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Abstract

This report gives a detailed description of the 10 ton per day low temperature CO_2 liquefaction and phase separation laboratory rig installed in SINTEF Energy's laboratories in Trondheim, including specifications for its main components. This is followed by experimental results from the first experimental test campaigns with gas chromatography (GC) instruments connected.

The phase separation part of the experimental set-up consists of two separator stages. A binary gas mixture first enters the main separator, where the bulk separation is performed. The liquid CO_2 is then heated and expanded to a lower pressure before it enters the second purification separator. The rig is well instrumented with pressure, temperature, mass flow and level sensors, as well as a gas chromatograph.

On 05.09.2018, experiments with a feed composition of approximately 63 mol% CO_2 and 37 mol% N_2 to the first separator were conducted. Tests were performed with liquid levels between 16 and 32 cm, and pressure levels between 30 and 35 bar in the first separator and 12 and 8 bar in the second separator. The final liquid CO_2 product purities were measured. Close to equilibrium conditions were achieved. The results show that the product purity can be controlled by the pressure level of the second separator stage.

On 10.09.2018 an experiment with 32 cm liquid level in both separators, 30 bar pressure in the first separator and 8 bar pressure in the second separator were performed for a feed gas composition of 76 mol% CO_2 and 24 mol% N_2. Measurements of the composition were performed at the inlet to the first separator and at the vapour and liquid lines from both separators. Close to equilibrium conditions are achieved in the two separators. The experimental results for purity and CO_2 capture ratio (CCR) are in good agreement with results from a HYSYS simulation of the process. A CCR of 83 % is estimated based on the experiments compared to 82 % in the simulations. It should be mentioned that the experimental rig and the simulation model thereof lacks an internal recycle stream which can increase the obtainable CCR considerably.

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1 LOW TEMPERATURE CO_2 LIQUEFACTION AND PHASE-SEPARATION LABORATORY RIG

A low-temperature CO_2 liquefaction and separation laboratory rig with capacity to capture approximately 10 tons of CO_2 per day has been built in the Thermal Energy laboratory (Varmetekniske laboratorier), a joint laboratory for SINTEF ER and NTNU in Trondheim. Descriptions of the rig, its components and functionalities are given in this chapter.

1.1 Description of the low temperature CO_2 separation rig

Figure 1.1 shows a principle schematic of the low temperature CO_2 liquefaction and separation laboratory rig. A more detailed process diagram is given in Appendix A.1.

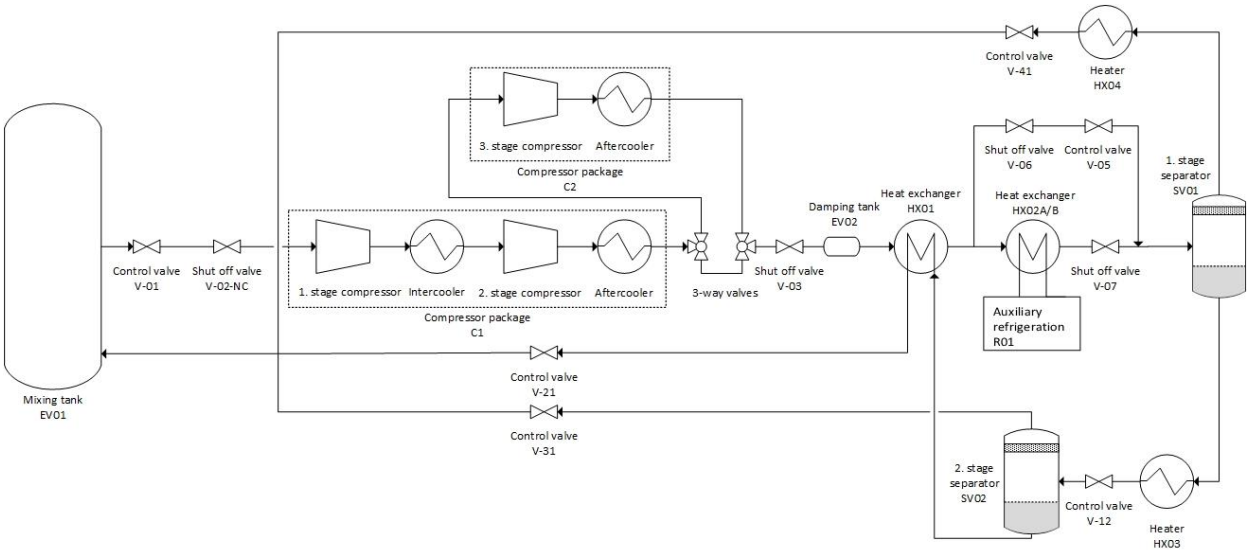

Figure 1.1 Principal schematic of the low temperature CO_2 separation laboratory rig.

The mixing tank (EV01) is filled from the laboratory's central gas system. CO_2 and N_2 supply lines are connected to the tank and a hydrogen supply line is ready to be connected for future needs. Filling of the tank is controlled by flow controllers installed on each gas supply line.

A gas mixture from EV01 flows through the two valves V-01 and V-02-NC towards the first stage compressor. V-01 is a pressure reducing valve controlled by the compressor inlet pressure, and V-02-NC is a shut-off valve. The gas mixture then enters the compressor packages consisting of two compressors (C1 and C2) with inter- and aftercoolers. The first compressor (C1) has two stages and is designed to compress the gas mixture from 7.5 bar(g) to about 45 bar(g). The gas can then either enter the second compressor (C2) for compression to 100-120 bar(g) in one compressor stage, or C2 can be bypassed by operating two three-way-valves. A shut off valve (V-03) is installed directly behind the compressor packages, followed by a damping tank (EV02) to reduce the fluctuations from the reciprocating piston compressors. The compressed gas flows through a Coriolis mass flow meter before entering the recuperator (HX01), where the gas mixture is cooled by the final liquid CO_2 stream. The gas mixture is then further cooled and expanded to the separation temperature and pressure through control valve V-05. At a later stage, an auxiliary refrigeration cycle (R01) will be installed, as seen in Figure 1.1. When R01 becomes operational, the gas can be cooled to the desired separation temperature without over-compressing and re-expanding the mixture to a lower pressure by Joule-Thomson throttling.

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After compression and cooling, the mixture is separated in the first separator stage (SV01) at temperatures below -50°C. Decarbonised gas leaving the separator gas outlet is heated by an electric heater (HX04), flows through a Coriolis mass flow meter and is expanded in the control valve (V-41) before it returns to the mixing tank. The liquid CO_2-rich phase is heated by an electric heater (HX03) and expanded to a lower pressure in control valve V-12, before it is separated in a second separator (SV02) to purify the CO_2 stream. Flashed gas returns to the mixing tank through a Coriolis mass flow meter and control valve V-31, while the liquid CO_2 stream enters HX01 before returning to the mixing tank through control valve V-21.

The instrumentation of the lab pilot rig is shown in the process diagram in Appendix A.1, including mass flow meters, temperature and pressure sensors, thermostats, level meters and sampling points for the gas chromatograph (GC). Currently, before the permanent GC is installed, a micro GC is used to measure the compositions of the streams.

1.2 Components

Specifications for the main components in the low-temperature CO_2 liquefaction and separation rig are given in this section.

1.2.1 Low-pressure mixing and expansion vessel EV01

The low-pressure mixing tank is used to store the gas mixture that is circulated in the separation rig. It can be refilled continuously using mass flow controllers or batch-wise during operation to make up for leakage in the reciprocating piston compressors. The tank is well insulated and trace heating wires are installed on its surface.

Table 1.1 EV01 specifications

Tag	**EV01**
Manufacturer	Heimdal Industriservice AS
Type	Pressure vessel
Connections	4 x 1 ½" OD Swagelok SS-2400-1-24W, 2 x 12 mm OD Swagelok SS-12M0-1-8W
Volume	250 l
Nominal pressure	30 bara
Material	1.4404
Isolation	50 mm Armaflex cryogenic isolation

1.2.2 Control valve V-01

The V-01 control valve is a Mankenberg DM 652 pressure reducing valve. The valve is a diaphragm-controlled spring-loaded and balanced proportional control valve. It is used to reduce the pressure from the mixing tank (EV01), to the desired compressor (C1) inlet pressure.

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Table 1.2 Specifications for control valve V-01

Tag	V-01
Producer	Mankenberg
Model	DM 652F DN50/PN40 KVS12
Type	Diaphragm-controlled spring-loaded and balanced proportional control valve
Diaphragm material	EPDM
Temperature range	-35 to 130 °C
Nominal pressure	40/16 bar

1.2.3 Shut-off valve V-02-NC

V-02-NC is used to close the pipe between EV01 and the compressor package. It can be used together with V-03 to shut out the compressor packages (C1 and C2). The valve is a normally closed, single-acting pneumatically operated ball valve.

