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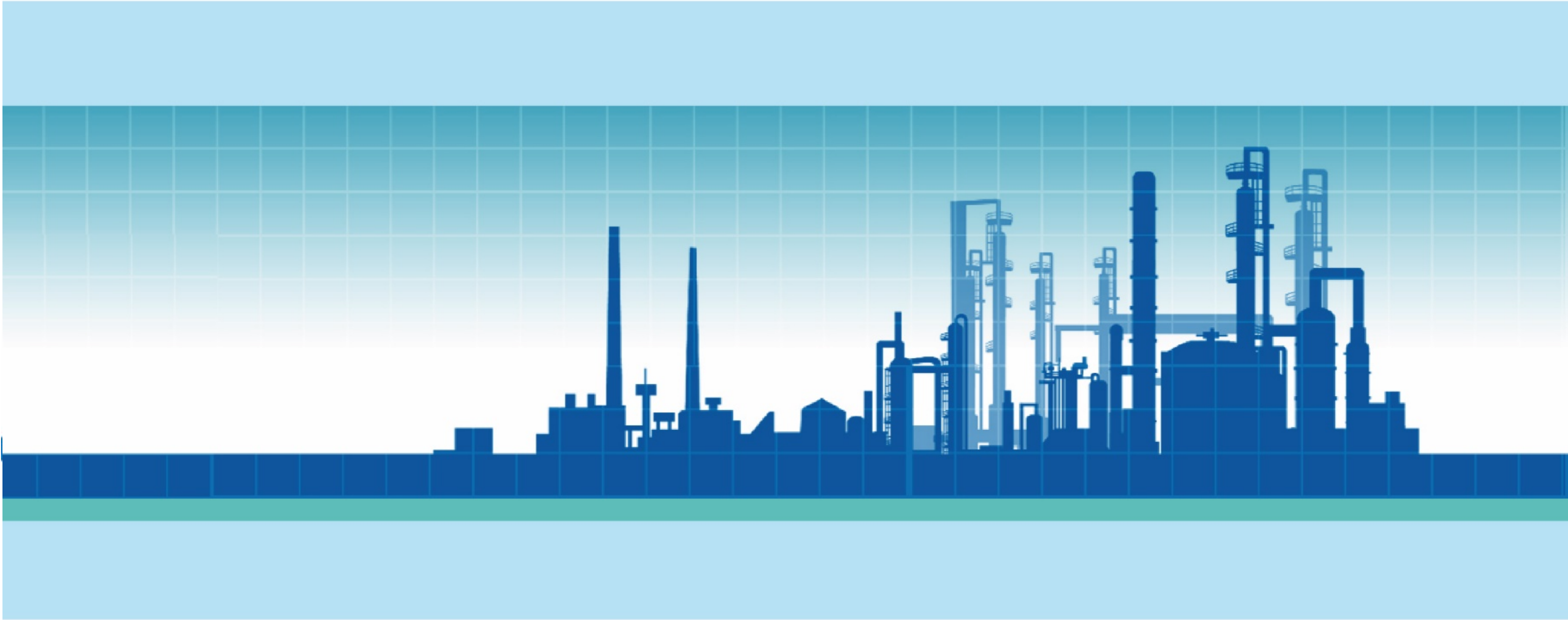
Demonstrating a Refinery-adapted cluster-integrated strategy
to enable full-chain CCUS implementation - REALISE

D2.2. Tiller plant modification and validation of CCLU

Thor Mejdell, Kai Hjarbo, Actor Chikukwa (SINTEF)



2022-06-13

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Document History

Revision History

This document has been through the following revisions:

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0.1	2021-11-02	A draft document summarizing the status sent out for internal review	Thor Mejdell
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Authorisation

This document requires the following approvals:

AUTHORISATION	Name	Signature	Date
WP Leader or QA	Juliana Monteiro	Juliana Monteiro	2022-05-30
Project Coordinator	Inna Kim	Inna Kim	2022-06-13

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Disclaimer

Executive summary

Deliverable D2.2. is defined as a document including the design and description of the bench-scale (10-15 kg/hr) CO_2 compression and liquefaction unit (CCLU) at the SINTEF's Tiller pilot plant.

The facility was designed with components like an industrial sized unit with three compressor stages including cooling and knock out drums. Out of the last drum the CO_2 gas at 35 - 40 bar is dried and then cooled down to about -5 to -10 °C by an external cooler. The liquefied CO_2 is then expanded to 15-16 bar through an expansion valve and stored at -26°C in a storage tank.

Samples of liquid can be taken from the knockout drums and samples of the gas before the external cooler. It is also possible to take a sample of the gas out of the storage tank.

Risk assessment measures that include HAZID, both classical and procedural HAZOP studies have been performed. These yielded a robust and optimal design that is characterized by safe operability and enhanced efficiency.

The unit has been tested and commissioned with CO_2 from the capture plan using 30 wt% MEA. The tests showed stable conditions in the whole unit and a gas cylinder with compressed CO_2. Gas and liquid samples from the knockout drums have been sent for analysis and will be used for developing analytical methods in Task 2.4.

Table of Contents

1 Introduction

The present work is a part of Work Package 2 dealing with technology demonstration. The objective of this work package is to demonstrate CO_2 capture from refinery stacks using the HS-3 solvent. Part of the demonstrations will take place at the Tiller plant in Trondheim, Norway.

1.1 SINTEF's Tiller CO_2Lab

The CO_2 laboratory at Tiller (Tiller CO_2Lab[1]) is a highly equipped test facility for development of post-combustion CO_2 capture technologies, as well as a research lab for flue gas pre-treatment analysis and emission research.

In the spring of 2010, a 30-meter-high indoor CO_2 absorber/desorber pilot rig was commissioned at Tiller (Figure 1-1). The pilot plant consists of a complete absorption and desorption plant with a CO_2 capacity of 50 kg CO_2/h. With more than 160 temperature sensors and 100 other sensors (flow, pressure, heat, analysers etc) the plant is very well instrumented.

The entire system is controlled by a Siemens PCS7 process control system and the plant can operate 24 hours a day. The plant is constructed for accurate measurements of key process variables including:

- Energy requirements
- Absorption capacity
- Emissions to air
- Degradation of solvents
- Other key process parameters

The results of these measurements combined with analyses of gas and liquid samples are important input parameters to SINTEF's simulation tool CO2SIM.[2] This simulation tool can be used for modelling and optimization of large-scale plants, based on pilot validation.

Flue gas from propane burner is used in the most of the tests. In 2016, the plant was equipped with a coal and biomass-burner with extra flue gas pre-treatment equipment. By proper understanding of the pre-treatment requirements of the flue gas prior to CO_2 removal, one can reduce the costs and prevent the CO_2 capture units from being damaged during continuous operation as well as to remove emission to a minimum.

Depending on the type of CO_2 capture technology utilized, there will be different pre-treatment requirements. The current pre-treatment system at Tiller CO_2Lab comprises a ceramic particle filter a cooling- and conditioning column and one column for removal of SO_2. In addition, a NOx removal unit has also been used.

In 2022 two new facilities have been commissioned: A desorber plant that can operate up to 20 bar and a CO_2 Compression and Liquifaction Unit (CCLU) which is described in the present report.

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[1] https://www.sintef.no/en/all-laboratories/co2-laboratory-tiller/

[2] https://www.sintef.no/en/software/co2sim-flowsheet-simulator-for-co2-absorption-proc/

Figure 1-1. CO_2 capture plant at Tiller CO_2Lab

1.2 Demonstration campaign at Tiller

The CO_2 plant at Tiller will be used to demonstrate the benefits of the solvent HS-3 – a blend of two amines that has been optimised in WP1. Solvent used in the demonstration campaing onsite Irving Oil Whitegate refinery during March-August 2022 using TNO's mobile pilot unit (20L) and conatining impurities from the oil refinery flue gases, as well as degradation products, will be brought to SINTEF and mixed with fresh solvent (600L). The composition of the solvent will therefore be similar to a partly reclaimed solvent. The 12 weeks long campain will be conducted from August to November 2022 and will include

- Finding the optimal L/G giving the minimum specific thermal reboiler duty in terms of MJ/kg CO_2
- Measuring solvent degradation during the campaign
- Measuring the degree of emissions of solvent and solvent degradation products to the air using various number of water wash sections (up to 4 sections).
- Generating dynamic step response data for validation of the dynamic model. In the last part of the campaign the Nonlinear Model Predictive Controller (NMPC) will be tested online in closed loop and provide valuable data for WP3.
- A unique part of the campaign is the use of a CO_2 Compressor and Liquefaction Unit (CCLU). These data are of high importance for the assessment and de-risking of CO_2 utilisation and CO_2 transport in WP3.
- Impurities in the compressed and liquefied CO_2 will quantified in Task 2.4.

The present deliverable D2.2 is a part of preparation for demonstration work in WP2.1 and describes the construction commissioning and testing of the new CO_2 Compressor and Liquefaction Unit at Tiller.

2 Design of the CCLU

Qualitly of the CO_2 coming out from the capture unit can be crucial for transportation, storage and utilisation of CO_2. In REALISE, the CO_2Lab pilot is equipped with a compact CO_2 compression and liquefaction unit (CCLU), enabling liquefying the CO_2 produced in the capture process. The main objective is to identify and quantify expected impurities in the CO_2 product when using HS-3 solvent in the refinery industry. The focus is on HS-3 amines and amine degradation products in the CO_2, but also componds like O_2 and NOx will be mesured.

For safety reasons the CCLU is built inside a cabinet with ventilation and CO_2 level alarm.

In the design of the CCLU it has been an important issue to be as close to a large scale standard unit as possible such that the results at Tiller are relevant for industrial cases.

2.1 Compressor train

The compressor train design is based on three compression stages with cooling of the gas to 20 - 25 °C and water separation after each stage. The design compressor ratio for each stage is 2.8. Assuming 1.8 bara pressure for the CO_2 rich gas from the top of the desorber this will give pressures of about 5, 14, and 40 bara after each stage. The PI&D of the compression train is shown in the Figure 2-1.

