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TNO report

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Basis of Design document for cl-HPAD of e-Methanol

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1 Introduction and Goal

Methanol, a vital chemical industry product, serves as a precursor for various chemicals like formaldehyde, acetic acid, and plastics. Currently, 98 million tonnes/year are produced globally, mainly from natural gas or coal. Shifting to renewable methanol, known as e-methanol, using CO₂ as feedstock, supports carbon-neutral goals, benefiting both chemical and fuel sectors. The interest in e-methanol arises from the urgent need to combat climate change by reducing CO₂ emissions, aligning with the goal of limiting the global temperature rise to 1.5 °C by achieving net carbon neutrality by 2050 (IRENA, 2021).

E-methanol is derived from CO₂ captured from renewable sources (e.g., direct air capture) and green hydrogen produced through water electrolysis with renewable electricity. In a high-TRL Carbon Capture and Utilization (CCU)-based chemical industry, CO₂-based methanol and methane replace fossil resources, offering the potential to produce up to 20 chemicals. This involves converting CO₂ and hydrogen into methanol and methane, with methanol further processed into olefins and BTX aromatics, serving as the basis for various chemicals. The high-TRL CO₂-neutral scenario is visually represented in Figure 1.

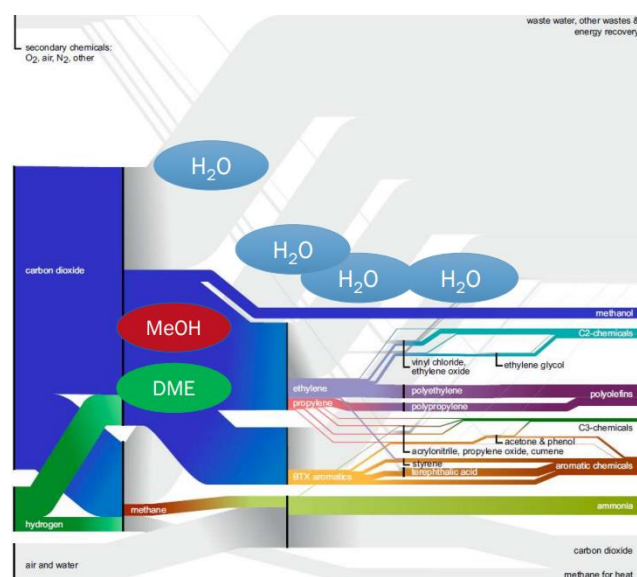


Figure 1: CCU-based chemical industry, chemicals derive from renewable methanol production (IRENA, 2021)

When producing methanol from CO₂ and hydrogen, water is produced as a by-product. Although distillation columns recover the product, distillation processes remain highly energy inefficient. Optimization and innovative distillation concepts, such as heat pump-assisted distillation (HPAD), can significantly reduce energy use, capital investment, and improve sustainability by electrifying the process. Closed-loop heat pump distillation (cl-HPAD) integrates a heat pump and a distillation column, providing the highest energy and cost benefits.

Simplified schematics of the e-methanol production process is shown in Figure 2. The heat produced in the methanol synthesis section is used in the distillation. The excess heat from synthesis reaction accounts for about 35% of total heat needed in

distillation. The use of cl-HPAD instead of traditional distillation can greatly reduce the heat requirement for separation, leading to elimination of external heat demand and full electrification of the overall process.

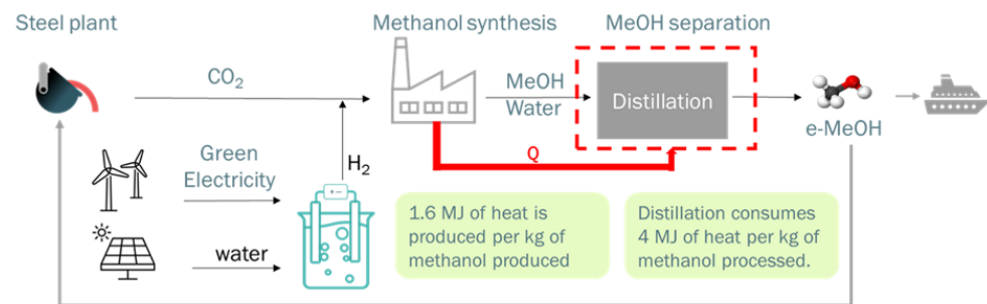


Figure 2: Simplified block diagram of e-methanol production process.

2 General information

2.1 Scope

The scope of the project is the process design and basic engineering of an advanced distillation unit that reduces the energy consumption of traditional distillation for the separation of methanol and water. This unit, called cl-HPAD, will follow the principle of heat integrated distillation column, where the pressure of rectifying section is elevated and heat is transferred from the rectifier to the stripping section. The basic engineering package consists of: preliminary mass & energy balance, process flow diagram (PFD), preliminary sized equipment list, control and safeguarding philosophies, P&ID, HAZOP, equipment layout, mechanical design and 3D model of cl-HPAD column.

The TRL6 cl-HPAD unit is designed for construction in the area of Rotterdam and its purpose will be to experimentally measure the: mass transfer, heat transfer and pressure drop performance when separating a methanol/water mixture and prove its superior performance over traditional separation via distillation. The installation will be placed in FLIE laboratory and it will be the downstream purification part of e-methanol produced via an 100kW electrolyzer, followed by a methanol synthesis reactor.

The remaining methanol plant which will produce the methanol-water mixture is outside the scope of this installation. The product mixture from the MeOH plant will be used as feed for cl-HPAD. The feed comes in at 65 °C and 1.5 bar(a). The separated MeOH and water will be disposed as per the rules of the location. The system operates between pressures of 1.11 and 6 bar (a) and temperature range between 65 and 120 °C. This installation works with an inlet flow of 143.2 kg/h.

2.2 Installation location, Climate and Geographical data

As the exact location is not known for the moment, the following assumptions are made:

Area	Rotterdam
Indoor/outdoor	Indoor
Existing / new location	New location
Climate and Geographical information:	
- Pressure, nominal, minimum, maximum	atmospheric
- Temperature, nominal, minimum, maximum	20°C, 0°C, 35°C
- Humidity, nominal, minimum, maximum	
- Rain, year total	not applicable
- Wind load	not applicable
- Snow load	not applicable
- Earthquake risk	No
- Site atmosphere: industrial / lab / other	Lab
- Requirements for winterisation	not applicable
- Requirements for Solar Radiation	not applicable

2.3 Results

TNO has successfully completed the design of a first-of-a-kind cl-HPAD for the methanol/water separation and the delivery of its basic engineering package. The

results are delivered in this report, accompanied by supporting material of a HAZOP and a P&ID diagram.