V-02-NC shares its control panel (P-01) with V-03. The panel consists of a 5/2 NC solenoid valve, 24 V DC, ¼" filter and manometer.

Table 1.3 Specifications for shut-off valve V-02-NC

Tag	V-02-NC
Producer	Zuercher Technik AG
Model	Procol A20D 3-piece DN40 PN64, reduced bore
Type	Ball valve
Actuator	Pneumatic, Air Torque AT251U, single acting

1.2.4 Compressor packages C1 and C2

Compressor package C1 consists of a two-stage reciprocating piston compressor with an inter- and aftercooler. C1 is designed for a suction pressure of 7.5 bar(g), an intermediate pressure of about 22 bar(g) and an outlet pressure of about 45 bar(g). There are relief valves and pressure sensors at the inlet, after the first stage and at the outlet of C1. The relief valves have set pressures of 9, 26 and 50 bar(g). Temperature sensors are installed at the outlet of each cylinder. There are two cylinders per stage.

Compressor package C2 consists of a one stage reciprocating piston compressor with an aftercooler. There are three cylinders in the stage. The compressor is designed for an inlet pressure of about 45 bar(g) and an outlet pressure of about 120 bar(g). There are safety valves and pressure sensors at the inlet and outlet of C1. The relief valves have set pressures of 50 and 132 bar(g). Temperature sensors are installed at the outlet of each cylinder.

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Table 1.4 Specifications for compressor package C1

Tag	**C1**
Supplier	LEWA AS
Compressor driver (motor)	
Manufacturer	ABB
Type	M3KP 225SMB 4
Voltage	400Y/230D VAC
Power	37.0 kW
RPM	1500
EX-protection	EExde IIC T4
IP	56
Fan driver	
Manufacturer	ABB
Type	M3HP 80MA 4
Voltage	400Y/230D VAC
Power	0.55 kW
RPM	1500
EX-protection	EExe IIC T3
IP	56
2 stage compressor	
Manufacturer compressor	RIX Industries
Type	4VX2B-23
Flow rated	200 SCFM
Design inlet pressure	7.5 Bar(g)
Design outlet pressure	45 Bar(g)
Intercooler	
Manufacturer	Funke
Type	CCFA 404-25
Aftercooler	
Manufacturer	Funke
Type	CCFA 305-63
Control cabinet and electrical wiring	
Manufacturer	Norse-Technology

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Table 1.5 Specifications for compressor package C2

Tag	**C2**
Supplier	LEWA AS
Compressor driver (motor)	
Manufacturer	ABB
Type	M3KP 180MLB 4
Voltage	400Y/230D VAC
Power	22.0 kW
RPM	1500
EX-protection	EExde IIC T4
IP	56
Fan driver	
Manufacturer	ABB
Type	M3HP 80MA 4
Voltage	400Y/230D VAC
Power	0.55 kW
RPM	1500
EX-protection	EExe IIC T3
IP	56
1 stage compressor	
Manufacturer compressor	RIX Industries
Type	4VX1BG-5.1
Flow rated	200 SCFM
Design inlet pressure	45 bar(g)
Design outlet pressure	120 bar(g)
Aftercooler	
Manufacturer	Funke
Type	CCFA 306-150
Control cabinet and electrical wiring	
Manufacturer	Norse-Technology

1.2.5 Shut-off valve V-03

V-03 is a shut-off valve located directly behind the compressor packages. It can be used together with V-02-NC to shut out the compressor packages. The valve is a double-acting pneumatically actuated ball valve. As stated in 1.2.3, V-03 and V-02-NC shares a control panel (P-01). The panel consists of a 5/2 NC solenoid valve, 24 V DC, ¼" filter and manometer.

Table 1.6 Specifications for shut-off valve V-03

Tag	**V-03**
Producer	Oliver valves
Model	B6FZ10
Type	Ball valve
Actuator	Pneumatic, Air Torque AT104 DA, double acting

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1.2.6 Damping tank EV02

The damping tank, EV02, is used to dampen the fluctuations from the reciprocating piston compressors.

Table 1.7 Specifications for damping tank EV02

Tag	EV02
Manufacturer	Swagelok
Model	DOT-3A 1800
Type	Double-ended sampling cylinder
Pressure rating	124 bar(g)
Volume	1 gal (3.785 l)
Material	304L SS

1.2.7 Heat exchanger HX01

HX01 is a process-to-process plate heat exchanger where the gas mixture from the compressors is cooled by the final liquid CO_2 stream from the outlet of SV02.

Table 1.8 Specifications for heat exchanger HX01

Tag	HX01
Manufacturer	Kaori Heat Treatment CO., LTD.
Model	C202-16
Type	Plate heat exchanger
Working pressure HP/LP side	140/30 bar
Number of plates	16

1.2.8 Heat exchanger HX02A and B

The heat exchangers HX02A and B are used to further cool the gas mixture coming from HX01 to the separation temperature. The cooling medium is CO_2 from the auxiliary CO_2 refrigeration cycle (R01). Hence, these heat exchangers are inactive in the present experimental runs.

Table 1.9 Specifications for heat exchanger HX02A and HX02B

Tag	HX02A	HX02B
Manufacturer	Kaori Heat Treatment CO., LTD.	Kaori Heat Treatment CO., LTD.
Model	C097-20	C097-16
Type	Plate heat exchanger	Plate heat exchanger
Working pressure HP/LP side	140/50 bar	140/50 bar
Number of plates	20	16

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1.2.9 CO_2 refrigeration cycle R01

A CO_2 refrigeration cycle with a cooling capacity of approximately 7 kW will be installed to cool the process gas down to the separation temperature. The process diagram for the refrigeration cycle is shown in Figure 1.2.

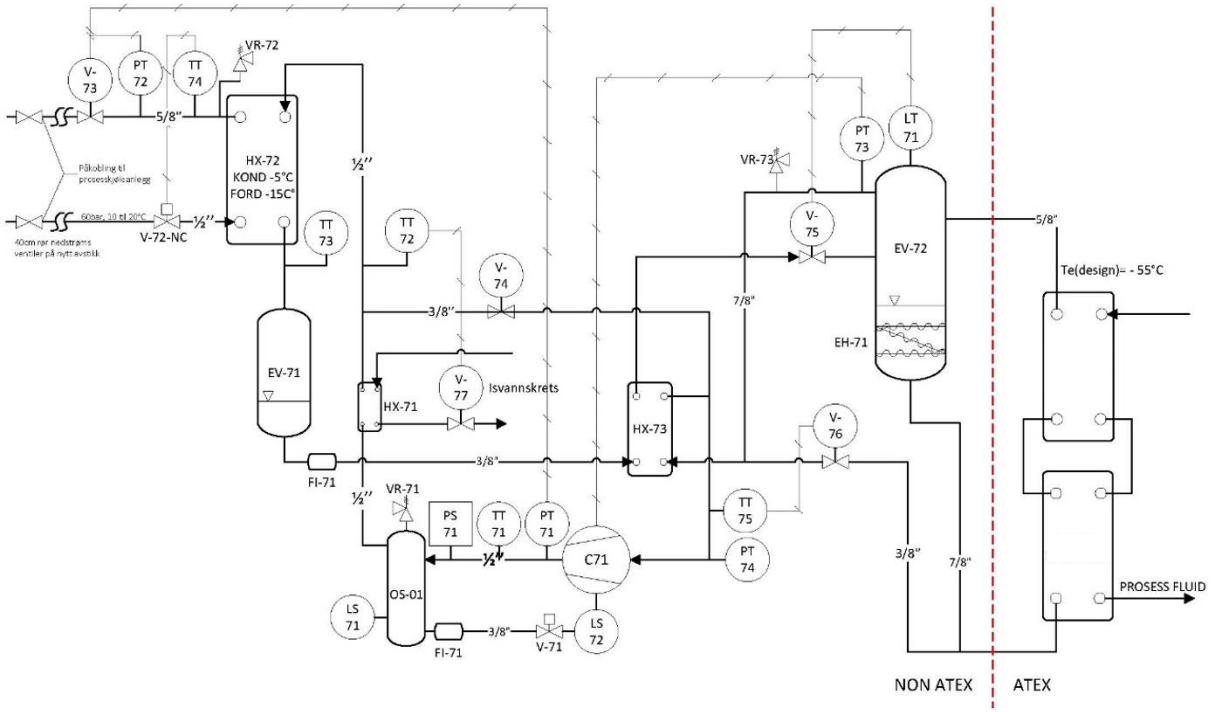

Figure 1.2 P&ID of CO_2 refrigeration cycle R01

1.2.10 Shut-off valve V-06 and V-07

The shut-off valves V-06 and V-07 are used to control if the flow will pass through HX02A/B to be cooled by the auxiliary refrigeration system (R01), or if HX02A/B are bypassed. In the latter position the final cooling is achieved by J-T expansion to a lower pressure in V-05. At this time R01 is not connected to the rig, and the inlet to HX02A/B is blanked off.

