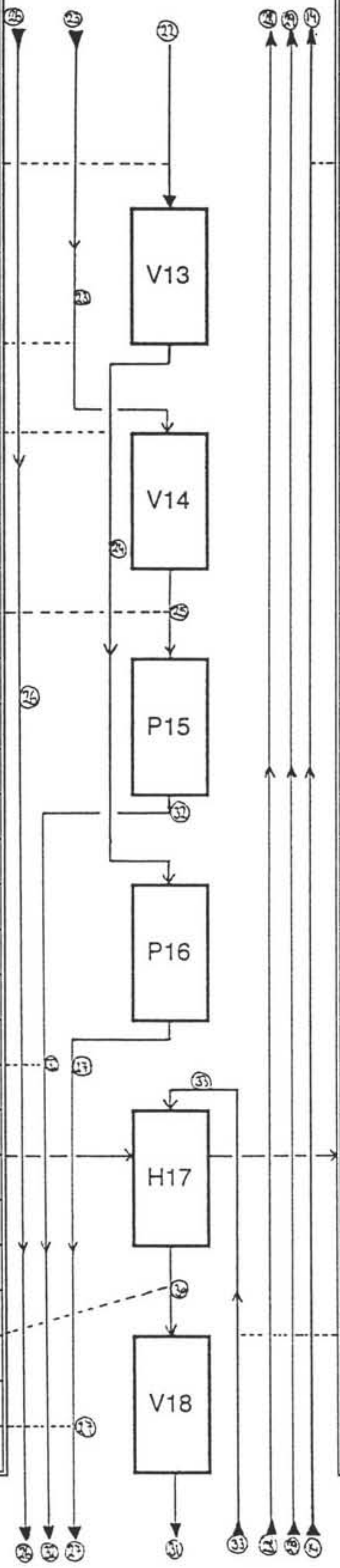
Mass in kg/hr

Heat in kW

FVO No. : 3170

Mass and heat balance

In		Forward	Back	OUT	
M	Q	M Q	M Q	M	Q
		1816.22 -1670.75	1154.63 -158.76		
		379.641 -315.50			
		1816.22 -1670.75			
		379.641 -315.50			
		379.995 -315.50			
4352.57	101.39			4352.57	152.17
		376.995 -314.36	376.641 -263.58		
		1816.22 -1670.75			



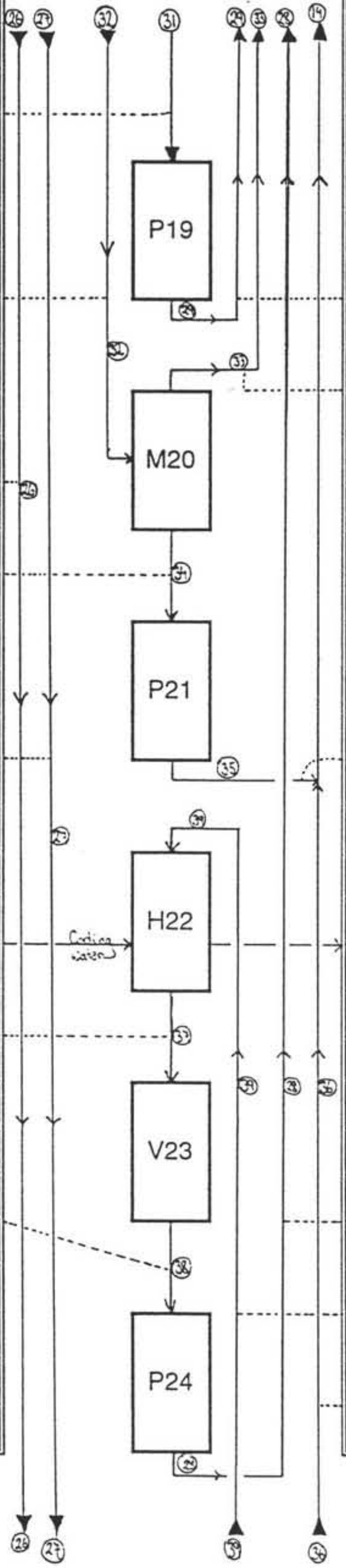
Mass in kg/hr

Heat in kW

FVO No. : 3170

Mass and heat balance

In M	In Q	Forward M	Forward Q	Back M	Back Q	OUT M	OUT Q
		376.995	-314.36				
		379.641	-315.50	376.995	-314.36		
				376.995	-263.58		
		167.617	-71.66				
		2.6483	-1.0954				
		1816.22	-1670.75	2.6483	-1.0953		
10712.57	249.54					10712.57	499.5
		665.26	-1450.57				
		665.26	-1450.56	664.194	-1450.53		
				664.194	-1200.61		
				1151.96	-157.71		



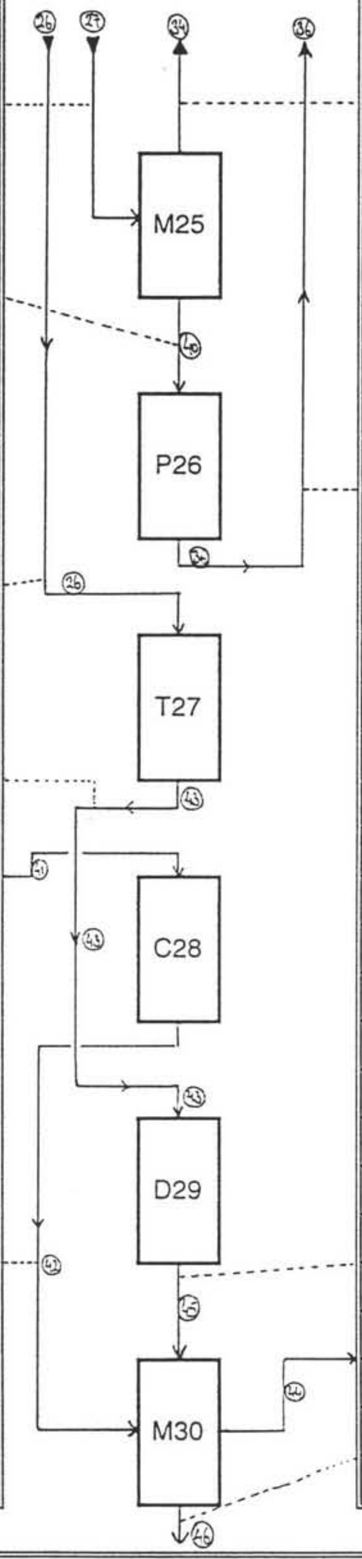
Mass in kg/hr

Heat in kW

FVO No. : 3170

Mass and heat balance

In M	In Q	Forward M	Forward Q	Back M	Back Q	OUT M	OUT Q
		1816.22	-1670.75	664.194	-1200.6		
		1151.96	-157.76				
				1151.96	-157.71		
		167.617	-71.66				
		167.617	-71.66				
1500	0.01822						
		1500	49.4222	165.6	-66.611		
						1502.02	44.3722
						165.6	-69.98



