Component commissioning of CO2 source (subsystem 1)

CCUS Innovation 2.0

Key Knowledge Deliverable 3.2

March 2024

Key Knowledge Deliverable Cover Sheet

This Key Knowledge Deliverable (KKD) has been produced by Cranfield University as part of the Department for Energy Security and Net Zero £1bn Net Zero Innovation Portfolio (NZIP) - CCUS Innovation 2.0 programme. The document is reflective of the status of the project at the time of writing. The material presented could have been subject to change as the project matured. These documents should not be considered a full representation of the final project.

Supercritical CO_2 power cycles are a novel process currently under consideration across the energy sector, including for carbon capture, utilisation, and storage (CCUS). Compared with conventional steam-driven systems, they boast higher cycle efficiencies, reduced emissions, and compact turbomachinery, resulting in reduced fuel and water consumption and lower capital expenditures. Through this project, John Crane—in collaboration with Cranfield University and the University of Edinburgh—aims to develop an innovative, uncooled high-temperature dry gas seal solution for supercritical CO_2 power cycles by using new simulations, material recipes and testing validations. This innovative dry gas seal, expected to work at high temperature and pressure values, will significantly reduce leakages and potentially enable the inclusion of an additional turbine expansion stage resulting to a higher efficiency CO2 cycle. Through a supercritical CO_2 cycle efficiency improvement, overall emissions will be significantly reduced, cutting the cost of CCUS and accelerating the adoption of supercritical CO_2 power cycles into existing and future power plants.

KKD 3.2 registers the process of ensuring that Subsystem 1 of the sCO_2 rig at Cranfield University was installed, tested, and operated correctly, providing assurance of its functionality and performance. This loop will be integrated into the test apparatus provided by John Crane. The primary objective of this stage is to ensure that the sCO_2 loop functions effectively and efficiently within the provided test apparatus.

KKDs to be released in full:

D2.2 Performance of 3D printed discontinuous fibre reinforced polymer composites.

D3.1 Conceptual design of the rig

D3.2 Component commissioning of CO_2 source (subsystem 1)

D3.3 Integrated sub-systems test - Shakedown test

D4.1 Commissioning of rig at John Crane

D4.2 Integrated system + seal rig commissioning at Cranfield University

KKDs to be released after being modified with critical information removed:

D3.4 Design and build of static/ dynamic test cell for 300 bar(g), 250 ºC

D3.5 Design and build of static/ dynamic test cell for 200bar(g), 600 ºC (CO2)

D4.3 Test of rig operating at 200 bar(g), 300 ºC (CO2)

D4.4 Testing to 200 bar(g), 250 ºC

D4.5 Testing to 200 bar(g), 500 ºC (CO2)

D5.1 Design/ build test seal with new seal head module/ bd-module

D5.2 Dynamic seal testing/ Benchmark testing, 140 m/s, to 200 bar(g), to 200 ºC

D5.3 Dynamic Seal Testing, 140 m/s, to 200 bar(g), to 200 ºC

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1. Introduction

1.1 Project Overview

Supercritical CO_2 (sCO_2) power cycles represent a cutting-edge development in the energy sector, with broad applications that extend to carbon capture, usage, and storage (CCUS). These cycles are gaining increasing attention for their potential to revolutionise power generation and environmental impact. Compared to conventional steam-driven systems, sCO_2 power cycles offer a range of compelling advantages, including high cycle efficiency, reduced emissions, compact turbomachinery, and reduced fuel and water consumption. In pursuit of these benefits and to overcome critical challenges, John Crane, in collaboration with Cranfield University and the University of Edinburgh, has embarked on a visionary project. The project's objective is the development of an innovative, uncooled high-temperature dry gas seal solution tailored specifically for supercritical CO_2 power cycles. This endeavour involves cutting-edge simulations, advanced material recipes, and rigorous testing validations.

1.2 Purpose

The purpose of this report is to provide details of the process followed to ensure that the equipment associated with Subsystem 1 of the sCO_2 rig was installed, tested, and operated correctly according to specifications.

1.3 Vision of the experimental activities at Cranfield University

Testing of the new balanced diameter sealing module will take place in two main stages: one at John Crane Slough and the other at Cranfield University. Within the activities planned at Cranfield University, there are two specific challenges involved in testing the sealing module:

1. Testing in CO_2 up to 300 °C.
2. Testing in CO_2 up to 500 °C

The objective of the test campaign is to produce performance maps that can verify and validate the results of Finite Element Analysis (FEA) and Computational Fluid Dynamics (CFD) studies. Temperature measurements will be taken at various locations to map the temperature profile throughout the sealing module. The test campaign will be successfully concluded if the extrusion resistance of the balance-diameter sealing module is within the design specifications for a Dry Gas Seal (DGS).