Table 1.10 Specifications for V-06 and V-07

Tag	V-06	V-07
Manufacturer	DIE ERSTE Industry Co., Ltd	DIE ERSTE Industry Co., Ltd
Model	Die Erste CLT-35 3-piece DN15 1/2"R	Die Erste CLT-35 3-piece DN20 3/4"R
Type	Cryogenic ball valve	Cryogenic ball valve
Actuator	Manual	Manual

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1.2.11 Control valve V-05

V-05 is mounted on the line that bypasses the auxiliary cooling system. It is used to expand the gas mixture to a lower pressure and cool it to the final SV01 separation temperature. The valve is an electro-pneumatically-controlled, fail open, globe valve, and the position is controlled by the temperature (T-05) downstream the valve.

Table 1.11 Specifications for V-05

Tag	**V-05**
Manufacturer	Samson
Model	Type 3251 DN25 PN160
Type	Globe valve
Body Material	1.4408
Packing material	PTFE
Actuator	Electro pneumatic positioner, Samson Type 3277, size 350 cm^2

1.2.12 Separator SV01 and SV02

SV01 is the main separator in the experimental rig, while SV02 is used for purification of the liquid CO_2 stream after heating in HX03 and expansion in V-12. SV01 and SV02 have an inner diameter and height of 10 cm and 140 cm, respectively. The two separators are identical except for the internals. SV01 has an 80 mm diameter, 100 mm high demister, while SV02 has a 65 mm diameter, 50 mm high demister. The separators are insulated with 50 mm Armaflex. Level meters (LT01 and LT02) are installed on the sides of the separators.

Table 1.12 Specifications for SV01 and SV02

Tag	**SV01 and SV02**
Manufacturer	Heimdal Industriservice AS
Working pressure	120 bar @ -55 C
Volume	~12 l
Material	316L

1.2.13 Electric heater HX03

The electric heater HX03 is an immersion heating system used to heat the liquid stream from SV01. This is to avoid formation of dry ice when the stream is expanded to a lower pressure in V-12. HX03 is controlled by the outlet temperature (TT10) from the heater.

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Table 1.13 Specifications for HX03

Tag	HX03
Supplier	Norske Backer AS
Manufacturer	Nemko
Model	Nemko 13 ATEX 1549X
Capacity	5 kW
Supply voltage	400 V
Current	7.2 A
Thermostat TSH	-30 °C
Temperature limiter TSHH	20 °C
Working pressure	120 bar
Immersion depth	750 mm
EX protection	II 2 G EX e d IIC T3 Gb
Material	AISI316L

1.2.14 Control valve V-12

V-12 is a control valve that is used to reduce the pressure of the liquid stream from SV01 before the SV02 purification separator. The valve is an electro-pneumatically-controlled, fail open, globe valve, and the position is controlled by the liquid level (LT01) in SV01 during operation.

Table 1.14 Specifications for V-12

Tag	V-12
Manufacturer	Samson
Model	Type 3251 DN25 PN160
Type	Globe valve
	Fail open
Body Material	1.4408
Packing material	PTFE
Actuator	Electro pneumatic positioner, Samson Type 3277, size 350 cm^2

1.2.15 Control valve V-21

V-21 is a control valve that is used to reduce the pressure of the purified CO_2 stream coming from HX01 before it returns to the mixing tank EV01. The valve is an electro-pneumatically-controlled, fail open, globe valve, and the position is controlled by the liquid level (LT02) in SV02 during operation.

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Table 1.15 Specifications for V-21

Tag	V-21
Manufacturer	Samson
Model	Type 3241 DN25 PN40
Type	Globe valve
Body Material	1.4408
Packing material	PTFE
Actuator	Electro pneumatic positioner, Samson Type 3277, size 350 cm^2

1.2.16 Control valve V-31

V-31 is a control valve that is used to reduce the pressure of the flashed gas from SV02 before it returns to the mixing tank EV01. The valve is an electro-pneumatically-controlled, fail open, globe valve, and the position is controlled by the pressure (PT10) in SV02 during operation.

Table 1.16 Specifications for V-31

Tag	V-31
Manufacturer	Samson
Model	Type 3241 DN25 PN40
Type	Globe valve
Body Material	1.4408
Packing material	PTFE
Actuator	Electro pneumatic positioner, Samson Type 3277, size 240 cm^2

1.2.17 Electrical heater HX04

HX04 is an electric trace heater used to heat the flashed gas stream from SV01. This is to avoid the risk of any formation of dry ice when the stream is expanded to a lower pressure in V-41. The heater is controlled by the inlet temperature (TT41) to V-41.

Table 1.17 Specifications for HX04

Tag	HX04
Supplier	Thorne and Derrick
Manufacturer	MICC Ltd.
Model	D-L2S374-3 2 core heating unit
Resistance	1.23 ohm/m
Heating cable length	20 m
Supply voltage	300 V
Safety temperature monitor (T-42)	40 °C
Safety temperature limiter (T-43)	50 °C
EX protection	II 2 G EX e IIC T1 to T6 Gb
Material	AISI 321

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1.2.18 Control valve V-41

V-41 is a control valve that is used to reduce the pressure of the flashed gas from SV01 before it returns to the mixing tank EV01. The valve is an electro-pneumatically-controlled, fail open, globe valve, and the position is controlled by the pressure (PT04) in SV01 during operation.

Table 1.18 Specifications for V-41

Tag	**V-41**
Manufacturer	Samson
Model	Type 3251 DN25 PN160
Type	Globe valve
Body Material	1.4408
Packing material	PTFE
Actuator	Electro pneumatic positioner, Samson Type 3277, size 350 cm^2

1.2.19 Shut-off valve V-45-NC

V-45-NC is located on the bleed-off line to atmosphere directly behind the compressor package. It can be used to bleed off the pressure on the compressor package. The valve is a normally closed, single-acting, pneumatically operated ball valve.

The control panel (P-02) for V-45-NC consists of a 3/2 NC solenoid valve, 24 V DC, ¼" filter and manometer.

Table 1.19 Specifications for V-45-NC

Tag	**V-45-NC**
Manufacturer	Flowserve Worcester Controls
Model	07A44-6666MYB BWGBWG
Type	3-piece ball valve
Material	316L
Actuator	Flowserve Norbro series 40R single acting pneumatic actuator

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1.2.20 Flow control M01 and M02

M01 and M02 are mounted on the supply lines from the CO_2 and N_2 central gas system, and are used to control the filling of mixing vessel (EV01).

Table 1.20 Specifications for M01 and M02

Tag	M01 and M02
Manufacturer	Bronkhorst
Model	F-201AV-70K-AGD-22-E Bronkhorst HI-TEC Mass Flow Controller
Type	El-Flow series mass flow controller
Connections	¼" Swagelok
Material	316L
Pressure rating	64 bar
Temperature range	-10 to 70 °C
Seals	EPDM

1.2.21 Mass flow meter MT00 and MT40

MT00 and MT40 are Coriolis mass flow meters. MT00 measures the mass flow after the compressor before HX01, while MT40 measures the mass flow in the flashed gas line after SV01.

Table 1.21 Specifications for MT00

Tag	MT00	MT40
Manufacturer	Rheonik	Rheonik
Model	RHM08-T1-P1-PFO-M1-R1-T1	RHM04-TA-P1-PFO-MO-R1-T1
Type	Coriolis mass meter	Coriolis mass meter
Connections	ANSI 1" 1500# RTJ	ANSI ½" 1500# RTJ
Material	1.4571 (SS316Ti)	1.4539 (904L) & 1.4571 (SS316Ti)
Temperature range	-20 to 120 °C	-45 to 120 °C
Pressure rating	According to flange rating	150 bar
Transmitter	RHE08-A1-IO-AT, IP65 for wall mount	RHE08-A1-IO-AT, IP65 for wall mount
EX protection	AT-ATEX [Ex ia Ga] IIC (Mounted in safe area)	AT-ATEX [Ex ia Ga] IIC (Mounted in safe area)

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1.2.22 Mass flow meter MT30

MT30 is a Coriolis mass meter that measures the mass flow in the flashed gas line from SV02.

