



Treball Final de Grau

Design of a methyl ethyl ketone production plant

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La ciencia se compone de errores, que a su vez son los pasos hacia la verdad.

Jules Verne

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SUMMARY

Methyl ethyl ketone is an organic compound whose main use in industry is as a solvent in the manufacturing processes of resins, nitrocellulose coatings, cellulose acetates and others.

The objective of the project is to design a production plant for methyl ethyl ketone that has a purity of 98%, with a production of 25,000 tons spread over 8,000 hours per year.

The different methyl ethyl ketone production processes have been studied and a catalytic dehydrogenation of 2-butanol in the gaseous phase has been carried out. This process has 3 main stages; vaporization of the raw material, the dehydrogenation reaction and, finally, a separation of the components, firstly between the liquid phase and the gas phase and then between the methyl ethyl ketone and the 2-butanol.

In the project, a basic design of the fixed-bed reactor, the liquid-gas separator, the plate distillation column and the cylindrical storage tanks has been carried out. In addition, the heat exchangers necessary for the correct operation of the main equipment have been designed and the pumps and the mixer have been selected according to the needs.

In addition to a basic design of the process equipment, the control and instrumentation of the process has been studied to ensure the correct functioning of the plant.

Keywords: Methyl ethyl ketone, basic equipment design, dehydrogenation of 2-butanol, control and instrumentation.

RESUMEN

La metil etil cetona es un compuesto orgánico cuyo principal uso en la industria es actuar como disolvente en los procesos de fabricación de resinas, revestimientos de nitrocelulosa y acetatos de celulosa entre otros.

El objetivo del proyecto es diseñar una planta de producción de metil etil cetona con una pureza del 98%, cuya producción sea de 25 000 toneladas repartidas en 8000 horas anuales.

Se ha estudiado los diferentes procesos de obtención de metil etil cetona y entre todos los procesos descritos se ha seleccionado la deshidrogenación catalítica del 2-butanol en fase gaseosa. Dicho proceso cuenta con 3 etapas principales; vaporización de la materia prima, la reacción de deshidrogenación y, por último, una separación de los componentes, primero de la fase líquida respecto a la fase gaseosa y a continuación, de la metil etil cetona respecto al 2-butanol.

En el proyecto, se ha realizado un diseño básico del reactor de lecho fijo, del separador líquido - gas, de la columna de destilación de platos y de los tanques de almacenamiento cilíndricos. Como añadido, se han diseñado los intercambiadores de calor necesarios para el correcto funcionamiento de los equipos principales y se han seleccionado las bombas y el mezclador acorde a las necesidades.

Además de realizar un diseño básico de los equipos del proceso, se ha estudiado el control junto con su instrumentación para garantizar el correcto funcionamiento de la planta.

Palabras clave: Metil etil cetona, diseño básico de equipos, deshidrogenación del 2-butanol, control e instrumentación.

1. INTRODUCTION

Over the years, the solvents industry has gone somewhat unnoticed, despite the fact that they are of great importance for the manufacture of multiple products in almost every sector. However, it is increasingly playing a more important role and gaining more recognition.

As far as the chemical industry is concerned, some of the most commonly used solvents are alcohols, ketones, halogenated solvents, amides, ethers [1]. Among the group of ketones there is the methyl ethyl ketone, a solvent which has been showing a favorable production trend in recent years.

Since solvents are so important for many processes, a methyl ethyl ketone production plant will be designed in this project.

1.1. METHYL ETHYL KETONE

Methyl ethyl ketone (MEK), also known as 2-butanone (Figure 1), is an excellent organic solvent, since it is miscible with the majority of organic compounds.

Its molecular formula is C_4H_8O , whereas its semi-developed formula is $CH_3COCH_2CH_3$.

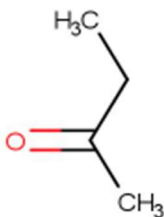


Figure 1. MEK molecule [2].

1.2. PHYSICAL PROPERTIES

The most significant physical properties are shown in Table 1.

Table 1. MEK physical properties [3][4].

Physical state	Liquid
Color	Colorless
Odor	Sweet
Molecular weight [g/mol]	72.107
Density [g/mL]	0.8050
Boiling point [°C]	79.64
Melting point [°C]	-86.9
Critical temperature [°C]	262.45
Critical pressure [MPa]	4.15

As it was mentioned above, MEK is miscible with the vast majority of the organic compounds, however, it is partially miscible with water. Table 2 shows the solubility on water at a temperature of 20 °C.

Table 2. Water – MEK solubility at 20 °C [4].

Substances	Mass fraction [%]
MEK in water	27.5
Water in MEK	12.5

MEK forms binary (Table 3) and ternary azeotropic mixtures in contact with water and other organic compounds.

Table 3. Binary azeotropic mixtures containing MEK [4].

Second component	Mass fraction of MEK	<i>bp</i> at 1013 hPa, °C
Water	88.7	73.4
Benzene	37.5	78.4
<i>n</i> -Hexane	29.5	64.3
<i>n</i> -Heptane	73.0	77.0
Cyclohexane	40	72
1,3-Cyclohexadiene	40	73
Methanol	30.0	63.5
Ethanol	60.9	74.0
Isopropyl alcohol	68.0	77.5
<i>tert</i> -Butyl alcohol	73	77.5
Ethyl acetate	18.0	77.0
Methyl propionate	52	79.25
Propyl formate	55	79.45
Chloroform	96.0	79.65
Carbon tetrachloride	71.0	73.8
Carbon disulfide	15.3	45.85
Propyl mercaptan	75	55.5
Thiophene	55.0	76.0
Ethyl sulfide	20	77.5

1.3. CHEMICAL PROPERTIES

MEK is a stable compound under normal conditions and in the absence of atmospheric oxygen, so that care must be taken in the presence of oxygen, as peroxides can be formed. It is also heat and light stable, as it only decomposes after prolonged UV exposure.

Methyl ethyl ketone peroxide is formed by oxidation with a 30% solution of hydrogen peroxide, whereas diacetyl is formed by oxidation with air in the presence of particular catalysts.

MEK exhibits different behaviors depending on the medium in which it is found (Figure 2). In an alkaline medium, MEK condenses with aldehydes to form highly unsaturated acetones. During base-catalyzed autocondensation in the liquid phase and during gas-phase condensation on alkalized copper catalysts, the carbonyl group reacts with the methyl group, whereas during acid-catalyzed condensation the methylene group in α -position to the carbonyl group is attacked [4].

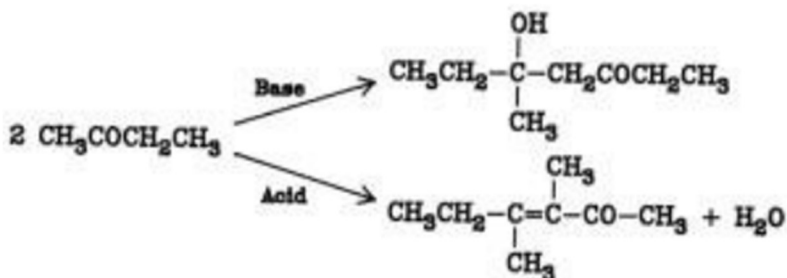


Figure 2. MEK reactions in acidic and basic medium [4].

When MEK reacts with primary and secondary alcohols, higher ketones are obtained, for example, the reaction with sec-butyl alcohol (SBA) gives ethyl amyl ketone. Whereas, when it reacts with polyoxy compounds or epoxides, it forms cyclic products.

1.4. CURRENT USES

MEK is mainly used as a solvent due to its good properties, and also it is used in the manufacturing processes of resins, cellulose acetates and nitrocellulose coatings [5]. Compared to other solvents with similar evaporation rates, MEK has the following advantages:

- Very high power of dissolution
- Favorable volume/mass ratio due to its low density
- Miscibility with a large number of hydrocarbons without impairing the solids content or viscosity
- High ratio of dissolved matter to viscosity

Over half of global MEK demand is derived from the paints and coatings industry as advantage low viscosity solutions can be obtained, having a high solids content, without affecting the film properties.

Moreover, its fast evaporation rate makes it popular for rubber-based industrial cements, low-temperature bonding agents and as an azeotropic separation solvent for printing inks [6].

As it is a highly effective solvent, it is used in the manufacturing process of many products. Among them, some of the most important are lubricants, greases, coatings, cooling liquids, plastics, textiles, antifreeze...

1.5. ANNUAL CONSUMPTION AND PRODUCTION

MEK production in 2017 was approximately 1.7 million tons [7], with the main consumers shown in Figure 3.

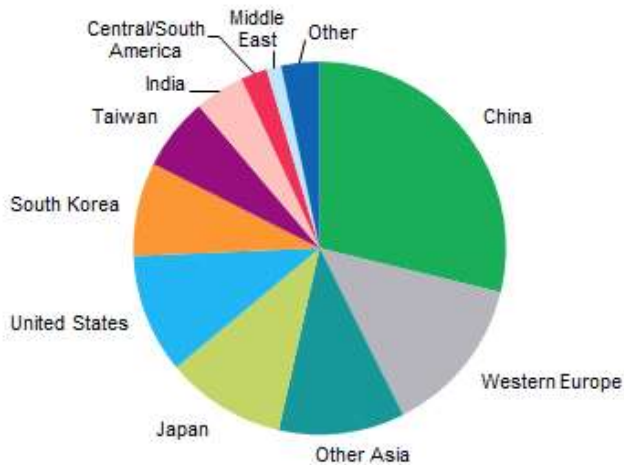


Figure 3. MEK world consumption in 2017 [8].

Between 2018 and 2019 the MEK exports went from \$898M to \$700M. As for MEK's exports in 2019, the countries that exported the most were China (\$161M), Japan (\$146M), United Kingdom (\$92M), Netherlands (\$67.1M) and South Africa (\$66.8M). And as for the imports in 2019, the top importers were South Korea (\$112M), United States (\$101M), Belgium (\$58.5M) Vietnam (\$42.3M) and Indonesia (\$41.8M) [9].

2. OBJECTIVES

The main objective of the project is to do the preliminary design of an industrial plant to produce 25000 tons per year of methyl ethyl ketone. In order to achieve this production, it is necessary to carry out the following tasks:

- Find the optimal production process and study its phases.
- Basic design of the equipment that make up the process.
- Control and instrumentation of the plant.
- Perform the diagrams; both process flow diagram (PFD) and the piping and instrumentation diagram (P&ID).

The project covers the design, automation and diagrams of the methyl ethyl ketone production process. The design of the pipes has not been studied in depth, although it has been taken into account.

3. PROCESS SELECTION

3.1. MEK MANUFACTURING PROCESSES

MEK is mainly obtained by two processes; an 88% is produced by dehydrogenation of sec-butyl alcohol (SBA), in a two-step process that uses sulfuric acid as the catalyst, or in a single-step process that adds water and uses acidic ion-exchange resins as the catalyst. Whereas the remaining 12% is obtained by catalytic oxidation of liquid butane [4].

3.1.1. Catalytic dehydrogenation of SBA in the gaseous phase

The catalytic dehydrogenation of SBA is an endothermic reaction, as its enthalpy has a value of $\Delta H=51$ kJ/mol, that takes place in gaseous phase in a multi-tube fixed-bed catalytic reactor. The most common catalysts used in a gas-phase dehydrogenation are copper, zinc and bronze.

The equilibrium constant for SBA is calculated with the equation 1, where MEK concentration increases as the temperature is hotter and reaches its maximum at approximately 350°C [4].

$$\log K_p = -2.790 \cdot T^{-1} + 1.51 \log T + 1.865 \quad \text{Eq.1}$$

Where the temperature is measured in Kelvins. Figure 4 shows the diagram for a plant producing MEK by catalytic vapor phase dehydrogenation.

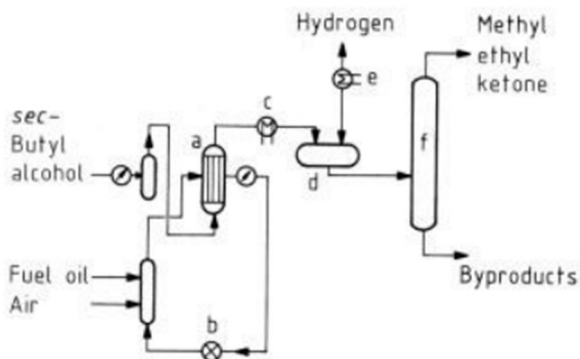


Figure 4. Gaseous-phase dehydrogenation of SBA (Deutsche Texaco AG process) [4].

The process begins vaporizing the SBA that is fed to the multi-tube catalytic reactor. Since it is an endothermic reaction, the reactor requires an energy input, which is provided by a closed thermal oil circuit (in the Deutsche Texaco AG process).

The output current leaves the reactor in gaseous phase and is fed to a cooler. This current is composed of MEK, unreacted SBA and hydrogen, which is extracted in a vapor-liquid separator.

Finally, the output liquid stream is fed to a distillation column, where MEK is obtained as a distillate.

3.1.2. Liquid-phase oxidation of n-butane

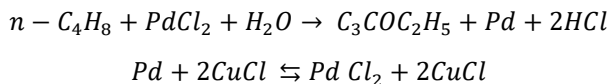
Autoxidation of n-butane takes place in the liquid phase according to a radical mechanism that produces acetic acid as the final product and MEK as an intermediate product. The continuous plug flow process allows the partial MEK intermediate collection.

MEK and acetic acid have a mass ratio of 0.15 - 0.23 : 1.0 and are obtained by a non-catalyzed liquid-phase oxidation at 5.3 MPa and 180 °C while mixing.

When continuous oxidation takes place under plug flow conditions at 150 °C, 6.5 MPa (65 bar), and a residence time of 2.7 min, MEK and acetic acid are formed at mass ratios of up to 3 : 1 [4].

3.1.3. Direct oxidation of n-butenes (Hoechst-Wacker process)

Direct oxidation of n-butenes is carried out in homogeneous aqueous phase in the presence of a redox salt pair, $PdCl_2/2CuCl$, which is later reoxidized.



The process gets to high conversions, around 95%, with the following selectivity distribution (Table 4) [4].

Table 4. Products distribution in the Hoechst-Wacker process.

Reaction products	Selectivity [mol %]
MEK	86
n-Butyraldehyde	4
Chlorinated products	6
Carbon dioxide	1

Although the process achieves a good yield and a favorable selectivity for the desired product, it presents some important disadvantages such as the corrosion caused by free acids and the formation of chlorinated butanones and n-butyraldehyde.

3.2. PROCESS SELECTION

The process that will be carried out in the following process is the catalytic dehydrogenation of SBA in the gaseous phase.

This alternative has been chosen as it is the process in which the highest MEK conversion is obtained and, in addition, it is the most widely used process in the industry due to its simplicity in the equipment requirements and operating conditions. Moreover, during reaction and subsequent treatments, practically no waste disposal problems arise.

4. PROCESS DESCRIPTION

4.1. CAPACITY OF THE PLANT

The plant is designed in order to produce 25000 tons of MEK per year and will be operated continuously for 8000 hours per year. The remaining hours will be spent on installation checks, cleaning and repairing the equipment...

4.2. PROCESS DESCRIPTION

MEK manufacturing process is mainly composed of 3 blocks; vaporization, reaction and separation, which are represented in the PFD diagram on page 18.