The design of cl-HPAD can separate the methanol/water mixture and collect methanol of 99.6 wt% purity. The specific energy consumption is lower than the traditional distillation column by 43%. More specifically, the heat demand in the reboiler is reduced by 83% and an distillation electrification of 71% is achieved with the use of the compressor. In the overall e-methanol process, that means that the excess heat of methanol synthesis can cover the total downstream requirement. Hence, the use of cl-HPAD in the downstream processing eliminates completely the external heat demand and leads to full electrification of the e-methanol production process.

The design of cl-HPAD is based on assumptions on the mass and heat transfer coefficients. Experimental validation is required to reduce the uncertainties around these values. A TRL4 cl-HPAD unit is present in TNO premises in Petten, and will be used for that reason in future projects. During the current project, the unit was started-up successfully, but no measurements were performed.

However, the design of TRL6 cl-HPAD that is provided in this project is flexible to variations in mass and heat transfer coefficients and it could adapt to different operation conditions in case the experiments show wide difference from the expected results. The expected values were derived by modifying values from existing experiments in the TRL4 cl-HPAD with different mixture of hydrocarbons.

3 Process description

3.1 CI-HPAD process

The principle of the heat-pump assisted distillation technology (HPAD) is shown in the Figure 3. The separation process in the conventional column on the left hand side is driven by the waste heat supplied to the reboiler after it has been upgraded to the required temperature using a heat pump. In the open-loop HPAD (ol-HPAD), whose common embodiment is mechanical vapour recompression (MVR), the distillate vapours are compressed to supply heat to the bottom reboiler. When part of the liquid distillate returns to the column, the concept would turn into a semi-open loop HPAD. On the right hand side, the closed-loop HPAD is presented. The rectifier (R) which normally operates at a lower temperature, is operated at a higher pressure than the stripper (S). Therefore, the heat of the condensing flow in the rectifier can be used to evaporate part of the mixture in the stripper. External heat exchange is limited to a small condenser to remove excess heat and if necessary at start-up the system. This final embodiment is thermodynamically most favourable. The concept of cl-HPAD and its excellent efficiency has been known for some time with calculated potential energy savings >60% compared to the conventional distillation process. For the structured packing cl-HPAD, was experimentally shown to have both excellent mass transfer and heat transfer properties at a similar pressure drop to conventional packing (Bruisma 2012). Simulation of an industrial scale plate-packing unit indicates that this equipment rightfully balances the required heat and mass transfer rates.

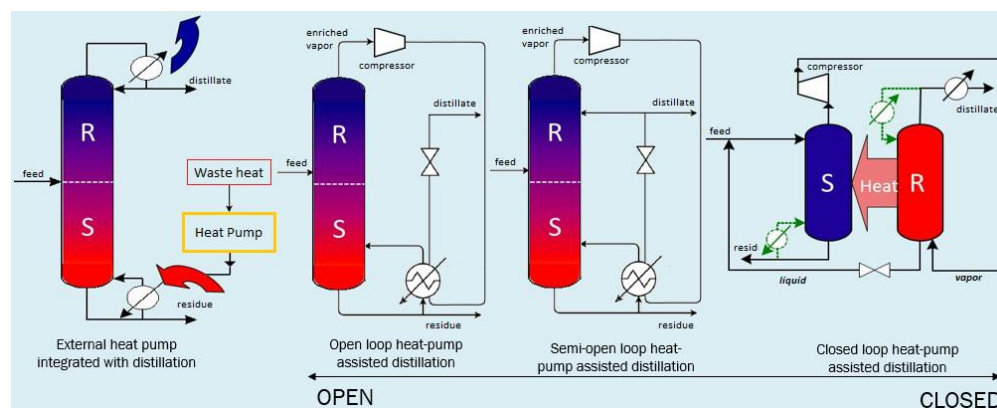


Figure 3: Principle of simple distillation (left) and heat integrated distillation (right).

Aspen simulations show a superior performance of cl-HPAD compared to traditional distillation. The specific energy consumption is lower in cl-HPAD by 43%. More specifically, the heat demand in the reboiler is reduced by 83% and an electrification of 71% is achieved.

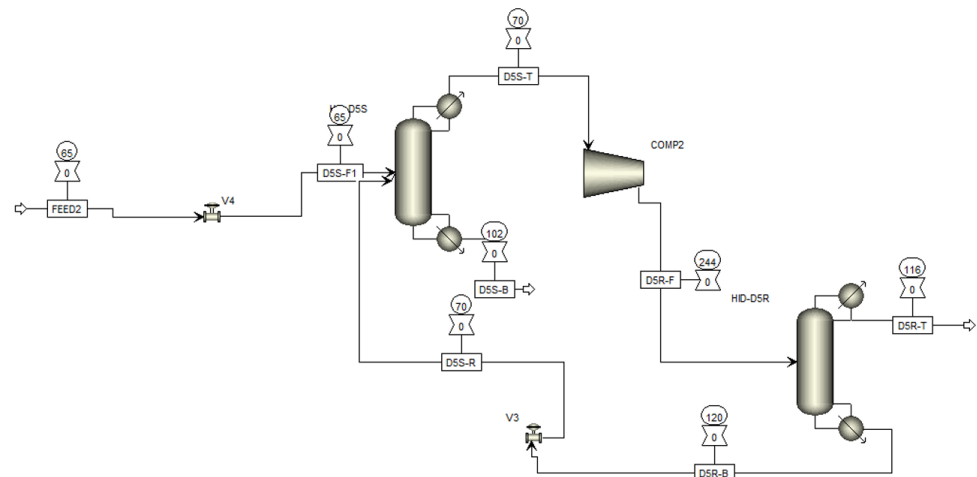


Figure 4: Aspen simulations of cl-HPAD. Duty is shown in Watts.

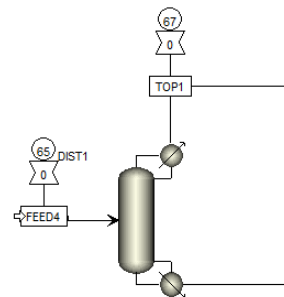


Figure 5: Aspen simulations of simple distillation column. Duty is shown in Watts.

The aspen simulations were performed along with adjustment of the heat transfer between the two columns in an excel file. The excel file calculates the actual heat transfer based on heat and mass transfer coefficients that were derived in previous projects in the TRL4 cl-HPAD experimental unit in Petten for a mixture of different hydrocarbons. The heat and mass transfer coefficients were adjusted to estimate the performance of methanol/water mixture, and they will be more accurately calculated in future project, in the TRL4 cl-HPAD.