In M	In Q		OUT M	OUT Q
25818.25	5216.81	Total	25818.26	5754.58

Mass in kg/hr

Heat in kW

FVO No. : 3170

Appendix V: Equipment specifications

Apparatus specification list for reactors, and vessels **FVO No. :3170**

APPARAAT NO.	M1	R4/R5	M20	M25
Name : Type :	Mixer Continous stirred mixer	Reactor Packed bed	L/V separator vertical cylinder vessel	L/V separator vertical cylinder vessel
Pressure [bar] Abs./Eff. :	1	1	1.2	1.2
Temp. [°C] :	60	160	100	40
Volume [m^3] : Diameter [m] : L or H [m] :	9 1.56 4.68	27.44 1.8 6.8	0.59 0.61 1.83	0.33 0.46 1.83
Packing : Tray(number): Fixed packing: Catalyst type: Catalyst form:		Zeolite H-beta Pellets		
Special material				
Number : - serial : - parallel :				
Other :				

Apparattus specification for pumps, blowers and compressors **FVO No.: 3170**

APPARATUS NO.	P2	P7	P10	P15	P16
Type :	Centrifugal pump	Centrifugal pump	Centrifugal pump	Centrifugal pump	Centrifugal pump
Medium :	N-Butylbenzene	N-Butylbenzene	N-Butylbenzene	Pentane	Water
Capacity : [kg/s]	1.071	1.045	1.045	0.105	0.504
Density [kg/m^3] :	860	860	860	626	1000
Pressure abs/eff [bar] suck/press [bar] :	1/2	1/2	1/2	1/2	1/2
Temp. In/Uit [°C] :	60/60	160/160	20/20	25/25	25/25
Power [kW] - theoretical: - practical :	0.122 0.243	0.122 0.243	0.122 0.243	0.0136 0.034	0.054 0.126
Number - serial : - parallel :					
Special material :					
Other :					

Apparattus specification for pumps, blowers and compressors **FVO No. :3170**

APPARATUS NO.	P19	P21	P24	P26	C28
Type :	Centri-fugal pump	Centri-fugal pump	Centri-fugal pump	Centri-fugal pump	Centri-fugal compressor
Medium :	Pentane	N-Butylbenzene	Water/pentane	N-Butylbenzene	Nitrogen
Capacity : [kg/s]	0.104	$7.341.10^{-4}$	0.185	0.319	0.4167
Density [kg/m^3] :	626	860	1000	680	1
Pressure abs/eff [bar] suck/press [bar] :	1/2	1/2	1/2	1/2	1/2.68
Temp. In/Uit [°C] :	25/25	40/40	25/35	100/100	25/25
Power [kW] **-theoretical:** **- practical :**	 0.0156 0.039	 $0.098.10^{-2}$ $0.392.10^{-2}$	 0.0248 0.062	 0.0372 0.093	 0.109 0.167
Number - serial : - parallel :					
Special material :					
Other :					

Apparattus specification for coolers

FVO No.: 3170

Equipment number	H6	H8	H17	H22
Tube side : T_1/T_2 **Shell side : t_1/t_2**	100/40 20/40	170.7/60 20/40	40/25 20/30	100/25 20/40
Shell passes **Tube passes**	1 2	1 2	1 2	1 2
LMTD (°C) F_t ΔT_m (°C)	36.41 0.8 29.13	76.6 0.94 72	7.21 0.78 5.63	22.13 1 22.13
U ($W/m^2.°C$)	248	261.3	228.4	270.7
A (m^2)	0.06	18.30	15.04	11.3
Q (kW)	1.76	329.4	50.78	250
Tubes: o.d (mm) i.d (mm) sheet thickn. (mm) length (m) eff. length (m) surf. area 1 tube (m^2)	 20 16 50 4.88 4.83 0.30	 20 16 50 4.88 4.83 0.30	 20 16 50 4.88 4.83 0.30	 20 16 50 4.88 4.83 0.30
number of tubes	1	60	50	37
K_1 n_1	0.249 2.207	0.249 2.207	0.249 2.207	0.249 2.207
Bundle diam. (mm) clearance (mm) shell diam. (mm)	37.6 0 37.6	240 52 292	221 52 273	192.8 50 242.8
Type of coolers	floating head	floating head	floating head	floating head

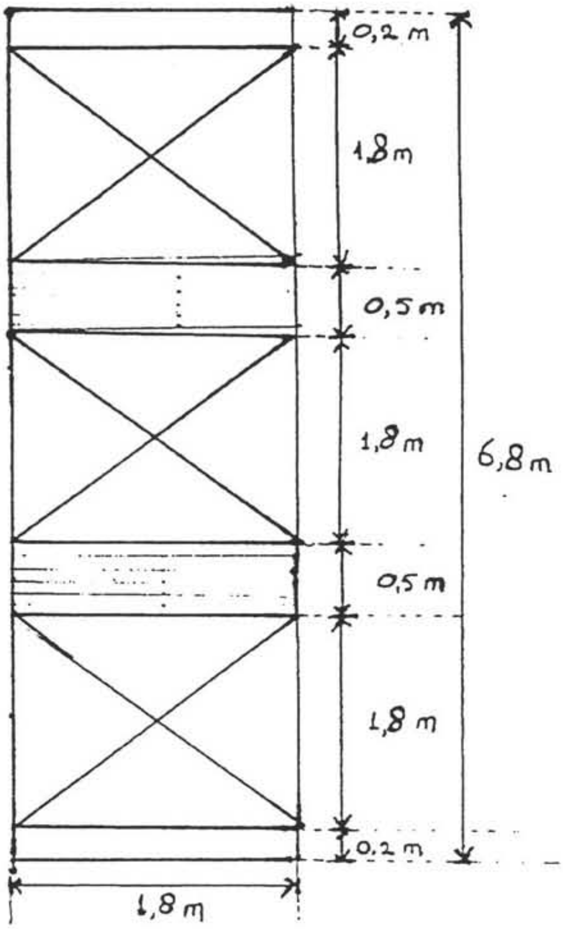
Apparatus No.	H3	H6	H8	H17	H22
Name **Type**	heater Internal Floating head with clamp ring	cooler Internal Floating head with clamp ring	cooler Internal Floating head with clamp ring	cooler Internal Floating head with clamp ring	cooler Internal Floating head with clamp ring
Medium **- tubes** **- shell side**	 LP steam feed	 cooling water water (vap)	 cooling water product	 cooling water pentane	 cooling water water (vap)
Capacity Heat transfer (kW)	-222.9	1.76	329.4	50.78	250
Heat transfer area (m^2)	9.02	0.06	18.30	15.04	11.30
Number **- serie** **- parallel**					
Abs./Eff. pressure (bar) **- tubes** **- shell side**	 2.0 3.0	 1.0 1.0	 1.0 1.0	 1.0 1.0	 1.0 1.0
Temp. in/out (° C) **- tube side** **- shell side**	 60/160 190/160	 100/40 20/40	 170.7/60 20/40	 40/25 20/30	 100/25 20/40
Used material	AISI 304	AISI 304	AISI 304	AISI 304	AISI 304
Others	Reactor feed heater	Cooling for vapour water from reactor	Product stream cooling to crystallisator	Recycle pentane (washer) cooling	Recycle waste water cooling