4.2.1. Vaporization block

The aim of this block is that the input stream to the reactor is in the vapor phase and at 240°C. For this purpose, a mixer and several heat exchangers are used, which will be described below.

First, SBA at 25 °C is fed to the mixer M-1 together with the bottoms stream coming from the distillation column. In this way, an output stream consisting of SBA and very little MEK is obtained at a temperature of 29 °C, which is then pumped to a pressure of 12 atm using the P-1 pump. This stream is heated to the mixture boiling temperature (192 °C) in the HE-1 heat exchanger, using the reactor outlet stream as heating fluid.

Then, the cold output stream from HE-1 enters the heat exchanger HE-2 in which a phase change takes place, where the mixture of SBA and MEK leaves the equipment at the reaction temperature (240 °C) .

4.2.2. Reaction block

This block is very simple and consists only of the fixed-bed reactor R-1.

The reactor has a feed stream at 12 atm consisting of the vapor mixture from the heat exchanger HE-2 and an output stream at 10 atm consisting of MEK, hydrogen and unreacted SBA.

4.2.2.1. Reaction

The reactor R-1 carries out the dehydrogenation reaction of SBA at a temperature of 240 °C, a conversion rate of 94.2% and a MEK selectivity of 99.9% [10]:



As the reaction is endothermic ($\Delta H=51$ kJ/mol), a heating fluid is needed to circulate through the casing to provide the necessary heat demanded by the reaction. A thermal oil has been used as the heating fluid, as steam requires extremely high pressure conditions.

4.2.2.2. Catalyst

The catalyst that has been used is CuO/SiO₂, i.e. a copper catalyst supported on silica. It was chosen because of its improved activity, selectivity and stability compared to other catalysts.

Various processes carry out the preparation of the catalyst by co-precipitation techniques. However, in this case, the catalyst used has been prepared by the sol gel method, as it provides an optimal surface area and a good dispersion of the active metals.

The main characteristics of the catalyst are shown in Table 5 [10].

Table 5. Main properties of the CuO/SiO₂ catalyst.

Surface area [m ² /g]	Bulk density [g/mL]	Effective pore size [Å]	CuO/SiO ₂ mol ratio	Space velocity [h ⁻¹]
575	0,55	20	0,139	5

4.2.2.3. Reaction kinetics

The kinetics of the reaction were first studied by Perona and Thodos in 1957 [11], who described the stages of the reaction.

The stages are:

1. Absorption of alcohol over two active sites.
2. Decomposition of the alcohol-active site complex to form MEK in the vapour phase and molecular hydrogen adsorbed on the surface of the catalyst.
3. Molecular hydrogen desorption.

Regarding reactions with copper catalysts supported on silica, in the article published by Keuler, Lorenzen and Miachon [12], the effect of temperature, copper percentage and particle size is studied.

The mechanism proposed in the article is a double active site reaction, being this the controlling stage and whose kinetic equation at temperatures between 190 °C and 250 °C is as follows:

$$r'_a = \frac{8.29 \cdot 10^5 \cdot e^{-\frac{6903}{T}} \cdot \left(p_A - \frac{p_B \cdot p_C}{3.538 \cdot 10^9 \cdot e^{-\frac{7100}{T}}} \right)}{\left(1 + 8.804 \cdot 10^5 \cdot e^{-\frac{2298}{T}} \cdot p_B \right)^2} \quad \text{Eq.2}$$

Where sub-indices A, B and C refer to SBA, MEK and hydrogen respectively.

After studying the reaction, the optimum values of the different variables were determined, being those a 15% copper in particles with a diameter between 300 and 850 μm and at temperatures close to 240°C.

Therefore, the choice and conditions chosen for the catalyst are appropriate, as they are close to the optimum values.

In this case, a percentage of 13.9% of copper with respect to silica has been used, so that the optimum value of particle diameter is between 850 and 1180 μm [12].

4.2.3. **Separation block**

The reactor outlet stream is used as a heating fluid through the heat exchanger HE-1 where it is cooled down to a temperature of 120 °C.

Then, the stream enters the heat exchanger HE-3, where the organic compounds leave the heat exchanger in liquid phase and the hydrogen in gas phase, so that the phase separation can be carried out in the separator S-1.

Once they have been separated, the vapor phase, mainly composed of hydrogen is burned for energy, so that it can be used for other processes. The liquid stream containing MEK and SBA is depressurized to atmospheric pressure by the pressure reducing valves V-1 and V-2 and then heated to 81 °C by the heat exchanger HE-4, so that it can be fed to the distillation column C-1.

At the head of the column, a flow of steam with a MEK purity of 98% is obtained, which is passed through a condenser HE-5, so that the current turns into its liquid phase, and subsequently through a T-1 condensate receiver to prevent it from reverting to the vapor phase. From this stream, a part is recirculated to the column and the rest is sent to the heat exchanger HE-6 to be cooled down to 25°C and stored in the tank T-2, ready for MEK trading.

Finally, at the bottom of the column, a liquid stream is obtained which is passed through the reboiler HE-7 and, again, one part is recirculated to the column and the other part is pumped by the pump P-3 to feed the M-1 mixer. Theoretically, this stream should be purged so that by-products do not accumulate in the reactor, however, since the formation of by-products is very small, this has been ignored.

4.3. SERVICES

The auxiliary services used in the process, which are supplied outside the battery limit, are described below.

4.3.1. Electricity

The electricity required for the operation of the equipment and lighting is obtained from the high-voltage power grid of up to 125 000 V. This current passes through transformers to reach the factory.

The process equipment is three-phase, so it will work at 380 V.

4.3.2. Cooling water

The cooling fluid used is induced draught tower water, which enters the equipment at 30°C and is returned to a cooling column.

4.3.3. Water-ethylene glycol 30%

For cases where cooling below the cooling water temperature is desired, a water-ethylene glycol mixture with 30% ethylene glycol (EG) is used. The mixture will enter the equipment at a temperature of 10 °C [13].

4.3.4. Steam

Low pressure steam (3 atm; 134°C), which is generated in a pyro-tube boiler, has been used to vaporize the column inlet stream and for the column's reboiler.

4.3.5. Thermal oil

It has been used in cases where the temperature causes the water vapor to require very high pressures. The oil used is supplied at 260 °C by means of a natural gas burning cooker.

5. PROCESS BASIC DESIGN

This chapter will show the results of the calculations used to design the process equipment and, in addition, the control implemented in the process will be shown in detail.

5.1. DESIGN OF PROCESS EQUIPMENT

Detailed calculations for this chapter are provided in the respective annexes of each chapter.

5.1.1. Reactor

The reactor that has been implemented in the process is a multitube fixed-bed reactor in horizontal arrangement and working in a non-adiabatic way. The choice of the arrangement has been made in order to minimise pressure loss, thus increasing the passage area per unit volume of the bed.

Detailed calculations of the reactor design will be shown in Appendix 1.

5.1.1.1. Reaction feed and products

Table 6 shows the main characteristics of the reactor feed and output streams.

Table 6. Operating conditions of the reactor streams

Stream	P [atm]	T [°C]	Mass flow [kg/h]			
			Total	SBA	MEK	H ₂
Inlet	12	240	3461	3448	13	0.00
Outlet	10	240	3461	200	3173	88

5.1.1.2. Catalyst

It is located inside the tubes, so the feed stream will flow through the tubes as well.

The catalyst particle has been considered to have a cylindrical shape with a particle diameter to particle length ratio of 1:5. Following the optimums studied by Keuler [12], a d_p of 1180 μm and a L_p of 5900 μm have been used, so the effective particle diameter is 1609.1 μm [14].

To know the sphericity of the particle, it is necessary to make the quotient between d_p and the equivalent sphere diameter, which is calculated with Eq.3.

$$d_{sph} = \sqrt[3]{\frac{6 \cdot V_p}{\pi}} \quad \text{Eq.3}$$

Thus, a sphericity of 0.51 is obtained.

Knowing all the properties, all that remains is to know the required amount of catalyst, which is calculated as the quotient between the feed rate of the limiting reactant and the space velocity of the catalyst. This gives 681.92 required kilograms of copper catalyst supported on silica.

The catalyst calculations are summarised in Table 7.

Table 7. Catalyst design specifications.

W [kg cat]	d_p [μm]	L_p [μm]	V_p [m^3]	d_p' [μm]	d_{sph} [μm]	Φ
681.92	1180.00	5900.00	$6.45 \cdot 10^{-9}$	1609.10	2309.78	0.51

5.1.1.3. Bed

To know the porosity of the bed only the sphericity of the particle is needed, so Eq.4 gives a porosity of 0.64.

$$\varepsilon_L = 0.9 - \frac{\Phi}{2} \quad \text{Eq.4}$$

Knowing the mass of catalyst required and the bulk density of the bed, the reactor bed volume is 1.24 m^3 , and therefore the residence time is 2.3 s.

5.1.1.4. Tubes design

Commercial steel pipes were used, so that, following the dimensions according to EN 10 210-2 [15], an outer diameter, an internal diameter and a pipe thickness of 42.4 mm, 34.8 mm and 3.8 mm respectively have been used. However, it must be considered that the catalytic converter occupies a part of the tube, so by knowing the porosity of the bed, a net internal diameter of 27.9 mm is obtained.

The configuration of the tubes used is a 30° triangular arrangement, as it favors the heat transfer. In addition, it should be noted that the fluid has been passed through the tubes twice.

To determine the number of tubes, it has been necessary to iterate until a pressure loss of 2 atm is found, so that the reactor outlet current is at 10 atm. Assuming the number of tubes and considering that they are all identical, the amount of catalyst in each tube can be found and then the length of each tube by Eq.5.

$$L_t = \frac{4W_t}{\rho_{LB} \cdot \pi \cdot D_o^2} \quad \text{Eq.5}$$

Finally, to calculate the pressure loss (Eq.7), it is necessary to know the Reynolds number with respect to the effective particle diameter and the friction factor calculated by Ergun's correlation (Eq.6) [14].

$$f = \frac{1 - \varepsilon_L}{\varepsilon_L^3} \left(1.75 + 150 \frac{(1 - \varepsilon_L)}{Re'} \right) \quad \text{Eq.6}$$

$$\frac{(-\Delta P)}{L_t} = \frac{f \cdot u_s^2 \cdot \rho_f}{d_p'} \quad \text{Eq.7}$$

Thus, the result of the tubes design are shown in Table 8 and Table 9.

Table 8. Reactor tubes sizing.

D _o [mm]	d _{int} [mm]	t _t [mm]	d _{int,net} [mm]	L _t [m]	A _t [m ²]	V _t [m ³]
42.40	34.80	3.80	27.90	2.80	0.38	3.95 · 10 ⁻³

Table 9. Reactor tubes specifications.

N_t	Number of passes	Arrangement	W_t [kg cat/tube]	u_s [m/s]	Re'	$-\Delta P$ [atm]
314	2	Triangular 30°	2.17	5.54	17200.62	2.00

5.1.1.5. Reactor design

Knowing the arrangement of the tubes, and the number of times the stream passes through them, the shell diameter is obtained from Eq.8 [16].

$$D_s = 0.637 \sqrt{\frac{CL}{CTP}} \cdot \sqrt{\frac{A \cdot \left(\frac{Pt}{D_o}\right)^2 \cdot D_o}{L_t}} \quad \text{Eq.8}$$

With Pitch being 1.25 times the outer diameter of the tubes, the diameter of the shell 1.05 m. Single segmental baffles have been used, and in order to calculate how many are needed, a spacing of 0.5 times the diameter of the shell has been considered, so a total of 5 baffles have been obtained using Eq.9 [16].

$$N_B = \frac{L_t}{B} - 1 \quad \text{Eq.9}$$

As for the baffle cut, the heuristics have been followed and it has been chosen to be 0.225 times of the casing diameter [17]. Figure 5 shows a schematic of the baffles used.

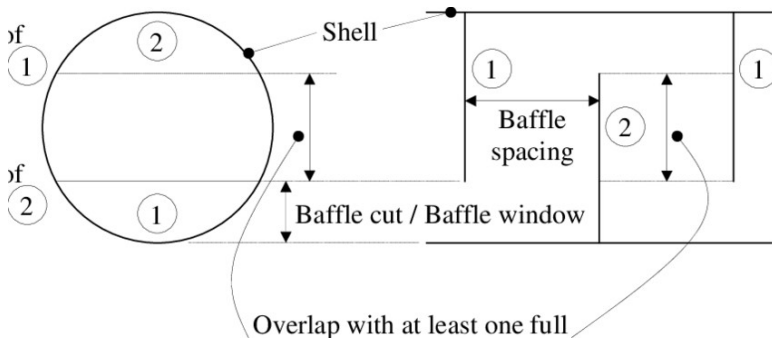


Figure 5. Schematic configuration of single segmental baffles.

Finally, knowing the dimensions of the tubes and the shell, the reactor can be dimensioned and thus the residence time in the shell can be calculated. The most important data are summarized in Table 10.

Table 10. Main reactor specifications.

Number of baffles	5
Baffle spacing [m]	0.52
Baffle cut [m]	0.23
Shell diameter [m]	1.05
Shell thickness [mm] [18]	6.4
Reactor length [m]	2.80
L/D_s	2.69
Heat transfer area [m²]	116.97
Reactor volume [m³]	2.39
Shell residence time [s]	57.31

The volume of the reactor is the volume necessary to obtain a conversion of 0.942. However, it cannot be guaranteed that the reactor will operate at 100% efficiency, so it has been oversized by 20% [19], thus obtaining a volume of 2.86 m³, so that if the reactor operates with a lower than expected performance, the desired conversion can be guaranteed.

As for the L/D_s ratio and the velocity in the tubes, values lower than those of the heuristics have been obtained. Nevertheless, improving these values mean increasing the pressure loss, so the priority has been to minimize the pressure loss.

5.1.1.6. Heating fluid

As mentioned before, the operation does not work adiabatically, so it will be necessary to supply a heating fluid to control the temperature of the reactor (240 °C), which will circulate through the shell.

The heating fluid used in this case is the mineral oil PIROBLOC HTF-MINERAL [20], as it is an economical oil with a very good stability, excellent resistance to oxidation and a high maximum operating temperature.

By simulating the process in ASPEN PLUS V11, a heat of 712.18 kW is obtained, so knowing that the oil enters at 260 °C, and having calculated the number of tubes and their area, it is obtained that the oil leaves at a temperature of 251.27 °C. The oil's most important physical properties are shown in Table 11 [21] [22].

Table 11. PIROBLOC HTF-MINERAL physical properties at operating temperatures.

T [°C]	Density [kg/m ³]	Specific heat [kJ/kg·K]	Thermal conductivity [kJ/m·s·K]
251	724	2.72	1.16·10 ⁻⁴
260	715	2.76	1.21·10 ⁻⁴

Finally, it is necessary to supply 30 kg/s of thermal oil to control the reactor temperature.

5.1.1.7. Headers

The reactor headers are torispheric due to their low cost and frequent use in industry. As the reactor works at pressures of 10 and 12 atm, the headers will be of the Korboggen type [23].

5.1.1.8. Material

With the exception of the tubes, which are made of commercial steel and the insulation that is made of glass wool, the rest of the reactor is made of AISI 316 stainless steel, as it is suitable for vessels working at high pressures [24], can withstand very high temperatures and has excellent corrosion resistance [25].