The column design resulted to the following column dimensions:

Height:	3 m
Width:	0.5 m
Depth:	0.03 m
Volume:	0.18 m ³
Heat transfer area:	6 m ² (Height x Width x 4)
Sh:	33.3 m ² /m ³ (Heat transfer Area / Volume)

The column is simulated for 10 steps of 0,6 m² of heating area each. The resulting F-factor is 1.7 This result derives from assumed heat transfer coefficient $U=410$ W/m²/K and HETP=0,35 m. The estimation of these coefficients is based on previous experiments in the same TRL4 unit for different mixture of hydrocarbons.

3.2 Heat and Material balance

The following table provides the material balance of the installation.

Table 1: Mass balances of cl-HPAD

	Feed	Stripper top to compressor	Stripper bottom	Feed to rectifier after compressor	Rectifier Top	Rectifier Bottom	Recycle feed to Stripper
Denotation on simulation	D5S-F1	D5S-T	D5S-B	D5R-F	D5R-T	D5R-B	D5S-R
Phase	Liquid Phase	Vapor Phase	Liquid Phase	Vapor Phase	Liquid Phase	Liquid Phase	mixed phase
Temperature [°C]	65	70.2	101.6	243.9	116.1	119.9	70.2
Pressure [bar]	1.5	1	1.11	5.8	5.7	5.81	1.1
Mass							
Mass flow -Total [kg/h]	143.2	318.9	53.1	318.9	90.1	228.8	228.9
Mass flow - Methanol [kg/h]	90.2	289.9	0.5	289.9	89.7	200.1	200.2
Mass flow - Water [kg/h]	53.0	29.1	52.6	29.1	0.4	28.7	28.7
Methanol [wt%]	63.00%	90.89%	1.00%	90.89%	99.60%	87.46%	87.46%
Water [wt%]	37.00%	9.11%	99.00%	9.11%	0.40%	12.54%	12.54%
Mass Density [kg/m ³]	804.9	1.0	913.7	4.0	673.2	688.1	6.7
Mole							
Mole Flow -Total [kmol/h]	5.76	10.66	2.94	10.66	2.82	7.84	7.84
Mole flow - Methanol [kmol/h]	2.82	9.05	0.02	9.05	2.80	6.25	6.25
Mole flow - Water [kmol/h]	2.94	1.61	2.92	1.61	0.02	1.59	1.59
Methanol [mol%]	48.91%	84.87%	0.56%	84.87%	99.29%	79.68%	79.68%
Water [mol%]	51.09%	15.13%	99.44%	15.13%	0.71%	20.32%	20.32%
Molar Density [kmol/m ³]	32.36	0.04	50.50	0.13	21.07	23.57	0.23
Volumetric flow [m ³ /h]	0.18	304.29	0.06	79.01	0.13	0.33	34.26

The heat balance is described below (the units are shown in Figure 6):

Heat exchanger-1 (electric preheater): this heat exchange is placed in the feed line to heat up the stream from room temperature to 65 °C. The capacity of this heat exchanger is calculated/estimated to be 15 kW.

Heat exchanger-2 (condenser): is the heat duty required for the condenser placed in rectifying column to heat the liquid contents to 116 °C. The capacity of this heat exchanger is calculated/estimated to be 120 kW (including 40% oversize). This heat exchanger duty is overestimated in order to use it for measuring flooding point.

Heat exchanger-3 (electric reboiler): is the heat duty required for the re-boiler placed in stripping column to heat its liquid contents to 102 °C. The capacity of this heat exchanger is calculated/estimated to be 105 kW (needed for startup - including 40% oversize). This capacity of the heat exchanger is overestimated in order to

work for reaching the flooding point also could be used for stating up the installation. This heat exchanger duty is overestimated in order to use it for measuring flooding point.

The compressor was modelled with 66 % isentropic efficiency and a pressure ratio of 5.8, $T_{in} = 69\text{ }^{\circ}\text{C}$ and Volumetric flow of $384\text{ m}^3/\text{h}$. The compressor duty is overestimated for 50 kW. Additional duty is taken into account to ensure operation at flooding point can be measured.

Heat exchanger-4 is the electric trim heater with the heat duty of 2 kW will be used during the startup to prevent any liquid going to the compressor from the top of stripping section.

Heat exchange-5: is the trim cooler with the heat duty of 2 kW will be used during the startup to prevent hot liquid going to the recycle and for the water product out.

3.3 Process flow diagram

In this chapter the Process Flow Diagram (PFD) is outlined. Main characteristics of the various processes are described. All the sections shall have sufficient flexibility to cover the operating condition ranges outlined in the test program. A simplified PFD is shown in Figure 6.

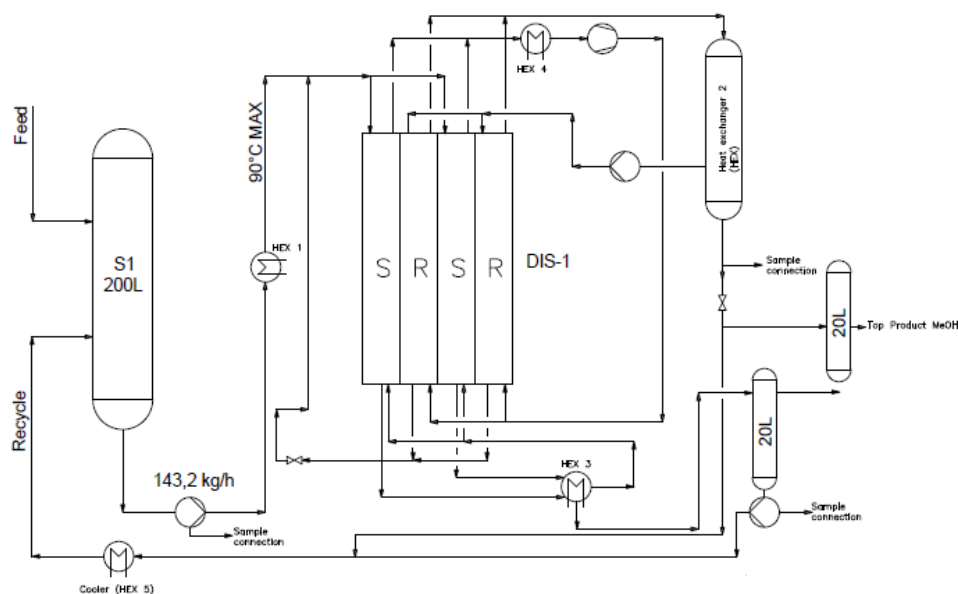


Figure 6: Simplified PFD of cl-HPAD installation.