Table 1.22 Specifications for MT30

Tag	MT30
Manufacturer	Endress+Hauser AS
Model	Promass 83 DN15, 83F08-CCVSBADEAEAT
Type	Coriolis mass meter
Connections	Swagelok 1/2" SS-8-VCO-4
Materials	316L/1.4404 904L/1.4539
Temperature range (sensor)	-50 to 200 °C
Pressure rating	40 bar
EX protection	Atex II 2G EEx de IIC T1 – T6 IP67

1.2.23 Electrical heater HX00

HX00 is an electric trace heater wrapped around the mixing tank (EV01). The heater is controlled by the inlet temperature (TT01) to compressor C1.

Table 1.23 Specifications for HX00

Tag	HX04
Supplier	Thorne and Derrick
Manufacturer	MICC Ltd.
Model	D-L2S583-4 2 core heating unit
Resistance	0.191 ohm/m
Heating cable length	50 m
Supply voltage	300 V
Safety temperature monitor (T-08)	40 °C
Safety temperature limiter (T-09)	50 °C
EX protection	II 2 G EX e IIC T1 to T6 Gb
Material	AISI 321

1.2.24 Thermostat T-08, T-09, T-42 and T-43

T-08 (Safety temperature monitor, alarm 40 °C) and T-09 (Safety temperature limiter, trip 50 °C) are the thermostats on HX00, while T-42 (Safety temperature monitor, alarm 40 °C) and T-43 (Safety temperature limiter, trip 50 °C) are the thermostats on HX04.

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Table 1.24 Specifications for T-08, T-09, T-42 and T-43

Tag	T-08, T-09, T-42 and T-43
Manufacturer	JUMO Gmbh & Co. KG
Model	JUMO exTHERM Type 605056
Temperature rating	-50 to 500 °C
EX protection	II 2G Ex d IIC Gb For explosive gas atmosphere Ex d IIC Gb
Material	Aluminium

1.2.25 Level meter LT01 and LT02

LT01 measures the liquid level in the main separator (SV01), while LT02 measures the liquid level in the CO_2 purification separator SV-02. The current level meters are not ATEX certified and must be replaced before experiments with H_2 is run.

Table 1.25 Specifications for level meters LT01 and LT02

Tag	LT01 and LT02
Manufacturer	HB Products
Model	HBLC-CO2-17-6
Length	1700 mm
Temperature rating	-55 to 30 °C
Pressure rating	150 bar
EX protection	No
Protection degree	IP65
Material – liquid parts	AISI 304/PTFE

1.2.26 Pressure transmitter PT00/PT01/PT02/PT03/PT04/PT10

Pressure transmitters are placed in the different pressure zones as shown in the process diagram in Appendix A.1.

Table 1.26 Specifications for PT00/PT01/PT02/PT03/PT04/PT10

Tag	PT00/PT01/PT02/PT03/PT04/PT10
Manufacturer	Keller
Model	PAA-33XEi / 40 bar / 81871.10
Type	Piezoresistive Pressure Transmitter
Compensated temperature range	-10 to 80 °C
Pressure	0 – 30/0 – 10/0 – 150/0 – 150/0 – 150/0 – 30 barA
Error band [%FS] digital	± 0.15
EX protection	KEMA 04 ATEX 1081 X Ex II 1 GD Ex ia IIC T4 ... T6 Ga Ex ia IIIC T130°C Da IECEx DEK 14.0070 X

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1.2.27 Temperature transmitter TT01-05/TT20/TT30/TT41

Thermocouples are placed as shown in the process diagram in Appendix A.1.

Table 1.27 Specifications for TT01-05/TT20/TT30/TT41

Tag	TT01/TT02/TT03/TT04/TT05/TT20/TT30/TT41
Manufacturer	Omega
Model	M12LCP-TSS-1/8-U-0600
Type	Thermocouple Probes with high temperature M12 Molded Connectors
Temperature range	-50 to 260 °C

1.2.28 Temperature transmitter TT06/TT07/TT12/TT13

Resistance Temperature Detectors (RTD) are placed in the separators (SV01 and SV02).

Table 1.28 Specifications for TT06/TT07/TT12/TT13

Tag	TT06/ TT07/ TT12/ TT13
Manufacturer	Omega
Model	P-M-A-1/8-6-0-T-33 PT100 1/8
Type	Ultra Precise RTD Sensors for Industrial Applications
Temperature range	-100 to 250 °C
Accuracy	±(0.15 + 0.002 |t|)°C

1.2.29 Temperature transmitter TT40/TTLT01/TTLT02/TT00A/TT00B

Self-adhesive surface thermocouples are placed as shown in the process diagram in Appendix A.1.

Table 1.29 Specifications for TT40/TTLT01/TTLT02/TT00A/TT00B

Tag	TT40/TTLT01/TTLT02/TT00A/TT00B
Manufacturer	Omega
Model	SA1XL-T-120-SRTC
Type	Self-Adhesive surface Thermocouples
Temperature range	-73 to 260 °C

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2 EXPERIMENTAL RESULTS

2.1 Experiments 2018-09-05

2.1.1 Start-up and pre-cooling

The first experimental tests with gas chromatography (GC) instruments connected were conducted 05.09.2018. The rig was started up from a state of 25 bar standstill pressure and room temperature. About one hour and forty minutes was used to cool down the rig, adjust cooling water flow rates and make other adjustments required to reach the desired steady-state operating conditions. This is illustrated in Figure 2.1, which shows the temperature development for the sensor TT04 located downstream of the heat recuperator HX01 on the feed line. As can be observed, the TT04 temperature first increases for a period up to around 50 °C, followed by a long period of decrease before it eventually stabilises in an interval between -45 °C and -50 °C.

The rig was replenished continuously with CO_2 and N_2 makeup gas to compensate for the compressor leakages and maintain constant inlet conditions to the low-temperature separation process. During the cool-down phase, V-21 is kept fully open to get a large mass flow through the recuperator. V-12 is set to 60 % open position, while the PID controllers are turned on for V-41 and V-31 to keep the pressure levels at 30 bar and 12 bar in SV01 and SV02, respectively. V-05 is positioned to maintain a pressure of 100 bar at the compressor outlet. When the temperature in SV02 closes in on -50 °C, the PID controller is activated for V-21 to a set point of 16 cm liquid level in SV02 (V-21 was downward constrained with a minimum allowed opening position of 5 %). When the rig runs steadily with this liquid level accumulated in SV02, the PID controller for V-12 is activated with a set point of 16 cm liquid level in SV01 (V-12 with a minimum allowed opening position of 20 %). The temperature set points of the heaters HX00, HX03 and HX04 are manually adjusted to suitable levels during the cool down process.

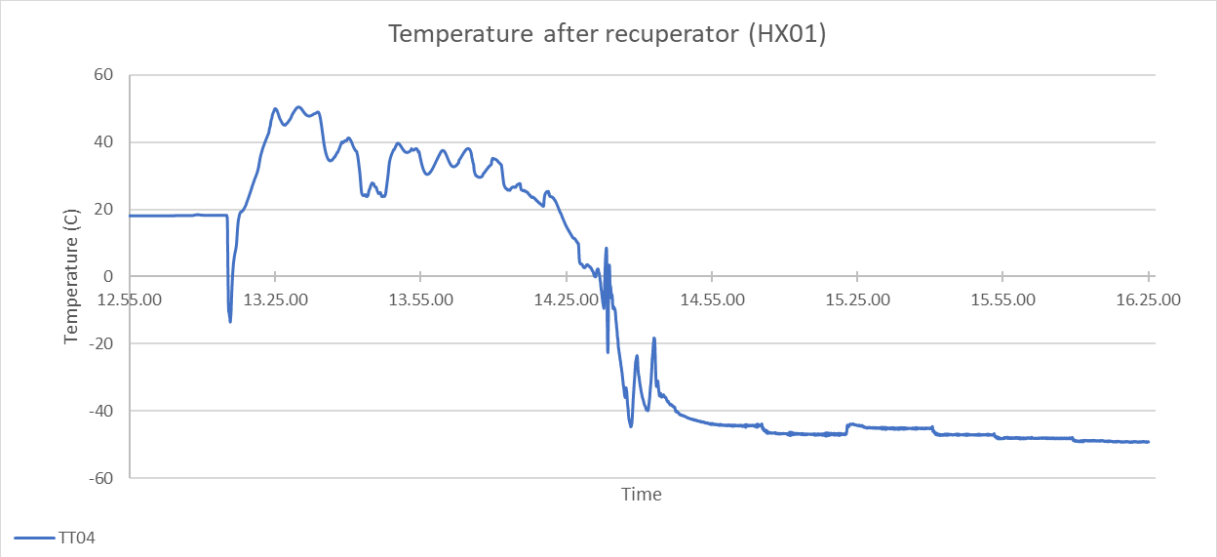

Figure 2.1:Temperature development of TT04 from start-up until stable operation

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2.1.2 Steady-state results

When steady-state conditions were reached with 16 cm liquid level in both separators, the composition of the binary gas mixture flowing in to SV01 was measured to 63.28 mol% CO_2 and 36.72 mol% N_2. The operating conditions in the different nodes of the rig at this point are shown in Table 2.1. It is assumed that the feed composition remained relatively constant through the remainder of the experimental run. If near-equilibrium conditions for vapour and liquid phase are assumed in the separators, the composition of the product liquid CO_2 stream will be close to unaffected by small changes in the composition of the gas mixture entering SV01 provided that the temperature and pressure levels are well controlled.