5.1.2. Distillation column

Since the boiling temperatures of MEK (79.6°C) and SBA (99.0°C) are very different, the separation of MEK and SBA can be carried out relatively easily using a distillation column. The contact will be made in stages, so a column of trays will be used.

Detailed calculations of the column design will be shown in Appendix 3.

5.1.2.1. Trays

There are multiple types of trays in the industry. However, the most commonly used are the following [26]:

- Bubble cap trays: It has a riser or chimney installed above each orifice and a plug covering the riser. The plug is mounted so that there is a space between the riser and the plug to allow steam to pass through. The vapor rises up the chimney and is directed down through the cap. eventually discharging through the slots in the cap, and finally bubbling through the liquid in the tray.
- Sieve trays: Sieve trays are basically metal plates with holes. The liquid flows through the pan and down through the downstream overflow.
- Valve trays: Perforations are covered by liftable caps. Vapor flows lift the caps, thus self-creating a flow area for the passage of vapor. The lifting cap directs the vapor to flow horizontally into the liquid.

Sieve trays have been used because of their low cost and the fact that their construction is easier, requiring the drilling of small holes in the tray.

Once the type of plates used by the column has been decided, the internal parameters are optimized following section 5.1.2.2. process.

Thus, the column will have trays that will each have 391 holes of 12.7 mm diameter. Tray thickness and hole diameter are related by a coefficient of 0.43. Therefore, the thickness of the tray has a value of 6 mm.

5.1.2.2. Simulation and optimization of the column

In order to design this column, ASPEN PLUS V11 has been used, in which the following specifications were introduced:

- The feed stream to the column, which comes from the vapor-liquid separator, has a flow rate of 3338 kg/h and is composed of 94% MEK and 6% SBA.
- Per column head, 3189 kg/h are obtained with a purity of 98% MEK.
- In terms of pressure, the column works at 1 atm and with a pressure drop of $6 \cdot 10^{-3}$ atm, as a pressure gradient is necessary for the fluid to flow upwards.

The column then, has been optimized by means of a sensitivity analysis in which, for a certain number of stages, the feed tray with the lowest reflux ratio (RR) has been searched for.

Once the optimal feed tray is found, RR is plotted against the number of stages. The number of stages where RR is at or near the minimum value is selected, since the fewer stages the column has, the fewer trays are needed and therefore the column will be more economical.

Finally, it is checked that the feed tray is again the optimal one for the column with different number of stages. Table 12 and Table 13 show the data obtained from the column optimization.

Table 12. Colum streams results.

Stream	Feed	Distillate	Bottoms
Flow [kg/h]	3337.61	3188.78	148.83
MEK %	94	98	9
SBA %	6	2	91
T [°C]	81.0	80.4	97.6

Table 13. Optimized column data.

Number of stages	Feed stage	Reflux ratio
14	4	0.763

The column has 14 theoretical stages, which corresponds to 12 theoretical trays, as it has a total condenser and a reboiler.

5.1.2.3. Number of trays

Once the theoretical stages are known, the real number of stages are calculated using the Bakowski equation (Eq.10).

$$E_o = \frac{1}{1 + \frac{3.7 \cdot 10^4 \cdot K \cdot M}{h' \cdot \rho_L \cdot T}} \quad \text{Eq. 10}$$

An efficiency of 67.0% is obtained, which is reasonable, since the tray's efficiency for hydrocarbon distillation is between (60 – 90)%. [27]

By knowing the efficiency, the actual number of trays can be known.

$$E_o = \frac{\text{Theoretical trays}}{\text{Real trays}} \quad \text{Eq. 11}$$

Thus, the column has 18 trays.

5.1.2.4. Column sizing

Section and diameter

For the calculation of the column diameter, the optimum velocity (Eq. 12) at which the steam flows through the plates has first been calculated.

$$u_{opt} = K_f \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} \quad \text{Eq. 12}$$

It should be noted that the lowest liquid density has been used, as the denser the fluid is, the more it tends to descend. K_f is the flooding constant, which depends on the distance between trays and the height of the liquid seal of the trays (Table 14).

Table 14. Approximate values of the K_f [28].

Distance between trays [cm]	Approximate K_f values [m/s]		
	Height of the liquid seal on the trays [mm]		
	12.5	25	50
15	0.006-0.012	-	-
30	0.028-0.035	0.020-0.028	0.015-0.020
60	0.055	0.050	0.048
90	0.061	0.059	0.057

A flooding constant of 0.028 m/s has been chosen. Since a distance between trays of 30 cm and a liquid height of 25 mm has been used. Thus, the optimum velocity has a value of 0.48 m/s.

Knowing the optimum velocity and the volumetric flow rate of distillate, the net area and diameter of the column can be obtained.

Although the steam only passes through the trays, they do not occupy the entire area of the column. So the total area takes into account the vapor passage area (net area) and the liquid overflow area, which occupies 8.8% of the total area. Thus, the diameters and areas obtained are shown in Table 15.

Table 15. Column dimensions

Net area [m ²]	Net diameter [m]	Total area [m ²]	Total diameter [m]
0.571	0.85	0.619	0.89

Although the most common tray spacing is 60 cm, 30.5 cm (12 inches) has been used to meet the heuristics shown in Table 16.

Table 16. Plate spacing heuristics.

Diameter [ft]	Tray spacing [inches]
< 3	6-12
3-5	18-24
5-6	24-30
6-12	30-36
12-24	36-48

The diameter of the column has a value of 2.91 feet, so the tray spacing used is correct.

Height

The 18-tray column will have a height of 5.48 m, as a tray spacing of 0.3048 m has been used. In addition, 1 m is required at both the head and end of the column to avoid possible flooding. Therefore, the total height of the column is 7.48m and the height/diameter relation (h/d) has a value of 8.16.

5.1.2.5. Accessories

Column headers

The column headers are torispheric due to their low cost and frequent use in industry. If none of the following conditions are met, Klopper type headers are generally used [23]:

- Design temperature above 350°C.
- Design pressure equal to or higher than 7 bar.
- Height/diameter ratio greater than 10.

Since none of the conditions are fulfilled, Klopper headers are a good choice.

Manholes

Three manholes of a 20 inches inside diameter have been implemented for the maintenance and cleaning of the column. These have been placed above the upper plate and below the lower plate and the feed plate [23].

Support

A cylindrical skirt has been chosen as a support, as the height/diameter ratio is greater than 5 and, being a column with a certain height, it is possible that vibrations may occur [23].

Stairs

The columns are equipped with a cat staircase with 40 cm wide steps and 30 cm apart with a protective cage, as the column is over 6 m long.

The stairs are equipped with several landing platforms located at the height of the manholes. The platforms are 1 meter wide and have a 1 meter high safety barrier [23].

5.1.2.6. Material

Table 17 shows the materials used for every part of the distillation column.

Table 17. Distillation column materials.

Column	Trays	Column heads	Manholes	Skirt	Cat staircase	Insulation
Carbon steel	Stainless steel 304	Carbon steel	Carbon steel	Carbon steel	Carbon steel	Glass wool

Generally, carbon steel has been used because of its high strength and hardness. However, the plates are in direct contact with fluids, so 304 stainless steel has been used, as it is more resistant to corrosion [29].

As for the insulation, glass wool has been used as it has a very good ratio between thermal resistance and economic viability.

5.1.3. Heat exchangers

The process has indirect heat exchangers, i.e. the hot and cold fluid are separated by a wall through which heat is transferred. Specifically, shell and tube heat exchangers have been used, as they are the most commonly used in the chemical industry due to their wide range of pressures and temperatures.

As for the fluids, they are made to flow in countercurrent and with a 1-2 configuration, i.e. 1 pass through the shell and 2 passes through the tubes. The different exchangers are presented in Table 18.

Table 18. Process heat exchangers description.

Nomenclature	Type	Cold fluid	Hot fluid
HE-1	Heater	Mixer outlet stream (SBA-MEK)	Reactor outlet stream (MEK-SBA-H ₂)
HE-2	Heater	HE-1 cold fluid outlet stream (SBA-MEK)	Thermal oil
HE-3	Cooler	Water – 30% ethylene glycol	HE-1 hot fluid outlet stream (MEK-SBA-H ₂)
HE-4	Reboiler	Separator liquid phase (MEK-SBA)	Low pressure steam
HE-5	Condenser	Cooling water	Column distillate stream (MEK-SBA)
HE-6	Cooler	Water – 30% ethylene glycol	MEK 98% purity
HE-7	Reboiler	Column bottoms stream (SBA-MEK)	Low pressure steam

The heat exchangers have been simulated using ASPEN PLUS V11, from which the heat has been obtained. Detailed calculations of design will be shown in Appendix 3.

5.1.3.1. Configuration

The Tubular Exchanger Manufacturers Association (TEMA) standards [30] have been followed for the choice of heads and casing. Table 19 shows the configurations used for each exchanger.

Table 19. Heat exchangers configuration.

Nomenclature	HE-1	HE-2	HE-3	HE-4	HE-5	HE-6	HE-7
Configuration	BEM	BEM	BEM	BKU	BEM	BEM	BKU

The distribution head type B has been chosen, as it is simple and economical for clean fluids. As for the shell, type E has been used, as it is the most common single-pass type and the easiest to seal the baffle panels. Finally, the type M return header has been used, as the temperature difference does not exceed 50 °C.

It can be seen that this configuration has been used for all the exchangers with the exception of the reboilers, which have a K-type casing, since it has a large space for the separation of the steam, and a U-type return header, since the tubes inside are arranged in a U-shape.

5.1.3.2. Heat exchangers design

The heat exchangers have been simulated using ASPEN PLUS V11, from which the heat has been obtained. Thus, knowing the temperatures of the streams and their overall heat transfer coefficient [31], the heat transfer area is obtained.

As for the tubes, AISI 304 stainless steel tubes have been used [32], the dimensions of which have been adjusted to obtain a good D_s/L ratio (between 0.066 and 0.2) and a velocity in accordance with the heuristics. This velocity should be 0.5 to 3 m/s for liquids, and in the order of 30 m/s for gases [16].

Once the dimensions have been decided, the number of tubes is obtained and Eq.13 gives the tube bundle diameter (d_b).

$$N_t = a \left(\frac{d_b}{d_o} \right)^b \quad \text{Ec.13}$$

Where a and b are coefficients that depend on the number of passes per tube and the arrangement of the passes per tube, which is squared for all. Thus, knowing the diameter of the tube bundle, the diameter of the shell [33] and ,consequently its thickness can be obtained [18].

Finally, by the same process as for the reactor, the number of baffles is obtained, together with their spacing and height.

The heat exchangers specifications are shown below in Table 20 and Table 21.

Table 20. Heat exchangers general and tubes specifications.

HE #	Q [kW]	A [m ²]	L [m]	d _o [mm]	d _{int} [mm]	t _t [mm]	N _t	u _s [m/s]	Fluid state
HE-1	469.68	34.99	3.96	15.87	10.33	2.77	178	13.96	Vapor-liquid
HE-2	473.33	32.73	3.05	19.05	13.51	2.77	180	2.18	Liquid
HE-3	-413.73	19.71	3.96	25.40	19.86	2.77	63	0.55	Liquid
HE-4	339.94	5.83	1.83	12.70	10.22	1.59	80	32.76	Vapor
HE-5	-704.92	28.07	3.05	25.40	21.18	2.11	116	29.92	Vapor
HE-6	-112.90	11.31	3.96	12.70	8.48	2.11	72	0.54	Liquid
HE-7	705.39	25.93	3.05	15.87	10.33	2.77	171	26.33	Vapor

Table 21. Heat exchangers shell specifications.

HE #	Pitch [mm]	d _b [m]	D _s [m]	t _s [mm]	D _s /L	N baffles	B [m]	B _c [m]
HE-1	19.83	0.34	0.35	3.2	0.089	22	0,18	0.080
HE-2	23.81	0.41	0.43	3.2	0.140	14	0.21	0.096
HE-3	31.75	0.35	0.36	3.2	0.091	22	0.18	0.081
HE-4	15.88	0.19	0.20	3.2	0.110	17	0.10	0.046
HE-5	31.75	0.46	0.47	3.2	0.153	13	0.23	0.105
HE-6	15.88	0.46	0.47	3.2	0.118	16	0.23	0.105
HE-7	19.83	0.34	0.35	3.2	0.114	17	0.17	0.078

As it can be seen, the exchangers are within the permissible ranges for both the flow velocity in the tubes and the ratio of shell diameter to tube length.

5.1.3.3. Material

The material used for both the tubes and the casing is stainless steel AISI 304, due to its good corrosion resistance, its economic price and the fact that it allows a wide range of working temperatures. As for the insulation, glass wool has been used.

5.1.4. Mixer

Since the mixture to be treated is liquid, a mixing tank has been used [34]. The most important data of the streams are shown in Table 22.

Table 22. Operating conditions and properties of the mixer streams.

Stream	P [atm]	T [°C]	q [m ³ /h]	ρ [kg/m ³]	Mass flow [kg/h]			
					Total	SBA	MEK	H ₂
SBA Feed	1	25.0	4.12	803.24	3312.69	3312.69	0.00	0.00
Recirculation	2	97.8	0.21	722.11	148.83	135.57	13.26	0.00
Mixture	1	28.3	4.33	799.86	3461.52	3448.26	13.26	0.00

A residence time of 5 minutes has been assumed, so knowing the volumetric flow rate, a minimum capacity of 0.38 m³ is required. Since the turbulence of the streams makes them mix easily, there is no need for a mixer with a large volume, therefore, a 500 L stainless steel industrial AISI 304 tank mixer has been used (Figure 6).



Figure 6. Agitator mixing tank [35].

5.1.5. Separator

The vapor-liquid separator is designed in such a way that hydrogen can be separated while losing as little MEK as possible. The operating conditions to achieve this are shown in Table 23.

Table 23. Operating conditions and properties of the separator streams.

Stream	P [atm]	T [°C]	q [m ³ /h]	ρ [kg/m ³]	Mass flow [kg/h]			
					Total	SBA	MEK	H ₂
Feed	10	25	112.85	30.66	3461.52	200.00	3173.18	88.34
Vapor	10	25	108.72	1.14	123.93	0.65	34.94	88.34
Liquid	10	25	4.13	808.31	3337.59	199.35	3138.24	0.00

In terms of sizing, the heuristics have been followed and a 0.5 m diameter separator with a height of 2 m and a thickness of 3.2 mm [18] has been designed. This is due to the fact that by having a large diameter, the velocity decreases and, therefore, phase separation is favored.

The headers are torispheric due to their low cost and frequent use in industry. As the separator works at pressures higher than 7 bar, the headers will be of the Korboggen type [23].

In terms of materials, AISI 304 stainless steel has been used for the entire separator.

5.1.6. Tanks

Cylindrical pressure tanks have been used [36], specifically, a horizontal tank for the T-1 tank and a vertical tank for the T-2 tank. Detailed calculations of the tanks design will be shown in Appendix 4.

A residence time has been assumed for each tank. In the case of the condensate tank T-1, the only interest is that the liquids do not return to their vapor state, so a residence time of 12 min has been chosen. However, with tank T-2, a residence time of 10 days has been chosen, since it is the final product of the plant and it is of interest to store it for a period of 10 days.

Once the residence times and volumetric flow rates are known, the tank volume can be calculated. It should be noted that this has been oversized by 20%, as this takes into account the volume that the fluid will occupy in the heads [37].