Tabel A3: Mixer summaries

Design parameter*)	Value
D_t (m)	1.56
H (m)	4.68
D (m)	0.52
N (1/s)	19.167
ρ (kg/m^3)	860
μ (Pa.s)	0.7917 10^{-3}
Re	5.629.10^6
N_p	0.25
P (kW)	57.5595

*): D_t=Agitator diameter H=Mixer height
D=Mixer diameter N=Agitator speed
ρ=liquid density μ=liquid viscosity
Re=Reynolds number N_p=Power number
P =Agitator power consumption

Tabel A4: Reactor summaries

Design parameter*)	Value	Design scheme
$\Phi_{v,\ in}$ [m^3/h]	4.483	
τ [h]	3	
$V_{reactor,fixed\ bed}$[m^3]	13.447	
ϵ [-]	0.4	
$V_{kat}{}^1$[m^3]	8.068	
$H_{void\ space\ between\ fixed\ beds}$ [m]	**0.5**×2=1	
$H_{void\ space\ inlet/outlet}$ [m]	**0.2**×2=0.4	
D [m]	**1.8**	
$H_{fixed\ bed,total}$ [m^3]	5.4 (1.8 m each)	
H [m]	**6.8**	
A [m^2]	2.544	
u_c [m/s]	$4.895.10^{-4}$	

^{1}) Vkat = (1-ϵ)×Vbed

*) $\Phi_{v,\ in}$ = feed volume debiet, m^3/h

τ = residence time, s

ϵ = bed porosity

V = column volume, m^3

D = column diameter, m

H = column height, m

A = column cross sectional area, m^2

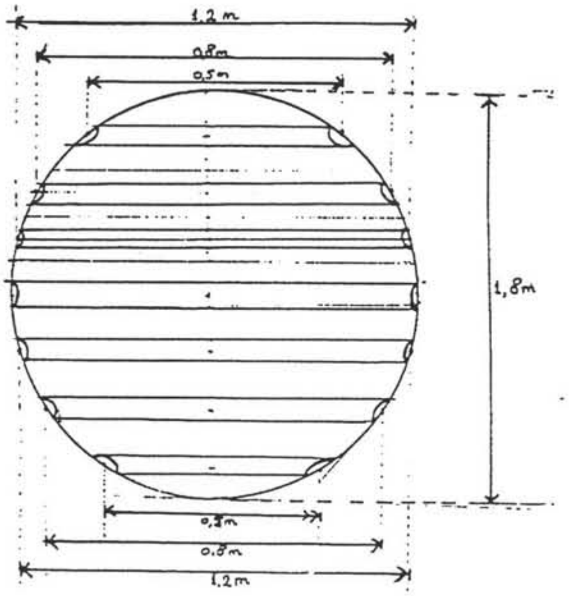
u_c = superficial velocity, m/s

Table A5: Pressure drop summaries

Parameter*)	Value	Formula	
		Symbol	Value
u_c (m/s)	$4.895.10^{-4}$	$Re_l = u_c\ \rho_{nbb} / [S(1-\epsilon)\eta]$	$Re_l = 0.6$
ρ_{nbb} (kg/m^3)	860		
S (m^2/m^3)	1500		
ϵ	0.4		
η (Pa.s)	$0.792.10^{-3}$		
l (m)	6.8	$R_l / \rho_{nbb}u = \Delta P\ \epsilon^3 / [l*S(1-\epsilon)\ u_c^2\ \rho_{nbb}]$	$R_l / \rho_{nbb}u = 10$
ΔP (bar)	1.97.10-3		

*) ρ_{nbb} = liquid density, kg/m^3
S = specific surface area, m^2/m^3
η = liquid viscosity, Pa.s
l = bed height, m
ΔP = pressure drop, bar

Table A6: Reactor heater summeries

Design parameter*)	Value	Design scheme
Q [kW]	31.665	
U [W/ m^2 °C]	750	
$T_{s,av}$-Tr [°C]	17.5	
$A_{total\ each\ heater}$ [m^2]	2.412	
$A_{total\ each\ layer}$ [m^2]	0.534	
l [m]	6.8	
N_t [-]	5	
Φ_m [kg/h]	2171.31	

*) Q = Heat required

U = Overall heat transfer cofficient

$T_{s,av}$ = Average in/out steam temperature

T_r = Isoterm reactor temperture

$A_{total\ each\ heater}$ = total heat transfer area each heater

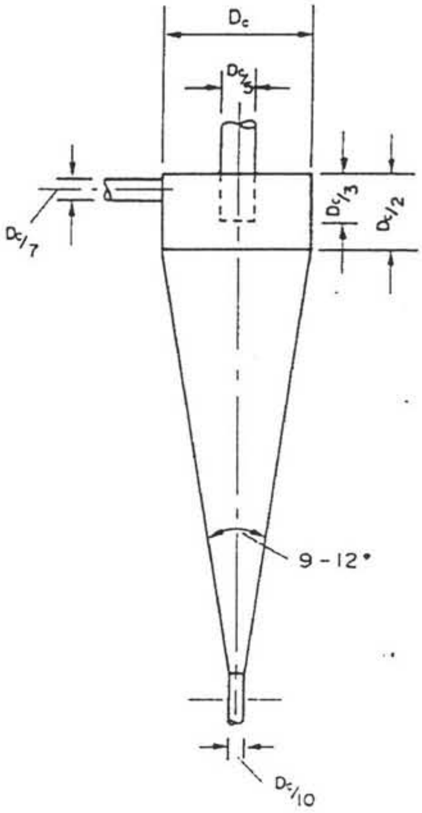
$A_{total\ each\ layer}$ = total heat transfer area each layer

N_t = number of layer each heater

l = total length of tube each layer

Φ_m = steam flow

Table A7: Hydrocyclone summaries

Design parameter*)	Value	Compound	
d_p [μm]	500	DHBP	
d_{50} [μm]	350	DHBP	
ρ_l [g/cm^3]	0.86	N-butyl benzene	
ρ_s [g/cm^3]	1.1	DHBP	
η [m Ns/m^2]	0.79	N-butyl benzene	
L [l/min]	72.94		
Dc [cm]	100		
Dc/2 [cm]	50		
Dc/3 [cm]	33		
Dc/5 [cm]	20		
Dc/7 [cm]	14.3		
Dc/10 [cm]	10		
angle	9-12°		

*) d_p = particle diameter,
L = feed flow rate(L/min)= (feed in (kg/h) / ρ_l(kg/m^3))*1/60,
d_{50} = the particle diameter for which the cyclone is 50% efficient,
ρ_l = liquid density,
ρ_s = solid density,
η = liquid viscosity,
Dc = cylone diameter.