In reality, the cl-HPAD is two units, each one of them being a Rectifier section (R) with $\frac{1}{2}$ Stripper section (S) in each side. An accurate illustration of cl-HPAD is shown in the more detailed PFD of Figure 7.

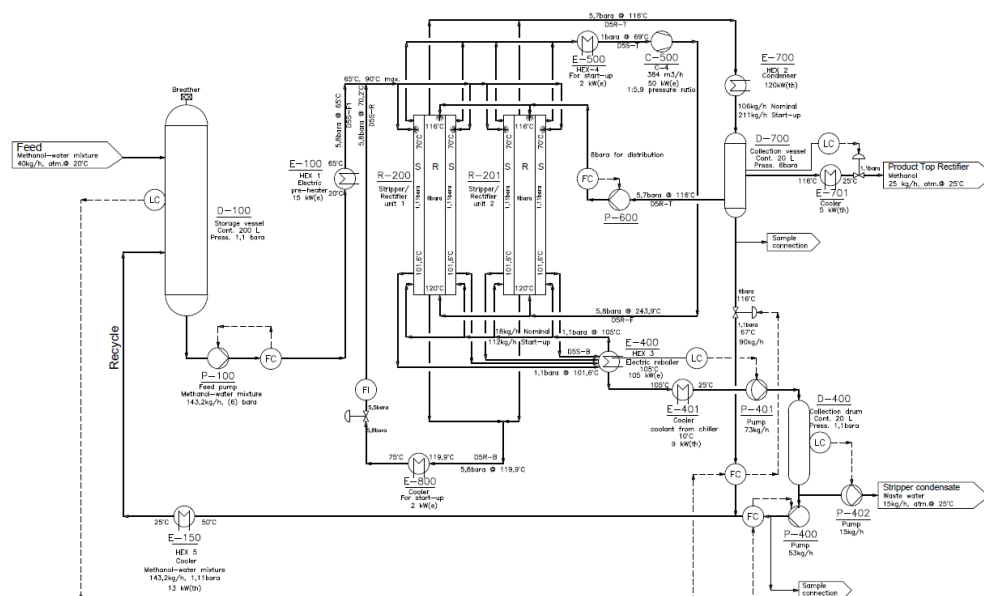


Figure 7: Detailed PFD of cl-HPAD installation.

The unit is divided into 8 sections:

1. Feed section
2. Stripper sections
3. Top product Stripper sections
4. Bottom product Stripper sections
5. Compressor section (intercooling might be needed)
6. Rectifier sections
7. Top product rectifier sections
8. Bottom product rectifier (incl. recycle)

3.4 Description of individual sections

Feed and stripping sections

In this section the liquid feed mixture (output stream from the methanol production plant), is fed to the column. A pump is used to feed the pre-mixed liquid to the top of the stripping column with a flow rate of 143.2 kg/h (2.39 L/min) at 1.11 bar (a). The temperature of the feed is raised to 65 °C using a heat exchanger with capacity 15 kW and the stripping section of the column operates at near atmospheric pressure (max 1.11 bar). A liquid distributor is needed to equally distribute liquid over the width of stripping sections.

Top product stripping sections

The top vapor product from the stripping sections is collected and sent to the compressor. The stream is at 70 °C and atmospheric pressure.

Bottom product stripping sections

The liquid bottom product of the stripping section is high boiling liquid rich in water. The stream is at 101.6 °C and 1.11 bar. This stream is further cooled to 70°C and then collected in a 20 L vessel before redirecting it to the feed section (recycle). A part of the product is removed to prevent accumulation of liquids in continuous

process. A sampling point is also placed in this section for liquid sampling and can be used for further analysis.

Compressor section

The compressor is used to pressurize the top vapor product from stripping sections to 6 bar. By compressing to 6 bar the temperature of the stream increases to approx. 240 °C. There is no risk of condensation in the compressor section as only vapor will enter this section. A trim heater (electric, max. 2kW) will be used for heating the vapors during the start-up.

Rectifier Sections

The gaseous stream from the compressor enters the bottom of the rectifier column and vapor stream is distributed over the rectifying sections. The column operates at 6 bar pressure and the temperature profile is 120 °C (bottom) and 116 °C (top).

Top product rectifier section

The liquid top product of the rectifying section is low boiling liquid rich in methanol . The stream is at 116 °C and at 6 bar pressure. This stream is expanded to 1.1 bar(a) collected in a 20 L vessel before redirecting it to the feed section (recycle). A part of the product is removed to prevent accumulation of liquids in continuous process. A sampling point is also placed in this section for liquid sampling and can be used for further analysis.

Bottom product rectifier sections

The liquid bottom product at 120°C and at 6 bar pressure, is redirected to the top of stripping section, after being expanded to 1.1 bar(a) with a valve.

3.5 Material selection

The materials are be selected based on further detailed engineering. Presently SS-316 L is used for selection by default.

4 Utility specifications

4.1 Electricity

The compressor has a heat duty of 50 KW with pressure ratio of 5.8 , $T_{in}= 69\text{ }^{\circ}\text{C}$ and volumetric flow of 384 m³/h.

4.2 Air for instrumentation

Standard Air supply is adequate to run the pneumatic instruments such as valves.

4.3 Cold utility

Industrial water could be used for heat exchangers for additional cooling if required depending on the availability. The heat exchangers should be designed in a way that the T difference of the cooling water should be less than 5 °C.

4.4 Other

Steam could be used for reboilers but depending on availability otherwise electric heaters would be used.

N₂ will be used for flushing, start-up and shut down (normal and emergency shut-down)

5 Battery limit specifications

The installation will be constructed in Rotterdam, but the exact location is presently unknown. The battery limits required at ambient conditions are:

- Feed liquid : 143.2 kg/h from the Methanol Plant
- Pressurised air for compressor (6 bar) also to operate valves
- Industrial water
- Nitrogen
- Product liquid: Methanol and water
- Electricity

Feed liquid

Typical composition and flow: please refer to chapter 3.2. The Feed should be typically delivered by the methanol plant (which is outside the scope of this document)

Pressurised air

The minimum required operating pressure of the compressor is 6 bar. The location owner will be responsible for the availability

Product liquid:

The product liquid Methanol and water will be drawn out from the stripping and the rectifying sections depending on the flow and composition of the input feed. This feed will be either used for further processing and/or disposed with the available procedure available at the location. The liquid products will be stored in the storage vessels for both MeOH and water separately.