To meet the testing needs of the project, adjustments were required for the current sCO_2 test loop situated in Building 205A of the Gas Turbine Laboratories at Cranfield University. This setup will function as an open-loop system, delivering the necessary carbon dioxide stream to a test apparatus produced by John Crane. The test rig or mini-plant was previously discussed in the Key Knowledge Deliverable (KKD) report: CCUS2108-KKD-D3.1 [1]. It consists of the following equipment:

- CO_2 storage.
- CO_2 conditioning
- CO_2 pump
- CO_2 pre-heater
- CO_2 heater
- CO_2 expansion
- Control of the test rig

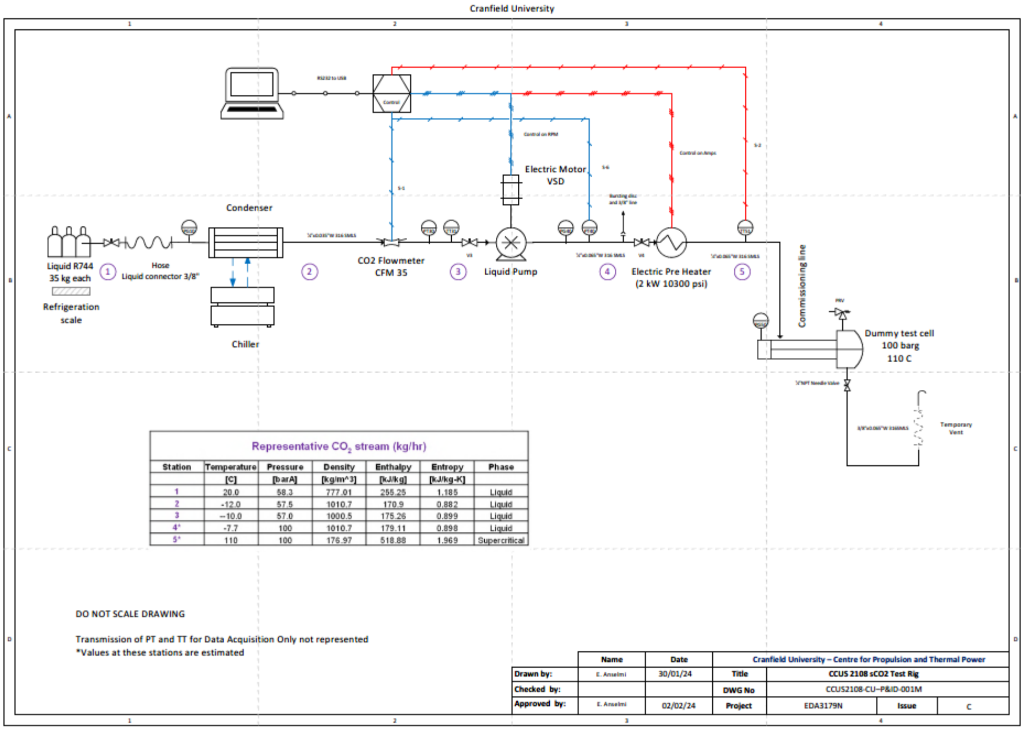
However, for the purpose of testing the sealing module up to 300 °C, it was identified early on that a subsystem assembly without a CO_2 heater was sufficient. Therefore, the configuration of Subsystem 1 of the sCO_2 mini plant refers to the previously mentioned list excluding the CO_2 heater.

A comprehensive process flow diagram of Subsystem 1, incorporating details regarding tubing, measurement, control, and materials, is illustrated in the diagram CCUS2108-CU-P&ID-001M version C, shown in Figure **1**. This drawing represents the current modification of the piping and instrumentation diagram (P&ID) CCUS2108-CU-P&ID-001 version A (November 2023), designed to guide the specific procedures followed during the commissioning process. Additionally, it assists in formulating steps for installation, startup, functional testing, and performance verification.

The principle for commissioning was to operate Subsystem 1 against a dummy test cell, represented by a rated pressure vessel. During commissioning activities, the working fluid is CO_2, which is supplied to the test rig from 22.8 kg cylinders connected to the condenser through a flexible hose. The liquid CO_2 exiting the condenser is measured using a Coriolis flow meter and pumped to the high-pressure side of the test rig using a positive displacement pump connected to a variable speed drive. The high-pressure liquid CO_2 is then heated in an electric heater and directed to the pressure vessel before being throttled back to atmospheric pressure. To produce the throttle, a needle valve was manually operated to serve as a back pressure control valve.

Two types of dummy test cells were utilised: firstly, a mechanical assembly with a capacity of 20 litres and CE rated at 100 bar(g) @ 100°C, equipped with a pressure relief valve; secondly, a non-stirred reactor, essentially a high-pressure/high-temperature vessel with a capacity of 1.25 litres, CE rated at 289 bar(g) @ 600°C.

Figure 1: **P&ID CCUS2108-CU-P&ID-001M**



Representative CO_2 stream (kg/hr)						
Station	Temperature	Pressure	Density	Enthalpy	Entropy	Phase
	[C]	[barA]	[kg/m^3]	[kJ/kg]	[kJ/kg-K]	
1	20.0	58.3	777.01	255.25	1.185	Liquid
2	-12.0	57.5	1010.7	170.9	0.882	Liquid
3	--10.0	57.0	1000.5	175.26	0.899	Liquid
4*	-7.7	100	1010.7	179.11	0.898	Liquid
5*	110	100	176.97	518.88	1.969	Supercritical

Transmission of PT and TT for Data Acquisition Only not represented
*Values at these stations are estimated

	Name	Date	Cranfield University – Centre for Propulsion and Thermal Power			
Drawn by:	E. Anselmi	30/01/24	Title	CCUS 2108 sCO2 Test Rig		
Checked by:			DWG No	CCUS2108-CU–P&ID-001M		
Approved by:	E. Anselmi	02/02/24	Project	EDA3179N	Issue	C

The instruments used in this test loop are described in Table 1. The flow rate, pressure and temperature have been measured at various locations to monitor the performance of the pump and the preheater and ensure the system's ability to satisfy the test requirements using various control strategies.