Table A8: The parameters of the vessels *)(L/V separator)

	M20	M25
P_i [bar]	1.2	1.2
T [°C]	40	100
D_i [m]	0.46	0.61
H [m]	1.83	2.31
ρ_L [kg/m^3]	708.38	809.52.
t [min]	5	5
V [m^3]	0.33	0.73
u_v [m/s]	0.59	0.97
Q [MJ/h]	-57.92	-470.2
Head type	vertical cylinder	vertical cylinder

*)The material of the vessels are all from carbon steel

Table A9: Coolers and heater summaries

	H3 (heater)			H6			H8			H17			H22		
stream number	6	7		8	9		11	12		33	30		39	37	
Temperature [°C]	60	160		100	40		107.7	60		40	25		100	25	
Pressure [bar]	2	2		1	1		2	2		1	1		1	1	
Heat duty [kW]	-837.5	-614.6		-108.6	-110		-346.5	-675.9		-263.6	-314.4		-1200.6	-1450.6	
Mass flow rate [kg/h]	3855	3855		90.6	90.6		3764.3	3764.3		377	377		664.2	664.2	
Viscosity [mNs/m^2]	0.99	0.31	0.65	0.28	0.68	0.48	0.27	0.70	0.48	0.01	0.91	0.46	0.01	0.29	0.15
Density [kg/m^3]	862.1	771.4	816.7	957.8	992	975	756.4	858	807.2	1.05	927.19	464.1	2.47	633.6	318
Specific heat [kJ/kg/°C]	1.9	2.3	2.1	4.2	4.2	4.2	2.4	1.9	2.1	1.8	3.2	2.5	1.8	2.4	2.1
Conductivity [W/kg/K]	0.12	0.10	0.11	0.68	0.63	0.65	0.10	0.12	0.11	0.02	0.31	0.17	0.02	0.12	0.07

Table A10: Pump summaries

Pump number	Flow rate [kg/s]	Fluid density [kg/m^3]	Flow rate [m^3/s]	Efficiency [%]	ΔP [Pa]	Power [kW]
P2	1.071	$860^{*)}$	$1.25.10^{-3}$	50	10^5	0.249
P7	1.045	$860^{*)}$	$1.22.10^{-3}$	50	10^5	0.243
P10	1.045	$860^{*)}$	$1.22.10^{-3}$	50	10^5	0.243
P15	0.105	$626^{**)}$	$1.68.10^{-4}$	40	10^5	0.034
P16	0.504	$1000^{***)}$	$5.05.10^{-4}$	40	10^5	0.126
P19	0.104	$626^{**)}$	$1.67.10^{-4}$	40	10^5	0.039
P21	$7.341.10^{-4}$	$743^{+)}$	$0.988.10^{-6}$	25	10^5	$0.392.10^{-3}$
P24	0.185	$743^{+)}$	$2.48.10^{-4}$	40	10^5	0.062
P26	0.319	$860^{*)}$	$3.72.10^{-4}$	40	10^5	0.093

*) = nbb density
**) = pentane density
***) = water density
+) = average nbb and pentane density

Table A11: Tanks summaries

	V13/V23	V14/V18
P_i [bar]	1.2	1.2
T [°C]	25	25
D_i [m]	0.6	0.5
H [m]	1.8	1.4
e_s,e [mm]	5	5
shell	cylinder	cylinder

Appendix VI:

Table A12 : HAZOP analyse of reactor section

Stream nr. Guide word	Subject.	Reason	Consequence	Management
Not, No	No flow	Valve closed in error.	1)No feed to the reactor, thus no reaction occur.	1. Ensure good communication with intermediate storage operator 2.Check lines and valves and replace it if necessary.
		The breaking of pipeline	2)Explosion danger, flammable	3a. shut down the flow temporarily 3b.Ventilation must be beter. 3c.install new pipeline
	P	High pressure drop causes corrosion will take place.	2)Flow is decreasing	3a. 4. clean up the pipeline
	T	Unit defect, and the reactor outlet blocked.	2) 3) Higher concentration and heat accumulation. 4)Pressure increases in the reactor. 5)Clogging	3a, 3b. 5.check the reactor inlet and outlet pipeline. 6.controll the heat exchanger and replace it if necessary.
More	Flow	Flow Controller (FC) doesn't work	6)Overflow 7)higher pressure, excess liquid, less efficient.	3a, 7. install automatic shut down/by-pass
	P	Pressure Controllers (PC) defect, higher T, FC doesn't work.	7) 8)Explosion danger, flammable	3a, 8. install an alarm sys. closes unit with quenching
	T	Heat exchanger defect,higher T, plug in pipeline.	3), 8)	3a, 6, 8
Less	Flow	Pump defect, FC doesn't work,	2), 8)	1, 2, 4, 8
	P	High pressure drop, corrosion will take place, flow overcooled.	2), 5)	5, 4
	T	Friction, higher T, heat exchanger defect, plug in pipeline,flow overcooled.	3) 5)	6
Reverse	Flow	Leakage	Possible explosion danger, flammable	3b, 3c, 8

Table A13: Calculation of the Dow Fire and Explosion Index

	Unit 1 (reaction resorcinol & benzoic acid)	Unit 2 (washing with pentane & drying)
Material Factor (MF)	4	21
<u>1. General process hazards</u>		
Base factor	1.00	1.00
B. Endothermic reactions	0.20	0.40
C. Material Handling & Transfer	0.25	0.85
D. Enclosed or indoor process units	0.30	0.30
F. Drainage and spill control	0.25	0.25
GENERAL PROCESS HAZARDS FACTOR (F_1)	2.00	2.80
<u>2. Special process hazards</u>		
Base factor	1.00	1.00
A.Toxic material(s)	0.40	0.20
B.Process temperature	0.30	0.30
C.Operation or near flammable range	0.30	0.30
F. Rotating equipment	0.50	0.50
SPECIAL PROCESS HAZARDS FACTOR (F_2)	2.50	2.30
UNIT HAZARD FACTOR (F_1 x F_2 = F_3)	5.00	6.44
FIRE AND EXPLOSION INDEX (F_3 x MF = F & EI)	20	135.24

1 a. From the first unit the Unit Damage Factor is 0.125.
   b. The area of exposure is determined as 16.

2 a. The determine of Unit Damage Factor from the third unit is 0.78.
   b. The area of exposure is determined as 116.

CAS-nummer: [65-85-0]
benzeencarbonzuur

C_6H_5COOH

BENZOËZUUR

FYSISCHE EIGENSCHAPPEN

Kookpunt, °C	249
Sublimatiepunt, °C	>100
Smeltpunt, °C	122
Vlampunt, °C	121
Zelfontbrandingstemperatuur, °C	570
Relatieve dichtheid (water = 1)	1,3
Relatieve dampdichtheid (lucht = 1)	4,2
Relatieve dichtheid bij 20 °C van verzadigd damp/luchtmengsel (lucht = 1)	1,0
Dampspanning, mbar bij 20 °C	<0.1
Oplosbaarheid in water, g/100 ml	0,3
Relatieve molecuulmassa	122,1
Log P octanol/water	1,9
Brutoformule:	$C_7H_6O_2$

BELANGRIJKE GEGEVENS

WITTE VASTE STOF IN DIVERSE VORMEN MET TYPERENDE GEUR
Stofexplosie mogelijk indien in fijn verdeelde vorm gemengd met lucht. Reageert heftig met oxidatiemiddelen en met sterke basen en reducerende stoffen.