The Industrial water:

It is the responsibility of the location to have industrial water and/or tap water close to the composition mentioned below

Typical composition:

- Conductivity ($\mu\text{S}/\text{cm}$): 544
- Chloride (mg/l): 90
- Total hardness (oD): 8.6
- pH: 8.2

Nitrogen:

It is the responsibility of the location to have bulk N_2 close to the composition mentioned below

Typical composition:

- N_2 (vol%) > 99.9
- O_2 (vpm): <1000
- Water (vpm): <1000

Electricity:

240-600 VAC

6 Operation

6.1 Description of the normal operating control narrative

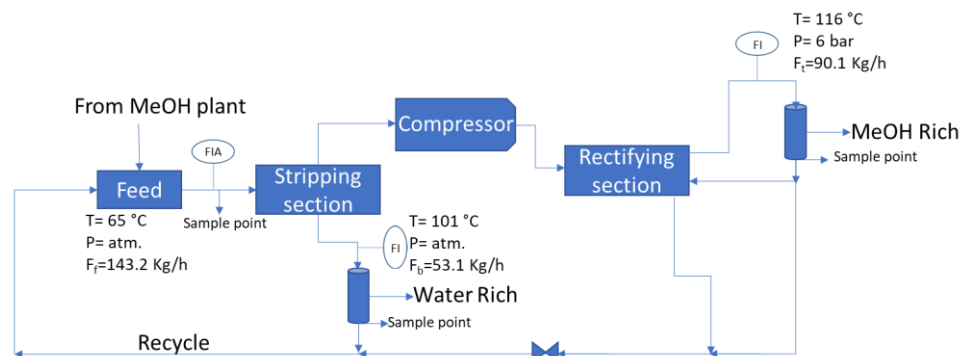


Figure 8: block diagram of the installation

Critical:

- The feed flow to the HPAD column is critical. It has to be maintained by a flow controller.
- The feed level in the feed tank has to be maintained at certain level to ensure continuous operation.
- To ensure steady state operation, the MeOH rich product and the water rich product have to be coupled with flow from the MeOH plant (to avoid any accumulations).
- During process startup, the inverse heat transfer from the stripping to the rectifying sections must be avoided, because it can lead not only to consumption of extra energy, but also risks of potential operation problems. Therefore, it is crucial to raise a certain level of pressure difference between the two sections as soon as possible after startup operation begins. To enhance the pressure difference, the operator needs to start the overhead condenser at a time later than the bottom reboiler.

General:

The flow for cl-HPAD column comes from the MeOH plant which is further placed upstream and is outside the scope of this document. The production of MeOH (and water) depends on H_2 produced from 100 kW electrolyzer (which is even further placed upstream) , also outside the scope of this document.

Feed section:

Feed is basically MeOH-water mixture either coming from a MeOH plant .The flow required for cl-HPAD column is 143.2 kg/h (as per the requirements by Sulzer). In order to ensure stable operation, the product streams are recycled and mixed again to the feed vessel. This feed is heated up to $65\text{ }^{\circ}\text{C}$ using a heat exchanger-1 with a capacity of 15 kW. This heat exchanger can be electrical or heated with steam (if present in location). This feed is pumped using industrial grade pump. A sample can also be taken for mass balance calculations.

Stripping section:

The flow from feed (at $T = 65\text{ }^{\circ}\text{C}$ and $P = \text{atm.}$) is redirected to stripping section. The temperature profile of stripping section is $101.6\text{ }^{\circ}\text{C}$ at the bottom and $70\text{ }^{\circ}\text{C}$ at the top. The reboiler of the stripping section would be either electrically heated or with available steam (depending on location). The top stream is further processed, and the bottom stream mainly contains water. The bottom stream is collected in a 20 L tank. Most of the bottom stream is recycled back to the feed section using an industrial grade pump. Part of this stream is removed to avoid any accumulation (because of the input from MeOH reactor).

Compressor section:

The top feed from stripping section is compressed to 6 bar before redirecting to the rectifying section. The capacity of this Compressor is 50 kW. The temperature will rise from $70\text{ }^{\circ}\text{C}$ to $242\text{ }^{\circ}\text{C}$ because of compression.

Rectifying section:

The flow from the compressor is redirected to the rectifying section. The temperature profile of Stripping section is $109\text{ }^{\circ}\text{C}$ at the bottom and $106\text{ }^{\circ}\text{C}$ at the top. The top stream is collected in a 20 L tank. Most of the top stream is recycled back to the feed section using an industrial grade pump. Part of this stream is sent back to the rectifier, part removed to avoid any accumulation.

6.2 Piping and Instrumentation Diagram (P&ID)

The PI&D is constructed.

6.3 Description of start-up procedure

Pre-startup:

- The installation has to be properly flushed with Nitrogen before starting up.
- The installation needs to be pressurised with N_2 to check for any leakages.
- Check if the MeOH production plant is already producing the mixture.
- Pre-heat all the trace line to ensure no heat loss while transport of the mixture.
- Heat-up the columns and heat exchanges to required value.

Control Philosophy

1. To start with the feed flow rate and the composition of feed from the Methanol reactor has to be monitored.
2. The level on the Feed tank has to be monitored by the level control from the feed input.
3. Feed is introduced into the feed stage by a pump with a controlled flow rate.
4. The feed liquid is preheated before introducing to the feed stage of the stripper.
5. the reboiler in the stripper supplies required heat to create enough vapor and also maintains a certain volume as hold-up.
6. The compressor needs to start- as the flow in the top stripping section begins to flow, for the start-up a trim heater will supply the heat to avoid any condensations.

7. The pressure in the rectifying section reaches a pre-specified level by the help of the compressor.
8. The pressure is maintained by the condenser duty and level control starts to work. The process is run at total reflux until the vapor flow rate from the overhead equals to a specified value.
9. Most of the flow is recycled as the column as to run with specified flow rates (Feed flow must be 143.2 kg/h). This recycle flow is coupled with feed from Methanol reactor to ensure level in the feed tank to be constant.
10. The products are drawn out from the rectifier and stripper sections. The flow of the products which are drawn out are coupled with the feed input from the methanol reactor.
11. Meanwhile, the reflux and reboiler rates are reduced to zero gradually on the rectifier.
12. The continuous operation starts in this manner.