Table 1: Measuring devices during the commissioning

Process Variable	Sensor type	Number of	Tag / Location
Flow rate	Coriolis flow meter (FM350)	1	CFM35 / Pump suction side
Pressure	Mechanical pressure gauge Range: 0 to 10,000 psig	1	PG50 / Dummy test cell
Pressure	High pressure transmitter (MEAS model M5251-0000005-10KPG) Range: 0 to 10,000 psig	1	PT40 / Pump discharge side
Pressure	Low pressure transmitter (PRESS XDCR M3501-000005-05KPG) Range: 0 to 5,000 psig	1	PT30 / Pump suction side
Temperature	Thermocouple (type ICS type J)	2	TT31 /Pump suction side. TT51 / Preheater outlet

The compactness of the core equipment of Subsystem 1 can be observed in Figure **2**, where the following equipment – condenser, liquid pump, and preheater – can be seen housed within a compact metallic frame measuring 900 mm (W) x 800 mm (L) x 650 mm (H).

Figure 2**: Main equipment of Subsystem 1 – sCO_2 test rig**

The commissioning team was comprised of a group of specialists, including electrical and mechanical technicians, tubing and fittings experts, a process engineer, health and safety advisors, and personnel from the manufacturer.

Pre-commissioning and commissioning activities were spread in a period of 3 months, between January and March 2024.

2. Methodology

Commissioning, a crucial phase in any project, involves the introduction of design process fluids into the rig. This stage encompasses operating the mini plant, making necessary adjustments for the rig's satisfactory operation, and conducting "Functional tests". These tests or checks ensure that mechanical equipment and control systems function correctly. The following activities were pursued for the commissioning of Subsystem 1 of the test rig:

1. Establish general procedures for operation of pump and pre-heater.
2. System checks/inspection
    a. Electrical
    b. Mechanical
        i. Coupling alignment
        ii. Motor rotation
        iii. Vibration levels

        c. Instrumentation
            i. Flow and pressure calibration
3. Visual inspections/ Leak tests.
4. Safety devices/ safety procedures checks
    a. Functional test of ESD loop testing
    b. Functional test of alarms
    c. Functional test of gas detectors
5. Functional test related to delivering CO_2 at certain flow, pressure and temperature to the target values.
    a. Control modes of operation
6. Offline testing and calibration of instruments as reflected in P&IDs
7. Documentation and training personnel
8. Generate input for next stage.

After completion of the build, pre-commissioning activities were conducted. The pre-commissioning activities included: checking for the status of electrical, mechanical and instrument installation, flushing tubes, and pipes, and producing a generic method statement.

2.1 Commissioning activities

As indicated in the preceding section "Vision of the Experimental Activities", the primary objective of Subsystem 1 (as standalone system) is to deliver a consistent stream of CO_2 up to 300 °C to the sealing module within John Crane's test apparatus. The requested stream process conditions (variable flow rate) are outlined in Table 2:

Table 2: Carbon dioxide representative conditions requested for initial testing

Process Variable	**Value [Unit]**
Pressure	≤ 200 bar(g).
Temperature	≤ 300 ºC
Purity	As per R744 refrigerant – (purity of 99.995%)

A plan was developed to address all major pre-commissioning activities by packages, facilitating initial testing. These activities included electrical supply, tubing fitting, gas detection upgrade, chiller installation, and production of a generic method of operation. Once these pre-commissioning tasks were successfully completed, the sCO_2 system depicted in Figure **1** was passed-off. The individual packages were:

- Bottles of liquid R744 (21.4 or 22.8 kg presentations), secured and connected to subsystem 1.
- Condenser connected to Chiller for CO_2 conditioning.
- Control panel of subsystem 1 (liquid pump, flowmeter, and pre-heater) electrically connected.
- Dummy test cell (vessels of 20 or 1.5 litres) connected to subsystem 1 and to a needle valve, followed by venting tube.

As generic method of operation, a Safe Operation Procedure (SOP) was developed. The scope of this document encompasses the installation of the equipment and the necessary preparatory actions, the pressurisation and depressurisation of a CE vessel, and references to health and safety during the activity. It does not include instructions for operating the chiller, pump, or pre-heater of the rig, as these instructions were provided by the manufacturers and considered during the functional tests.

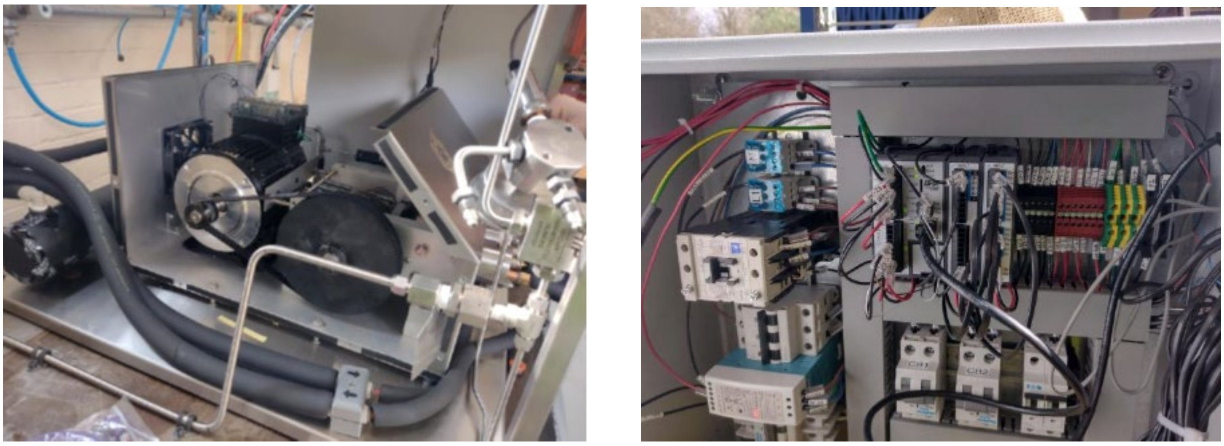
2.1.1 Installation verification

For installation verification, the API 700 Plant Completion Checklist [2], published by the American Petroleum Institute (API) in 1981 and currently withdrawn, served as a guide for the manufacturer and Cranfield University in defining their respective roles during the completion of work assignments for the rig, systems, and facilities. Specifically, sections 3 and 4, which include general procedures and a specific procedures checklist, were customised for this installation verification.