Table 2.1 Operating conditions in the rig during the first GC measurements

	Experimental results
Inlet gas mixture composition	CO_2: 63.28 mol% N_2: 36.72 mol%
EV01	
Pressure (PT00)	6.36 bar
C1 and C2	
Pressure (PT01)	6.21 bar
Temperature (TT02)	20.4 °C
PT02	100.0 bar
TT03	19.5 °C
Mass flow (MT00)	3.44 kg/min
HX01	
Temperature (TT04)	-44.2 °C
Pressure (PT03)	99.96 bar
SV01	
Temperature (TT05)	-52.35 °C
Temperature (TT06)	-54.05 °C
Temperature (TT07)	-56.31 °C
Pressure (PT04)	29.96 bar
Liquid level (LT01)	16 cm
Liquid retention time	50 s
Liquid composition	CO_2: N_2:
Vapor composition	CO_2: N_2:
Mass flow (MT40)	1.252 kg/min
HX03	
Temperature (TT10)	-46.32 °C
SV02	
Temperature (TT11)	-49.59 °C
Temperature (TT12)	-28.4 °C
Temperature (TT13)	-52.7 °C
Pressure (PT10)	12.00 bar
Liquid level (LT02)	16 cm
Liquid retention time	55 s
Liquid composition	CO_2: 99.30 mol% N_2: 0.70 mol%
Vapor composition	CO_2: N_2:
Mass flow (MT30)	0.122 kg/min

After GC measurements of the CO_2 liquid product purity at these conditions, the pressure level set points were changed to 35 bar in SV01 and 10 bar in SV02. At these conditions, a new GC measurement of the product purity was performed. The liquid level in SV02 was thereafter

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increased to 32 cm, and the pressure level in SV02 was increased to 12 bar and reduced stepwise down to 9 bar. GC measurements of the liquid CO_2 product purity were performed for 12, 10 and 9 bar separation pressure in SV02 while keeping the pressure in SV01 constant at 35 bar. The liquid level in SV01 was then increased to 20 cm and the pressure set to 8 bar in SV02 for a new measurement. Results are shown in Table 2.2, and a plot of the product purities vs. separation pressure for the measurements with 32 cm liquid level in SV02 are given in Figure 2.2. All measured compositions are average values of multiple GC measurements performed over a short time interval.

Table 2.2 SV02 liquid stream composition and conditions in SV01 and SV02.

SV02 Liquid composition		**Separator SV01**				**Separator SV02**			
N_2	CO_2	PT04 (bar)	TT07 (°C)	LT01 (%)	RT* (s)	PT10 (bar)	TT13 (°C)	LT02 (%)	RT* (s)
0.70	99.30	29.96	-56.31	20.16	50	12.01	-52.69	20.60	55
0.56	99.44	35.02	-55.84	19.80	47	9.93	-52.73	20.66	56
0.81	99.19	35.03	-54.16	20.24	41	11.98	-52.88	39.82	80
0.53	99.47	35.04	-55.51	19.77	36	9.99	-53.33	39.39	75
0.36	99.64	34.99	-56.06	20.14	37	8.92	-53.28	40.52	79
0.15	99.85	34.90	-56.79	25.24	43	8.04	-53.29	39.39	80

* Estimated retention time in seconds

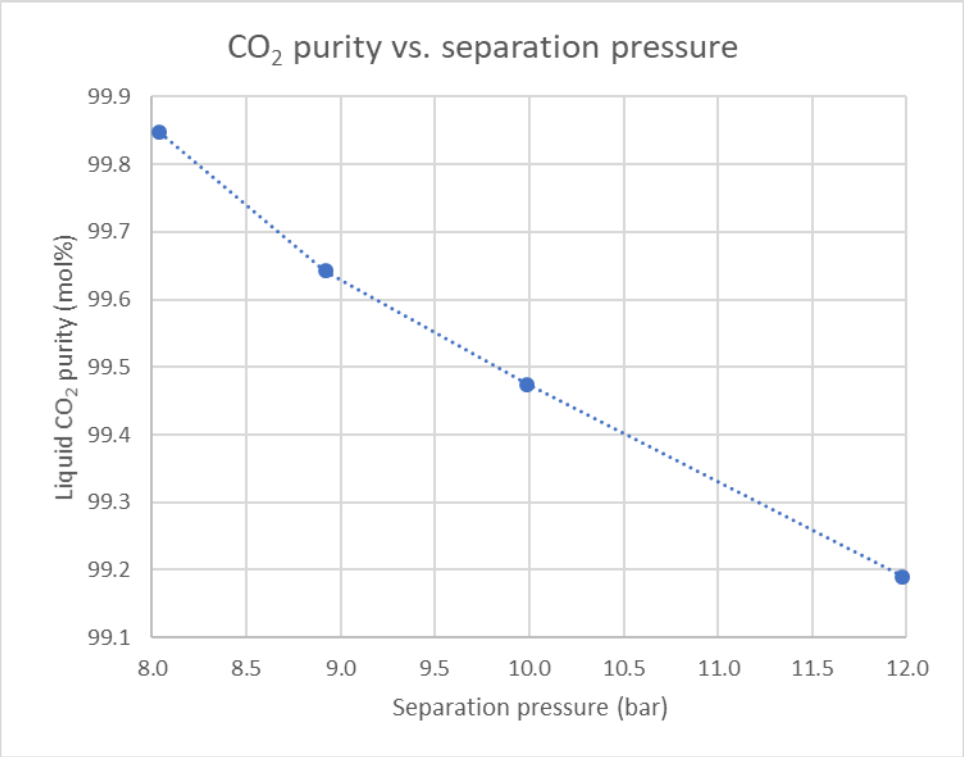

Figure 2.2: CO_2 purity vs. separation pressure in SV02

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2.2 Experiments 2018-09-10

2.2.1 Start-up and pre-cooling

The rig was started up from 26 bar standstill pressure and room temperature. A cool-down procedure similar to the one described in section 2.1.1 Start-up and pre-cooling were performed, and stable conditions and operation with 32 cm liquid level in both separators were achieved after approximately one hour and twenty minutes. The temperature development of TT04, TT07 and TT13 and the liquid levels in the separators are shown in Figure 2.3, Figure 2.4 and Figure 2.5, respectively.