6.4 Description of shut-down procedure

Actual shutdown procedure will be described after the P&ID is developed

1. Stop the feed flow from methanol plant.
2. Ensure the exhaust sections are properly ventilated (not to risk any spillage of methanol).
3. The temperatures have to be reduced.
4. Pressures to be released to atmospheric.
5. Continuously run the flows till temperatures and pressures are ambient.
6. Switch off the flow pumps.
7. Remove the rest liquids from the installation by flushing the installation with N₂.

6.5 Description of emergency shut-down (ESD) procedure

Ensure there is always N₂ supply to the system. During emergencies, N₂ is ensured to be supplied to the installation and temperature, pressures will be set to ambient. All the liquid flow to the installation will be stopped from the methanol reactor. The rest liquid from the installation will be flushed out by N₂.

6.6 Waste streams definition

6.6.1 *Liquid waste streams*

The liquid waste streams are mainly water from the stripping section and methanol from the rectifying section. Both of these streams will be treated as chemicals and a due procedure will be followed as per the location for waste disposal.

6.6.2 *Gaseous waste streams*

There are no gaseous waste streams in this part of the process. The gaseous streams are separated from liquid mixture upstream and this is outside the scope of the installation. It could be that during start-up the pure MeOH and water could be in vapor form but, they will be recycled.

6.6.3 *Solid waste streams*

n.a

7 HAZOP analysis

A Hazard and Operability study (HAZOP) is a formal, qualitative, systematic and rigorous examination of a plant, process or operation to identify credible deviations from the design intent in the context of the complete system that can contribute to hazards or operability problems, by applying the experience, judgement and imagination, stimulated by key words, of a team.

A HAZOP was performed by reviewing the PFD and P&ID. The PFD was divided in nodes. A node is a section of the study subject suitable for HAZOP study. Typically a section where the plant, process or operation does not change in that way deviations cannot be examined consequently. The HAZOP resulted in recommendations for the design and future engineering, commissioning and operation.

8 3D design

8.1 Illustration of the column

The HPAD design consists of two units each containing 1/2S-R-1/2S as depicted in Figure 9. In that way heat transfer area between Rectifier and Stripper is maximized, which can lead to higher F-factor, and hence, higher and more optimum operating capacity.

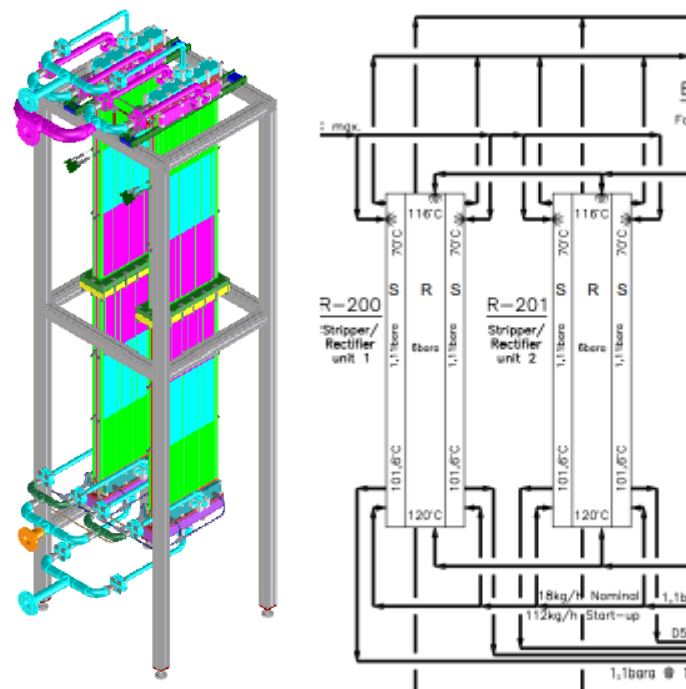


Figure 9: 3D illustration of cl-HPAD.

The dimensions of the column are:

Height:	3 m
Width:	0.5 m
Depth:	0.03 m

8.2 Fluid distribution

The During pre-design it was concluded that for this small unit it is not feasible to make the header for R & S including fluid distributors on top. The reason being that the stripper is at a working pressure of 5 barg (PS=6 barg). The unit being 500mm wide and 30mm thick is not resistant to this pressure if not almost everywhere fins are brazed between both plates to transfer the pressure force from one side to the other.

The design proposed is a tube of relative large diameter, with many small holes to distribute the fluid over the width of the unit a pressurized fluid distribution system. The small holes provide the main resistance so the fluid will be distributed even

over the 4 sub units and over the width within this sub-unit. Pressure drop calculations are made to get an sense on dimensioning of these distribution tubes.

Calculations (for $\frac{1}{2}$ S) showed that a 6mm OD pipe with a wall thickness of 1mm and 50 holes of 0.2mm gives a pressure drop of app. 3.7 bar. The pressure drop is very sensitive to the hole diameter, a 0.15mm hole gives a pressure drop of 21 barg.

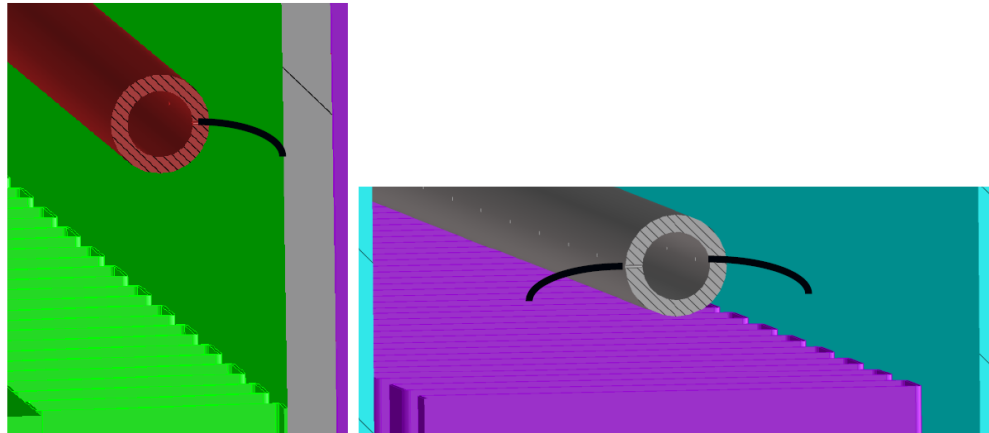


Figure 10: Fluid distribution in 1/2 stripper (left) and rectifier (right)

In the rectifier, the breaker plate is used to prevent flow through the middle fins because they are in line with the central exhaust connection. The fluid distribution tube is also stuck all through. This equalizes the flow resistance along the width and gives a better position accuracy and support of the tube in the unit.