General procedures that were consulted and agreed upon included manufacturer service assistance, leak and pressure testing, inspection of pressure safety relief devices, purging, vessel packing, housekeeping, and noise surveys. Additionally, specific procedures for operating vessels, shell, and tube exchangers (condensers), pumps, piping systems, instrument systems, and waste disposal were discussed. The key procedures during the installation verification stage were chiller installation, electrical connections, and leak and pressure testing.

The chiller installation proved to be a relatively straightforward process, primarily focused on selecting the appropriate fittings, determining hose lengths, and choosing the suitable coolant media. The electrical inspection primarily focused on ensuring all connections were securely fastened and checking continuity across the control panel. Figure 3 shows examples of electrical inspection.

Figure 3: Installation verification of pump and control panel



Pressurisation and leak testing was accomplished without operating the pumps, given the high pressure of the R744 liquid bottles. Gradual pressurisation of the assembly allowed for easy identification of leaks, given the low temperatures developed by the expansion of CO_2 liquid towards ambient. The following recommendations were followed:

1. Mark out a no-go zone for staff safety and inform people on site not to enter the area.
2. Prior to undertaking the test, all joints will be inspected to ensure they are complete and installed correctly.
3. Compression type fittings should be tight and undamaged.
4. Tubes should be in the correct place and securely fastened.
5. Position the test gauge in a safe and visible location or use a remote viewing device.
6. Use a "soapy bubble" trace solution during the tightness test.
7. Pressurisation should be always carried out in a controlled manner, with pressure applied gradually.
8. The pressure source should never be left unattended when open to the system.
9. Throughout the process, at least two people should be in attendance.
10. The system should never be approached if the pressure exceeds 10% of the rated pressure unless the pressure has been reduced by at least 10% from the previous highest pressure.
11. Never attempt to tighten anything (e.g., screwed fittings) with the system under pressure. Always isolate and depressurise.
12. Venting should be monitored, avoiding release into closed spaces where the leakage could pose an asphyxiation hazard or in areas where wind conditions may expose personnel to persistent low concentrations of CO_2.

Figure 4 shows examples of the leak testing and pressure testing inspections, operations that were repeated each time the commissioning activities were carried out.

Figure 4: Leak and pressure testing of subassembly 1 and dummy test cell

2.1.2 Health and safety considerations

Two main sources of hazards were considered: working with carbon dioxide [3] [4] and working with pressurised vessels [5] [6]. Hazards were summarised in the SOP as follows:

Hazards

- **Exposure to fail pressurised systems may cause**

Impact from the blast of an explosion or release of compressed CO_2 *(liquid or gas)*

Impact from parts of equipment, pipes and fitting that fail or any flying debris.

Contact with released liquid or gas – Liquid splashes may cause freeze burns.

- **Exposure to a high concentration of CO_2 may cause asphyxiation.**

- **Exposure to a low concentration of CO_2 causes increased respiration and headache.**

- **Exposure to a controlled release of CO_2 may cause**

Contact with liquid or solid CO_2 - severe freeze burns.

- **Exposure to high and low temperature pipes may cause**

Burns

As mitigation of these Hazards (safety considerations):

Subsystem 1 has one rupture (bursting) disc (a device designed to relieve excess pressure in a system by rupturing at a predetermined pressure threshold), and the 20-litre dummy cell was equipped with a pressure relief valve.

While CO_2 is not highly toxic or flammable, it poses hazards associated with prolonged exposure or high concentrations. The practical limit for CO_2 exposure is 0.1 kg/m³ (56,000 ppm), with workplace inhalation exposure limits set at 5,000 ppm for an 8-hour period and 15,000 ppm for 15 minutes [3].

Warning signs of high concentration of CO_2 are:

- Tingling fingers
- Numbness
- Dizziness
- A strange mouth taste

The room where the rig is located is equipped with four infrared-type, fixed-point gas detectors, connected to a control panel capable of activating five audible/visual alarms (blue) when the CO_2 levels rise:

- **Pre-alarm at 1% (5,000 ppm)**
- **Main alarm at 2% (10,000 ppm)**

Oxygen (O_2) depletion will not be monitored as long as:

- Subsystem 1 is operated with open access doors
- Nominal content of bottles in the room are less than 65 kg of CO_2 – when main test lab room access is closed.

It is not recommended to carry out activities while the test lab room is closed.

Personal protective equipment (PPE) required:

- Safety glasses EN 166

- Ear defenders EN 352 (when depressurising gases)
- Protective gloves (includes high temperature gloves EN 398)

A list of authorised personnel for performing the commissioning is issued before / during the activity.