The volume of the T-2 tank is too large, so it has been decided to split it into two tanks with identical characteristics. In addition, for safety and flexibility, it is advisable to use one tank for loading and the other for unloading, instead of feeding and withdrawing product at the same time.

To calculate the dimensions, a h/D ratio of 1.25 [38] has been used for tank T-1, whereas a ratio of 2 has been used for tank T-2 as it is a vertical vessel. Thus, knowing that the tanks are cylindrical, both the height and the diameter can be obtained.

As mentioned above, the tank has been oversized so that the liquid does not overflow, in this case, the limit is 80%. This way, the liquid level and therefore the liquid pressure can be known. Thus, the most important data of the tanks are shown in Table 24.

Table 24. Tanks main specifications.

Tank #	q [m ³ /h]	t [h]	V [m ³]	h [m]	D [m]	h_{liq} [m]	P_{liq} [atm]
T-1	7.24	0.2	1.74	1.51	1.21	1.21	1.09
T-2a	3.96	240	570.38	14.27	7.13	11.41	1.89
T-2b	3.96	240	570.38	14.27	7.13	11.41	1.89

The headers will be torispherical Kloppe type as none of the three conditions mentioned above are met and, as far as the materials are concerned, AISI 304 stainless steel has been used.

5.1.7. Pumps

They have been used to pump fluids, mainly to reach certain heights, i.e. to overcome the head loss (Δh), as the process is designed for fluids to move by gravity. The impulsion pumps that have been used are all centrifugal, as they are the most common in the industry, easy to maintain and the most efficient.

The pumps have been simulated using ASPEN PLUS V11 with an efficiency of 75% and the data shown in Table 25 have been obtained.

Table 25. Pumps specifications.

Nomenclature	$(-\Delta P)$ [atm]	q [m ³ /h]	Power [HP]	NPSH available [m]
P-1	11.00	4.33	1.797	12.422
P-2	1.00	3.24	0.122	0.130
P-3	0.99	0.21	$7.77 \cdot 10^{-3}$	$8.09 \cdot 10^{-3}$

As for the available NPSH, it shall be higher than the required NPSH, as this avoids cavitation problems. The pump model used is the CPK-SY (Figure 7), a cast steel pump often used in the chemical industry that can work at high temperatures.



Figure 7. Centrifugal pump CPK-SY [39].

5.1.8. Pressure reducing valves

Since the pressure had to be reduced from 10 atm to atmospheric pressure, two pressure reducing valves have been used instead of one, as the pressure ratio 1:10 is high. Thus, it has been halved, which makes it easier to find a suitable valve model.

The model used for the pressure reducing valves will be specified in section 5.2.3.

5.2. CONTROL OF THE PROCESS

A process control system has been implemented in order to ensure a stable and safe operation of the plant, thus avoiding possible disturbances in the installation. For this purpose, a feedback control system has been proposed, whose corrective action is based on the error, i.e. the

difference between the actual value and the setpoint value of the controlled variable. The P&ID diagram will be shown in the Appendix 5.

5.2.1. Elements of a control system

As mentioned above, the control loops will be feedback loops, due to their simplicity and the fact that they are cheaper. The control loops consist mainly of the following elements:

- Sensor: It is used to measure the variable to be controlled.
- Transmitter: It translates the signal generated by the sensor and sends it to the controller.
- Controller: It receives the signal from the transmitter and compares the input value with the set point. Finally, it calculates the deviation and sends an output to the final control element in order to adjust the values.
- Final control element: It is used to produce a change in the controlled variable. The final control element used is the pneumatically actuated control valves. Such a valve performs the control action by regulating a flow rate.

In addition, alarms have been used for when the deviation of the variable to be controlled causes it to be in a value outside the allowed range.

5.2.2. Equipment control [40]

In this section, the control implemented in the equipment of the process is explained, describing both its loops and additional elements.

5.2.2.1. Heat exchangers

- Loops 101, 102 and 106: They are located in the HE-1, HE-2 and HE-4 heat exchangers respectively and measure the temperature of the cold fluid outlet stream by regulating the heating fluid flow rate.
- Loops 104, 108 and 112: These are located in the HE-3, HE-5 and HE-6 heat exchangers and measure the temperature of the hot fluid outlet stream by regulating the cooling fluid flow rate.
- Loop 113: It is located in the heat exchanger HE-7 and it measures the reboiler liquid level by manipulating the outlet flow to the mixer.

5.2.2.2. Reactor

Loop 103 is located in the R-1 reactor, which measures the temperature of the outlet stream by regulating the thermal oil feed flow.

With regard to the reactor pressure, instead of establishing a control loop, a pressure indicator and a system of high and low pressure alarms have been implemented to be able to act against disturbances and thus avoid possible overpressure on the walls that could cause the equipment explosion.

5.2.2.3. Separator

If the liquid drain is not able to handle the volume of liquid being collected, the separator will not function properly as it needs an air gap to be effective. Therefore, the level loop 107 has been implemented, which measures the liquid level by regulating the liquid flow rate of the outlet stream.

5.2.2.4. Distillation column

- Loop 107: the distillate flow rate is controlled by regulating the pressure at the head of the column, so that the higher the pressure, the higher the flow rate extracted.
- Loop 110: The current that is recirculated through column head C-1 is affected by a flow control loop, which consists of establishing total reflux during start-up and, once the column has stabilized, the required reflux ratio is imposed, in this case 0.763.
- Loop 112: The column temperature is controlled by the heating vapor flow rate of the HE-7 reboiler, so that the higher the flow rate, the more evaporated liquid is recirculated to the column, thus increasing the column temperature. It should be noted that the temperature will be controlled on plate number 7, since ASPEN PLUS V11 has detected that this is where it varies most sharply, so that if there is a variation, it will be detected on plate 7 and the disturbance will be corrected quickly.

In addition, a level indicator with respective high and low level alarms has been implemented to be able to react to disturbances and to avoid possible flooding of the column.

5.2.2.5. Tanks

Loop 109 has been implemented in the T-1 condensate tank, in which the liquid level is controlled by regulating the venting of gases, i.e. to fill the tank, any air present is evacuated and, once the tank is full, the vent valve is closed.

As for the T-2 tank, whose function is to store the final product, a control loop has not been implemented, but as in other cases, an indicator and a system of high and low level alarms have been implemented, so that it can act in the event of disturbances and thus prevent the liquid from exceeding the 80% established in its design.

5.2.2.6. Pumps

As control elements, only a pressure sensor has been implemented at the outlet of the pumps. However, preventive measures have been taken at the pumps, which will be detailed in section 5.2.4.

The control loops of the different process equipment are summarized in Table 26 and Table 27.

Table 26. Description of control loops 101-105.

Loop number	Location	Variable to control	Measured variable	Manipulated variable	Final control element
101	Heat exchanger HE-1	Cold fluid outlet temperature	Cold fluid outlet temperature	Heating fluid flow rate	Valve V-1008
102	Heat exchanger HE-2	Cold fluid outlet temperature	Cold fluid outlet temperature	Heating fluid flow rate	Valve V-1012
103	Reactor R-1	Reactor outlet stream flow rate	Reactor outlet stream flow rate	Heating fluid flow rate	Valve V-1016
104	Heat exchanger HE-3	Hot fluid outlet temperature	Hot fluid outlet temperature	Cooling fluid flow rate	Valve V-1020
105	Separator S-1	Liquid level	Liquid level	Outlet liquid flow rate	Valve V-1024

Table 27. Description of control loops 106-113.

Loop number	Location	Variable to control	Measured variable	Manipulated variable	Final control element
106	Heat exchanger HE-4	Cold fluid outlet temperature	Cold fluid outlet temperature	Heating fluid flow rate	Valve V-1028
107	Distillate stream	Distillate stream flow rate	Distillate stream flow rate	Column head pressure	Valve V-1032
108	Heat exchanger HE-5	Hot fluid outlet temperature	Hot fluid outlet temperature	Cooling fluid flow rate	Valve V-1036
109	Condensate tank T-1	Liquid height in the condensate receiver	Liquid height in the condensate receiver	Gases venting	Valve V-1040
110	Reflux stream	Reflux ratio	Recirculated distillate flow rate	Recirculated distillate flow rate	Valve V-1050
111	Heat exchanger HE-6	Hot fluid outlet temperature	Hot fluid outlet temperature	Cooling fluid flow rate	Valve V-1054
112	Column C-1	Column temperature	Column temperature	HE-7 steam flow rate	Valve V-1058
113	Heat exchanger HE-7	Liquid level	Liquid level	Outlet liquid flow rate	Valve V-1068

5.2.3. Items

The different valves and sensors that have been used to control the process will be detailed below.

5.2.3.1. Valves

Industrial valves have been used to allow, regulate or prevent the flow of a fluid through a pipe or machine. There are different types of valves, but only the following have been used [41]:

- Globe valves: These are the valves in charge of variable control and are used to control the flow of the fluid, modifying the pressure drop according to the signal that the controller sends to the valve. The model used is the Neles® valve (Figure 8), a self-acting valve made of stainless steel that can work at very high temperatures.



Figure 8. Neles® globe valve [42].

- Gate valves: They are used to allow or prevent the passage of fluid by opening their gate. The model used is Cast Steel Gate Valve API 600 ANSI Class 150 (Figure 9), a valve with a very high temperature and size range.



Figure 9. Cast Steel Gate Valve API 600 ANSI Class 150 [43] [44].

- Non-return valves: They allow the fluid to flow in one direction only, i.e. the valve is open when the fluid is flowing in the right direction, and when the fluid loses pressure or velocity, the valve closes to prevent the fluid from flowing backwards. The model used is Belgicast BV-05-91 Ruber-Check valve (Figure 10), a manually operated AISI 316 stainless steel and cast iron valve.



Figure 10. Belgicast BV-05-91 Ruber-Check valve [45].

- Pressure reducing valves: They are responsible for reducing the pressure of a stream. The model used is the VPR-6L-36-50-00-00 valve (Figure 11), which is made of AISI 316 stainless steel and can work in a wide temperature range.



Figure 11. VPR-6L-36-50-00 pressure reduction valve [46].

5.2.3.2. Sensors

Various sensors have been used to measure the different process variables; temperature, pressure, level and flow.

The temperature sensors will be of the RTD type, which base their operation on the temperature resistance of the material of which they are composed [47]. In this case, the PT100 (Figure 12) has been chosen; a platinum sensor with high sensitivity and precision that has a wide temperature range (from -200 to 850 °C).



Figure 12. PT100 RTD sensor [47].

As for the pressure sensors, the AEP TP3 amplified industrial pressure sensor (Figure 13) made of stainless steel with IP65 protection has been chosen, which has an amplified signal with a linearity of 0.5% [48] and a wide pressure range from 0.5 bar to 2000 bar.



Figure 13. AEP TP3 pressure sensor [48].

In order to reduce the response time, the level measurement is carried out by means of differential pressure, i.e. the pressure is measured at two points and the control action is carried out according to the difference between these points. The level sensor model Rosemount™ 3051S (Figure 14) has been used, which has a wide temperature range and reduces installation costs, as it eliminates the need for heat tracing and impulse piping.



Figure 14. Rosemount™ 3051S level sensor [49].

Finally, the AISI 304 stainless steel P214 flow sensor (Figure 15) has been used, which works in a wide range of flow rates, from 0.3 to 600 L/min. It has been chosen because of its good accuracy and its frequent use with hydrocarbons.



Figure 15. P214 flow sensor [50].

5.2.4. Prevention measures

In order to minimize risks, preventive measures have been proposed for both valves and pumps [40]. Regarding the valves, a system consisting of four valves has been implemented as shown in Figure 16.

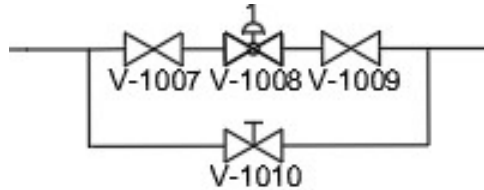


Figure 16. Valves prevention measures.

The automatic regulation globe valve V-1008 is in charge of the control of the variable of interest. On both sides of it, there are the gate valves V-1007 and V-1009 and, in parallel, there is a manual regulation valve V-1010. The advantage of this valve system is that in case the automatic control fails, it can be carried out manually.

For the pumps, a parallel pump system has been implemented (Figure 17) in order to continue the process in case of a pump failure.

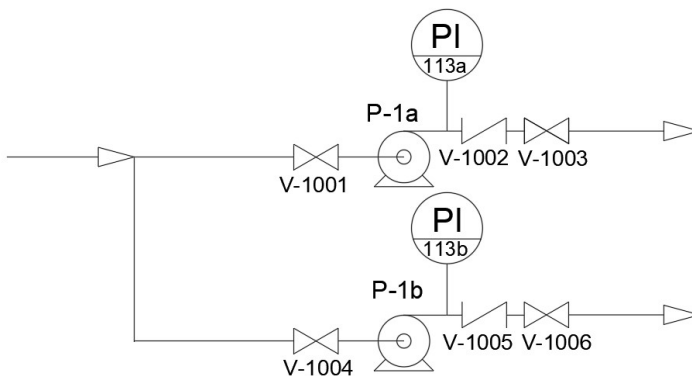
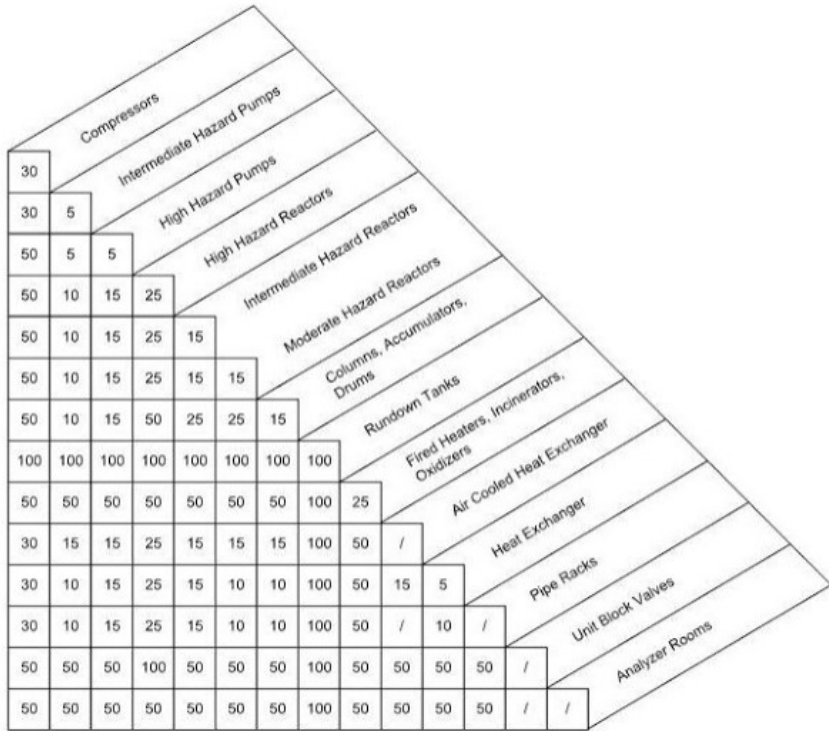


Figure 17. Pumps prevention measures.