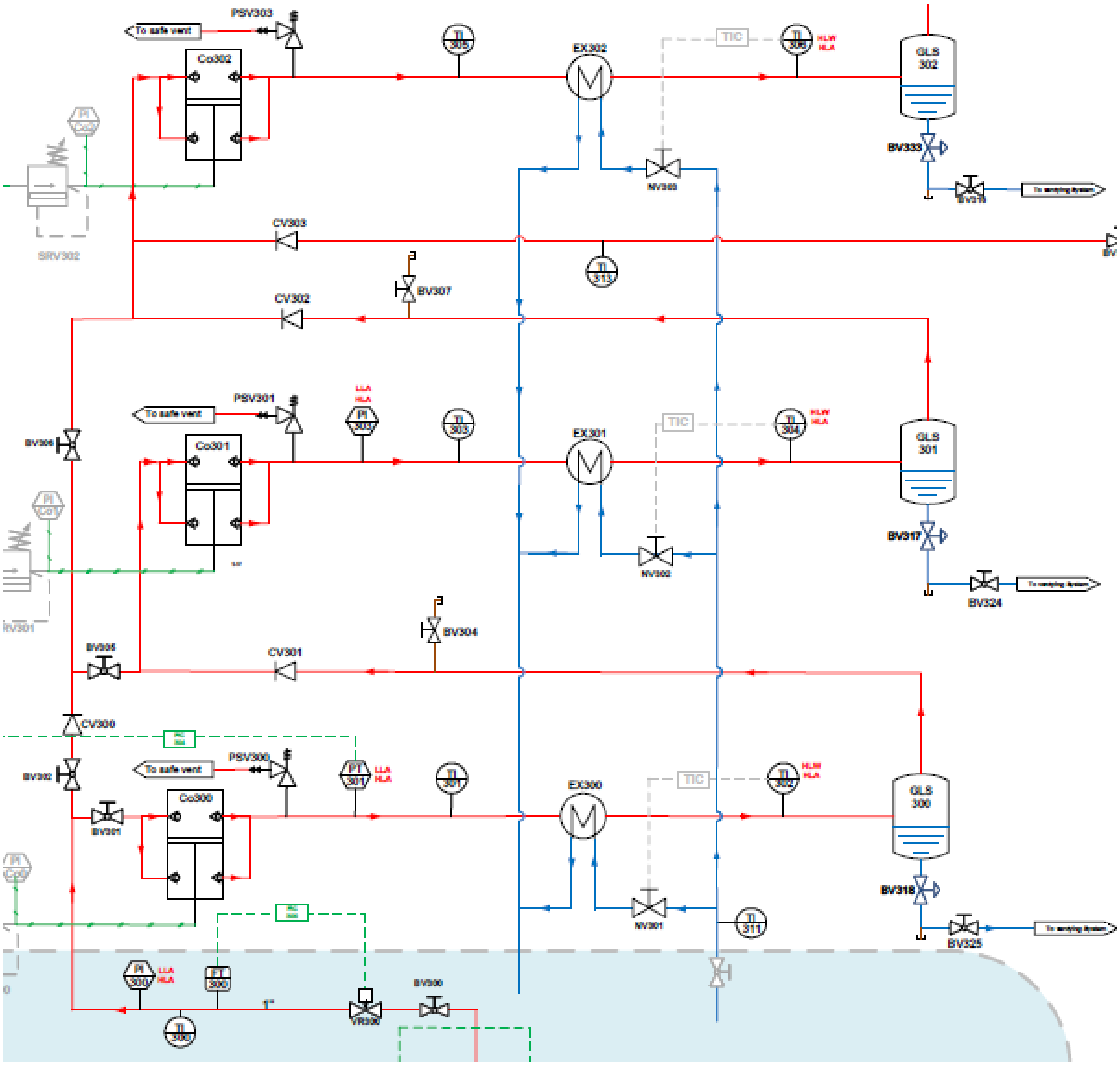
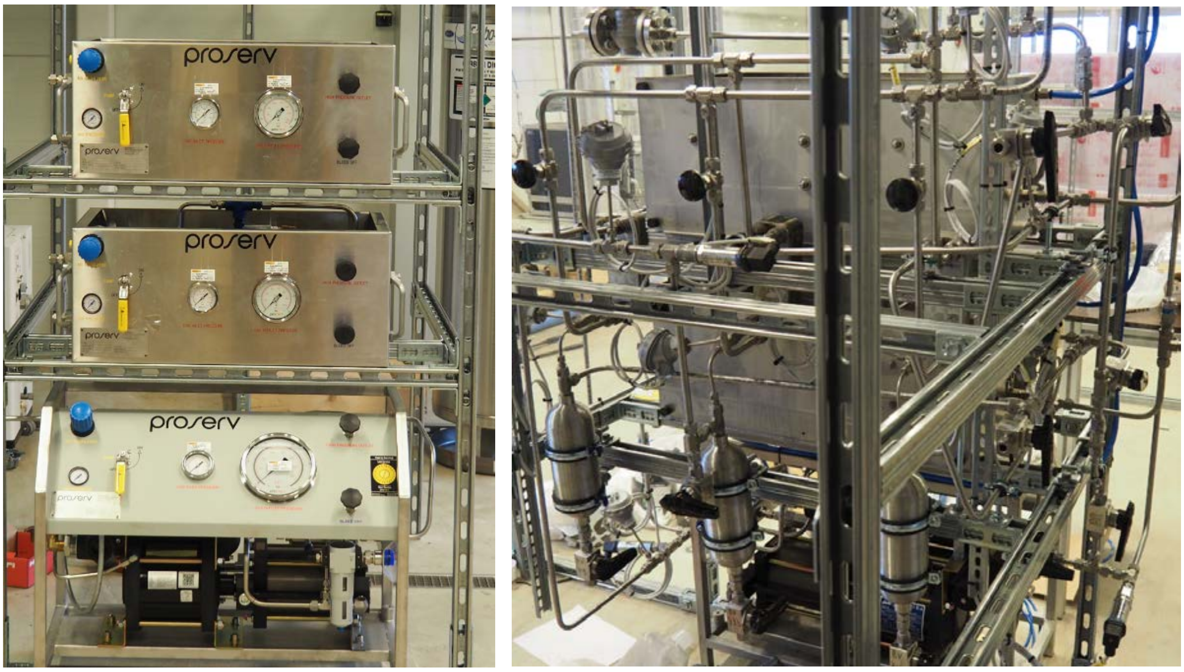


Figure 2-1. PI&D Compression part of the CCLU. The CO_2 from the stripper enters the figure at the bottom and leaves at the top.

The compressor stages are implemented using Haskel gasboosters from Proserv. These use compressed air to drive the piston in the boosters. At SINTEF Tiller such air is available for the whole area from a central compressor. Even if the design compression rato is 2.8 they have some flexibility for increasing/decreasing this ratio.

The amount of CO_2 taken from the stripper CO_2 product stream is measured by a Coriolis flow meter (FT300). Also, the inlet temperature is measured (TI300). The gas then enters the first compression stage at approximately 1.5-1.8 bar and leave it at approximately 5 bars by using the first Haskel gas booster. The gas is then cooled down to 15-20 °C and the condensed component (water or other condensate) is separated out in a knockout drum. The gas is then sent to a second Haskel booster which increases the pressure to about 14 bar and then to the third booster which gives about 35-40 bar. There are knock-out drums after each of these two compressors as well. It is possible to take liquid samples from the drums.



a) b)

Figure 2-2. The three compressors a) Front view b) back view with pipes, valves and knock out drums

Figure 2-2a shows the front panel for the three boosters. These are accessible through cut outs in the cabinet. Figure 2-2b shows the piping and knock out drums inside the cabinet.

2.2 Dryers

After the third knock out drum the compressed CO_2 gas is at 35 - 40 bar and 15 - 25 °C. The pressure is controlled by a control valve. The gas at this stage will have 400 – 600 ppm of water. To get down to 20 – 30 ppm that often is required, the gas is dried in a cylinder filled with molecular sieve 3Å beads. Two such cylinders are mounted in parallel (see Figure 2-2) to increase the flexibility of the system.

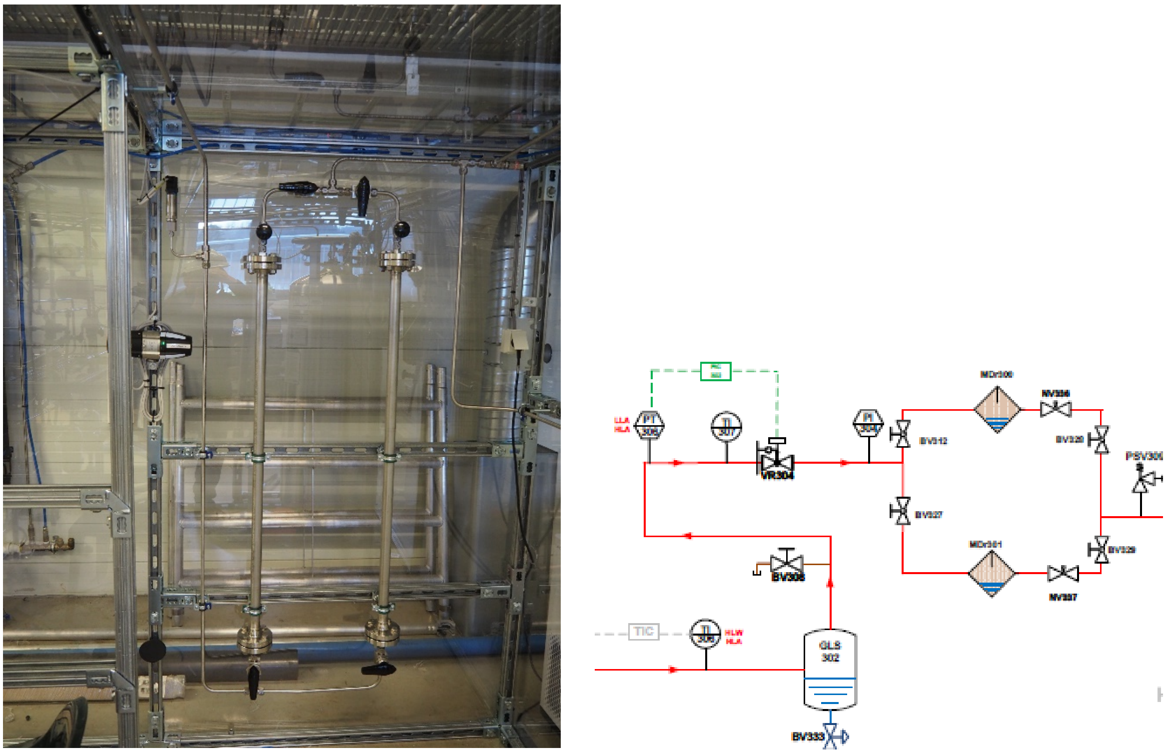


Figure 2-3. The drying system of the CCLU

2.3 Liquefaction

An external Lauda Integral IN 250XTW cooler (See Figure 2-4) provides a cooling medium (ethanol) at typically -5 to -20 °C. The CO_2 gas is liquified with this ethanol inside a plate-and-frame heat exchanger. The temperature and pressure of the liquid CO_2 out of the exchanger is typically -5 to -15 °C and 30 – 40 bar.

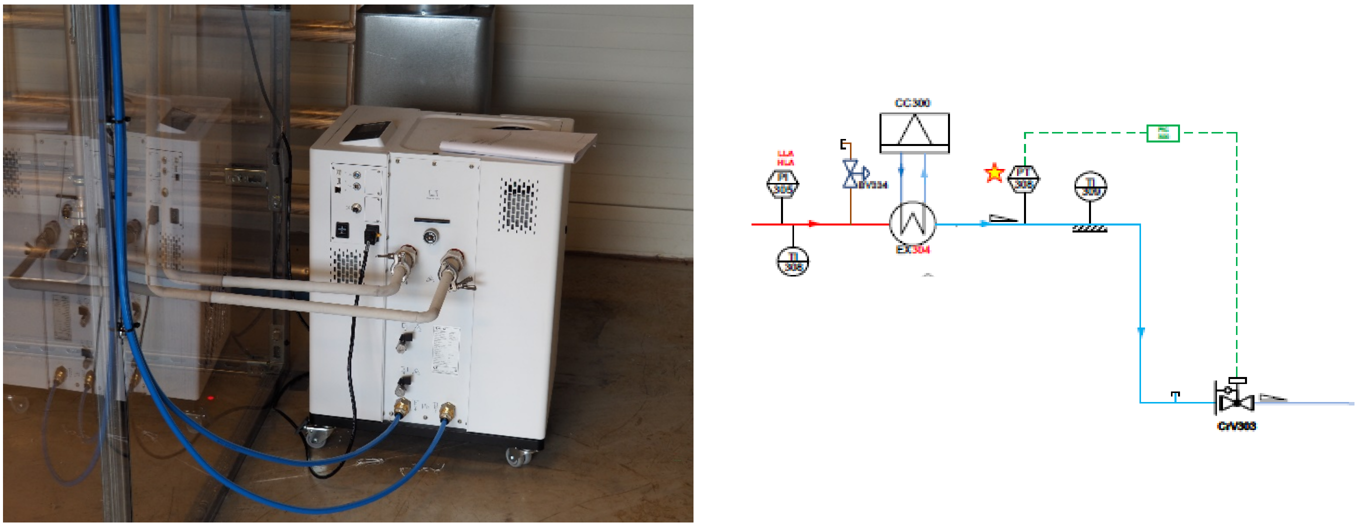


Figure 2-4. a) The external cooler Lauda. *b) Cooling and expansion of CO_2*

Afterwards the liquid is expanded through an expansion valve CrV303 to the desired pressure (15-16 bar). This will produce a two-phase stream at about –26 °C that enters a CO_2 storage tank (Carbo-Max450) produced by Linde (Figure 2-5).