MAC-waarde niet vastgesteld

Wijze van opname / Inademingsrisico: De stof kan worden opgenomen in het lichaam door inademing van stofdeeltjes, door de huid en door inslikken. Deze stof verdampt bij 20 °C praktisch niet; als poeder kan bij (ver)stuiven echter snel een irriterende concentratie in de lucht ontstaan.
Directe gevolgen: De stof werkt irriterend op de ogen, de huid en de ademhalingsorganen. Inademing van de stof kan astma veroorzaken (zie Opmerkingen). De stof kan inwerken op het centraal zenuwstelstel, met als gevolg onwillekeurige trillingen en stuiptrekkingen.
Gevolgen bij langdurige, herhaaldelijke blootstelling: Herhaaldelijk of langdurig contact kan overgevoeligheid veroorzaken. De stof kan op de lever en darmen inwerken, met als gevolg orgaanafwijkingen. Contact met de huid met 0.25% benzoëzuuroplossing kan allergische aandoeningen veroorzaken.

DIRECTE GEVAREN / SYMPTOMEN	PREVENTIE	BLUSSTOFFEN / EERSTE HULP
Brand: brandbaar.	geen open vuur en niet roken.	sproeistraal water, poeder.
Explosie: fijn verdeeld stof met lucht explosief.	stofafzetting voorkomen, gesloten systeem, stofexplosie-veilige electrische apparatuur en verlichting.	bij brand: tanks/vaten koel houden door spuiten met water.
	VERSPREIDEN VAN STOF VOORKOMEN! STRENGE HYGIENE!	
Inademen: keelpijn en hoesten, brandend gevoel, misselijkheid, moeizaam ademen, braken.	ventilatie of ruimtelijke afzuiging (indien niet in poedervorm), plaatselijke afzuiging, ademhalingsbescherming (filter-type P2).	frisse lucht, rust, half zittende houding, naar arts verwijzen.
Huid: *wordt opgenomen*, roodheid, pijn.	handschoenen (butylrubber, PVC).	verontreinigde kleding uittrekken, huid spoelen en wassen met water en zeep, naar arts verwijzen.
Ogen: roodheid en pijn, slecht zien.	stofbril.	eerst langdurig spoelen met veel water (contactlenzen verwijderen mits makkelijk mogelijk), dan naar arts brengen.
Inslikken: buikkramp, hoofdpijn, misselijkheid, braken, zwaktegevoel, diarree.		mond laten spoelen, twee glazen water laten drinken, en onmiddellijk naar ziekenhuis vervoeren.

OPRUIMING / OPSLAG

Opruimen gemorst produkt: Draag handschoenen, filtermasker met filtertype P2 en stofbril.
Gemorst produkt afdekken, vervolgens zorgvuldig opzuigen/opscheppen (explosieveilige stofzuiger) en eventueel hergebruiken.
Restant zorgvuldig verzamelen en opslaan in vaten. *Eventuele laatste resten* verwijderen met water.
Spoelwater afvoeren naar riool.
Resten etiketteren en afvoeren volgens BAGA/KCA regels.
Opslag: gescheiden van oxidatiemiddelen, gescheiden van sterke basen, gescheiden van reducerende middelen, ventilatie langs de vloer.

ETIKETTERING

Afleveringsetiket: vraag leverancier

NFPA:

BAGA: C 8
KCA : III

OPMERKINGEN

Wie die overgevoeligheidsverschijnselen heeft gekregen door blootstelling aan benzoëzuur, dient in de toekomst blootstelling aan deze stof te vermijden. De verschijnselen van astma openbaren zich veelal pas na enige tijd; rust en opname in een ziekenhuis is daarom noodzakelijk. Iemand die astma verschijnselen heeft vertoond, mag nooit meer met deze stof in contact komen.

CAS-nummer: [109-66-0]
n-pentaan

PENTAAN

FYSISCHE EIGENSCHAPPEN

Kookpunt, °C	36
Smeltpunt, °C	−130
Vlampunt, °C	−40
Zelfontbrandingstemperatuur, °C	260
Relatieve dichtheid (water = 1)	0,6
Relatieve dampdichtheid (lucht = 1)	2,5
Relatieve dichtheid bij 20 °C van verzadigd damp/luchtmengsel (lucht = 1)	1,8
Dampspanning, mbar bij 20 °C	573
Oplosbaarheid in water, g/100 ml	0,03
Explosiegrenzen, volume% in lucht	1,4-8
Minimum ontstekingsenergie, mJ	0,22
Soortelijke geleiding, pS/m	$2x10^4$
Relatieve molecuulmassa	72,2
Brutoformule:	C_5H_{12}

BELANGRIJKE GEGEVENS

KLEURLOZE, ZEER VLUCHTIGE VLOEISTOF, MET ZWAKKE GEUR
De damp is zwaarder dan lucht en verspreidt zich over de grond met kans op ontsteking op afstand. Ten gevolge van het geringe geleidingsvermogen van de vloeistof kunnen elektrostatische ladingen worden opgewekt bij stroming, beweging enz. Bij vullen, aftappen of verwerken geen persluchtals drijfgas toepassen.

MAC-waarde 600 ppm 1800 mg/m³

Wijze van opname / inademingsrisico: De stof kan worden opgenomen in het lichaam door inademing en inslikken. Een voor de gezondheid gevaarlijke concentratie in de lucht kan door verdamping van deze stof bij ca. 20°C vrij snel worden bereikt; bij vernevelen nog sneller.
Directe gevolgen: De damp van de stof werkt irriterend op de ogen en de ademhalingsorganen. Na inslikken van de vloeistof kunnen druppeltjes in de longen terecht komen (aspiratie), waardoor longontsteking kan optreden. De stof kan inwerken op het zenuwstelsel. Blootstelling kan bij hoge concentraties verlaging van het bewustzijn veroorzaken.
Gevolgen bij langdurige, herhaaldelijke blootstelling: De vloeistof ontvet de huid.

DIRECTE GEVAREN / SYMPTOMEN	PREVENTIE	BLUSSTOFFEN / EERSTE HULP
Brand: zeer brandgevaarlijk.	geen open vuur, geen vonken en niet roken.	poeder, A.F.F.F., schuim, koolzuur, (halonen).
Explosie: damp met lucht explosief.	gesloten apparatuur, ventilatie, explosieveilige elektrische apparatuur en verlichting, aarden, vonkarm handgereedschap.	bij brand: tanks/vaten koel houden door spuiten met water.
Inademen: keelpijn, hoesten, kortademigheid, duizeligheid, sufheid.	ruimtelijke afzuiging, plaatselijke afzuiging, ademhalingsbescherming (filtertype AX).	frisse lucht, rust, en arts waarschuwen.
Huid: droge huid, roodheid.	handschoenen (polyvinylalcohol, nitrilrubber).	verontreinigde kleding uittrekken, huid spoelen met veel water of douchen.
Ogen: roodheid, pijn.	veiligheidsbril.	eerst langdurig spoelen met veel water (contactlenzen verwijderen mits makkelijk mogelijk), dan naar arts brengen.
Inslikken: buikpijn, misselijkheid, zwaktegevoel.		mond laten spoelen, GEEN braken opwekken, en naar ziekenhuis vervoeren.