The pumps have a gate valve on each side (V-1001, V-1003 for pump P-1a and V-1004, V-1006 for pump P-1b), the purpose of which is to prevent fluid from reaching the pump during pump maintenance. In addition, non-return valves (V-1002 and V-1005) have been used on the discharge to prevent water hammer, i.e. when the fluid flows in the opposite direction due to overpressure, it does not hit the pump, causing a failure.

5.3. EQUIPMENT SEPARATION

Safety distances between equipment are an important aspect to be taken into account, as they can reduce the impact in case of an accident. Figure 18 has been used to determine these safety distances.



1 ft = 0.305 m
 / = no spacing requirements

Figure 18. Intra-unit spacing recommendations [52].

It should be noted that a moderate hazard reactor has been considered, since the reaction components show good chemical stability. Therefore, the safety distances established between process equipment are shown in Table 28.

Table 28. Spacing between process equipment.

Equipment 1	Equipment 2	Spacing [m]
Pump	Pump	1.52
Pump	Heat exchanger	3.05
Pump	Column	3.05
Pump	Reactor	3.05
Pump	Tank	3.05
Heat exchanger	Heat exchanger	1.52
Heat exchanger	Reactor	3.05
Heat exchanger	Column	3.05
Heat exchanger	Tank	3.05
Column	Reactor	7.62
Column	Tank	4.57

6. CONCLUSIONS

After carrying out this project, the conclusions reached are the following:

- Among all the methyl ethyl production processes, the catalytic dehydrogenation of 2 - butanol in the gas phase is the most suitable, since it yields the highest conversion of methyl ethyl ketone.
- The copper catalyst supported on silica is a good choice, as it allows near-total conversions with virtually no by-products.
- In order to carry out production under optimal conditions, the essential equipment includes 6 heat exchangers, 1 fixed bed reactor, 1 phase separator, 1 distillation column and 2 storage tanks.
- The pre-distillation heat exchanger and the mixer are not indispensable, however, they have been used to reduce energy consumption and to exploit the bottoms stream of the column respectively.
- The control system is closed loop, since the output is involved in the control action.
- The process is simple to control, as the control is based solely on feedback loops and indicators with their respective alarms.

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ACRONYMS

A: Heat transfer area [m²]

B: Baffle spacing [m]

B_c: Baffle cut [m]

CL: Tube configuration constant [-]

C_p: Heat capacity [kJ/kg·K]

CTP: Tube counting constant [-]

D: Diameter [m]

d_b: Bundle tube diameter [m]

d_{int}: Inner tube diameter [m]

D_o: Bed diameter/ tubes outer diameter in a multitube reactor [m]

d_o: Outer tube diameter [m]

D_s: Shell diameter [m]

d_{sph}: Equivalent sphere diameter [m]

d_p: Particle diameter [m]

d_p[!]: Effective particle diameter [m]

E_o: Efficiency of the trays [-]

F: Correction factor [-]

f: Friction factor [-]

h: Tank height [m]

h': Effective liquid depth [-]

h_{lq}: Liquid level [m]

- K: MEK equilibrium distribution coefficient [-]
- K_f: Flooding constant [m/s]
- K_p: Equilibrium constant for SBA
- LHSV: Catalyst space velocity [h⁻¹]
- L_p: Particle length [m]
- M_{MEK}: MEK molecular weight [kg/kmol]
- N_B: Number of baffles [-]
- NPSH: Net positive suction head [m]
- N_{pt}: Number of passes per tube [-]
- N_t: Number of tubes [-]
- P: Pressure [atm]
- P_A: SBA partial pressure [atm]
- P_B: MEK partial pressure [atm]
- P_C: H₂ partial pressure [atm]
- P_{liq}: Pressure exerted by the fluid [atm]
- Pt: Pitch [m]
- Q: Heat [kW]
- q: Volumetric flow rate [m³/h]
- q_e: Inlet volumetric flow rate [m³/h]
- r_a': Reaction velocity [mol/L·s]
- Re': Reynolds number with respect to the effective particle diameter [-]
- S: Section [m²]
- S_{net}: Net cross-section [m²]
- T: Temperature [K]
- T₁: Hot fluid inlet temperature [°C]
- T₂: Hot fluid outlet temperature [°C]
- t₁: Cold fluid inlet temperature [°C]
- t₂: Cold fluid outlet temperature [°C]

t_s : Shell thickness [m]

t_t : Tube thickness [m]

U: Overall heat transfer coefficient [$\text{kW}/\text{m}^2\cdot\text{K}$]

u_{opt} : Optimal velocity [m/s]

u_s : Velocity through the tubes [m/s]

V: Volume [m^3]

V_b : Bed volume [m^3]

V_p : Particle volume [m^3]

W: Catalyst mass [kg]

w_{SBA} : SBA mass flow rate [kg/h]

w_{oil} : Oil flow rate [kg/s]

W_t : Catalyst mass per tube [kg]

$(-\Delta P)$: Pressure loss [atm]

ΔT_{ml} : Logarithmic mean temperature difference [K]

ε_L : Bed porosity [-]

λ : Latent heat [kJ/kg]

μ_f : Fluid viscosity [kg/m·s]

ρ_f : Fluid density [kg/m^3]

ρ_L : Liquid density [kg/m^3]

ρ_{LB} : Bed bulk density [kg/m^3]

ρ_{oil} : Oil density [kg/m^3]

ρ_v : Vapor density [kg/m^3]

τ : Tank residence time [s]

τ_b : Bed residence time [s]

τ_s : Shell residence time [s]

Φ : Sphericity [-]

APPENDICES

APPENDIX 1: REACTOR DESIGN

Catalyst particle volume

Considering that the particle is cylindrical:

$$V_p = \pi \cdot \frac{d_p^2}{2} L_p \quad \text{Eq 1.1}$$

V_p : Particle volume [m^3]

L_p : Particle length [m]

d_p : Particle diameter [m]

$$V_p = \pi \cdot \frac{(1.18 \cdot 10^{-3} m)^2}{2} \cdot 5.90 \cdot 10^{-3} m = 6.45 \cdot 10^{-9} m^3$$

Effective particle diameter

$$d'_p = \frac{3 \cdot d_p}{2 + \left(\frac{d_p}{L_p}\right)} \quad \text{Eq 1.2}$$

$$d'_p = \frac{3 \cdot 1.18 \cdot 10^{-3} m}{2 + \left(\frac{1.18 \cdot 10^{-3} m}{5.90 \cdot 10^{-3} m}\right)} = 1.61 \cdot 10^{-3} m$$

Equivalent sphere diameter

$$d_{sph} = \sqrt[3]{\frac{6 \cdot V_p}{\pi}} \quad \text{Eq 1.3}$$

$$d_{sp} = \sqrt[3]{\frac{6 \cdot 6.45 \cdot 10^{-9} m^3}{\pi}} = 2.31 \cdot 10^{-3} m$$

Sphericity

$$\Phi = \frac{d_p}{d_{sph}} \quad \text{Eq 1.4}$$

$$\Phi = \frac{1.18 \cdot 10^{-3} m}{2.31 \cdot 10^{-3} m} = 0.511$$

Bed porosity

$$\varepsilon_L = 0.9 - \frac{\Phi}{2}$$

$$\varepsilon_L = 0.9 - \frac{0.511}{2} = 0.644$$
Eq 1.5

Catalyst mass

$$W = \frac{W_{SBA}}{LHSV}$$
Eq 1.6

W: Catalyst mass [kg]

W_{SBA} : SBA mass flow [kg/h]

LHSV: Catalyst space velocity [h^{-1}]

$$W = \frac{3409.61 \text{ kg/h}}{5 \text{ h}^{-1}} = 681.92 \text{ kg}$$

Bed volume

$$V_b = \frac{W}{\rho_{LB}}$$
Eq 1.7

V_b : Bed volume [m^3]

ρ_{LB} : Bed bulk density [kg/m^3]

$$V = \frac{681.92 \text{ kg}}{550 \text{ kg}/m^3} = 1.24 \text{ m}^3$$

Bed residence time

$$\tau_b = \frac{V_b}{q_e}$$
Eq 1.8

τ_b : Bed residence time [s]

q_e : Inlet volumetric flow rate [m^3/h]

$$\tau_b = \frac{1.24 \text{ m}^3}{1937.95 \frac{m^3}{h} \cdot \frac{1h}{3600s}} = 2.30 \text{ s}$$

Number of tubes

It is necessary to assume a number of tubes in order to perform the rest of the calculations. By means of an iterative process, a total of 314 tubes are obtained.

Tube catalyst mass

$$W_t = \frac{W}{N_t} \quad \text{Eq 1.9}$$

W_t : Catalyst mass per tube [kg/tube]

N_t : Number of tubes [tubes]

$$W_t = \frac{681.92 \text{ kg}}{314 \text{ tubes}} = 2.17 \text{ kg/tube}$$

Bed cross-section

Tubes with an outer diameter of 42.4 mm and a thickness of 3.79 mm have been used.

$$S = \frac{\pi \cdot D_o^2}{4} \quad \text{Eq 1.10}$$

S : Bed cross-section [m^2]

D_o : Tube diameter [m]

$$S = \frac{\pi \cdot (0.0424 \text{ m})^2}{4} = 9.52 \cdot 10^{-4} \text{ m}^2$$

Bed net cross-section

It should be noted that the tube contains a part occupied by the catalyst, so the cross-section will be as follows:

$$S_{net} = S \cdot \varepsilon_L \quad \text{Eq 1.11}$$

$$S_{net} = 9.52 \cdot 10^{-4} \text{ m}^2 \cdot 0.644 = 6.14 \cdot 10^{-4} \text{ m}^2$$

Velocity

$$u_s = \frac{N_{pt} \cdot q_e}{N_t \cdot S_{net}} \quad \text{Eq 1.12}$$

N_{pt} : Number of passes per tube [-]

$$u_s = \frac{2 \cdot 1937.95 \frac{\text{m}^3}{\text{h}} \cdot \frac{1 \text{ h}}{3600 \text{ s}}}{314 \text{ tubes} \cdot 6.14 \cdot 10^{-4} \text{ m}^2} = 5.54 \text{ m/s}$$

Reynolds number

$$Re' = \frac{d'_p \cdot u_s \cdot \rho_f}{\mu_f} \quad \text{Eq 1.13}$$

Re' : Reynolds number defined with respect to effective particle diameter [-]

ρ_f : Fluid density [kg/m³]

μ_f : Fluid viscosity [kg/m·s]

$$Re' = \frac{1.61 \cdot 10^{-3} m \cdot 5.54 \frac{m}{s} \cdot 24.23 \frac{kg}{m^3}}{1.25 \cdot 10^{-5} \frac{kg}{m \cdot s}} = 17200.62$$

Friction factor

$$f = \frac{1 - \varepsilon_L}{\varepsilon_L^3} \left(1.75 + 150 \frac{(1 - \varepsilon_L)}{Re'} \right) \quad Eq 1.14$$

$$f = \frac{1 - 0.644}{0.644^3} \left(1.75 + 150 \frac{(1 - 0.644)}{17200.2} \right) = 2.38$$

Tube length

$$L_t = \frac{4 \cdot W_t}{\rho_L \cdot \pi \cdot D_o^2} \quad Eq 1.15$$

$$L_t = \frac{4 \cdot 2.17 \frac{kg}{tube}}{550 \frac{kg}{m^3} \cdot \pi \cdot (0.03482 m)^2} = 2.80 m$$

Pressure loss

$$(-\Delta P) = \frac{f \cdot u_s^2 \cdot \rho_f}{d_p'} \cdot L_t \quad Eq 1.16$$

$$(-\Delta P) = \frac{2.38 \cdot 5.54 \frac{m}{s} \cdot 24.23 \frac{kg}{m^3}}{1.61 \cdot 10^{-3} m} \cdot 2.80 m \cdot \frac{1 atm}{101325 Pa} = 2.002 atm$$

Heat transfer area

$$A = \pi \cdot N_t \cdot L_t \cdot D_o \quad Eq 1.17$$

$$A = \pi \cdot 314 tubes \cdot 2.80 m \cdot 0.0424 m = 116.97 m^2$$

Oil outlet temperature

$$Q = U \cdot A \cdot \Delta T_{ml} \quad Eq 1.18$$

Q: Heat [kW]

U: Overall heat transfer coefficient [kW/m²·°C]

ΔT_{ml} : Logarithmic mean temperature difference [°C]

The heat is obtained by simulating the reactor in aspen plus V11 and the overall heat transfer coefficient is chosen depending on the system fluids [53]. Thus:

$$\Delta T_{ml} = \frac{712.18 \text{ kW}}{0.4 \frac{\text{kW}}{\text{m}^2 \cdot \text{°C}} \cdot 116.97 \text{ m}^2} = 15.22 \text{ °C}$$

Knowing that the reactor temperature must remain constant at 240°C and the oil is supplied at 260°C, the oil outlet temperature can be obtained.

$$\Delta T_{ml} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln \left(\frac{(T_1 - t_2)}{(T_2 - t_1)} \right)} \quad \text{Eq 1.19}$$

T₁: Oil inlet temperature [°C]

T₂: Oil outlet temperature [°C]

t₁: Fluid inlet temperature [°C]

t₂: Fluid outlet temperature [°C]

$$\Delta T_{ml} = \frac{(260 - 240) \text{ °C} - (T_2 - 240) \text{ °C}}{\ln \left(\frac{(260 - 240) \text{ °C}}{(T_2 - 240) \text{ °C}} \right)} = 15.22 \text{ °C}$$

From Eq 1.18, the oil outlet temperature, T₂, is 251.27 °C.

Oil flow rate

$$Q = w_{oil} \cdot C_p (T_1 - T_2) \quad \text{Eq 1.20}$$

w_{oil}: Oil flow rate [kg/s]

C_p: Heat capacity [kJ/kg·K]

$$w_{oil} = \frac{712.18 \frac{\text{kJ}}{\text{h}}}{2.74 \frac{\text{kJ}}{\text{kg} \cdot \text{K}} \cdot (260 - 251.27) \text{ °C}} = 29.78 \frac{\text{kg}}{\text{s}}$$

Shell diameter

$$D_s = 0.637 \sqrt{\frac{CL}{CTP}} \cdot \sqrt{\frac{A \cdot \left(\frac{Pt}{D_o}\right)^2 \cdot D_o}{L_t}} \quad \text{Eq 1.21}$$

D_s : Shell diameter [m]

P_t : Pitch [m]

CL: Tube configuration constant [-]

CTP: Tube counting constant [-]

Pitch is considered to be 1.25 times the pipe diameter. As for the constants, CI has a value of 0.87 for triangular arrays and CTP has a value of 0.9 for two tube passes.

$$D_s = 0.637 \sqrt{\frac{0.87}{0.9}} \cdot \sqrt{\frac{116.97 \text{ m}^2 \cdot \left(\frac{0.053 \text{ m}}{0.048 \text{ m}}\right)^2 \cdot 0.048 \text{ m}}{2.80 \text{ m}}} = 1.04 \text{ m}$$

Baffles

$$N_B = \frac{L_t}{B} - 1 \quad \text{Eq 1.22}$$

N_b : Number of baffles [-]

B: Baffle spacing [m]

The baffle spacing is 0.5 times the diameter of the shell, so:

$$N_B = \frac{2.80 \text{ m}}{0.52 \text{ m}} - 1 = 4.36 \approx 5 \text{ baffles}$$

Furthermore, the baffle cut has been considered to be 0.225 times the diameter of the shell, so it is 0.23 m.