The gas phase will leave the tank through a control valve that keeps the pressure at 15 -16 bar. The liquid will be stored in the tank. The tank is well insulated, and any heat loss will be compensated by evaporation of liquid CO_2, which is then released through the control valve.

The gas out of the tank may either leave the unit to ventilation or be led back to the last stage compressor.

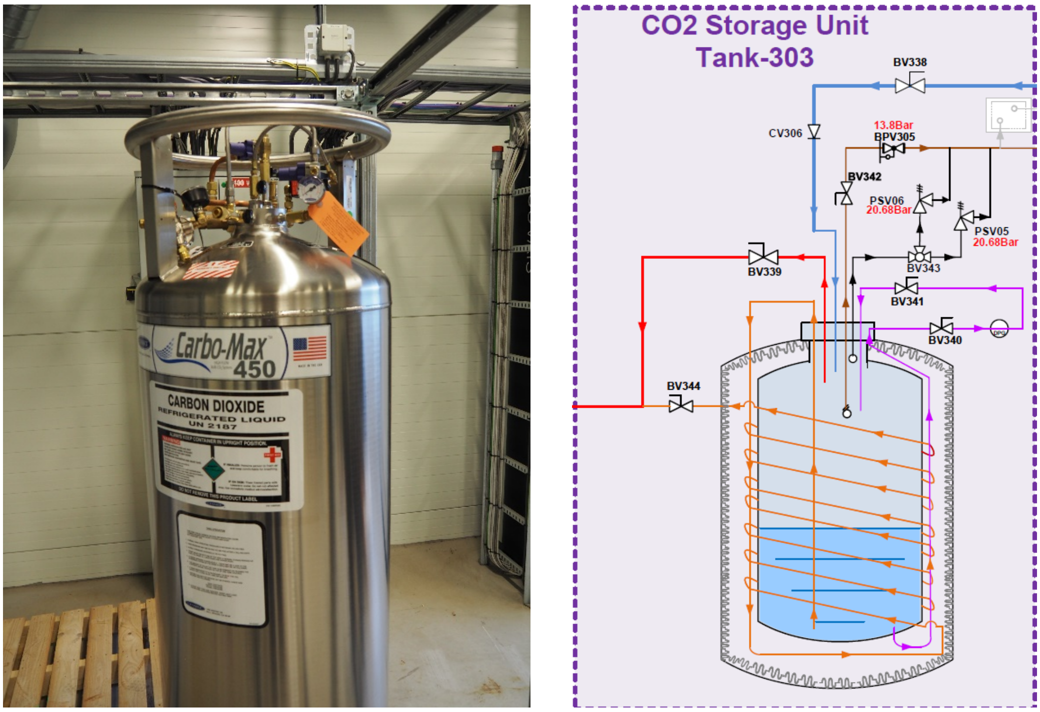


Figure 2-5. CO_2 storage tank CCLU.

The complete PI&D for the CCLU is shown in Figure 2-6. The sensors and control loops are implemented into the Siemens PC7 system of the pilot plant.

It is possible to take a sample of the gas after the dryers and of the gas stream out of the storage tank. Liquid samples can be taken from the knockout drums.

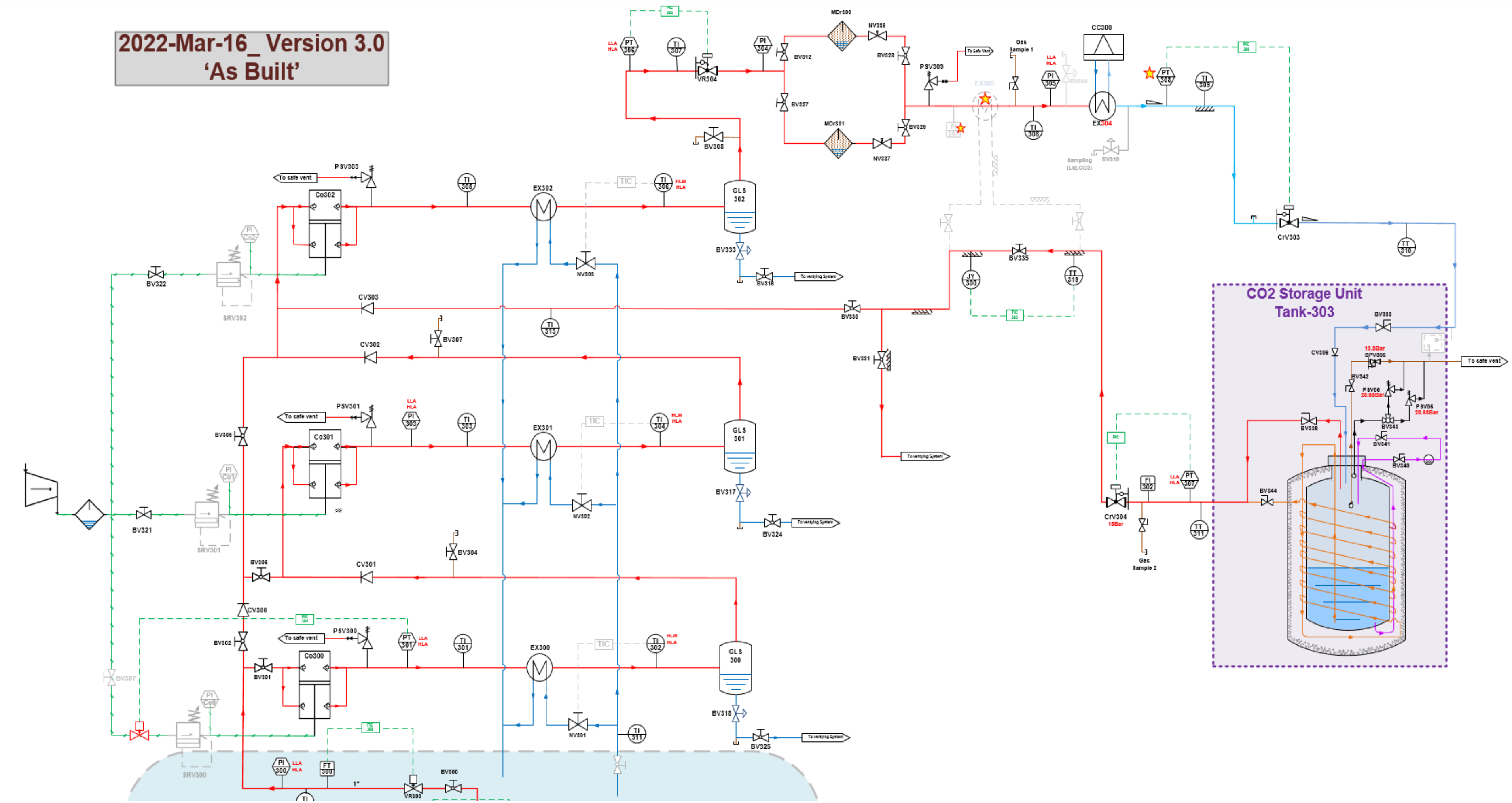


Figure 2-6: CCLU PI&D

3 Risk assessment with HAZOP/HAZID

3.1 Background

A holistic approach to ensure a robust technical design as well as safe operation was considered. Both classical- and Procedural-HAZOP studies were conducted to ensure quality as well as safe operation. Classical HAZOP mainly focussed on design optimization whilst the procedural study paid particular attention to operational safety.

3.2 HAZID/HAZOP (Classical) studies

This section concerns a concise summary of the combined HAZID/HAZOP studies that were conducted to improve the design as well as technical robustness & efficient operation of a CO_2-Compression-rig built SINTEF's CO_2Lab (Tiller, Trondheim) in the REALISE project.

HAZID - this aspect was conducted to qualitatively identify and evaluate potential hazards associated with the operation of the system/process that could cause harm to operators or affect safety of operation. This included, among other things, analysis of: design basis and design philosophy, base information review, identification of any hazards that could result in the loss of containment, injury to persons or damage to equipment/ structures. The intended goal was to make and implement appropriate safeguards, measures as well as appropriate recommendations in order to ensure high safety standards. The hazard- and operability-indices were assessed according to SINTEF’s reference safety criteria.

HAZOP - in the classical (and technical) sense, involves the review of a process in a formalised and systematic way by a multidisciplinary team of engineers & technical specialists. Typical Hazop sessions are focused on how system operation and ranges may deviate from that intended by design.

The design basis was improved by iterating the initial Piping and Instrumentation Diagrams (P&IDs) in a progressive manner based on HAZOP iteration results. The HAZOP philosophy comprised of analytical study sessions of the process and design refinements, in which case, the desired status of the system/process was scrutinzed against potential deviations from the intended behaviour potentially representing a hazard or operability problem.

Process scope – the study was based on a design for a new rig. The scope therefore naturally took into consideration the entire process chain from design, HAZID-HAZOP studies, testing, commissioning and the anticipated modes of operation.

Since SINTEF's CO_2Lab at Tiller is a complex facility with several rigs, some of which could be running at the same time, physical boundaries were considered in the context of potential hazards in a manner that took into account neighbouring rigs/facilities/activities. This included consideration of simultaneous operations that might take place in the vicinity as well as interaction through shared utilities like N_2, cooling H_2O, pressurized instrument air, etc.

As a general approach, progressive development of the P&ID was largely based upon the findings and recommendations from successive HAZOP study series as well as subsequent technical discussions in some of the project meetings. The resultant suggestions, changes and improvements were incorporated into the P&ID yielding a refined version each time, until it was considered to contain adequate detail deemed satisfactory by the technical team. Figure 3-1 shows a schematic illustration of the developmental phases of the P&ID based on HAZOP analysis studies.