2.1.3 Commissioning procedures

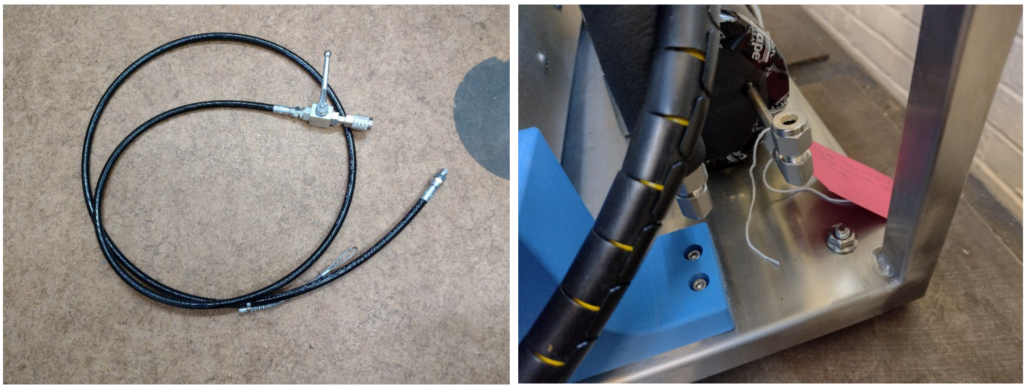
The following paragraphs outline three specific procedures followed during the commissioning process.

Procedure for filling the system with R744: When connecting R744 bottles to Subsystem 1, the following steps need to be observed:

1. Begin by attaching one end of the black CO_2 hose (or its equivalent) to the bottles and the other end to the condenser of Subsystem 1, ensuring not to tighten it yet.
2. Before opening the bottle valve, ensure that the lever of the needle valve on the black hose is closed and verify its hermeticity.
3. Gently open the needle valve to purge the hose briefly.
4. Subsequently, close the needle valve of the feeding hose and securely tighten the ¼” tubing end.
5. Label the needle valve of the feeding hose as "open" and ensure that Subsystem 1 receives pressure readings.
6. Finally, follow instructions from manufacturer and open valves v3 and v4 as indicated on the P&ID CCUS2108-CU-P&ID-001M (Figure 1).

In Figure 5, the hose and condenser connection for this procedure can be seen.

Figure 5: High pressure hose (left), condenser feed connection (right)



The Procedure for pressurising the dummy test cell using Subsystem 1 involves**:**

1. Flushing gas from the Subsystem 1 skid towards the test cell.

2. Controlled pressurisation of the assembly using the "Control Calculation Mode," with expected pressures of 60, 80, and 90 bar(g) for the 20-litre vessel, and 90, 150, 200, and 250 bar(g) for the 1.25-litre vessel.

3. Controlled pressurisation of the assembly using the "Control Pressure Mode," with pressures expected as before.

4. Controlled pressurisation of the assembly using the "Control Flow Mode," with pressures expected as before.

5. Back pressure control of all these activities will be conducted by manipulating the ½" NPT needle valve, wearing high-temperature-resistant gloves, and avoiding touching the assembly with the body.

6. Repeat steps 2 to 4, but using the CO_2 preheater, set at temperatures between 60 to 300 ºC upon vessel rating.

The procedure for depressurisation of the system:

1. Stop the pump remotely keeping all valves open.
2. Ensure that any hot surfaces are clearly identified and signposted to prevent accidental burns.
3. Securely close the valves on the R744 bottles and disconnect the feeding hose from the bottle end.
4. Return the bottles to secure storage or chain them against a stable base to prevent any unintended movement.
5. De-energise all electrical equipment, including the skid and chiller, to reduce the risk of electrical hazards.
6. Leave the needle valve fully open until the next day to safely release any remaining pressure. These safety measures are essential for maintaining a safe working environment and minimising the risks associated with depressurisation.

2.2 Functional testing

Functional testing is closely linked to the requirement for a test loop that can operate either intermittently or continuously, delivering supercritical CO_2 as required. Consequently, it is imperative to test the operation and performance of both the CO_2 liquid pump and the electrical preheater, ensuring the controlled delivery of supercritical CO_2 into the pressure vessel.

To assess the capabilities of the control system in flow and pressure modes, a beta tests of the control system was performed. The system components and instruments are connected to a graphical user interface, which allows the user to set the control parameters and monitor the cycle performance during the test, as shown in Figure 6. In Figure 6, the parameters' main interface screen is displayed, highlighting the pressure and flow rate of the pump discharge indicated by blue and green circles, respectively, along with the time history of the parameters. The lower blocks denote the control of the system components, indicating the condenser pressure, flow meter measurements, pump control modes and set points, preheater outlet temperature and set point, as well as a block named 'vessel 1', dedicated to the test article to be added. Figure 7 illustrates the alarms and events page of the graphical interface, indicating any potential issues with the system components or control. Additionally, this page displays a time history of the components' control actions taken to converge to the user input set-values.