Reactor volume

Treating the reactor as a cylinder, the volume is calculated by equation 1.22:

$$V = \pi \cdot L_t \cdot \left(\frac{D_s}{2}\right)^2 \quad \text{Eq 1.23}$$

$$V = \pi \cdot 2.80 \text{ m} \cdot \left(\frac{1.04 \text{ m}}{2}\right)^2 = 2.39 \text{ m}^3$$

As the reactor has been oversized by 20%, its actual volume corresponds to 2.86 m³.

Shell residence time

$$\tau_s = \frac{V \cdot \rho_{oil}}{w_{oil}} \quad \text{Eq 1.24}$$

ρ_{oil} : Oil density [kg/m³]

τ_s : Shell residence time [s]

$$\tau_s = \frac{2.86 \text{ m}^3 \cdot 715 \frac{\text{kg}}{\text{m}^3}}{29.78 \frac{\text{kg}}{\text{s}}} = 57.31 \text{ s}$$

APPENDIX 2: DISTILLATION COLUMN DESIGN

Efficiency

$$E_o = \frac{1}{1 + \frac{3.7 \cdot 10^4 \cdot K \cdot M_{MEK}}{h' \cdot \rho_L \cdot T}} \quad Eq\ 2.1$$

E_o : Efficiency

K : MEK equilibrium distribution coefficient [-]

M_{MEK} : MEK molecular weight [kg/kmol]

h' : Effective liquid depth [m]

ρ_L : Liquid density [kg/m³]

T : Temperature [K]

$$E_o = \frac{1}{1 + \frac{3.7 \cdot 10^4 \cdot 1.23 \cdot 72.11 \frac{kg}{kmol}}{0.025\ m \cdot 739.30 \frac{kg}{m^3} \cdot 359.47\ K}} = 0.67$$

Real number of trays

$$E_o = \frac{\text{Theoretical stages} - 2}{\text{Real stages} - 2} \quad Eq\ 2.2$$

$$\text{Real stages} = \frac{14\ \text{stages} - 2 + 2 \cdot 0.67}{0.67} = 19.91\ \text{stages} \approx 20\ \text{stages}$$

So, counting the reboiler and the total condenser, there are 18 real trays.

Optimal velocity

$$u_{opt} = K_f \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} \quad Eq\ 2.3$$

u_{opt} : Optimal velocity [m/s]

K_f : Flooding constant [m/s]

ρ_v : Vapor density [kg/m³]

The flooding constant has been obtained with Table 14 by knowing the height of the liquid seal of the trays and the distance between them.

$$u_{opt} = 0.028 \text{ m/s} \cdot \sqrt{\frac{739.30 \frac{\text{kg}}{\text{m}^3} - 2.55 \frac{\text{kg}}{\text{m}^3}}{2.55 \frac{\text{kg}}{\text{m}^3}}} = 0.476 \text{ m/s}$$

Column diameter

Given the cross-section and net diameter of the column and knowing that the weirs occupy 8.8% of the column, the cross-section of the column is obtained by equation 2.4:

$$S = S_{net} \cdot (1 + 0.088) \quad \text{Eq 2.4}$$

S: Column section [m²]

S_{net} : Column net section [m²]

$$S = 0.571 \text{ m}^2 \cdot (1 + 0.088) = 0.619 \text{ m}^2$$

Thus, taking the column as a cylinder, the diameter of the column is obtained:

$$S = \pi \cdot \left(\frac{D}{2}\right)^2 \quad \text{Eq 2.5}$$

$$D = \sqrt{\frac{4 \cdot 0.619 \text{ m}^2}{\pi}} = 0.889 \text{ m}$$

Column height

For the column height, the number of plates has been multiplied by the distance between the plates and then, the required distance to avoid flooding in the head and bottom of the column has been added. Thus, the column height is 7.486 m.

APPENDIX 3: HEAT EXCHANGERS DESIGN

In this section, calculations will only be performed for the heat exchanger HE-1. The design process of the other heat exchangers will be identical except for a couple of points, which will be discussed.

Cooling/heating fluid flow rate

$$Q = w_f \cdot C_p \cdot (T_{f2} - T_{f1}) \quad \text{Eq 3.1a}$$

w_f : Heating/cooling fluid mass flow [kg/h]

C_p : Heat capacity [kJ/kg·K]

T_{f1} : Inlet temperature of the heating/cooling fluid [°C]

T_{f2} : Outlet temperature of the heating/cooling fluid [°C]

$$w_f = \frac{413.73 \frac{\text{kJ}}{\text{s}}}{3.73 \frac{\text{kJ}}{\text{kg} \cdot \text{K}} \cdot (30 - 10) \text{ } ^\circ\text{C}} = 5.55 \text{ kg/s}$$

In cases where there is a change of state, it is necessary to take into account the latent heat:

$$Q = w_f \cdot \lambda \quad \text{Eq 3.1b}$$

Where λ is the latent heat [kJ/kg].

Heat transfer area

First the correction factor is calculated using Eq 3.2.

$$F = \frac{\left[\ln \left(\frac{1 - P}{1 - P \cdot R} \right) \right] \cdot \sqrt{R^2 + 1}}{\ln \left[\left(\frac{2 - P \cdot (R + 1) - \sqrt{R^2 + 1}}{2 - P \cdot (R + 1) + \sqrt{R^2 + 1}} \right) \right] \cdot ((R - 1))} \quad \text{Eq 3.2}$$

P and R are shown in Eq 3.3 and 3.4.

$$P = \frac{t_2 - t_1}{T_1 - t_1} \quad \text{Eq 3.3}$$

$$R = \frac{T_1 - T_2}{t_2 - t_1} \quad \text{Eq 3.4}$$

T_1 : Hot fluid inlet temperature [°C]

T_2 : Hot fluid outlet temperature [°C]

t_1 : Cold fluid inlet temperature [°C]

t_2 : Cold fluid outlet temperature [°C]

Substituting the data gives a correction factor of 0.965. It should be noted that in heat exchangers where there is a phase change, $F=1$ is considered. The logarithmic mean temperature difference is then calculated:

$$\Delta T_{ml} = \frac{(T_1 - t_2) - (T_2 - t_1)}{\ln\left(\frac{(T_1 - t_2)}{(T_2 - t_1)}\right)} \quad \text{Eq 3.5}$$

$$\Delta T_{ml} = \frac{(120.42 - 30)^\circ\text{C} - (25 - 10)^\circ\text{C}}{\ln\left(\frac{(120.42 - 30)^\circ\text{C}}{(25 - 10)^\circ\text{C}}\right)} = 41.98^\circ\text{C}$$

Finally, the heat transfer area is obtained by means of Eq. 3.6.

$$Q = A \cdot U \cdot F \cdot \Delta T_{ml} \quad \text{Eq 3.6}$$

$$A = \frac{413.73 \text{ kW}}{500 \frac{\text{W}}{\text{m}^2 \cdot ^\circ\text{C}} \cdot 0.965 \cdot 41.98^\circ\text{C}} = 19.71 \text{ m}^2$$

Number of tubes

AISI 304 stainless steel pipes have been used, so they have been dimensioned according to their standards. In this way, the area of a tube is calculated.

$$A_t = \pi \cdot d_o \cdot L_t \quad \text{Eq 3.7}$$

A_t : Area of a tube [m²]

d_o : Tube outer diameter [m]

L_t : Tube length [m]

$$A_t = \pi \cdot 0.0254 \text{ m} \cdot 3.962 \text{ m} = 0.316 \text{ m}^2$$

Therefore the number of tubes are:

$$N_t = \frac{A}{A_t} \quad \text{Eq 3.7}$$

$$N_t = \frac{19.71 \text{ m}^2}{0.316 \text{ m}^2} = 62.34 \text{ tubes} \approx 63 \text{ tubes}$$

Tube bundle diameter

First of all, information about Pitch is needed:

$$\text{Pitch} = 1.25 \cdot d_o \quad \text{Eq 3.8}$$

This gives a Pitch of 0.03175 m with a rectangular layout. Eq 3.9 is used to obtain the tube bundle diameter.

$$N_t = a \left(\frac{d_b}{d_o} \right)^b \quad \text{Eq 3.9}$$

d_b : Tube bundle diameter [m]

Coefficients a and b are obtained from Table 29.

Table 29. Constants used for equation 3.9 [33].

Number of passes		1	2	4	6	8
Triangular pitch	a	0.319	0.249	0.175	0.0743	0.0365
	b	2.142	2.207	2.285	2.499	2.675
Square pitch	a	0.215	0.156	0.158	0.0402	0.0331
	b	2.207	2.291	2.263	1.617	2.643

Knowing that the arrangement is square and that there are 2 passes per tube, the constants a and b are 0.156 and 2.291 respectively. Therefore, the diameter of the tube bundle is 0.349 m.

Shell diameter

Figure 19 is used to obtain the difference between the tube bundle diameter and the shell diameter.

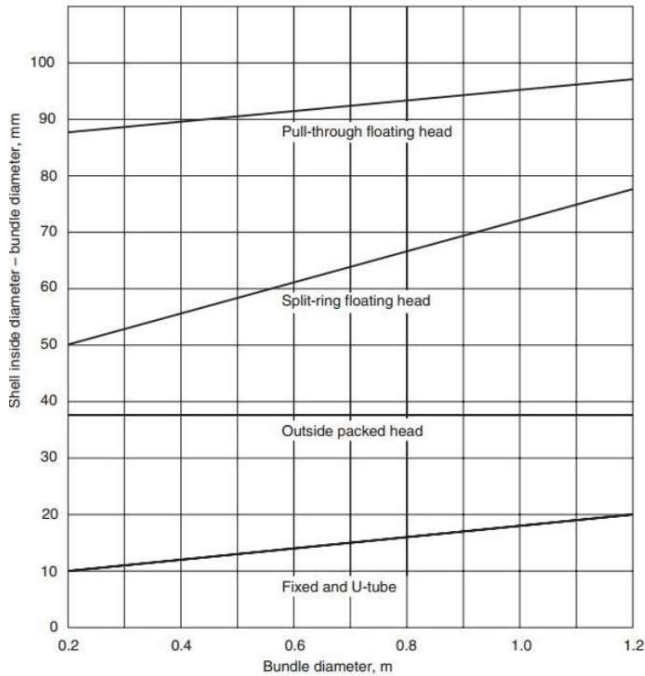


Figure 19. Shell-bundle clearance [33].

So following the graph for fixed and U-tube gives a difference of 12 mm and therefore a shell diameter of 0.360m.

Shell thickness

Table 30 has been used to obtain the thickness of the shell.

Table 30. Minimum shell thickness [18].

Nominal shell dia., mm	Carbon steel		Alloy steel
	pipe	plate	
150	7.1	—	3.2
200–300	9.3	—	3.2
330–580	9.5	7.9	3.2
610–740	—	7.9	4.8
760–990	—	9.5	6.4
1010–1520	—	11.1	6.4
1550–2030	—	12.7	7.9
2050–2540	—	12.7	9.5

Since the material is an alloy steel, the thickness of the shell has a value of 3.2 mm.

Baffles

$$N_B = \frac{L_t}{B} - 1 \quad \text{Eq 3.10}$$

N_B : Number of baffles [-]

B: Baffle spacing [m]

The baffle spacing is 0.5 times the diameter of the shell, so:

$$N_B = \frac{3.962 \text{ m}}{0.18 \text{ m}} - 1 = 21.00 \approx 21 \text{ baffles}$$

Furthermore, the baffle cut has been considered to be 0.225 times the diameter of the shell, so it is 0.081 m.

Velocity

$$u_s = \frac{N_{pt} \cdot w_f}{\rho_f \cdot N_t \cdot \pi \cdot \left(\frac{d_{int}}{2}\right)^2} \quad \text{Eq 3.11}$$

N_{pt} : Number of passes per tube [-]

ρ_f : Cooling fluid density [kg/m³]

d_{int} : Tube inner diameter [m]

$$u_s = \frac{2 \cdot 5.55 \frac{\text{kg}}{\text{s}}}{1035 \frac{\text{kg}}{\text{m}^3} \cdot 63 \text{ tubes} \cdot \pi \cdot \left(\frac{0.019862 \text{ m}}{2}\right)^2} = 0.549 \text{ m/s}$$

It should be noted that the flow rate and density used will vary depending on the fluid circulating through the tubes, in this case, the water-ethylene glycol mixture 30% (cooling fluid).

APPENDIX 4: TANKS DESIGN

In this section, calculations will only be performed for the tank T-1. The design process of the other tank will be identical except for a couple of points, which will be discussed.

Tank volume

$$V = \frac{q}{\tau} \quad \text{Eq 4.1}$$

V: Tank volume [m³]

q: Volumetric flow rate [m³/h]

τ : Residence time [h]

$$V = \frac{7.236 \frac{m^3}{h}}{0.2 h} = 1.447 m^3$$

The tank is oversized by 20%, as the volume that the fluid will occupy in the heads has been taken into account, so that the volume of the tank will be 1.2 times the volume obtained, i.e., 1.737 m³.

The volume of the T-2 tank is too large, so it has been decided to split it into two tanks with half the volume obtained with Eq 4.1.

Tank height and diameter

It has been imposed that the height of the T-1 tank be 1.25 times the diameter of the tank, whereas for the T-2 tank it is twice the diameter of the tank. Knowing that it is a cylinder, the diameter is obtained with equation 4.2.

$$V = \pi \cdot h \cdot \frac{D^2}{4} = \pi \cdot 1.25 D \cdot \frac{D^2}{4} \quad \text{Eq 4.2}$$

$$D = \sqrt[3]{\frac{4 \cdot 1.447 m^3}{1.25 \cdot \pi}} = 1.209 m$$

Therefore, the relationship between height and diameter gives a tank height T-1 of 1.512 m.

Liquid pressure

The oversizing has been carried out so that the maximum fluid level inside does not exceed 80%.