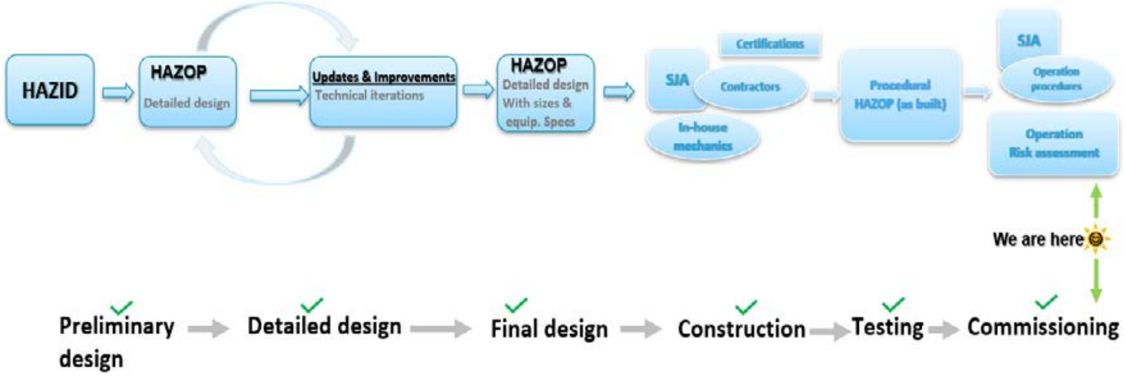


Figure 3-1: Schematic showing the evolvement of the P&ID based on HAZOP analysis studies

3.2.1 HAZOP Objectives

The objectives of the HAZOP session were to:

- Identify potential hazards, causes, and their potential consequences
- identify design deficiencies with potential to cause operability/safety problems
- Find solutions to identified operational problems
- Find appropriate safeguards to HAZARDS

3.2.2 HAZOP Methodology

The HAZOP sessions were based on parametric deviation analysis. The method relies on establishing sets of commonly applied deviations (i.e., from normal operation design range) on typical and relevant parameters/properties/operations. Each element (i.e., parameter, property, etc.) is subjected to scrutiny by assigning relevant deviations like: High, Low, No, etc. Put together, the resultant deviations effectively form a "library of deviations" which can be repetitively used, depending on the equipment type or node/section being 'HAZOP-ed'.

Different types of HAZOP exist, albeit parametric-deviation based HAZOP (methodology found in IEC Standard 61882, Hazard and Operability Studies Application Guide) is the commonly used form of HAZOP in SINTEF today.

It is simple to use, and if properly executed, usually yields reasonably consistent outcomes with an appreciable degree of quality.

Nevertheless, the HAZOP team must be cognizant of the following aspects:

- not to overlook certain interactions or special case deviations
- not to process more deviations than necessary (this can be time consuming and expensive)

- Take into consideration other activities going on in same building during the test period.

3.2.3 HAZOP Analysis

The study was characterized by subdividing the P&ID into appropriate 'nodes' (compression, chiller and storage). Nodes were organized by steps of an operation/activity focusing on a specific process section under study, in which case: parameters, inputs and outputs for each stage/node were specified 1st. To enhance intuitive visualization of the scenarios during the HAZOP studies, the anticipated change in conditions along the main flow line are figuratively indicated by (differently) coloured streams. After an introduction of the project intentions, historical progress with pertinence to HAZOP, objectives as well as a walk-through of the new P&ID changes/updates (by the designers), HAZOP analysis iterations were performed.

There are no firm rules regarding this organization of nodes nor the sequence applied during HAZOP, albeit, the analysis was carried out in sequential fashion to ensure inclusion of the full study scope in a logical and structured manner.

The essence is to be logical and clear such that all participants understand the area, operation and or activity in which the study attention is currently focused.

3.2.4 Main HAZOP (Classical) Recommendations and findings

The major constituent components that play important roles and therefore essentially govern HAZOP considerations during the sessions included: the buffer tank, the compression train, the chiller and the CO_2 storage tank. It should be emphasized that HAZOP-studies (parametric deviation based) focused on design improvement and optimization fused with HAZID.

The main points can be summarized as follows:

- Ensure materials of correct pressure rating are to be used. System parts that are not already approved by a vendor should be pressure tested to 60 bar before the commissioning stage.
- HAZOP study Analysis done for compression stage 1 is valid for stage 2 & 3.
- Procedural HAZOP to be conducted for the as-built design. Periodic pressure test was recommended.
- Certification must comply with regulations.
- All operators should be well trained and must wear full safety gear.
- Safety or technical risk management layers were included in the design based on main parameters i.e., pressure (e.g., LLA, HLA, etc), Temperature, Flow etc.
- The CO_2-compression rig should be installed in its own enclosure with proper ventilation, but operators must work from outside.

3.2.5 Action points & Closing-out

Discussion and results were recorded by exception in HAZOP-log sheets from which the required action points were then summarized in a simplified easy-to-follow table. Monitoring and fulfillment of the close-outs was be based on action-number. The action-numbers are organized and numbered in conjunction with reference nodes used in during the HAZOP iterations.

The standard way of dealing with close-outs involves preparation of separate and specific HAZOP-action sheets for each action-number item. For convenience, this can be further simplified into an Action Control Sheet (ACS), which is essentially, a concise list used to monitor and check individual close-out items in order to ensure that each case is properly addressed/solved to the expected standards. This is achieved by listing all the action-numbers on a single ACS with provision for columns that show close-out status, outstanding issues, commenting and dates where applicable. The custodian(s) of the HAZOP study monitors the completion of the ACS based on progress guided by proposed close-out dates.

For HAZOP studies with not so many action requirements as in this case, separate sheets for close-out are not necessary. The usual practice in SINTEF-KPMT in such cases is to assign ACS-custodian whose task is to ensure fulfilment and compliance of the all the items on the ACS.

3.3 Procedural HAZOP

There is great value in stress-testing new process designs and their corresponding procedures (or even just revise existing ones) to ensure they will work/perform as intended and will not generate unintended adverse consequences. Procedural HAZOP provide is one way of accomplishing this.

In the context of the CO_2 liquification rig design and development, procedural HAZOP was conducted in a fashion that considered a combination/interaction of hardware and software as well as procedures/practices of other systems/rigs within the CO_2Lab including people (operators). In other words, the activity required a holistic approach in order for it to produce an appropriate sequence that would result in simultaneous (& safe) operation of the liquefication process and other rig-facilities located within the Tiller CO_2Lab. In such a setting, interaction between hardware, software and people typically results in a complex environment, in which case, identification of the inherent issues with relevance to interaction, utility sharing and sequence pauses a significant challenge if not handled properly.

While classical HAZOP mainly focusses on the P&ID details, procedural HAZOP basically follows the same philosophy, albeit based mainly on the 'procedures document', because coordination and execution of complex processes largely rely on reliable procedures in order to ensure that activities are undertaken consistently and to levels that are compliant with expected standards and regulations. A Procedural HAZOP study is, therefore, essentially a form of risk assessment applied to procedures. The scrutiny (on procedures) provides a check on a procedure’s resilience by testing how it responds to stresses or excursions outside normal conditions, as represented by the guidewords.

Stress testing is achieved by asking questions based on a set of relevant guidewords, in which case, the guideword is practically a combination of words that describe a critical property or parameter of the process/procedure that might deviate from normal or expected behaviour. In a procedural HAZOP, 'action' is a key parameter. So, combining deviations with the parameter ‘action’ generates guidewords like ‘no action’, ‘more action’, ‘less action’, ‘wrong action’ etc.

3.3.1 Objectives

To stress-test the new CO_2 liquefaction process design and the corresponding procedures to ensure safe operation, expected performance and quality of the intended product.

3.3.2 Methodology

Typically, procedural HAZOP was preceded by technical meetings that established the initial draft of the procedures. Each session started with a quick review and update to the latest version of the procedures document. The appropriate guideword library (only choosing deviations with pertinence to subject/section understudy) would then be used to prompt risk identification iteration. In each case, the team discussed:

- potential causes relevant to the circumstances described by the guideword
- nature and extent (severity & probability of occurrence) of the consequences if that situation occurred
- controls in place to reduce/eliminate the probability of that situation arising or the consequences if it did arise
- any further safeguards and priorities that would be required to eliminate the risk

3.3.3 HAZOP Outcomes

The outcome of the procedural HAZOP sessions were amendments to the procedure draft, (resulting in a refined version each time) together with ancillary actions. All this was tailored to reduce the risk and ensure the successful achievement of the objectives. The amendments and actions mainly included:

- The re-phrasing (or re-wording) of instructions for steps in the procedure
- The re-ordering, addition and or removal of steps or instructions
- Highlighting 'warnings' or ‘watch-out’ remarks to the procedure

3.4 Summary of the HAZOP studies

The classical HAZOP study successfully produced a set of required action points which, after proper implementation, yielded to a robust final P&ID design that enabled efficient operation (i.e., operability) of the rig in a manner that produced the intended product, liquefied CO_2. On the other hand, procedural HAZOP analysis produced a well-structured and -sequenced set of procedures that enabled safe operation of the rig.

As a general comment, it is worthwhile to note that the technical team was composed of participants with a diverse and appropriate background that exhibited (during the HAZOP sessions) comprehensive competence and adequate experience whose integration traversed all the pertinent aspects of the subject matter at stake. The integrated efficacy of the combination i.e., technical team, both classical and procedural HAZOP studies, was evidenced by the successful commissioning of the CO_2 liquefication rig.

4 Commissioning and testing

4.1 Compression train

The first part of the CCLU that was ready for testing and commissioning was the compressor train with all parts from the gas entrance and up to the control valve VR304 before the dryer section. This part was tested and commissioned in January 2022 and included the Haskel boosters, coolers, knock out drums, pressure sensors and temperature sensors.

First the compression train was tested with air. All the three compressors performed well. The pressure increase could be adjusted from the operator panel outside the cabinet and was set according to the design levels. A small leakage was detected by a decrease in pressure over time. By tightening some connections these leakages were fixed and the whole train was gas tight.

The pilot plant was then started to produce CO_2 the 17th of January using 30 wt% of MEA.

4.1.1 Test performed 18th of January

The next day, when the whole pilot plant was in steady state, the CO_2 from the top of the desorber was led into the compression train. Figure 4-1 shows the pressure at each stage during the test.

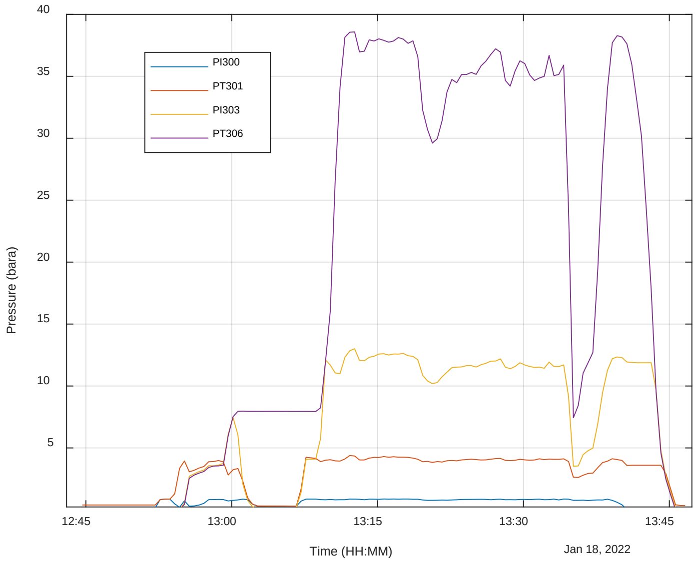


Figure 4-1. Pressures during the first test with the gas boosters

The pressure after the first compressor (PT301) was around 4 bar, the pressure after the second compressor (PT303) was typically 12 bar and out of the last compressor 35 bar. All in absolute values.

In Figure 4-2 the temperature measurements before and after the three coolers are shown.