OPRUIMING / OPSLAG

Opruimen gemorst produkt:
Draag handschoenen, laarzen en verse luchtkap/persluchtmasker.
Bij meer dan 50 liter gevarenzone ontruimen. Explosiegevaar. Extra ventilatie.
Gemorst produkt indammen en afdekken, vervolgens zorgvuldig opzuigen (explosieveilige stofzuiger) en eventueel hergebruiken.
Restant opnemen in inert absorptiemiddel en dit zorgvuldig verzamelen en opslaan in vaten (hermetisch afsluiten). *Eventuele laatste resten* verwijderen met zeepoplossing. *Spoelwater* afvoeren naar riool.
Vaten etiketteren en afvoeren volgens BAGA/KCA regels.
Opslag: brandveilig, koel.

ETIKETTERING

Afleveringsetiket:

🔥
Licht ontvlambaar

R: 11
S: (2-)9-16-29-33
Nota C

BAGA: D 5
KCA : VI

NFPA:
4
1 0

OPMERKINGEN

Gebruik van alcoholische dranken versterkt de schadelijke (bedwelmende) werking. De geur waarschuwt onvoldoende bij overschrijding van de MAC-waarde. Bij hoge concentraties in de lucht, bijvoorbeeld in een slecht geventileerde ruimte, ontstaat zuurstofgebrek met kans op bewusteloosheid.
Zie voor inertisering van explosieve gas, damp/luchtmengsels het Chemiekaartenboek, hoofdstuk 'Tabellen en formules'.
Pas een stevige gebruiksverpakking toe; plaats kwetsbare gebruiksverpakking in een stevige houder.

Transport Emergency Card TEC(R)-30G30

GEVI: 33; UN-nummer: 1265

CAS-nummer: [108-46-3]
1,3-dihydroxybenzeen
m-dihydroxybenzeen
resorcine

$C_6H_4(OH)_2$

RESORCINOL

FYSISCHE EIGENSCHAPPEN		BELANGRIJKE GEGEVENS
Kookpunt, °C	280	**WITTE KRISTALLEN, WORDT ROSE AAN DE LUCHT** Reageert heftig met oxidatiemiddelen. Kan elektrostatische lading opwekken bij werveling, pneumatisch transport enz.; stofexplosies mogelijk bij fijne verdeling in de lucht.
Smeltpunt, °C	110	
Vlampunt, °C	127	
Zelfontbrandingstemperatuur, °C	600	
Relatieve dichtheid (water = 1)	1,3	MAC-waarde 10 ppm 45 mg/m³
Relatieve dampdichtheid (lucht = 1)	3,8	
Relatieve dichtheid bij 20 °C van verzadigd damp/luchtmengsel (lucht = 1)	1,0	**Wijze van opname / inademingsrisico:** De stof kan worden opgenomen in het lichaam door inademing, inslikken en via de huid. Een voor de gezondheid gevaarlijke concentratie in de lucht zal door verdamping van deze stof bij ca. 20°C niet of slechts zeer langzaam worden bereikt; bij stuiven echter veel sneller. **Directe gevolgen:** De stof werkt irriterend op de ogen, de huid en de ademhalingsorganen. De stof werkt op het zenuwstelsel. In emstige gevallen kans op toevallen en dodelijke afloop. **Gevolgen bij langdurige, herhaaldelijke blootstelling:** Bij intensief huidcontact kans op huidaandoeningen. De stof kan aanleiding geven tot bloedveranderingen. Vorming van methaemoglobine. Lever-, nier- en hartbeschadigingen kunnen optreden.
Dampspanning, mbar bij 20 °C	0,01	
Oplosbaarheid in water, g/100 ml bij 20 °C	140	
Explosiegrenzen, volume% in lucht	1,4-?	
Relatieve molecuulmassa	110,1	
Log *P* octanol/water	0,8	
Brutoformule:	$C_6H_6O_2$	

DIRECTE GEVAREN / SYMPTOMEN	PREVENTIE	BLUSSTOFFEN / EERSTE HULP
Brand: brandbaar.	geen open vuur en niet roken.	sproeistraal water, poeder.
Explosie: fijn verdeeld stof met lucht explosief.	stofafzetting voorkomen, stofexplosieveilige apparatuur en verlichting, aarden.	
	STRENGE HYGIËNE	
Inademen: keelpijn, hoesten, kortademigheid, duizeligheid, krampen, bewusteloosheid, blauwe huid, zwaktegevoel.	ventilatie (indien niet in poedervorm), plaatselijke afzuiging of ademhalingsbescherming (combinatiefiltertype A/P2).	frisse lucht, rust, en naar ziekenhuis vervoeren.
Huid: *wordt opgenomen*, roodheid, zie verder 'inademen'.	handschoenen (butylrubber), beschermende kleding.	verontreinigde kleding uittrekken, huid wassen met water en zeep, en arts waarschuwen.
Ogen: roodheid, pijn, slecht zien.	gelaatsscherm, of oogbescherming in combinatie met ademhalingsbescherming.	eerst langdurig spoelen met veel water (contactlenzen verwijderen mits makkelijk mogelijk), dan naar arts brengen.
Inslikken: buikpijn, zie verder: 'inademen'.		mond laten spoelen, braken opwekken en onmiddellijk naar ziekenhuis vervoeren.

OPRUIMING / OPSLAG	ETIKETTERING
Opruimen gemorst produkt: Draag chemicaliën-pak uitrusting, filtermasker met filtertype A/P2 en gelaatscherm. *Gemorst produkt* afdekken, vervolgens zorgvuldig opzuigen/opscheppen (explosieveilige stofzuiger) en eventueel hergebruiken. *Restant* zorgvuldig verzamelen en opslaan in vaten. *Eventuele laatste resten* verwijderen met water. *Spoelwater* afvoeren naar riool. Vaten etiketteren en afvoeren volgens BAGA/KCA regels. **Opslag:** gescheiden van oxidatiemiddelen.	**Afleveringsetiket:** ☒ Schadelijk **NFPA:**  R: 22-36/38 S: (2-)26 **BAGA:** B 19 **KCA** : III

OPMERKINGEN

Iemand die overgevoeligheidsverschijnselen heeft gekregen door blootstelling aan resorcinol, dient in de toekomst elke blootstelling aan deze stof te vermijden. Bij vergiftiging door resorcinol is specifieke eerste hulp noodzakelijk. De benodigde middelen (Methyleenblauw) met gebruiksaanwijzing moeten beschikbaar zijn.

Transport Emergency Card TEC(R)-61G11BC

GEVI: 60; UN-nummer: 2876

CAS-nummer: [7727-37-9]

N_2

STIKSTOF
(vloeibaar, gekoeld)

FYSISCHE EIGENSCHAPPEN	
Kookpunt, °C	−196
Relatieve dichtheid (water = 1)	zie opm.
Relatieve dampdichtheid (lucht = 1)	0,97
Relatieve molecuulmassa	28,0
Brutoformule:	N_2

BELANGRIJKE GEGEVENS

KLEURLOZE VLOEISTOF MET ZEER LAGE TEMPERATUUR
Het koude gas is zwaarder dan lucht, en kan zich op laaggelegen plaatsen ophopen met aldaar ka
op zuurstofgebrek (bewusteloosheid).