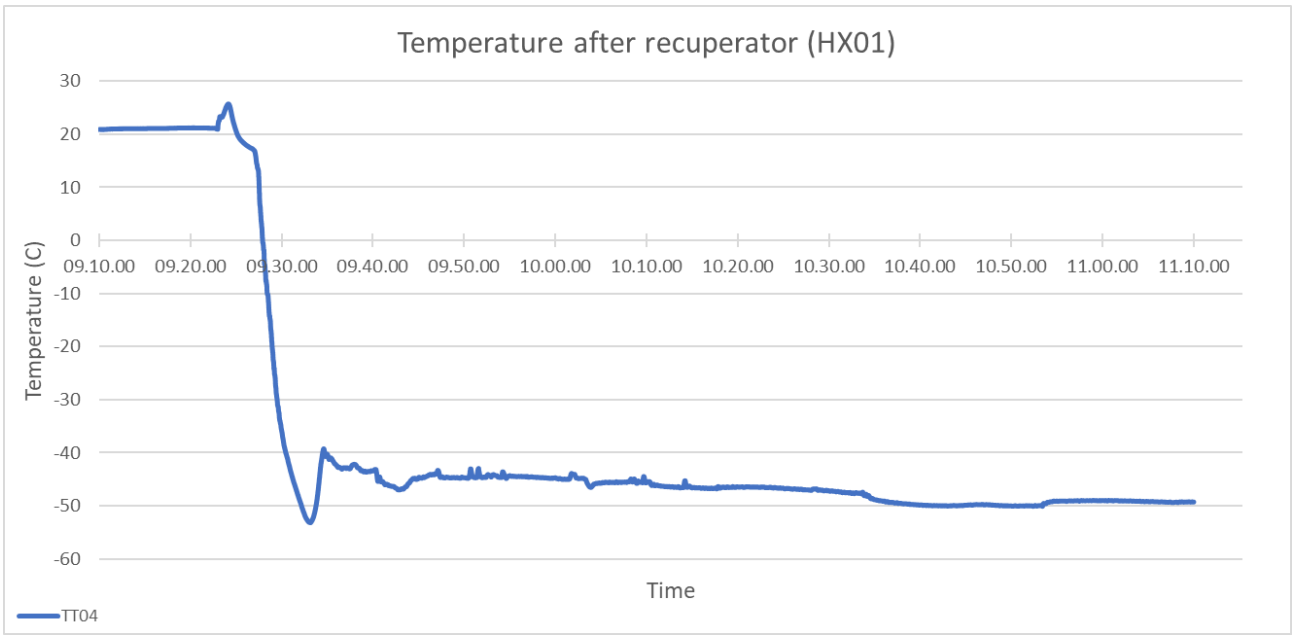

Figure 2.3:Temperature development of TT04

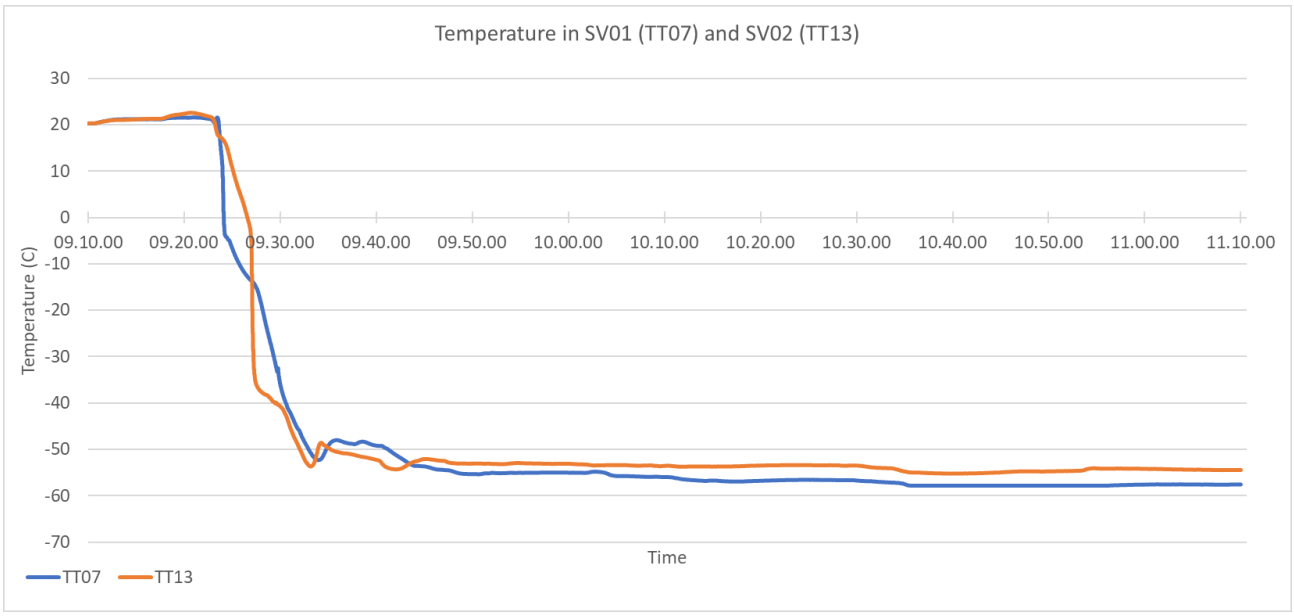

Figure 2.4:Temperature development of TT07 and TT13

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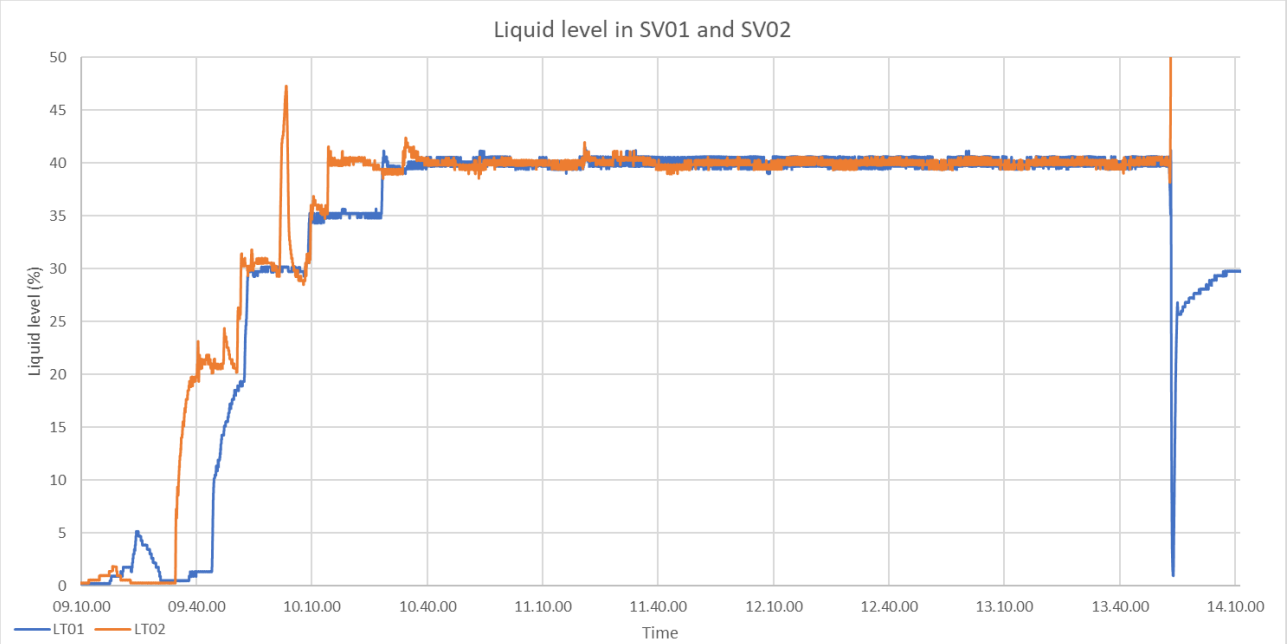

Figure 2.5:Liquid level in SV01 (LT01) and SV02 (LT02)

2.2.2 Steady-state results

At steady-state conditions with pressures of 30 bar and 8 bar in SV01 and SV02 respectively, and 32 cm liquid level in both separators, the composition of the feed stream entering SV01 and the vapor and liquid streams from SV01 and SV02 were measured. The composition measurements at the five sample points are performed sequentially over the span of 32 minutes. The conditions in the rig are kept as constant as possible during these measurements, but there are some variations as can be seen in Appendix A.2. The experimental measurement data are given in Table 2.3 together with corresponding results predicted from a HYSYS simulation of the process. The CO_2 capture ratio (CCR) is defined as the ratio between CO_2 flowrate in the liquefied CO_2 product and the CO_2 flowrate in the feed stream entering SV01. The CCR is estimated to 83 % based on measurement data obtained from the experimental run, and correspondingly 82 % from the HYSYS simulation run. The results from the experiment are thus close to expected values for CCR and purity. It should be mentioned that the experimental rig and the simulation model thereof lacks an internal recycle stream which will be present in a pilot- and full-scale process. The absence of the recycle stream reduces the obtainable CCR considerably for the simplified experimental rig.