Thus, the liquid level is calculated with the equation 4.3:

$$0.8 \cdot V = h_{liq} \cdot \pi \cdot \left(\frac{D}{2}\right)^2 \quad Eq\ 4.3$$

$$h_{liq} = \frac{0.8 \cdot 1.737\ m^3}{\pi \cdot (1.209\ m)^2} = 1.209\ m$$

Finally, the pressure exerted by the fluid is calculated.

$$P_{liq} = h_{liq} \cdot g \cdot \rho_{liq} + P_e \quad Eq\ 4.3$$

P_{liq} : Pressure exerted by the fluid [Pa]

g : Gravity [m/s^2]

ρ_{liq} : Density of the fluid [kg/m^3]

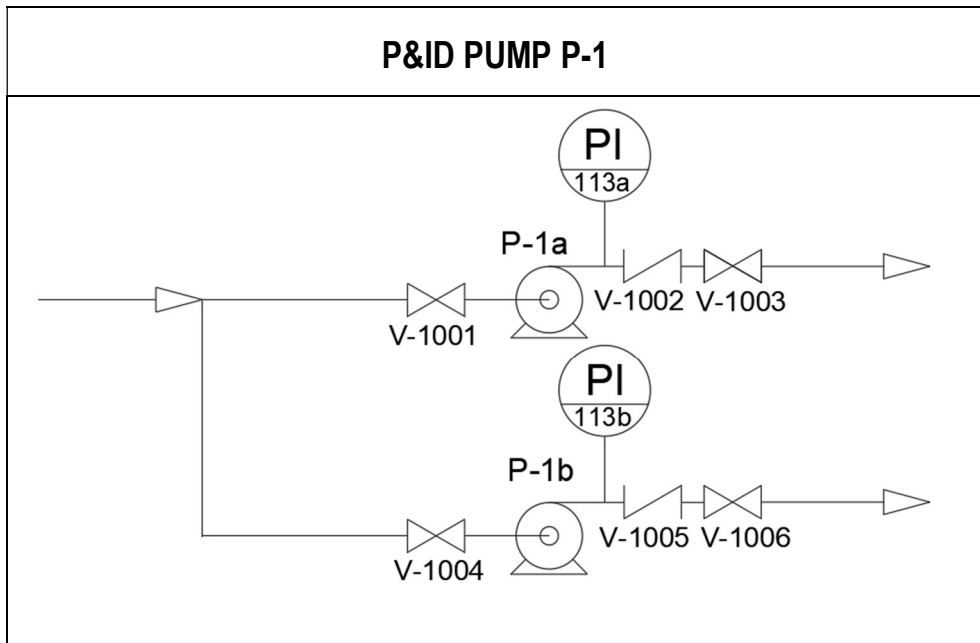
P_e : Tank inlet stream pressure [Pa]

$$P_{liq} = 1.209\ m \cdot 9.81\ \frac{m}{s^2} \cdot 805.020\ \frac{kg}{m^3} + 101325\ Pa = 110876\ Pa$$

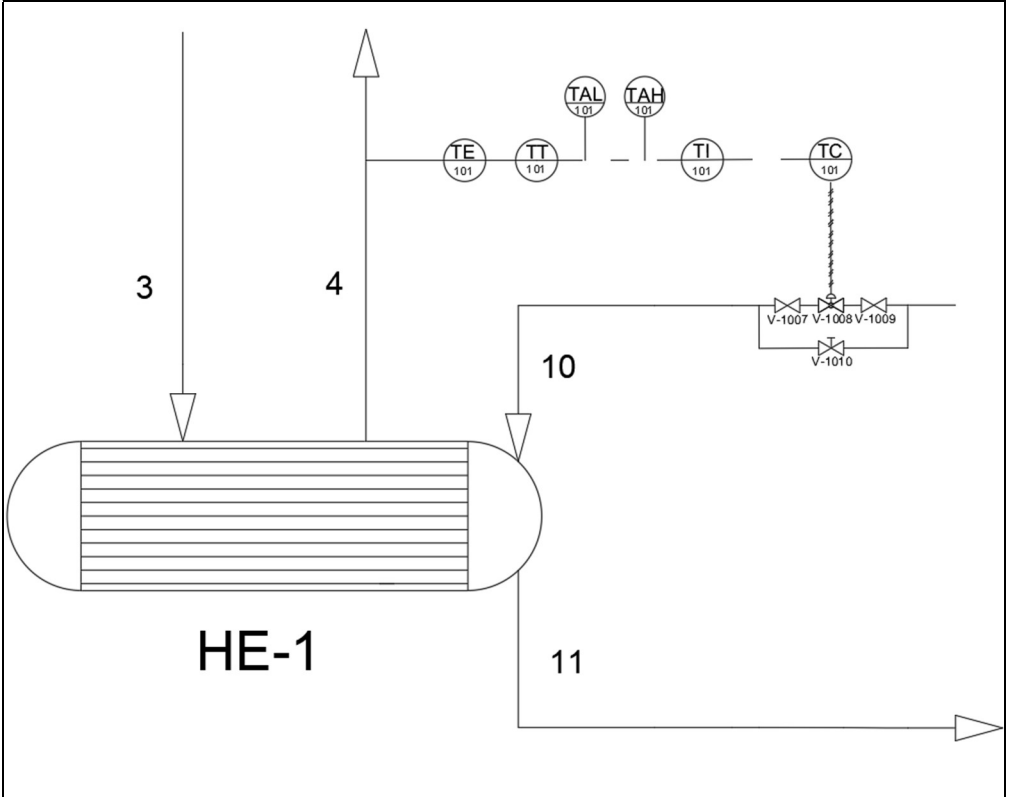
$$110876\ Pa \cdot \frac{1\ atm}{101325\ Pa} = 1.094\ atm$$

APPENDIX 5: P&ID DIAGRAM

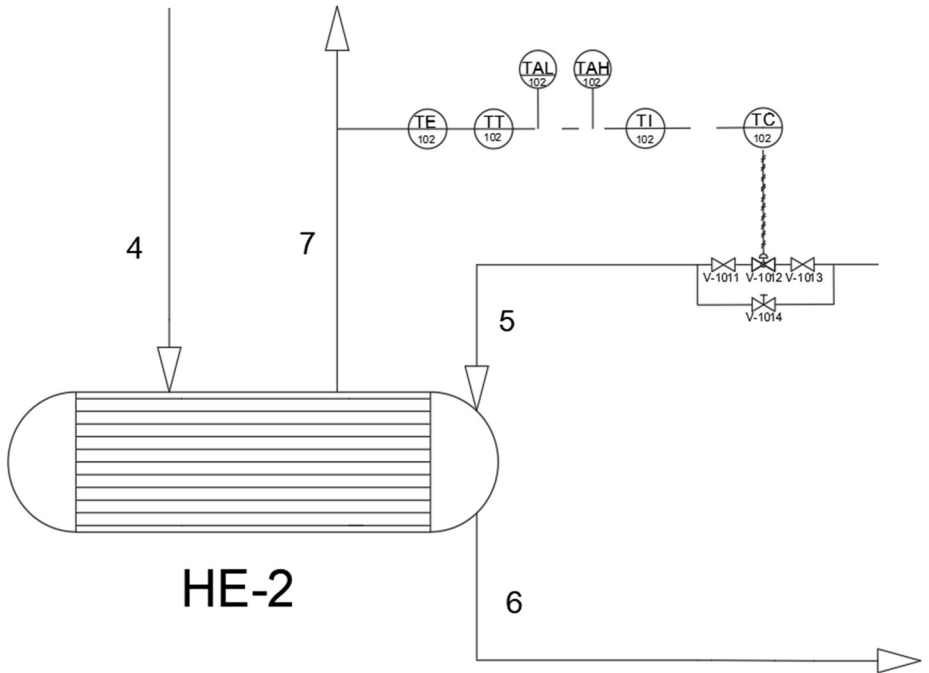
The elements with their corresponding P&IDs will be shown in order of their appearance in the process, and the stream numbers will be the PFD diagram ones.