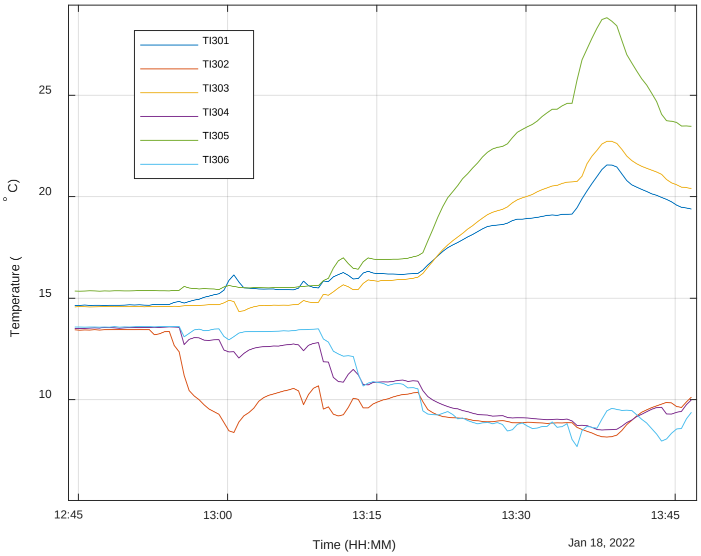


Figure 4-2. Temperatures during the first test with the gas boosters. TI301 after first booster, TI302 after first cooler, TI303 after second booster, TI304 after second cooler, TI301 after third booster, TI302 after third cooler.

The cooling was very efficient, and the gas had typically 8°C after each cooler.

4.1.2 Test performed morning 19th of January

A new test was performed the next day and the corresponding pressure measurements are shown in Figure 4-3

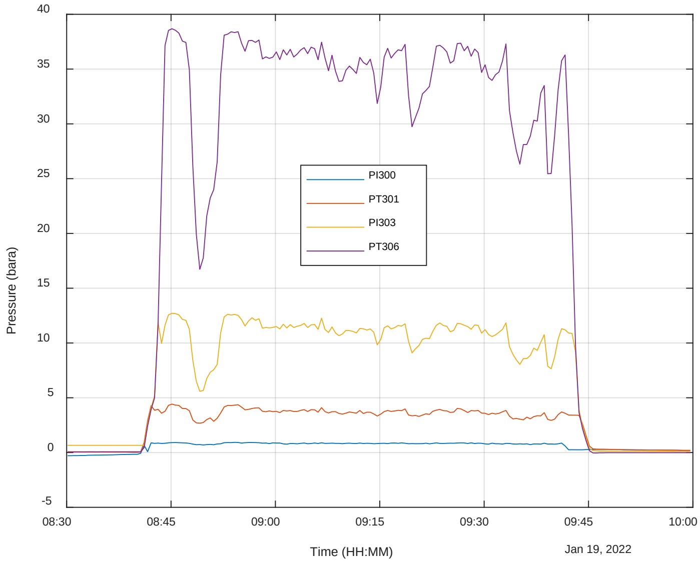


Figure 4-3. Pressures during the second test with the gas boosters

The pressures levels were quite similar as the first day with the first compressor (PT301) around 4 bar, the pressure after the second compressor (PT303) around 12 bar, and the last stage pressure at 35 bar. Also, the cooling temperatures were at the same level (Figure 4-4)

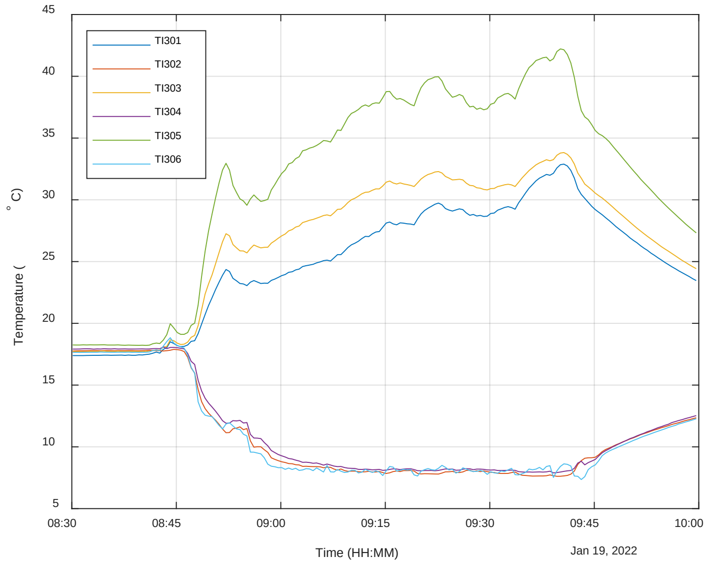


Figure 4-4. Temperatures during the second test with the gas boosters

4.1.3 Test performed afternoon 19th of January

A new test was performed later the same day with pressures shown in Figure 4-5.

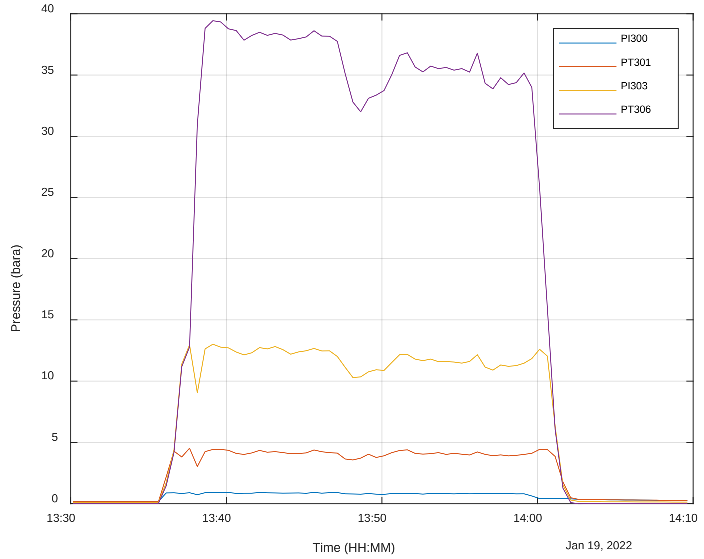


Figure 4-5. Pressures during the third test with the gas boosters

The pressure levels were about the same, but they varied less with time. Smoother curves were also seen with the temperatures shown in Figure *4-6*.

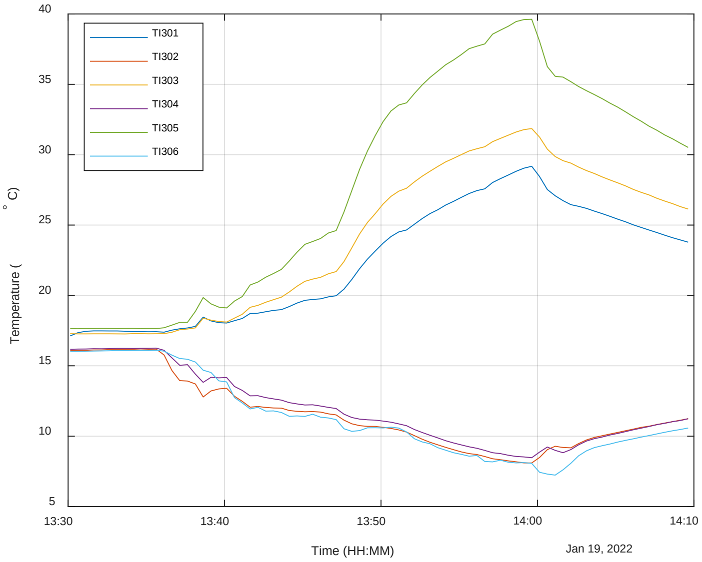


Figure 4-6. Temperatures during the third test with the gas boosters

4.2 The complete CCLU

In the beginning of March 2022, the rest of the CCLU with driers, valves, pressures, temperatures, heat exchanger and external cooler was in place. Before the testing with CO_2 the pipeline from VR304 to CrV303 equipment was pressure tested with Nitrogen. This was performed the 3rd of March and some leakages was detected and fixed.

The pilot plant was then started the 7th of March and run to 16th of March. There were some problems with the pilot plant such that the first day of testing was the 10th of March.

4.2.1 Test performed 10th of March

The test with CCLU was started when the whole pilot plant had been in steady state for several hours. The CO_2 from the top of the desorber was then led into the compression train. The Coriolis flow sensor FT300 was at this time in operation such that the total amount of gas could be measured. In Figure 4-7 the flow rate of the gas is shown. The flow was typically around 5 kg/h but with some frequent variation.

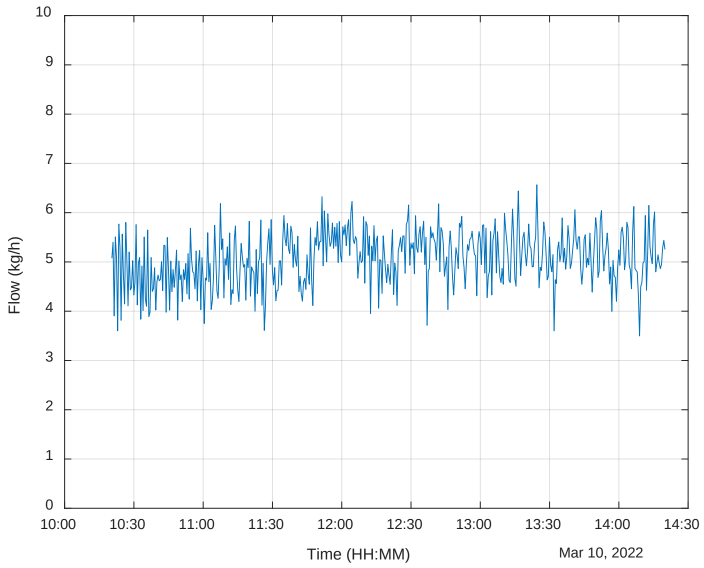


Figure 4-7.Gas flow rate into the compression train during the 10th of March test.

The pressure levels are shown in Figure 4-8. As can be seen they are very constant during the almost 4 hours of testing.

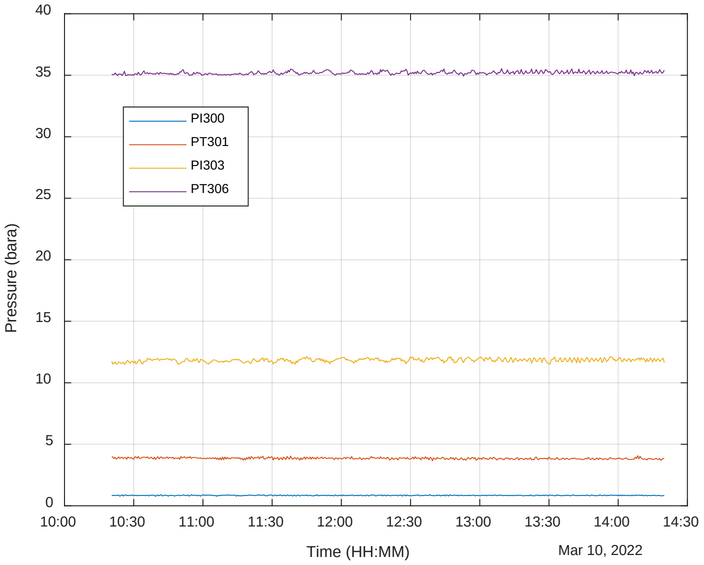


Figure 4-8. Gas booster pressures levels during the 10th of March test

This is also the case for the temperatures before and after the knockout drums shown in Figure 4-9.