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Table 2.3 Conditions in the rig during measurements and results from a HYSYS simulation

	Experimental results	**Simulation**
Inlet gas mixture composition	CO_2: 76.30	CO_2: 0.76
	N_2: 23.70	N_2: 0.24
EV01		
Pressure (PT00)	6.8 bar	7.3 bar
C1 and C2		
Pressure (PT01)	6.7 bar	6.7 bar
Temperature (TT02)	19.2 °C	18.3 °C
Pressure (PT02)	90.2 bar	89.5 bar
Temperature (TT03)	18.3 °C	18.3 °C
Mass flow (MT00)	4.015 kg/min	4.015 kg/min
HX01		
Temperature (TT04)	-50.0 °C	-50.7 °C
Pressure (PT03)	90.0 bar	89.5 bar
SV01		
Temperature (TT05)	-52.8 °C	-57.0 °C
Temperature (TT06)	-55.1 °C	-57.0 °C
Temperature (TT07)	-57.0 °C	-57.0 °C
Pressure (PT04)	30.0 bar	30.0 bar
Liquid level (LT01)	32 cm	32 cm
Liquid retention time	62 s	57 s
Liquid composition	CO_2: 95.87	CO_2: 96.03
	N_2: 4.13	N_2: 3.970
Vapor composition	CO_2: 22.11	CO_2: 22.07
	N_2: 77.89	N_2: 77.93
MT40	0.847 kg/min	0.854 kg/min
HX03		
Temperature (TT10)	-36.5 °C	-36 °C
SV02		
Temperature (TT11)	-51.0 °C	-53.1 °C
Temperature (TT12)	-47.8 °C	-53.1 °C
Temperature (TT13)	-54.4 °C	-53.1 °C
Pressure (PT10)	8.0 bar	8.0 bar
Liquid level (LT02)	32 cm	32 cm
Liquid retention time	72 s	67 s
Liquid composition	CO_2: 99.78	CO_2: 99.65
	N_2: 0.22	N_2: 0.35
Vapor composition	CO_2: 73.10	CO_2: 74.46
	N_2: 26.90	N_2: 25.54
MT30	0.376 kg/min	0.418 kg/min

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3 DISCUSSION AND CONCLUSIONS

The experiments carried out in the laboratory test pilot represent a somewhat simplified, but highly relevant, version of low-temperature CO_2 purification and liquefaction of CO_2-enriched flue gas which can be produced from polymeric membranes.

The experiments have been carried out in a scale of up to 6 ton-per-day feed mass flowrate, which is highly relevant with respect to understanding the performance of an industrial-scale pilot plant and eventually a full-scale plant. A higher feed mass flowrate, up to around 10 ton per day could have been achieved if the compressors were run on 100 % load, but would still represent the same order of magnitude as the flowrate achieved in these experiments.

The dimensions and achieved liquid retention time in the gas-liquid separators are of a scale relevant to understanding the operational and process control challenges in industrial-scale and full-scale plants.

Some simplifications are conceded in the current experimental set-up, which can be further understood and controlled in a modified and/or scaled-up set-up. The most obvious simplification in the performed experiments concern the lack of integration with a front-end membrane separation process. A principal layout of a fully integrated industrial pilot plant is given in CEMCAP deliverable D11.4 [1]. Another major simplification relative to an on-site plant connected to a cement flue gas line is the composition of the synthesized membrane permeate gas. In the experiments, a binary mixture between CO_2 and N_2 was used. Although the presence of other volatile components such as O_2 and Ar would influence the purity and composition of the final CO_2 liquid product, the experiments still gave strong indications that purities very close to those predicted from controlled vapour-liquid equilibrium measurements are obtainable.

The process design of the experimental set-up is somewhat simplified from a scaled-up process. Demonstrating the maximum energy efficiency and full process integration opportunities of the CO_2 liquefaction process were not the main priorities. Instead, the main focus was on demonstrating the CO_2 separation cut and the liquid CO_2 product purity obtainable by low-temperature separation taking place at temperature levels close to, and possibly somewhat below, the freeze-out temperature of CO_2. Moreover, demonstration of the operability and controllability of the process were key findings from the experimental campaign. The process control of the pilot facility showed to be well manageable. Several control loops were able to stabilize the process with respect to multiple temperature-based, pressure-based and liquid-level set-points.

Since the experimental setup comprises a closed-loop cycle with a more or less constant inventory of CO_2 and N_2, there is a considerable feedback in the process. Changes in liquid levels and pressure levels in the separators therefore affect the composition and flowrates in the feed-end of the process, which makes the process control more demanding than for an open cycle without this feedback. Since the closed-loop process could be controlled and stabilized by the control system, it is reasonable to conclude that an open process can be controlled by the same control structure as that used in the experimental runs.

To avoid excessive complexity and even more feedback loops in the closed cycle, the process layout of the experimental rig comprises some additional simplifications and short-cuts compared to the envisioned pilot-scale [1] or full-scale design, for which energy efficiency should be maximized.

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The cold vapour product stream from the bulk separator SV01 has not been heat-integrated with the incoming feed stream, which would require an additional heat exchanger. Instead, this stream is heated using an electrical heater along the vapour return line. Similarly, the reheat of the liquid separation product from SV01 is provided by electric heating instead of process-to-process heat transfer with the feed stream in an additional heat exchanger. In a scaled-up process these heat integration measures should be taken since they will reduce the refrigeration duty and thus also the net power requirement of the process.

The energy recovery potential from expanding the SV01 vapour product stream is not utilized in the experimental rig. In sufficiently large scale, energy can be recovered using a gas expander with power recovery by either direct coupling with a compression stage (when using centrifugal compressors) or as electrical energy from a generator.

As given in section 2.2.2, the estimated CO_2 cut rate, or CCR, was estimated to around 83 %. This figure would be considerably higher if an internal recycle of the vapour product from the purification separator SV02 is added to the design. The simulation-based estimate for CCR with the recycle included is 91 %. Since the experimental CCR results are close to the predicted results, it is also expected that the increased CCR could be demonstrated if the recycle were included.

The experiments were conducted without using the auxiliary refrigeration cycle R01 described in section 1.2.9. Instead of relying on auxiliary refrigeration, the equal separation state in SV01 could be obtained by compressing the feed stream to an excessive pressure level, 90–100 bar, and throttling it in the control valve V-05 after internal recuperation in HX01. The use of external refrigeration, however, will reduce the power requirement since the feed stream would be compressed only to approximately 30 bar instead of 90–100 bar and cooled at constant pressure in the heat exchangers HX01 and HX02A/B. In this case the 3rd compression stage would not be used.

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4 FURTHER WORK

4.1 Upgrades of the experimental rig

There are currently a few upgrades of the rig that is planned in the coming months. Firstly, the experimental rig will be connected to a permanent gas chromatograph which will enable simultaneous and more frequent measurements of the compositions at the different sample points. This will lead to more accurate results as there will be no, or a minimal, time lag between the measurements. Furthermore, a mass flow meter will be installed on the product liquid CO_2 line from SV02. Hence, the need to assume steady-state conditions for determining this mass flow will be eliminated. This leads to more accurate results, particularly for the CCR.

As previously mentioned, an auxiliary CO_2 refrigeration unit is currently being constructed, and will be connected to the rig by the end of the year. This will eliminate the need for over compressing the gas and re-expanding it to obtain the desired separation conditions.

4.2 Future experimental campaigns

The rig has currently been operated with a binary CO_2/N_2 gas mixture to simulate the membrane permeate gas. A real cement flue gas will contain significant amounts of oxygen and water, as well as other impurities after the first stage membrane separation. Experiments with a more realistic feed gas composition would be beneficial to determine the influence these components will have on the final CO_2 liquid product. Operation with small amounts of moisture in the gas would also give valuable operational experience related to possible problems with freeze-out and potential plugging in the rig.

It is also desirable to perform experiments with different CO_2 concentrations in the feed gas. Tests where the rig is operated with a stepwise increase of the CO_2 concentration from 50 to 90 mol% CO_2 would be beneficial, to see how this affects purities and CCR.

Experiments with different retention times (controlled by the compressor speed), as well as additional temperature and pressure levels in the separators would be beneficial to find the optimal operating conditions for the process.

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REFERENCES

[1] Trædal S., Berstad D., Stang J. Membrane-assisted CO_2-liquefaction scale up to TRL7–8. CEMCAP D11.4