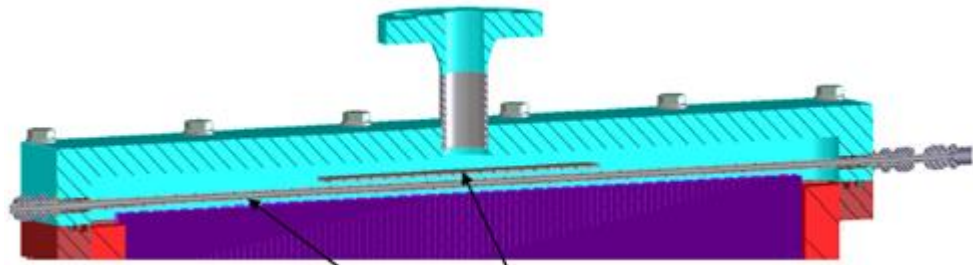


Figure 11: Rectifier header with fluid distribution tube and breaker plate.

8.3 Fins

The fins chosen are shown in Figure 12. The fin pitch is 5mm. The fin height is 30mm for the rectifier and 15mm for the 1/2 stripper. The wall thickness of the fins is 0.2mm made of stainless steel material (1.4404 PED certified).

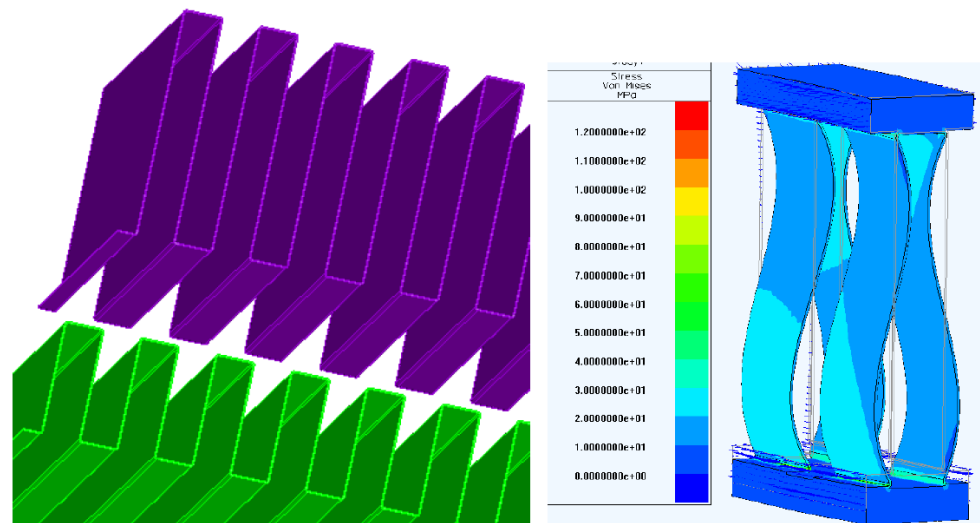


Figure 12: Illustration of the fins (left) and strength tests (right).

FEM analysis is used to determine:

- if the fins are strong enough for the 6 barg design pressure
- if a 5mm gap between fins for pressure distribution or inserting a thermocouple is feasible
- if operation is feasible when small parts of the fins are not brazed.

These calculations are made for pressure only. It needs to be determined what the influence of temperature difference is and how this relates to extra (shear)stress in the fins are the brazed connections.

Staggered fins are necessary to ensure sufficient packing. In that way, the chances of a droplet falling down to the bottom of the column are eliminated.

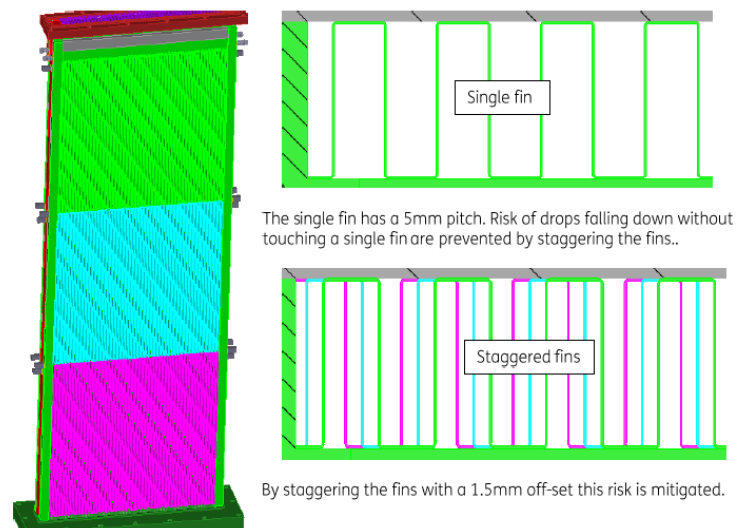


Figure 13: Side view of the fins (left) Top-down view of single and staggered fins (right).

8.4 Connections to units

The connection lines have identical lengths and fitting in order to minimize the difference in pressure drop between two units or between four half stripper sections.

This applies to the vapour/gas streams as well as the fluid streams. Although PN2.5 and PN10 are sufficient for the stripper and rectifier (Figure 14), parts with pressure ratings of PN06 and PN40 are more commonly available (Figure 16).

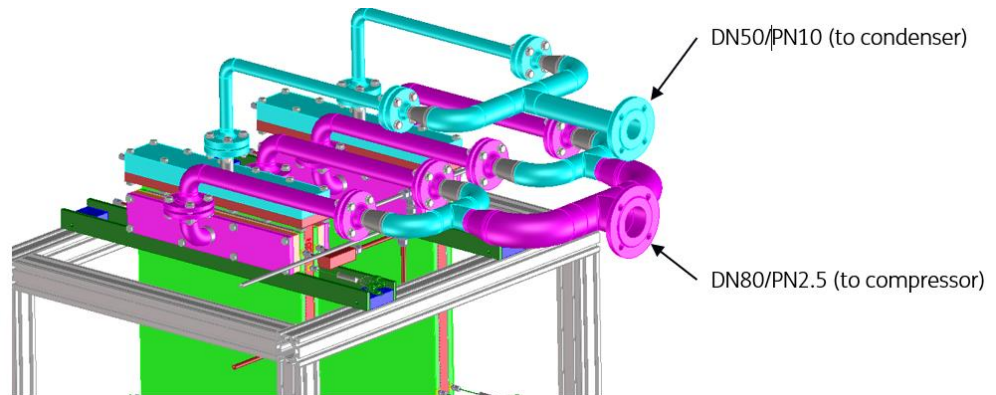


Figure 14: Connection lines on the top of the column.