Figure 6: Graphic user interface of control system for Subsystem 1

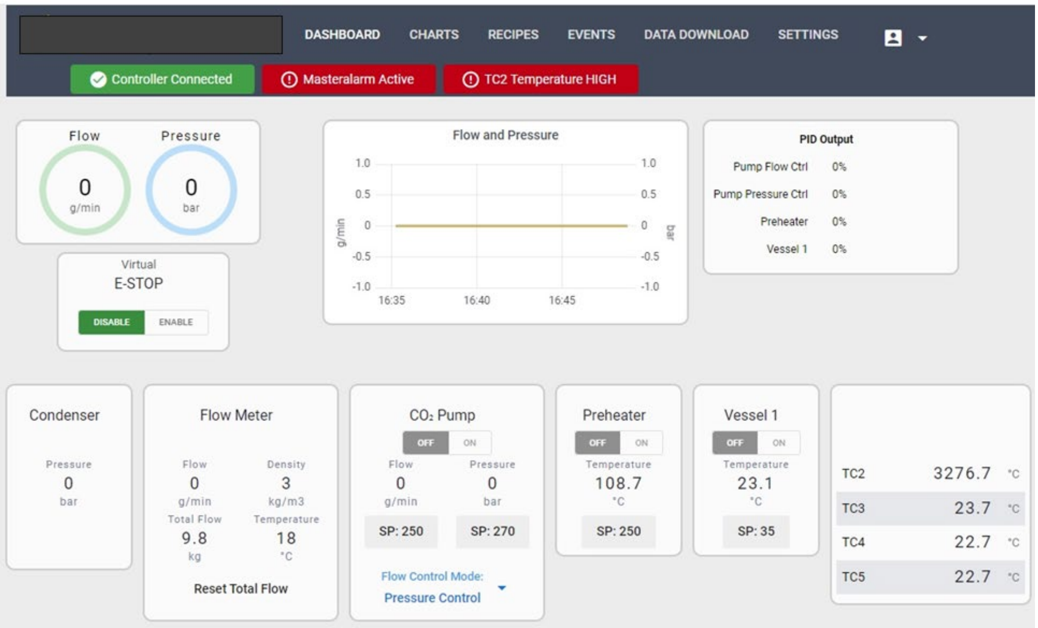
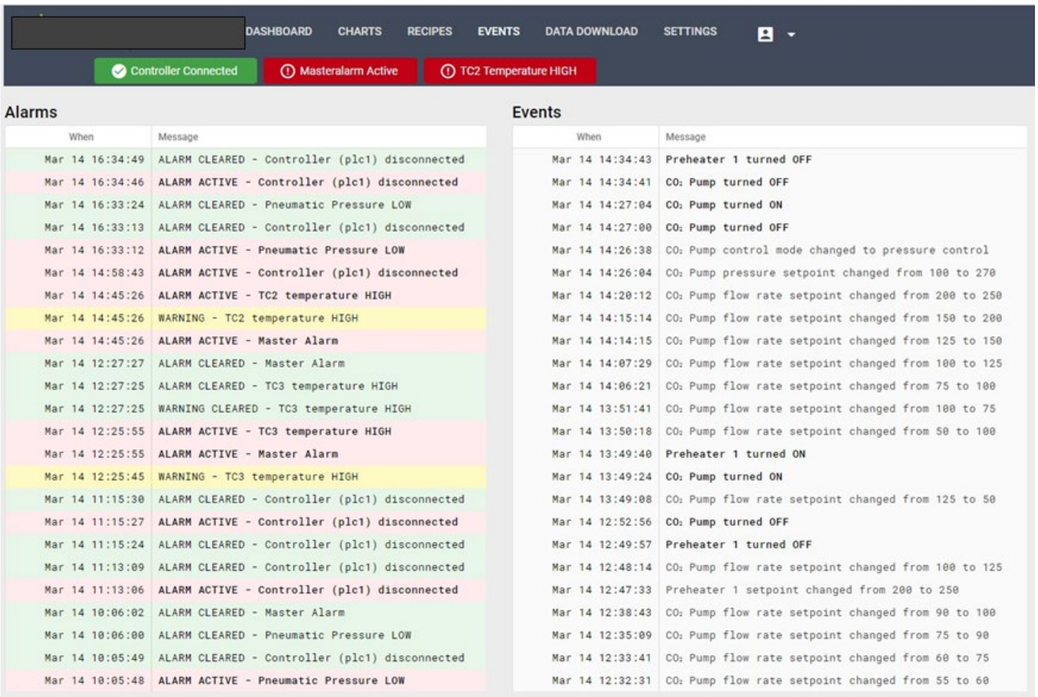


Figure 7: Alarms and events system (datalogger) for Subsystem 1



Alarms

When	Message
Mar 14 16:34:49	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 16:34:46	ALARM ACTIVE - Controller (plc1) disconnected
Mar 14 16:33:24	ALARM CLEARED - Pneumatic Pressure LOW
Mar 14 16:33:13	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 16:33:12	ALARM ACTIVE - Pneumatic Pressure LOW
Mar 14 14:58:43	ALARM ACTIVE - Controller (plc1) disconnected
Mar 14 14:45:26	ALARM ACTIVE - TC2 temperature HIGH
Mar 14 14:45:26	WARNING - TC2 temperature HIGH
Mar 14 14:45:26	ALARM ACTIVE - Master Alarm
Mar 14 12:27:27	ALARM CLEARED - Master Alarm
Mar 14 12:27:25	ALARM CLEARED - TC3 temperature HIGH
Mar 14 12:27:25	WARNING CLEARED - TC3 temperature HIGH
Mar 14 12:25:55	ALARM ACTIVE - TC3 temperature HIGH
Mar 14 12:25:55	ALARM ACTIVE - Master Alarm
Mar 14 12:25:45	WARNING - TC3 temperature HIGH
Mar 14 11:15:30	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 11:15:27	ALARM ACTIVE - Controller (plc1) disconnected
Mar 14 11:15:24	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 11:13:09	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 11:13:06	ALARM ACTIVE - Controller (plc1) disconnected
Mar 14 10:06:02	ALARM CLEARED - Master Alarm
Mar 14 10:06:00	ALARM CLEARED - Pneumatic Pressure LOW
Mar 14 10:05:49	ALARM CLEARED - Controller (plc1) disconnected
Mar 14 10:05:48	ALARM ACTIVE - Pneumatic Pressure LOW