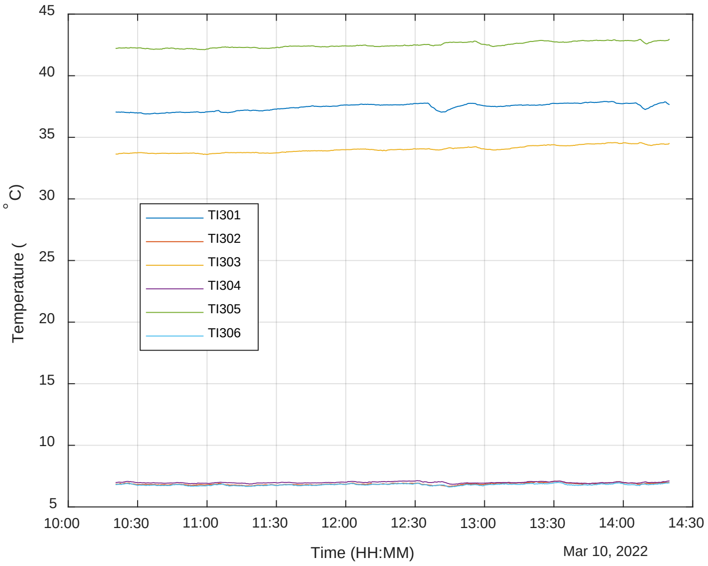


Figure 4-9. Temperatures in the compression train part during the 10th of March test

In Figure 4-10 the pressure before and after the expansion valve is shown.

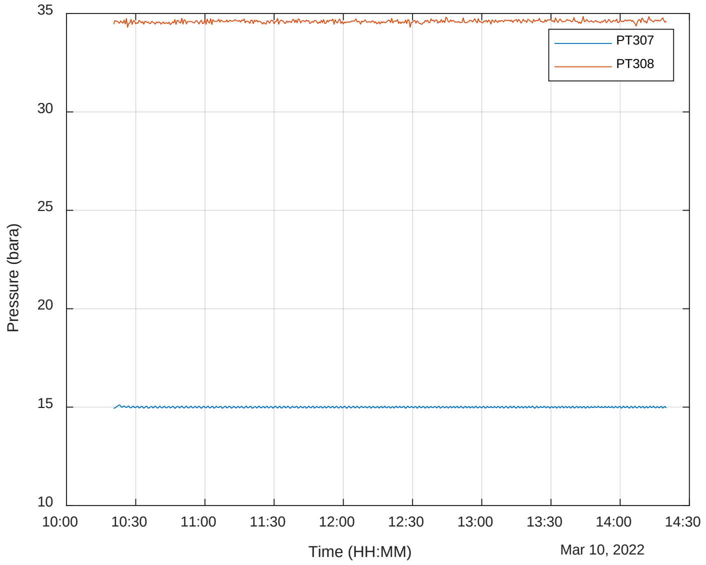


Figure 4-10. Pressures before and after the expansion valve during the 10th of March test.

They are also very constant during the test period. In Figure 4-11 the temperatures in the liquefaction part are shown. The temperature sensor after expansion (TT310) showed a value that was above what was expected from the phase diagram. The span for this temperature sensor had to be changed because it's preset minimum of -21.4 °C was exceeded in the test.

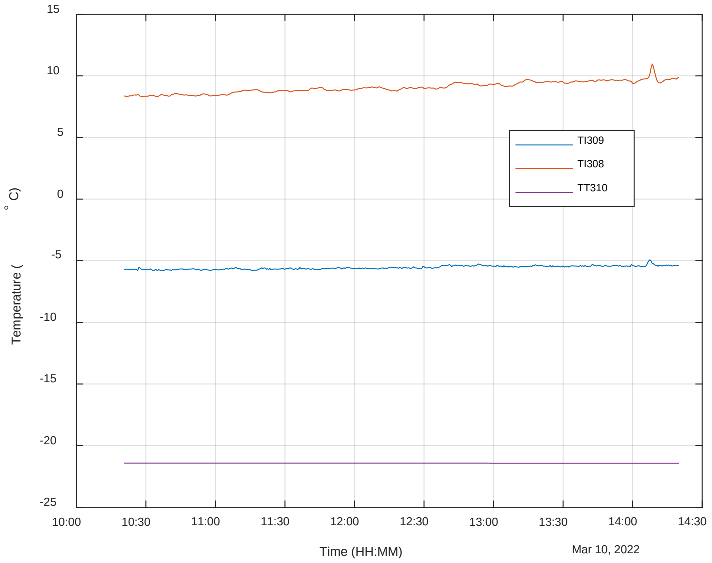


Figure 4-11. Temperatures before (TI308) and after external cooler (TI309) and after expansion valve (TT310)

4.2.2 Test performed 14th of March

Before the next test at 14th of March the span for the temperature sensor (TT310) was changed together with some other minor changes in the unit. One of the issues that was not satisfactory in the first test was the amount of CO_2 in the CCLU, and an increased capacity was wanted.

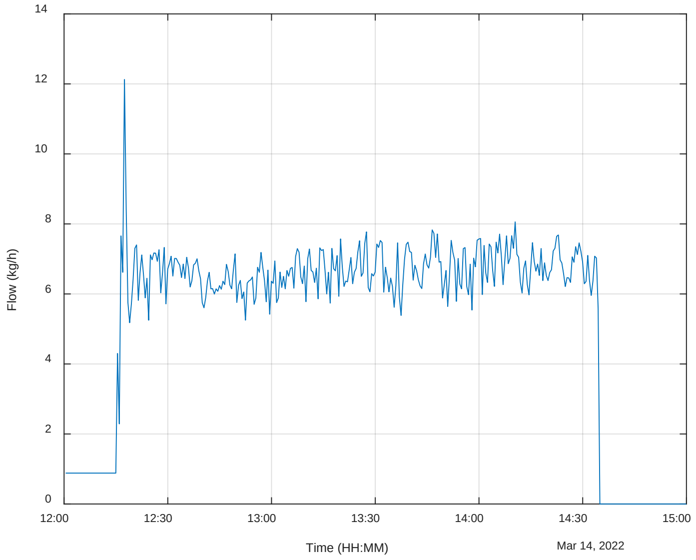


Figure 4-12.Gas flow rate into the compression train during the 14th of March test.

Figure 4-12 shows that the flow had increased, but not up to 10 kg/h, as wanted.

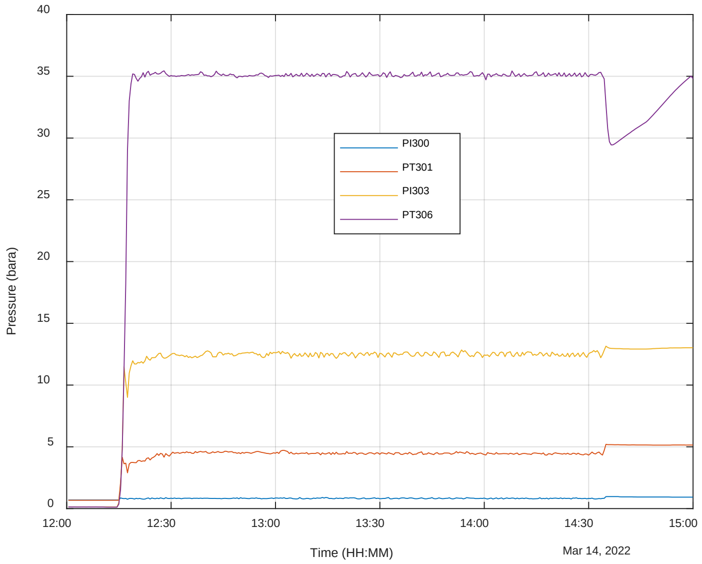


Figure 4-13. Gas booster pressures levels during the 14th of March test

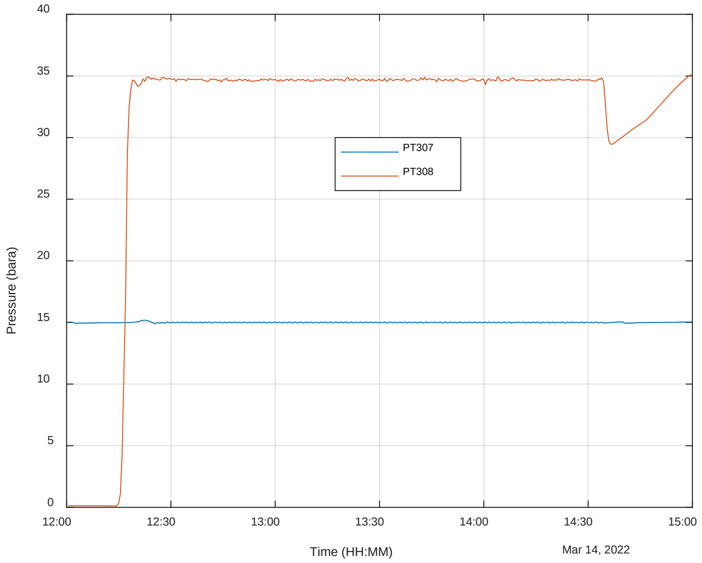


Figure 4-14. Pressures before and after the expansion valve during the 14th of March test.

Again, the pressure levels (Figure 4-13, Figure 4-14) and the temperatures after cooling (Figure 4-15) were constant during the test.

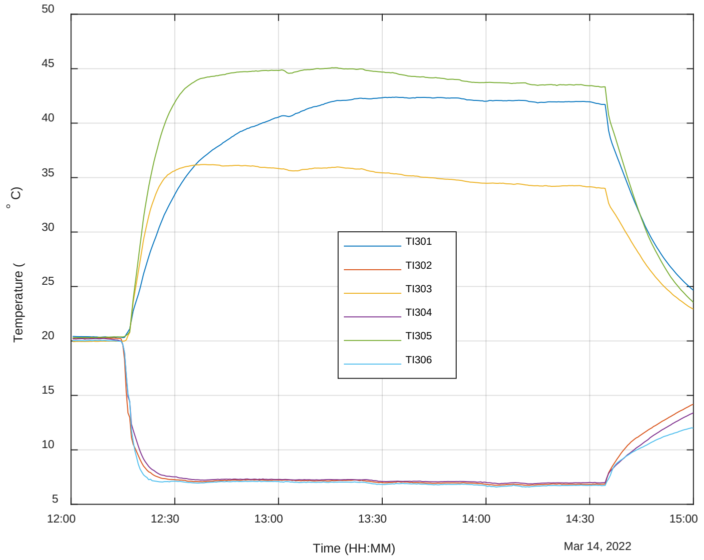


Figure 4-15. Temperatures in the compression train part during the 14th of March test

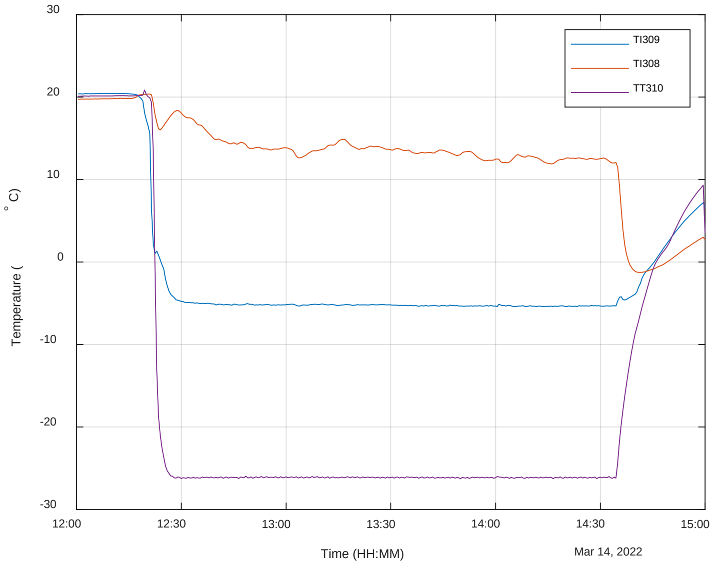


Figure 4-16. Temperatures before (TI308) and after external cooler (TI309) and after expansion valve (TT310) during the 14th of March test.