The fluid feed tubes are all 12x1.5mm (Figure 15).

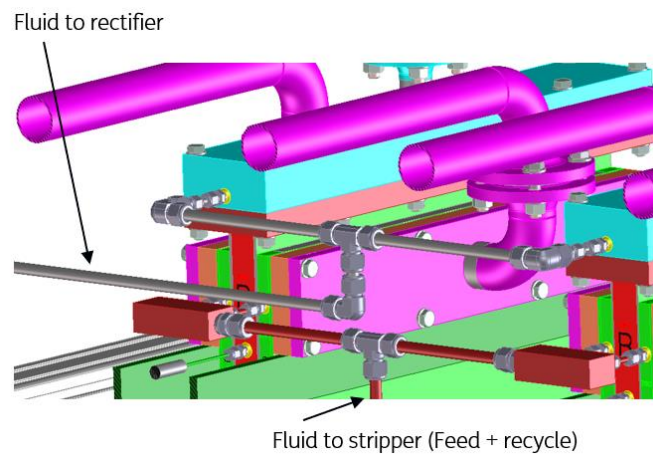


Figure 15: Connection lines in the feed of the column sections (top).

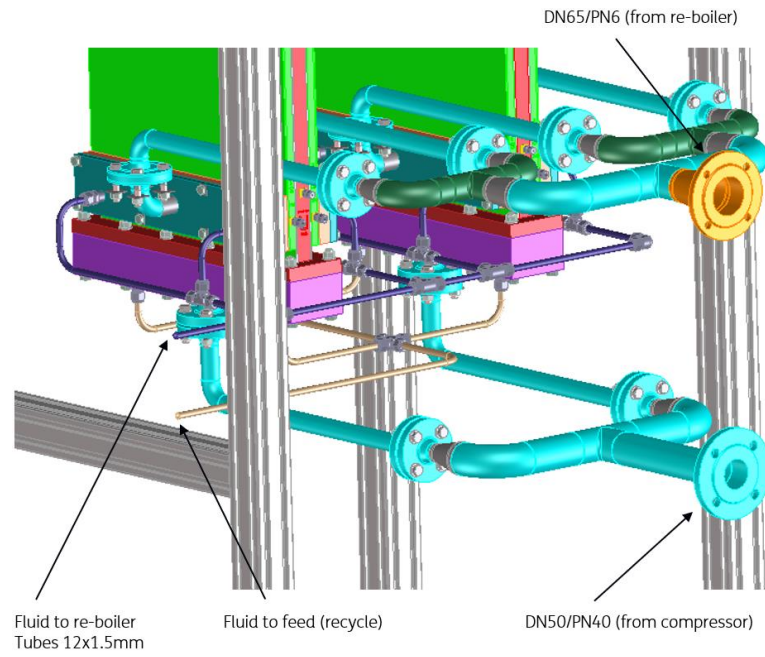


Figure 16: Connection lines in the bottom of the column.

9 Sampling and analysis

Sampling procedure:

A Dopak sampling system Figure 17 is used for sampling. The sampling points are at P-100 (feed), P- 600 (rectifying under pressure) and P- 400 (stripping sections). The samples are taken when the whole installation is in stable operation (such as flows in certain range, temperatures and pressures). It's important to sample within as short as possible time frame (1-3 minutes).

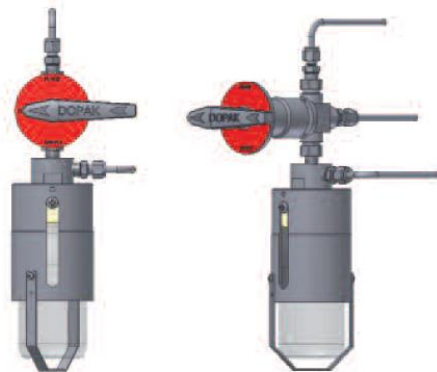


Figure 17: Dopak liquid sampling system

Actual sampling process is performed in 4 steps:

Step-1: Turn the Dopak handle to off position (see Figure 18). In this step, we provide a new septum on your sample bottle. Insert the bottle, with cap and septum into the sleeve until the septum is pierced by the needles. Put the bottle retaining clip in place.

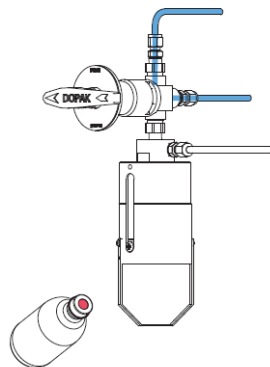


Figure 18: Off position (follow the blue line for the stream in and out)

Step-2: Turn the handle to the “bypass” position (see Figure 19), allowing a continuous flow of liquid through the sampler to ensure representative sampling. This position can be held for any required time.

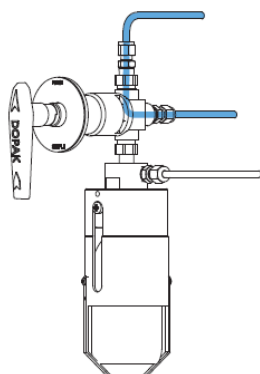


Figure 19: By pass position (follow the blue line for the stream in and out)

Step-3: Turn the handle to the “sample” position (see Figure 20), allowing the product to flow into the sample bottle. When the required amount has been taken, turn the handle to the “off” position.

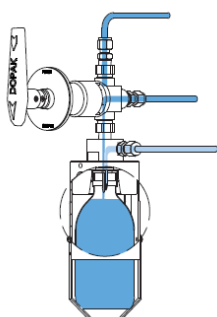


Figure 20: Sampling position (follow the blue line for the stream in and out)

Step-4: Check if the valve is turned to off position and remove the bottle retaining clip and pull the bottle out from the sleeve. The septum reseals automatically.

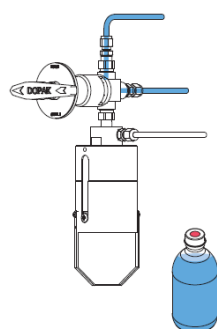


Figure 21: After sampling (follow the blue line for the stream in and out)

After collecting these liquid samples, they are analysed for Methanol and water. This is either done by Karl-Fischer titration equipment which gives estimation for water or using a refractive index, which gives information on the concentrations Methanol and water depending on the refractive index measured.

10 Costing

A preliminary cost analysis is conducted. The total cost of hours + materials excluding VAT is estimated at 461k Euros. The total cost of the equipment excluding VAT is estimated at 466k Euros. The total projects costs including VAT is estimated at 1,12 Million Euros.