Events

When	Message
Mar 14 14:34:43	Preheater 1 turned OFF
Mar 14 14:34:41	CO_2 Pump turned OFF
Mar 14 14:27:04	CO_2 Pump turned ON
Mar 14 14:27:00	CO_2 Pump turned OFF
Mar 14 14:26:38	CO_2 Pump control mode changed to pressure control
Mar 14 14:26:04	CO_2 Pump pressure setpoint changed from 100 to 270
Mar 14 14:20:12	CO_2 Pump flow rate setpoint changed from 200 to 250
Mar 14 14:15:14	CO_2 Pump flow rate setpoint changed from 150 to 200
Mar 14 14:14:15	CO_2 Pump flow rate setpoint changed from 125 to 150
Mar 14 14:07:29	CO_2 Pump flow rate setpoint changed from 100 to 125
Mar 14 14:06:21	CO_2 Pump flow rate setpoint changed from 75 to 100
Mar 14 13:51:41	CO_2 Pump flow rate setpoint changed from 100 to 75
Mar 14 13:50:18	CO_2 Pump flow rate setpoint changed from 50 to 100
Mar 14 13:49:40	Preheater 1 turned ON
Mar 14 13:49:24	CO_2 Pump turned ON
Mar 14 13:49:08	CO_2 Pump flow rate setpoint changed from 125 to 50
Mar 14 12:52:56	CO_2 Pump turned OFF
Mar 14 12:49:57	Preheater 1 turned OFF
Mar 14 12:48:14	CO_2 Pump flow rate setpoint changed from 100 to 125
Mar 14 12:47:33	Preheater 1 setpoint changed from 200 to 250
Mar 14 12:38:43	CO_2 Pump flow rate setpoint changed from 90 to 100
Mar 14 12:35:09	CO_2 Pump flow rate setpoint changed from 75 to 90
Mar 14 12:33:41	CO_2 Pump flow rate setpoint changed from 60 to 75
Mar 14 12:32:31	CO_2 Pump flow rate setpoint changed from 55 to 60

The control modes of subsystem 1 were tested according to matrices shown in Table 3. Both dummy test cells were pressurised under cold and hot conditions, following a combination of set pressures and temperatures. Each control mode (calculated, pressure, or flow mode) was tested using needle valves instead of back pressure controllers. Figure 8 show these needle valves.

Table 3: Example matrix for cold and hot pressurisation of the dummy test articles.

Target Pressure [bar(g)]	Temperature [C]	Variables to record
90	60	Time, needle valve position, temperature surface of tubing and vessels
	80	
	100	

Figure 8: Needle valve vessel 20-l (left), needle valve vessel 1.25-lt (right)

> ‘Needle valves were used as mechanism to regulate either pressure or flow during the commissioning activities.’

3. Performance verification

Due to the complexity of the commissioning phase, the discussion of test results is primarily focused on the tests conducted and to ensure that the test equipment meets specified performance criteria. In this context, particular attention was given to achieve the pressure and temperature levels near the target condition of 200 bar(g) and 300 ºC. These parameters are critical indicators of the system's ability to operate effectively and reliably within the desired range, and thus, essential to verify the functionality and performance of the equipment.

The test has been conducted at various pump and preheater control modes, including pump pressure control without heating, pump flow control without heating, pump pressure control with heating, and pump flow control with heating. Initial runs showed the pump's ability to handle mass flow rates up to 24 kg/hr against a back pressure of up to 100 bar(g) while the preheater element was turned off. During these runs, the pump was controlled either by setting a fixed pump discharge pressure or a fixed mass flow rate. In the fixed discharge pressure control mode, the mass flow rate is controlled by adjusting the opening of the needle valve connected to the pressure vessel, while in the fixed mass flow rate control mode, the needle valve opening affects the discharge pressure.

The preheater was activated to measure the maximum achievable temperature at various mass flow rates. During this run, the temperature was recorded at different points of the cycle using a thermocouple connected to a portable temperature reader, as shown in Figure 9. The temperature readings of this test run are recorded in Table 4. It can be seen from the table that the temperature at the outlet from the R744 cylinder is much lower than the ambient temperature, although at the beginning of the test, the cylinder was in thermal equilibrium with the surrounding environment. Consequently, the cylinder pressure drops from nearly 57 bar(g) at the atmospheric temperature to 42.7 bar(g) at the measured temperature after a few hours of operation. The temperature drop across the condenser is estimated to be 8°C, corresponding to around 100W of cooling conducted by the chiller, as shown in Figure 1.

Figure 9: Portable thermocouple

The fluid temperature is increased from 2.5 °C to 14.1 °C as the liquid CO_2 is compressed from 42.7 bar(g) to 260 bar(g) by the pump. The preheater heats the liquid until it reaches 222.4 °C, after which it is directed to the pressure vessel and the venting nozzle. It can be observed from the table that the liquid CO_2 heats up from 0.5 °C to 2.5 °C as it flows from the condenser outlet to the pump inlet due to heat gain from the ambient environment. Similarly, the gas loses temperature as it travels from the preheater outlet to the pressure vessel inlet due to heat exchange with the surroundings. This heat loss is estimated to be nearly 11 °C across a 1 m long stainless steel 316 tube with a nominal diameter of 0.25". The significant heat exchange with the surroundings is attributed to the low mass flow rate of the CO_2 through the test loop, as well as the lack of pipe insulation. The pipe geometry and insulation options should be carefully considered in the final test rig design to mitigate large temperature drops and ensure the test requirements are met.