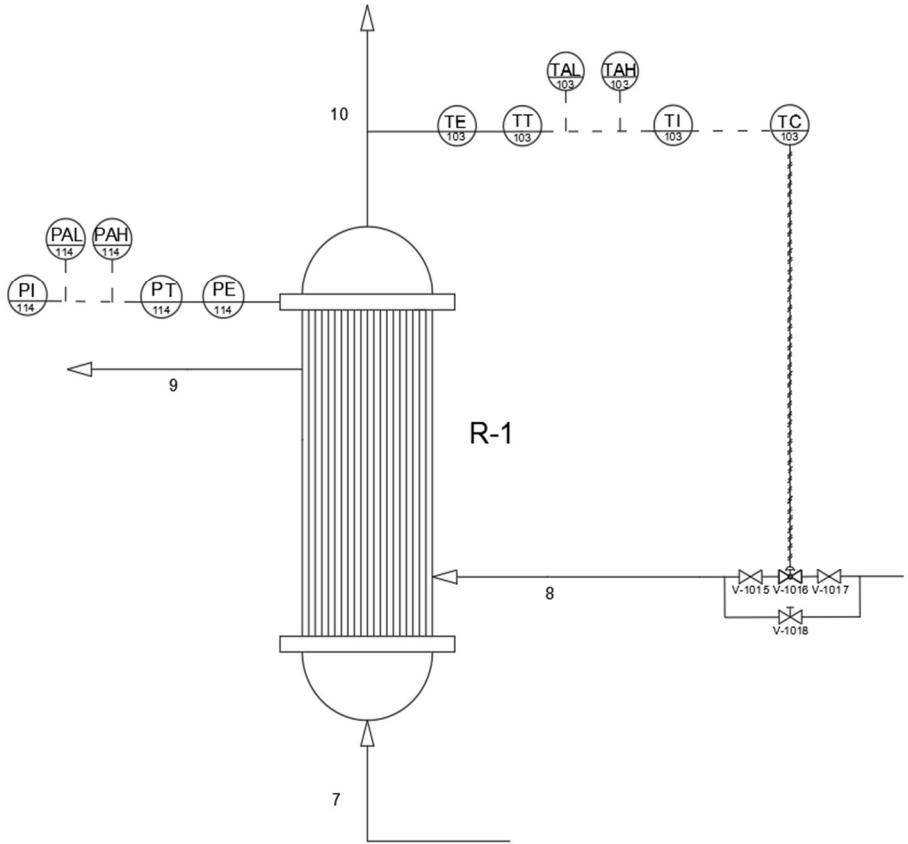
P&ID HEAT EXCHANGER HE-1



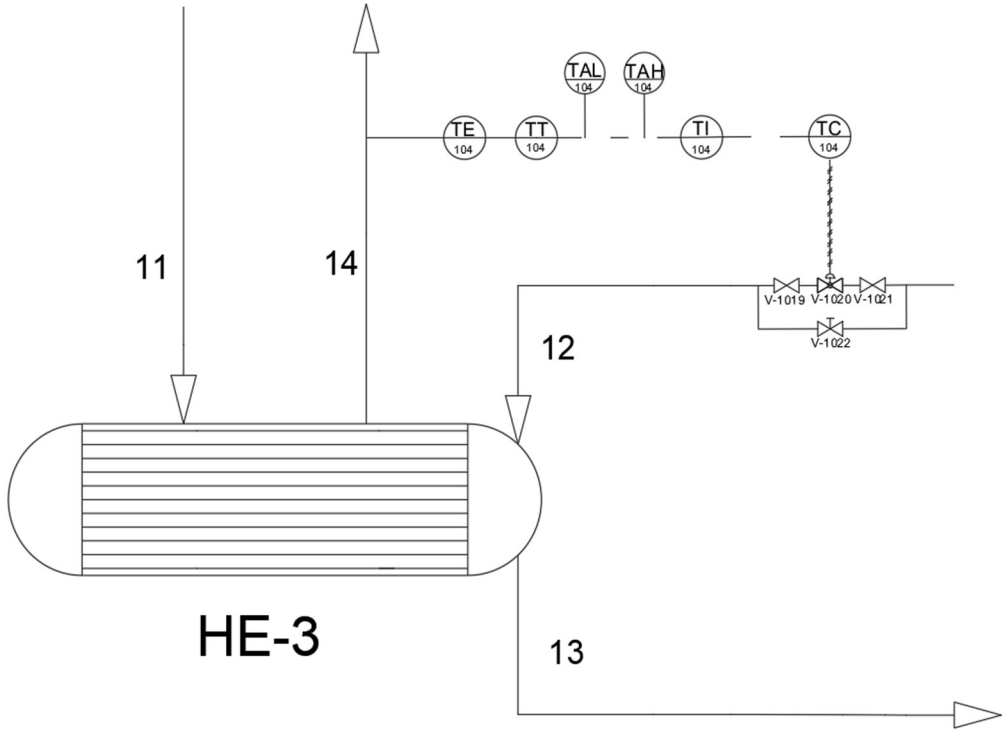
P&ID HEAT EXCHANGER HE-2



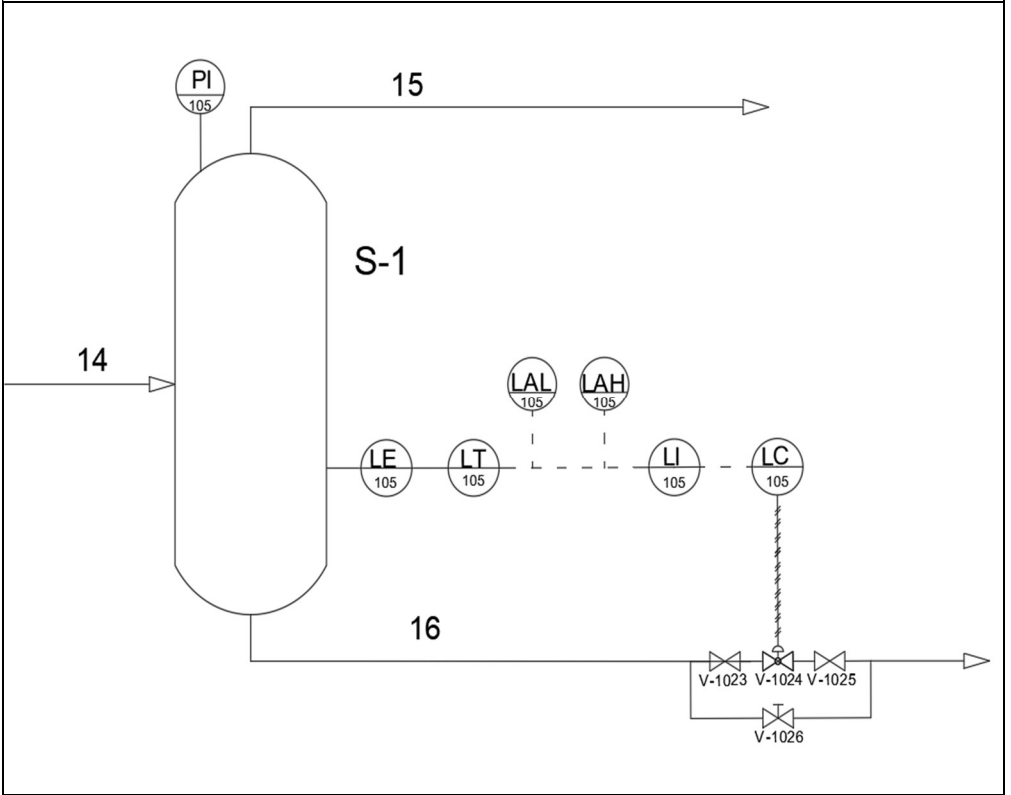
P&ID REACTOR R-1



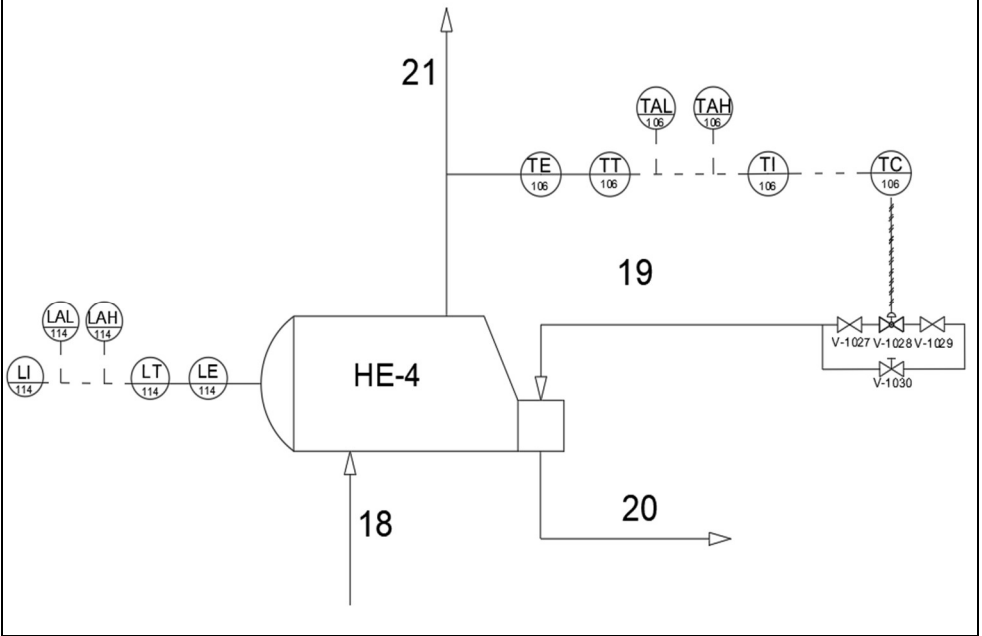
P&ID HEAT EXCHANGER HE-3



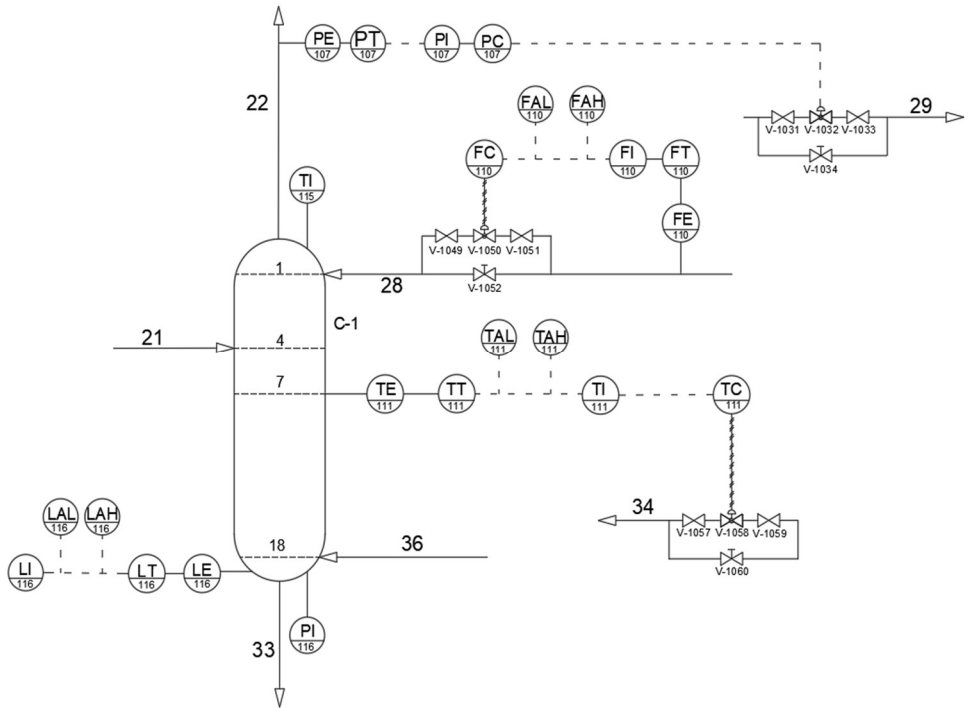
P&ID SEPARATOR S-1



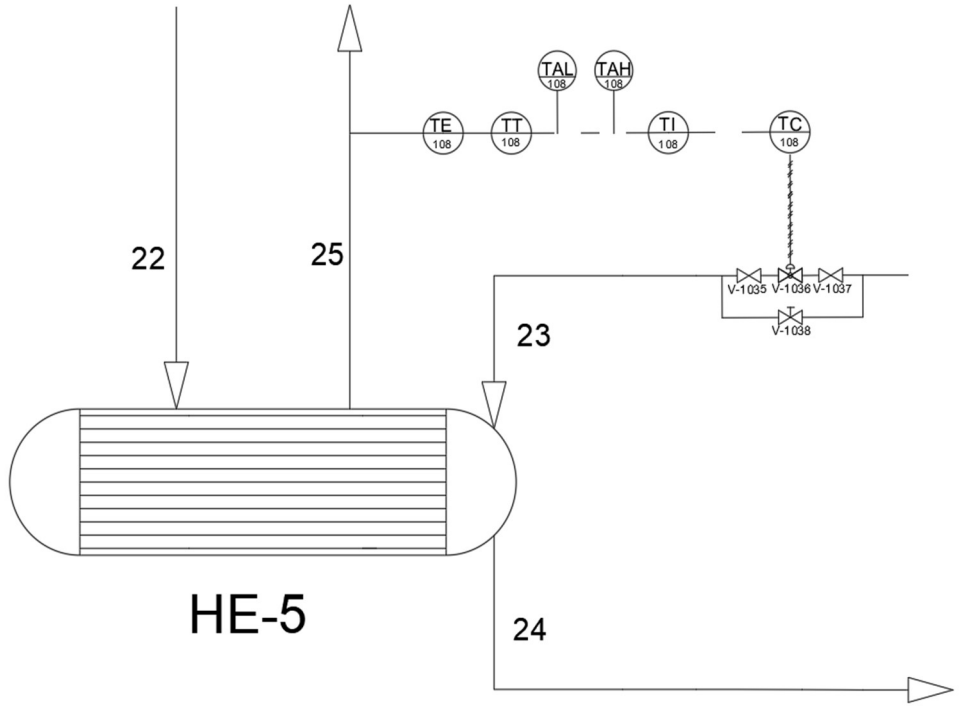
P&ID HEAT EXCHANGER HE-4



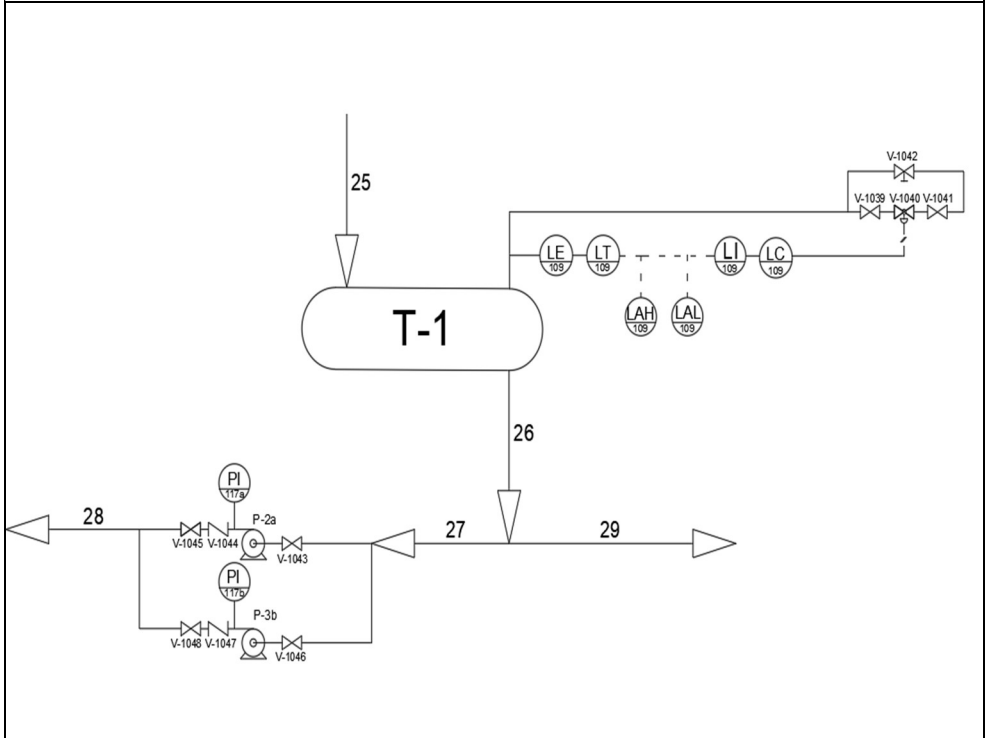
P&ID COLUMN C-1



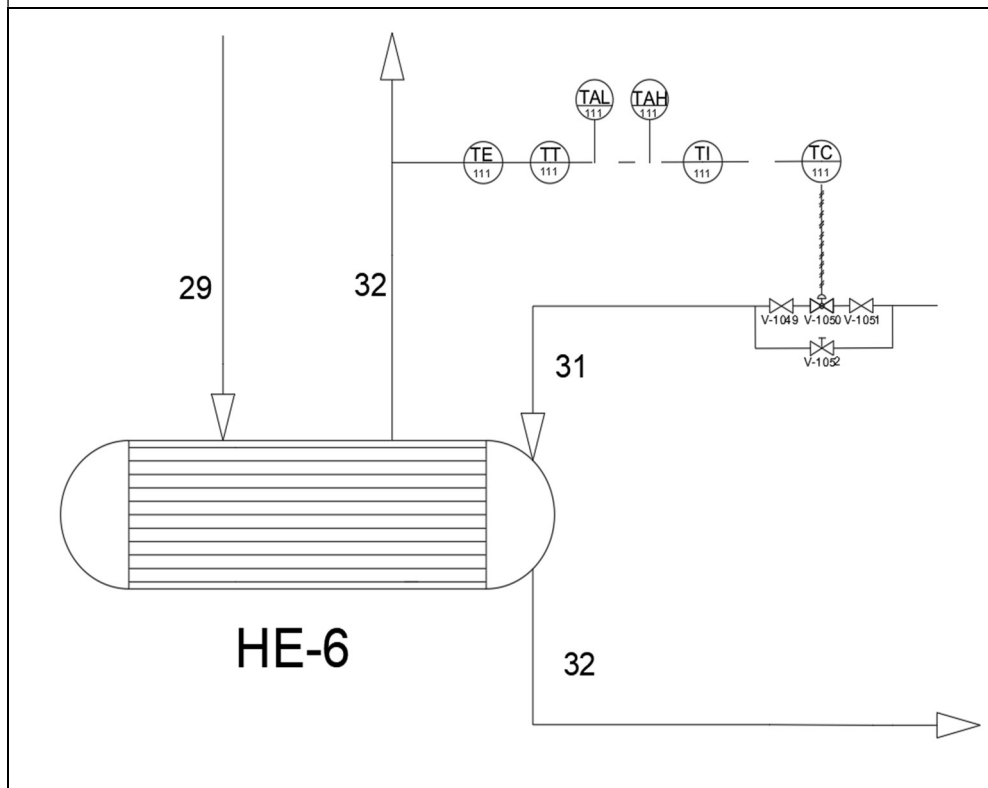
P&ID HEAT EXCHANGER HE-5



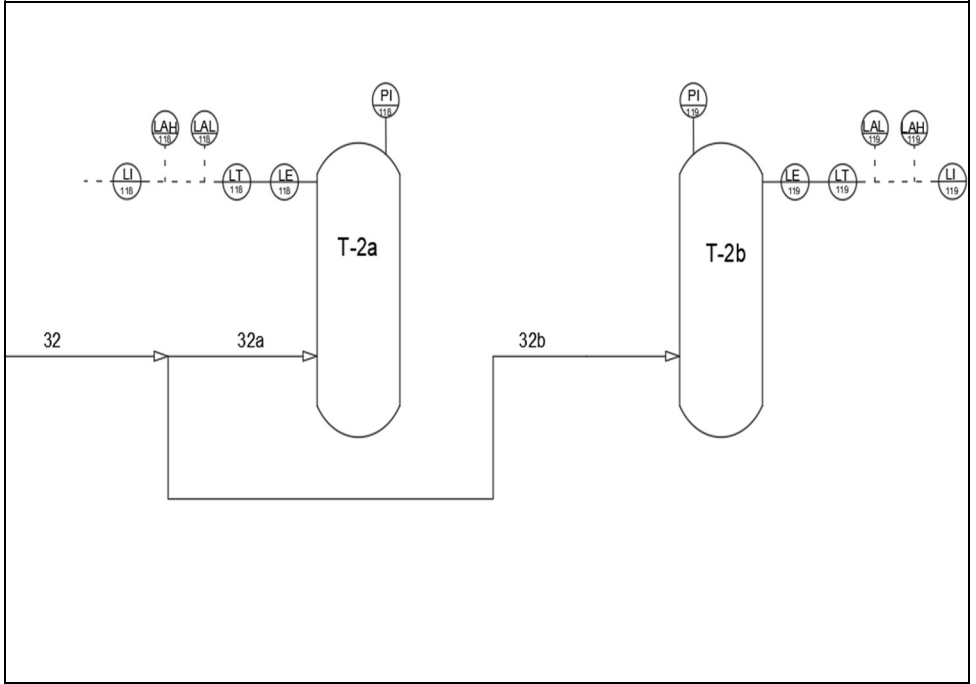
P&ID TANK T-1 AND PUMP P-2



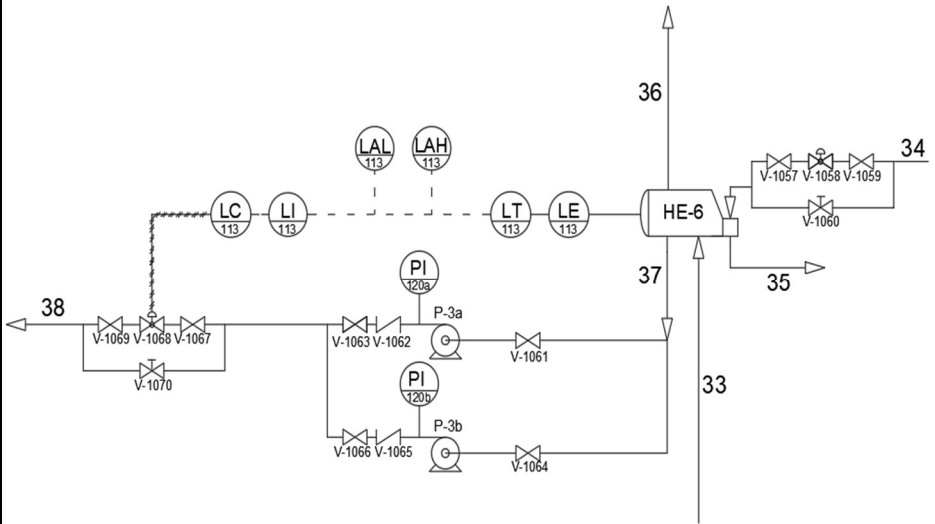
P&ID HEAT EXCHANGER HE-6



P&ID TANK T-2



P&ID HEAT EXCHANGER HE-7 AND PUMP P-3



For the the control elements used in the P&ID diagram, an "ABC" nomenclature has been followed, which is specified in Table 31 and Table 32.

Table 31. Letter A nomenclature in the P&ID diagram [53].



Nomenclature letter A	Variable
P	Pressure
T	Temperature
F	Flow
L	Level

Table 32. Letter B and C nomenclature in the P&ID diagram [53].

Nomenclature letters BC	Function
E	Measured element
T	Transmitter
AL	Low value alarm
AH	High value alarm
I	Indicator
C	Controller

In terms of the types of connections used in the diagram, a distinction can be made between those shown in Table 33.

Table 33. Types of connection used in the P&ID diagram [53].

Type of connection	Symbology
Mechanical	
Electrical	
Pneumatic	