Figure 4-16 shows that this time the temperature sensor after the expansion (TT310) showed a value of about -26 °C which is according to phase diagram reasonable.

4.2.3 Test performed 15th of March

The tests continued the next day. We wanted to test if we could get higher flow rates by changing the valves controlling the gas rate. The pressures are shown in Figure 4-17 and Figure 4-18. The resulting gas flow is shown in Figure 4-19.

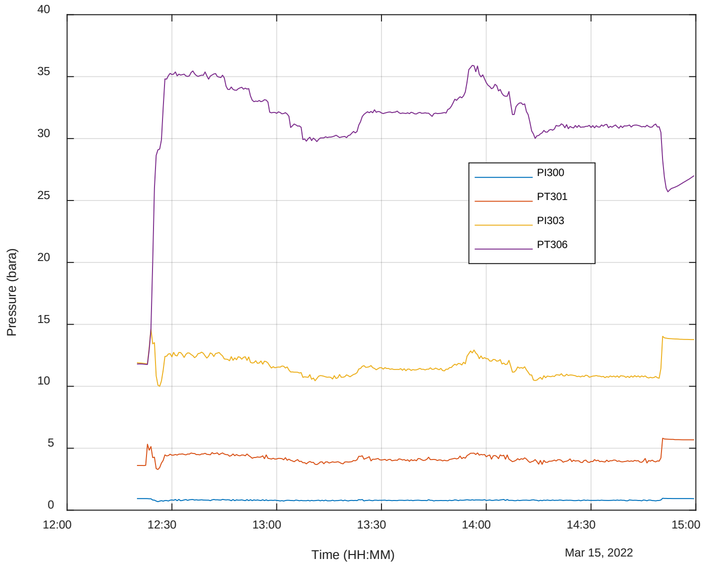


Figure 4-17.Gas boosters pressures levels during the 15th of March test

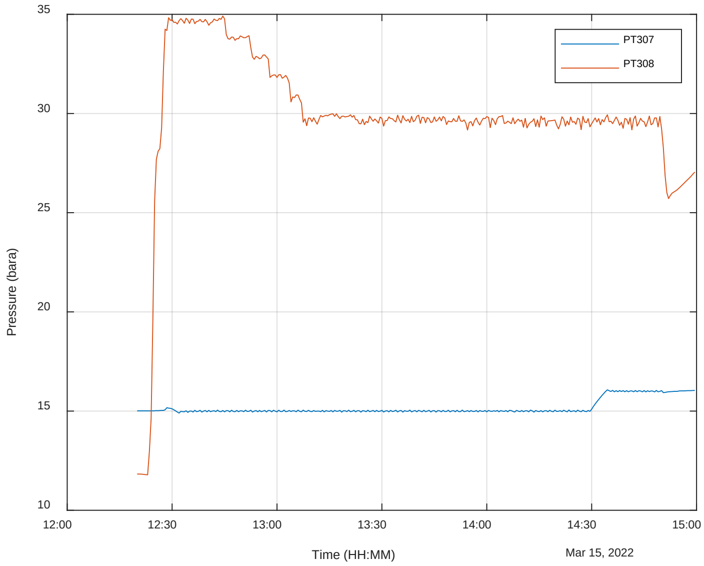


Figure 4-18. Pressures before and after the expansion valve during the 15th of March test.

The pressure at the top of the compression section (PT306) was first at 35 bar and then from 12:45 to 13:10 reduced in steps down to 30 bar. The pressure (PT308) before the expansion valve (CrV303) followed these changes but was slightly less due to the pressure drop in the pipes and the dryer. The pressure in the storage tank was kept at 15 bar by the control valve CRV304. As a result of the changes the gas flow rate increased from 7 to 11 kg/h as shown in Figure 4-19.

Afterwards the pressure PT308 was kept constant while the pressure PT306 was changed. At 3:20 the pressure was increased to 32 bar. The resulting flow rate was reduced only slightly from 11 to 10.5 bar. However, when the pressure was increased to 35 bar the flow rate turned back to 7 kg/h.

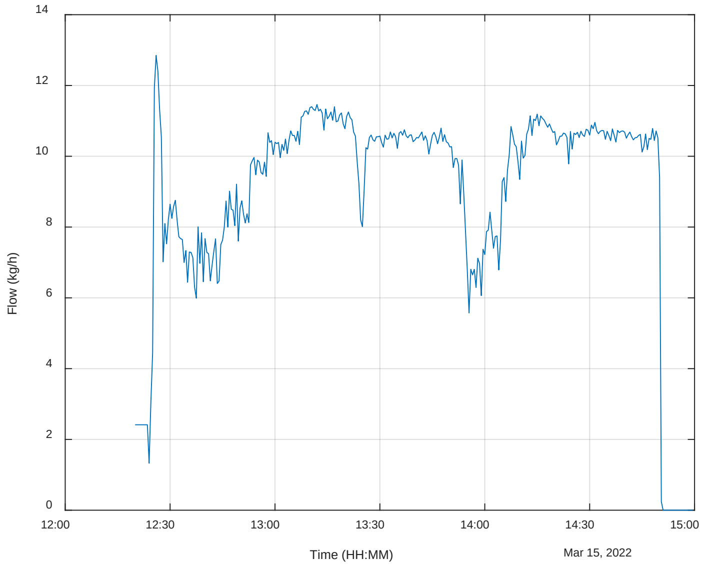


Figure 4-19.Gas flow rate into the compression train during the 15th of March test.

It can be concluded that the main resistance in the system was over the valve VR304 and not the drying section or the expansion valve CrV303.

At 14:30 the set point for the pressure in the storage tank (the control loop using CrV304) was increased from 15 to 16 bar. This had no effect on the on the flow. But as expected, the temperature TT310 increased from -26 to -24 °C as seen in Figure 4-20.

It was also found that the location for taking samples of the gas phase was located to close to the heat exchanger EX304 possibly allowing backflow of liquified CO_2 into the sample point.

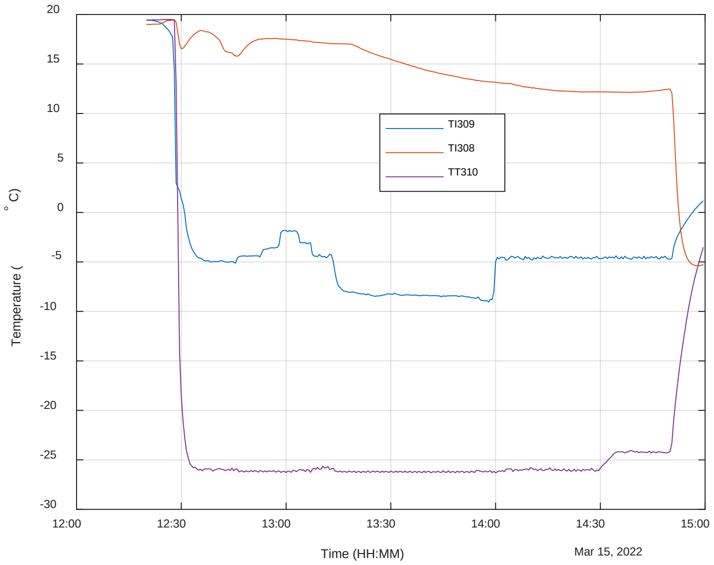


Figure 4-20. Temperatures before (TI308) and after external cooler (TI309) and after expansion valve (TT310) during the 15th of March test.

4.2.4 Test performed 5th of April

Two modification was done before the CCLU was tested again.

1) The location for gas sampling was moved to avoid any liquid coming into the sample cylinder.
2) The amount of air to the boosters was increased by increasing the diameter of a junction pipe in the supply system.

The last item was for increasing the capacity of the boosters. The plant was started again and the CCLU started the compression at 12:30. The pressure measurements are shown in Figure 4-21 and Figure 4-22. The PT306 was 35.6 bar and PT308 34.4 bar.

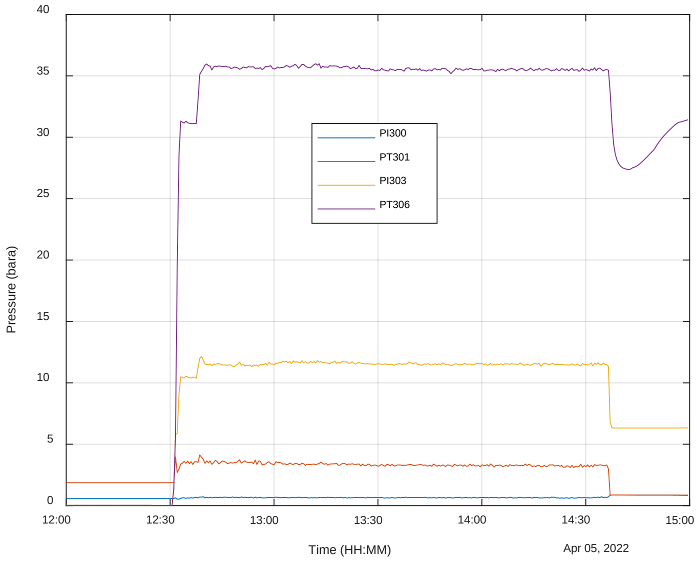


Figure 4-21. Gas boosters pressures levels during the 5th of April test

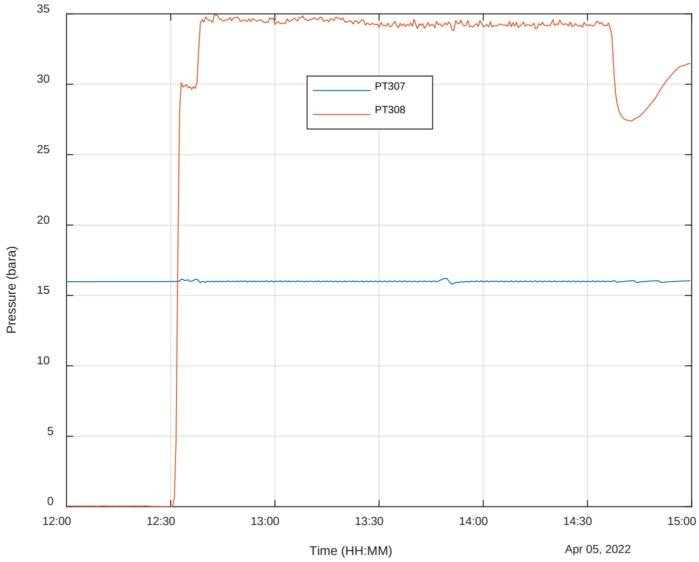


Figure 4-22. Pressures before and after the expansion valve during the 5th of April test.

The flow was 10.5 kg/h as seen in Figure 4-23.