MAC-waarde niet vastgesteld

Directe gevolgen: De koude vloeistof kan bevriezing veroorzaken.

DIRECTE GEVAREN / SYMPTOMEN	PREVENTIE	BLUSSTOFFEN / EERSTE HULP
Brand: niet brandbaar.		bij brand in directe omgeving: alle blusstoffen toegestaan.
Inademen: zie 'opmerkingen'.	ruimtelijke afzuiging, plaatselijke afzuiging, onder geen beding filtermaskers.	frisse lucht, rust, zonodig beademing, en arts waarschuwen, en zonodig naar ziekenhuis vervoeren.
Huid: *bij bevriezing:* roodheid, pijn, blaren, wonden.	koude-isolerende handschoenen.	*bij bevriezing:* GEEN kleding uittrekken, huid spoelen met veel water of douchen, en arts waarschuwen.
Ogen: *bij bevriezing:* roodheid, pijn, slecht zien.	gelaatsscherm.	eerst langdurig spoelen met veel water (contactlenzen verwijderen mits makkelijk mogelijk), dan naar arts brengen.

OPRUIMING / OPSLAG

Opruimen gemorst produkt:
Draag handschoenen en laarzen (koude-isolerend) en verse luchtkap/persluchtmasker.
Gevarenzone ontruimen. Extra ventilatie.
Vaten etiketteren en afvoeren volgens BAGA/KCA regels.

ETIKETTERING

Afleveringsetiket: vraag leverancier

NFPA:

KCA : VI

OPMERKINGEN

Bij hoge concentraties in de lucht, bijvoorbeeld in een slecht geventileerde ruimte, ontstaat zuurstofgebrek met kans op bewusteloosheid.
Dichtheid van de vloeistof bij het kookpunt: 0,8 kg/l.

Transport Emergency Card TEC(R)-112

GEVI: 22; UN-nummer: 1977

Bestelcode C-0365

57d

Appendix VII

The costs of base chemicals and utilities

Table A14: Costs of base chemicals

Component	Throughput (ton/yr)	Price* (Hfl/ton)	Total costs (kHfl/yr)
Resorcinol	961.44	10,558.6	10,151.46
Benzoic Acid	1066.32	2,306.74	2,459.72
n-Butylbenzene	0.7312	72,000	52.65
Pentane	18.5344	19,648.6	364.18
Water	18.7552	2.5	0.046
Nitrogen	12000	65	780
Zeolite H-beta	6.656	88,000	585.728
TOTAL			14,393.78

*: Ref: Chemical Marketing Reporter, Dec.1995 (Resorcinol & Benzoic Acid)
Saf Bulk Chemicals, Bornem (n-Butylbenzene & Pentane)
FVO Handleiding, Sept.1994 (Water)
Handouts of Chemische Fabriek (Nitrogen)
PQ Zeolites B.V.,Leiden (Zeolite H-beta)

Table A15: Costs of utilities

Apparatus	Type	Quantity	Unit	Price *	Cost (Hfl/yr)
M1	steam (LP)	0.037	kg/s	Hfl 30 per ton	32,184
H3		0.111			96,292.8
M20		0.008			6,950.4
M25		0.065			56,424
H6	cooling water	0.021	kg/s	Hfl 0.10 per m^3	60.34
H8		3.921			11,293.71
H17		0.605			1,741.03
H22		2.976			8,571.43
K9		0.697			2,006.73
P2		0.249	kWh/yr	Hfl 0.13 per kWh	258.96
P7	electricity	0.243			252.72
P10		0.243			252.72
P15		0.034			35.36
P16		0.126			131.04
P19		0.039			40.56
P21		0.000			0.41
P24		0.062			64.48
P26		0.093			96.72
C28		1,716,920			223,199.6
T27		0.320			322.80
M1		57.560			59,861
TOTAL					500,052

*: Prices from " FVO Handleiding ".

Investment costs calculations

Tabel A16: Investment costs of functional units (Zevnik - Buchanan-Jansen)

Functional unit	Complexity factor, C_{fi}	Throughput (kton/yr)	Investment costs (k$)
M1	2.18	30.840	650
R4/R5	3.013	30.840	898
K9	2.04	30.115	598
M11	2.00	30.115	588
W12	2.00	10.540	313
D29	2.15	1.341	98
TOTAL (I_B)			**314,5**

Tabel A17:Costliness index (f) estimation (Taylor)

		Throughput	Material of construction	Residence time	P & T	Score	Costliness index, f
Stor.&Hand.	Resorcinol	- 0.31	0	0	0	- 0.31	0.92
	Benzoic Acid	- 0.51	1	0	0	0.49	1.14
	n-Butylbenzene	- 3	0	0	0	- 3	0.46
	Pentane	- 3	0	0	0	- 3	0.46
	Water	- 3	1	0	0	- 2	0.60
	Recycle	5.86	0	0	0	5.86	4.65
Process	Reactor	6.02	0	0	0	6.02	4.85
	Crystallizer	5.97	0	0	0	5.97	4.79
	Product separation	5.97	0	0	0	5.97	4.79
	Filter & Wash	3.9	0	0	0	3.9	2.78
	Dryer	0.017	0	0	0	0.017	1.004
TOTAL							26.44

- The calculated costliness index (f) is 26.44.
- The plant capacity is 1.3248 kton/yr.
- The EPE cost index for 1995 is calculated as fallows: The EPE index for 1990 is 790. The price indices for 1990 and 1995 are, respectively, 105, 116 (Webci & Wubo, 1995). The EPE index for 1995 becomes 873 (= 790×116/105).
- The investment costs then become k$ 7,985.

IRR Calculations

The IRR can be calculated using the following formula:

$$\sum_{0}^{N} C_f D_i = 0$$

where C_f = cash flow (MHfl/yr)
D_i = discrete discount factor in year i = $1/(1+r)^n$
r = IRR/100
n = operational time span of the plant (years).

The cash flow is given by:
cash flow = Income - K_T + depreciation
= 26.635 - 24.54 +0.10×0.80×24.4 = MHfl 4.047 per year.
I_F = $0.06 \times K_I = 0.06 \times 24.4$ = 1.464 MHfl

$a = -K_I = 24.4$ $\quad b = C_f / (1+r)$ $\quad c = C_f / (1+r)^2$ $\quad d = C_f / (1+r)^3$
$e = C_f / (1+r)^4$ $\quad f = C_f / (1+r)^5$ $\quad g = C_f / (1+r)^6$ $\quad h = C_f / (1+r)^7$
$j = C_f / (1+r)^8$ $\quad k = C_f / (1+r)^9$ $\quad l = (C_f + I_W) / (1+r)^{10}$

$a + b + c + d + e + f + g + h + k + l + m = 0$

IRR = r×100 = 50%.