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APPENDIX

A APPENDIX A

A.1 Experimental test rig components

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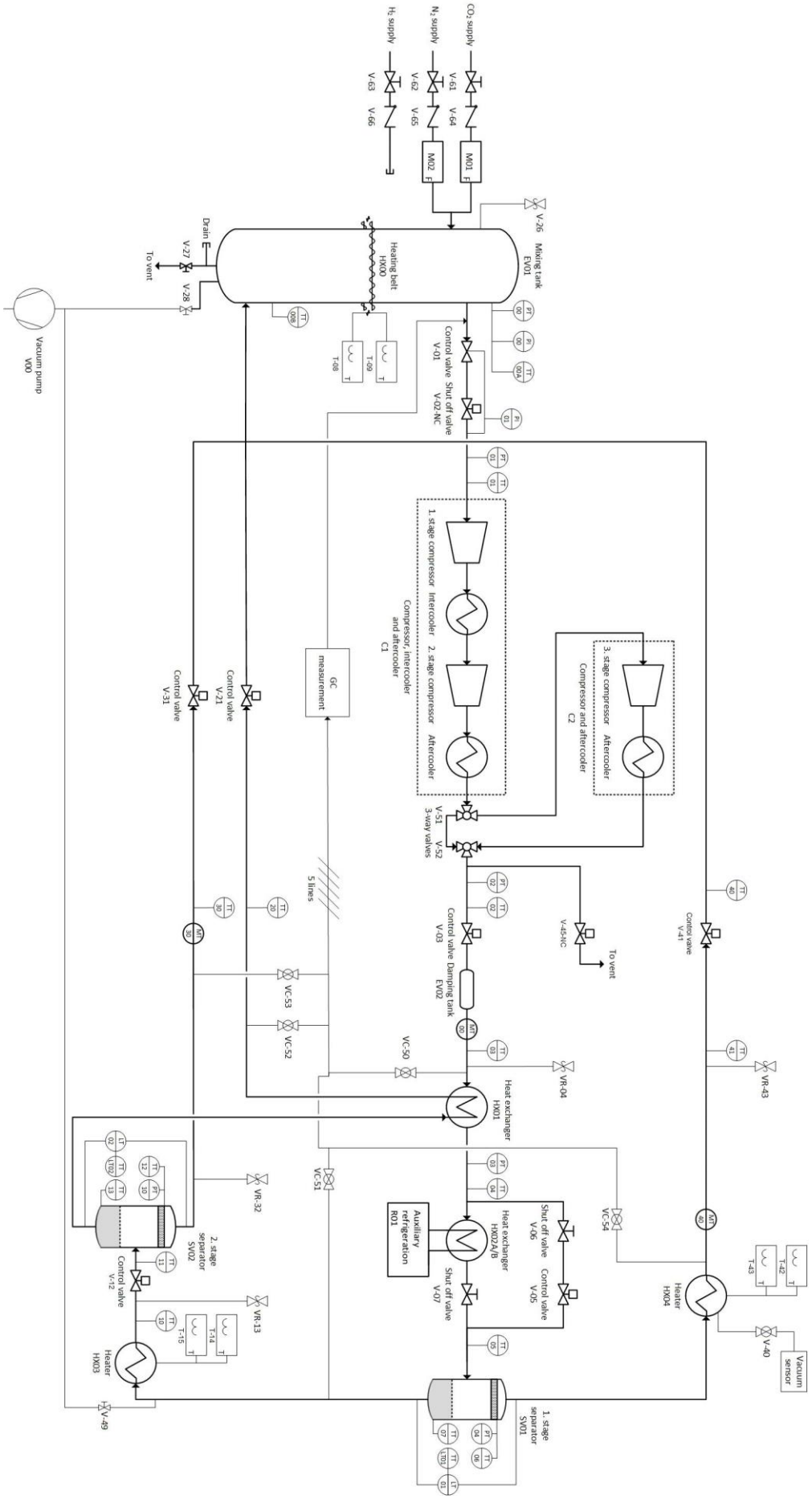

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A.2 Conditions in the rig during measurements 2018-09-10

Time	13:12	13:14	13:18	13:20	13:25	13:26	13:31	13:32	13:37	13:38	13:40	13:43	13:44	13:46	Average values
PT00	6.77	6.75	6.77	6.81	6.86	6.84	7.02	6.98	6.87	6.82	6.70	6.74	6.84	6.87	**6.83**
PT01	6.67	6.62	6.62	6.63	6.64	6.64	6.87	6.84	6.76	6.81	6.65	6.68	6.63	6.75	**6.70**
PT02	89.98	90.22	90.14	90.40	90.18	90.18	90.34	90.04	90.07	90.02	90.02	90.27	90.17	90.40	**90.17**
PT03	89.93	90.03	90.00	90.00	89.94	89.95	89.96	89.98	89.92	89.94	89.96	90.20	90.08	90.17	**90.00**
PT04	29.94	30.07	30.00	29.99	30.04	29.92	30.08	29.94	30.05	29.93	30.01	30.08	29.99	30.03	**30.00**
PT10	7.94	7.94	8.00	8.03	8.01	8.02	8.04	8.04	7.96	7.95	7.92	7.97	8.01	8.04	**7.99**
MT00	3.880	3.960	4.000	3.930	4.020	4.080	4.030	4.000	3.970	3.940	4.000	4.150	4.130	4.120	**4.015**
MT30	0.377	0.375	0.374	0.376	0.372	0.379	0.374	0.378	0.377	0.382	0.377	0.375	0.373	0.376	**0.376**
MT40	0.806	0.830	0.868	0.832	0.850	0.834	0.852	0.826	0.826	0.850	0.866	0.891	0.856	0.866	**0.847**
M01	3.285	3.358	3.441	3.434	3.441	3.438	3.441	3.433	3.434	3.437	3.524	3.523	3.515	3.530	**3.445**
M02	0.820	0.840	0.860	0.860	0.860	0.860	0.860	0.860	0.860	0.859	0.880	0.880	0.880	0.880	**0.861**
LT01	39.75	39.75	40.15	39.76	40.56	39.72	40.57	39.72	40.17	39.71	39.73	39.71	40.15	39.74	**39.94**
LT02	39.83	39.81	39.85	39.82	40.24	39.89	40.31	39.82	40.25	39.40	39.86	39.82	40.26	40.27	**39.96**
LT-TT02	-42.75	-42.77	-42.69	-42.65	-42.61	-42.58	-42.57	-42.52	-42.65	-42.68	-42.71	-42.74	-42.72	-42.64	**-42.66**
LT-TT01	-40.86	-40.83	-40.91	-40.92	-40.87	-40.92	-40.87	-40.87	-40.92	-40.93	-40.87	-40.92	-40.97	-40.96	**-40.90**
TT06	-55.06	-55.14	-55.08	-55.11	-55.12	-55.11	-55.06	-55.17	-55.15	-55.08	-55.20	-55.21	-55.15	-55.15	**-55.13**
TT07	-57.01	-57.03	-57.00	-56.96	-56.99	-57.01	-56.92	-56.91	-57.00	-57.03	-57.06	-57.05	-57.01	-56.98	**-57.00**
TT12	-47.88	-47.88	-47.80	-47.78	-47.88	-47.87	-47.75	-47.85	-47.92	-47.95	-47.91	-47.77	-47.83	-47.71	**-47.84**
TT13	-54.51	-54.49	-54.39	-54.36	-54.42	-54.42	-54.41	-54.44	-54.51	-54.47	-54.47	-54.35	-54.36	-54.35	**-54.43**
TT03	18.08	17.71	17.65	17.87	18.86	19.15	19.72	19.34	17.76	17.54	17.21	17.74	18.25	18.74	**18.26**
TT04	-50.17	-50.13	-50.02	-49.97	-49.96	-49.96	-49.95	-49.99	-50.15	-50.17	-50.16	-50.03	-49.99	-49.89	**-50.04**
TT10	-36.22	-36.60	-36.16	-36.71	-36.33	-36.46	-36.63	-36.88	-36.27	-36.22	-36.32	-36.84	-36.34	-37.25	**-36.52**
TT11	-51.03	-50.77	-51.39	-50.70	-51.48	-50.68	-51.41	-50.80	-51.21	-50.97	-50.85	-50.65	-51.18	-50.65	**-50.98**
TT05	-52.86	-52.84	-52.81	-52.77	-52.88	-52.82	-52.75	-52.78	-52.91	-52.92	-52.93	-52.88	-52.84	-52.79	**-52.84**
TT20	-44.02	-43.91	-43.75	-43.67	-43.78	-43.70	-43.72	-43.65	-43.92	-43.90	-44.07	-43.84	-43.82	-43.68	**-43.82**
TT30	-36.12	-36.13	-36.10	-36.10	-36.07	-36.08	-36.08	-36.12	-36.21	-36.25	-36.26	-36.25	-36.24	-36.23	**-36.16**
TT41	5.19	6.54	2.60	6.85	7.10	2.10	6.68	5.34	2.04	7.80	3.62	6.03	2.86	7.65	**5.17**
TT01	-2.84	-2.58	-0.20	0.58	0.39	-1.46	-1.95	-1.09	1.49	1.18	-0.22	-2.90	-2.81	-2.07	**-1.03**
TT02	18.76	18.37	18.80	19.08	20.10	20.35	20.57	19.91	18.46	18.22	17.97	19.13	19.49	20.00	**19.23**
TT00A	35.66	38.04	42.68	44.16	34.79	31.36	39.29	40.71	42.33	37.98	33.52	35.61	38.06	39.72	**38.14**
TT00B	26.63	26.67	26.76	26.76	26.65	26.65	26.78	26.81	26.81	26.76	26.70	26.73	26.75	26.79	**26.73**
TT40	27.54	27.55	27.57	27.59	27.58	27.64	27.62	27.64	27.63	27.64	27.67	27.63	27.63	27.65	**27.61**

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