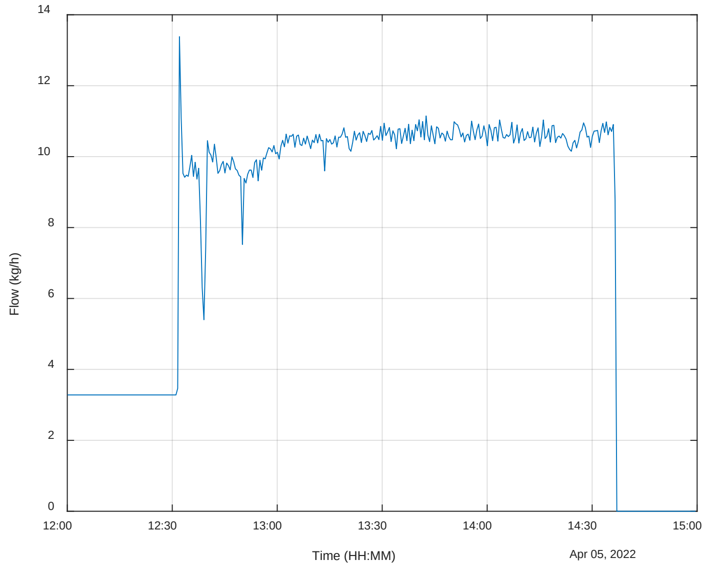


Figure 4-23.Gas flow rate into the compression train during the 5th of April test.

At 13:22 the temperature from the external cooler (Lauda) was changed and from 13:35 to the end of the test at14:35 the liquid CO_2 temperature was cooled down to -15.7 °C. This is shown in Figure 4-24. This change had almost no impact on the pressure conditions in the unit and neither on the temperature after the expansion valve (TT310).

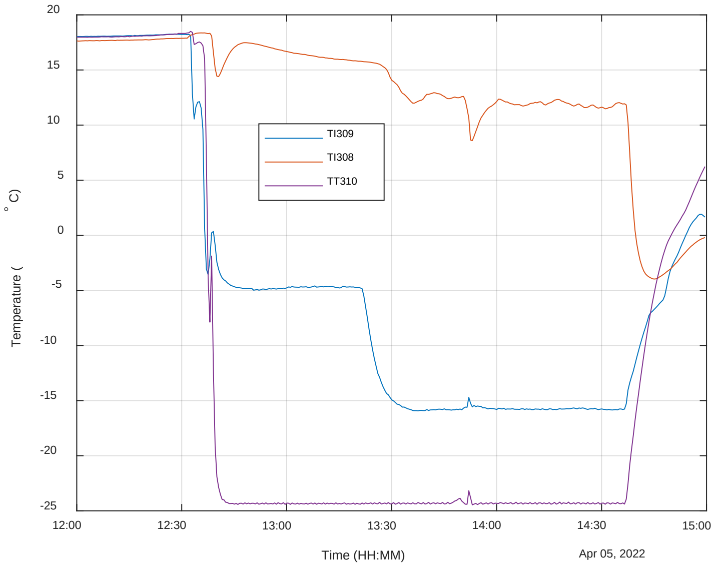


Figure 4-24. Temperatures before (TI308) and after external cooler (TI309) and after expansion valve (TT310) during the 5th of April test.

However, it influences the amount of gas leaving the storage tank. In Figure 4-25 the flow is shown. The flow varies a lot because the pressure drop over the valve CrV304 was large (15 bar) and the necessary valve opening was around 1-2%, making the regulation of the exit flow difficult. However, an average of the signal before and after the temperature change shows a decrease of the flow from 3.2 to 2.4 kg/h. The decreased temperature produced a larger liquid to gas ratio in the flow after the expansion valve CrV303.

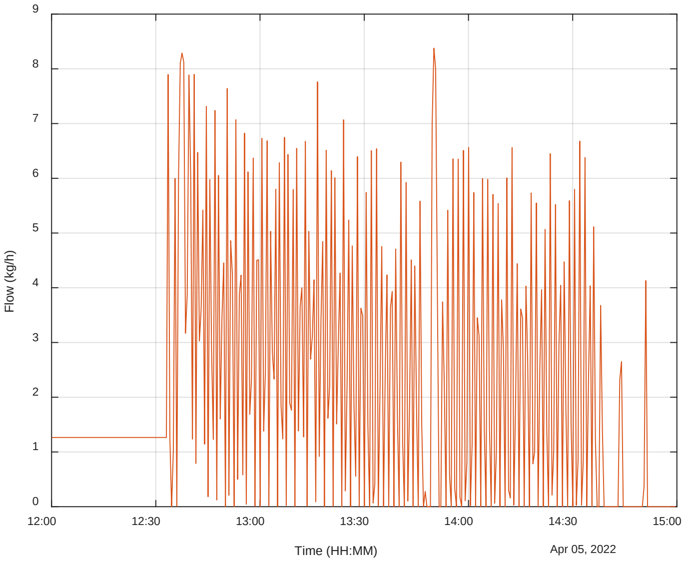


Figure 4-25.Gas flow rate FI308 out of the storage tank during the 5th of April test.

4.3 The Carbo Max storage tank

Between the testing periods the CO_2 in the Carbo Max 450 tank was stored. To illustrate a period where the CCLU was not running data from the 11th of March is shown in Figure 4-26.

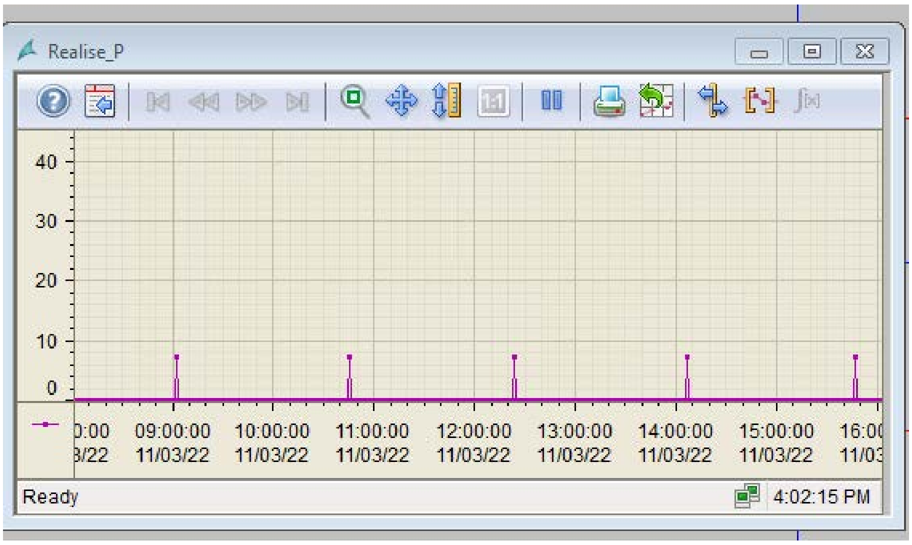


Figure 4-26.Gas flow rate from storage tank the 11th of March.

The figure shows the flow sensor FI308 out of the tank as shown in the control system. The flow is zero except from some spikes at regular intervals. The control valve CrV304 opens for a short period of time because the pressure has increased in the tank due to heat from the surroundings. In Figure 4-27 we see that it is about one hour and 40 minutes between the spikes.

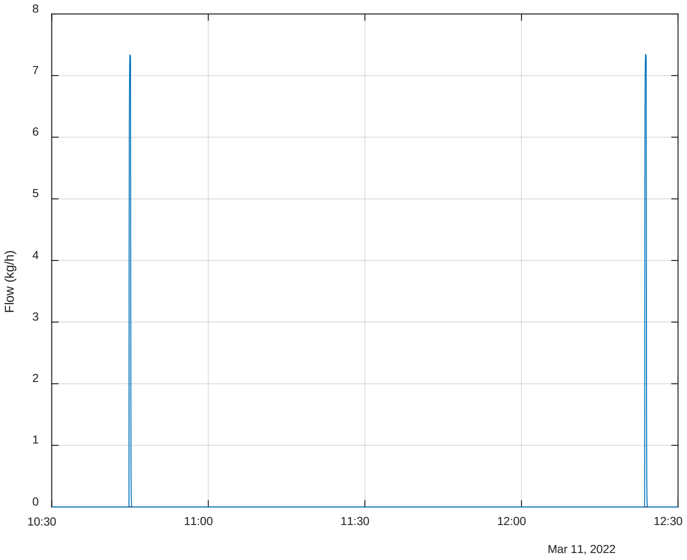


Figure 4-27.Gas flow rate from storage tank the 11th of March.

In Figure 4-28 a spike is enlarged and show that it last for about 30 seconds.

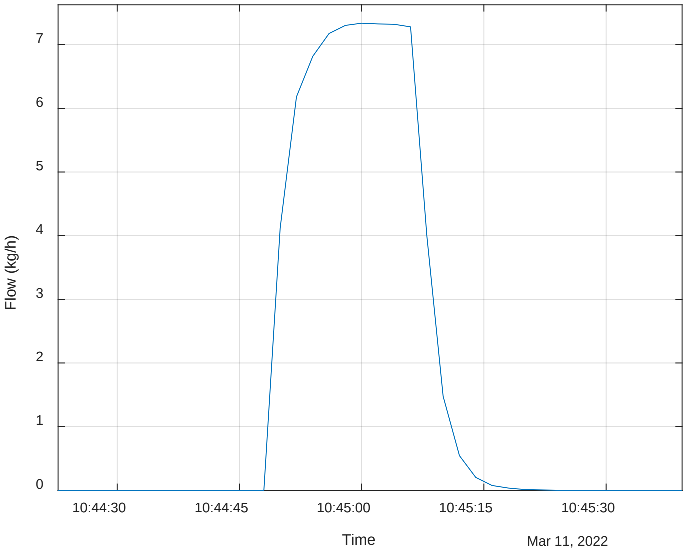


Figure 4-28.Gas flow rate from storage tank the 11th of March.

By using the spike frequency and integrating one spike the loss of CO_2 under storage is estimated to be 38 g/h or less than one kg pr day.

5 Summary and conclusions

A CO_2 Compression and Liquefaction Unit (CCLU) has been designed and built at the Tiller CO_2Lab. The CCLU was designed with components like an industrial sized unit with three compressor stages including cooling and knock out drums. Out of the last drum the CO_2 gas at 35 - 40 bar is dried and then cooled down to about -5 to -10 °C by an external cooler. The liquefied CO_2 is then expanded to 15-16 bar through an expansion valve and stored at -26°C in a storage tank.

Samples of liquid has been taken from the knockout drums and samples of the gas before the external cooler. It is also possible to take a sample of the gas out of the storage tank.

Risk assessment measures that include HAZID, both classical and procedural HAZOP studies have been performed. These yielded a robust and optimal design that is characterized by safe operability and enhanced efficiency.

The commissioning was done based on the procedural HAZOP. The compression part of the unit was tested and commissioned in January 2022 where the performance of the Haskel booster and any leakages was in focus. Both air and CO_2 from the desorber was used.

The last part of the unit was first tested with Nitrogen for leakage testing in March. Then the whole unit was tested with CO_2 during March and April 2022. Improvement of the unit was done such that the capacity could reach the design value of more than 10 kg/h.

The tests showed stable conditions in the whole unit and a gas cylinder with compressed CO_2 gas and liquid samples from the knockout drums have been sent for analysis.

The Lauda Integral could cool down the liquid CO_2 to less than -15 °C

The Carbo Max storage tank was tested both at 15 and 16 bar. The temperature was -26.3 and -24.4 °C respectively. The storage tank could store the CO_2 with a loss less than 1 kg of CO_2 per day.