APPENDIX VIII: Design/calculation of reactor and heat exchanger

Reactor

Pressure drop calculation

The Re_1 can be calculated by:
$Re_1 \quad = u_c \, \rho_{nbb} / [S(1\text{-}\varepsilon)\eta]$
$\quad =4.895.10^{-4} * 860/(1500*(1-0.4)*0.792.10^{-3})$
$\quad =0.6$
Extrapolated of this value in figure 4.1 Coulson & Richardson gives :$R_1 / \rho_{nbb}u = 10$.
Further calculation of pressure drop will be:
$R_1 / \rho_{nbb}u^2 = \Delta P \, \varepsilon^3 / [l*S(1-\varepsilon) \, u_c^2 \, \rho_{nbb}]$
$10 = \quad \Delta P * 0.4^3/(6.8*1500*(1-0.4)*(4.895.10^{-4})^2*860) \Rightarrow$ **$\Delta P = 1.97.10^{-3}$ bar.**

Heater calculation

Each section must delivered $Q_{total}/2 = 63.333 /2 \Rightarrow Q_{each\ heater} = 31.665$ kW

The total heat transfer area each section will be :
$A_{total\ each\ heater} = Q_{each\ heater} / (U*(T_{s,av} - T_r) = 31665/(750*(177.5-160) = 2.412$ m^2

The total heat transfer area each tube (id=25 mm, total length: 6.8 m $^{*)}$) :
$A_{total\ each\ layer} = \pi * d * l = \pi * 25.10^{-3} * 6.8 = 0.534$ m^2

Number of layer = $A_{total\ each\ heater} / A_{total\ each\ layerr} = 2.412/ 0.534 \approx 5$

Amount of steam needed for each heater can be calculated by:
$\Phi_m = Q_{each\ heater} / cp_{steam} * (Tin\text{-}Tout) = 31665 / 2.1. \, 10^3 * (190-165) = 0.613$ kg/s=2171.31 kg/h

$^{*)}$ total length of 'one layer' heater i.e. 1.8 + 2*0.5+2*0.8+2*1.2 = 6.8 m, see design scheme of heater.

Heater calculation

Heater H3

Heat capacity liquid = 2.1 kJ/kg.°C.
Heat load = -837.5-(-614.6) = -222.9 kW
Steam water flow = 222.9/ 4.2(190-160) = 1.77 kg/s = 6372 kg/h

$\Delta T_{lm} = (60-160)-(160-190)/\ln[(60-160)/(160-190)]$
$\quad = -58.14$ °C

Use one shell pass and two tube passes.
$R \quad = (T_1 - T_2)/(t_2 - t_1) = 3.33$
$S \quad = (t_2 - t_1)/ (T_1 - t_1) = 0.23$

From Fig. 12.19; $F_t \quad = 0.85$
$\Delta T_m \quad = 49.42$ °C

From Tab. 12.1; $U \quad = 500$ W/m^2.°C

Provisional area: A = $9\ m^2$

Choose 20 mm o.d., 16 mm i.d., 4.88-m-long tubes (¾ in. x 16 ft), carbon steel.
Allowing for tube-sheet thickness, take

L = 4.83 m
Area of one tube = $4.83 \times 20 \times 10^{-3} = 0.303\ m^2$
Number of tubes = $9/0.303 = 29.7 \approx 30$

The triangular pitch is 1.25 (for the clean shell-side fluid).

$P_t = 1.25 \times 20 = 25$ mm

Bundle diameter $D_b = 20\ (29.7/0.249)^{1/2.207} = 174.55$ mm
Number of tubes in centre row,

$N_{cent} = D_b\ /\ P_t = 7$

With a split-ring floating head type, from Fig. 12.10, bundle diameter clearance = 50 mm,
shell diameter, $D_s = 174.55 + 50 = 224.55$ mm.

The assumed overall coefficient is checked by the following calculations:

Tube-side coefficient

Mean water temp = (160 + 190)/2 = 175 °C
Tube cross-sectional area= $\pi/4 \times 16^2$ = $201\ mm^2$
Tubes per pass = 30/2 = 15
Total flow area = $15 \times 201 \times 10^{-6}$ = $0.003\ m^2$
Water mass velocity = 1.77/0.003 = $590\ kg/s.m^2$
Viscosity of water = $0.8\ mNs/m^2$
Thermal conductivity = 0.59 W/m. °C
Density water = $995\ kg/m^3$
Water linear velocity = 590/995 = 0.59 m/s

The coefficient can be calculated using equation 12.15.

$$Re = \frac{\rho u d_i}{\mu} = 995 * 0.59 * 16 * 10^{-3} / 0.8 * 10^{-3} = 11741$$

$$Pr = \frac{C_p\ \mu}{k_f} = 4.2 * 10^{3} * 0.8 * 10^{3} / 0.59 = 5.7$$

Neglect (μ/μ_w)

$L/d_i = 4.83 \times 10^3\ /\ 16 = 302$

From Fig. 12.23, $j_h = 3.9 \times 10^{-3}$

$h_i = \{0.59/16 \times 10^{-3}\} \times 3.9 \times 10^{-3} \times 11741 \times 5.7^{0.33} = 2998.75\ W/m^2\ .\ ^\circ C$

Shell-side coefficient

Choose baffle spacing = $D_s/5$ = 45 mm
Tube pitch P_t = 1.25 x 20 = 25 mm
Cross-flow area A_s = (25-20)/25x224.55x45x10^{-6} = 0.002 m^2 (eq. 12.21)
Mass velocity G_s = 3855/3600 x 1/0.002 = 535.42 kg/s.m^2
Equivalent diameter d_e = 1.1/20 (25^2 - 0.917 x 20^2) = 14.4 mm (eq. 12.23)
Mean shell-side temp = (60 + 160)/2 = 110 °C
Liquid density = (862.05 + 771.44)/2 = 816.74 kg/m^3
Liquid viscosity = (0.989 + 0.3114)/2 = 0.65 mNs/m^2
Heat capacity of liquid = (1.9 + 2.33)/2 = 2.12 kJ/kg.°C
Thermal conductivity = 0.11 W/m.°C

$$Re = \frac{G_s d_e}{\mu} = 535.42 * 14.4 \cdot 10^{-3} / 0.65 \cdot 10^{-3}$$

$$Pr = \frac{C_p \, \mu}{k_f} = 2.12 * 10^3 * 0.65 * 10^{-3} / 0.11 = 12.53$$

Choose 25 per cent baffle cut, from Fig. 12.29
j_h = 6 x 10^{-3}

Without the viscosity correction term
From eq. 12.25,
h_s = 0.11/14.4 x 10^{-3} x 6 x 10^{-3} x 11861.54 x $12.53^{1/3}$ = 1252.12 W/m^2.°C

Estimate wall temperature
Mean temp. difference = 110 - 175 = -65 °C
across all resistances
across the water film = U/h_0 x ΔT = 500 / 1252.12 x 65 = 26 °C

Mean wall temp. = 110 - 26 = 84 °C
μ_w = 0.37 mNs/m^2
$(\mu/\mu_w)^{0.14}$ = 1.08 $\approx$ 1

which shows that the correction for a low-viscosity fluid is not significant.

Overall coefficient

Fouling factors use 1000 W/m^2 °C from Table 12.2.
Conductivity of steel k_w = 45 W/m^2 °C (table 12.6)

1/U = 1/1252.12 + 1/1000 + {20x10^{-3} ln (20/16)}/2x45 + {20/16 x 1/1000} + {20/16x1/2998.75}
= 3.52.10^{-3}

U = <u>284.5 W/m^2 °C</u>