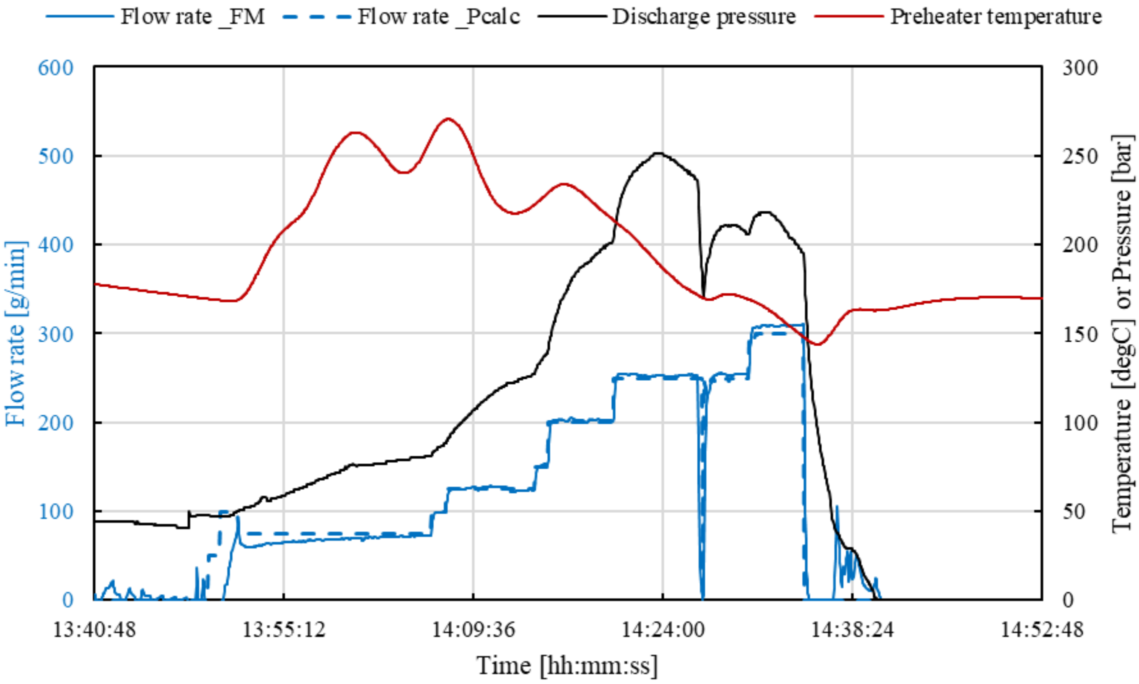
Table 4: Operating conditions and temperature readings at various locations of the test rig during the final test run.

Item	Location	Values
Operating conditions	Mass flow rate	300 g/min
	Suction pressure	42.7 bar(g)
	Pump discharge pressure	260 bar(g)
	Ambient temperature	20 ºC
Temperature readings	R744 cylinder outlet line	8.5 ºC
	Condenser outlet	0.5 ºC
	Pump suction line	2.5 ºC
	Pump discharge line	14.1 ºC
	Preheater outlet	222.4 ºC
	Pressure vessel inlet	211.6 ºC
	Venting nozzle outlet	29.7 ºC
	Pressure vessel bulk temperature	122.2 ºC

It should be noted that the bulk temperature of the pressure vessel, which weighs around 26 kg, affects the gas temperature at the venting nozzle. During the initial test runs, the CO_2 gas solidified from the Joule-Thompson effect of the expanding gas across the needle valve and the outlet nozzle, resulting in partial or total blockage of the flow path. However, as the temperature of the pressure vessel increased to around 122°C, the gas at the nozzle outlet became more stable, reaching an outlet temperature of 29.7°C. While ice formation downstream of the back pressure devices should not pose an issue to the polymer seal test, considering that the temperature requirement is 500°C, transient operation should be carefully planned.

The time history of various test parameters was recorded, as depicted in Figure 10. This figure illustrates the flow rate measured by the flow meter (CFM35), the flow rate calculated by the pump drive speed (Pcalc), the pump discharge pressure, and the preheater outlet temperature plotted against time for the test run when the heater is activated. During this test run, the mass flow rate was set at 80 g/min for 15 minutes while allowing the temperature to increase to prevent ice formation at the nozzle outlet. Subsequently, the mass flow rate was incrementally increased until reaching 250 g/min, recording a maximum pressure of 250 bar(g). Throughout this run, the preheater sensor recorded a maximum temperature of 270°C. However, the preheater temperature began to decrease again at mass flow rates exceeding 100 g/min. Although controlling the pump drive speed allowed for increased flow rates against high discharge pressure, larger flow rates affected the preheater's performance, failing to meet temperature requirements. This suggests the necessity for a second heater capable of attaining temperatures exceeding 500°C at a higher test flow rate, thus compensating for the temperature drop between the main heater and the test cell.

Figure 10: Time trends of the monitoring and control parameters recorded during testing subsystem in a vessel of 1.25-lt



Additional pictures of the commissioning and performance verification can be found in the Appendix.

4. Acceptance criteria

The initial commissioning of CO_2 test rig subsystem 1 has been completed, involving testing of the CO_2 pump, preheater, condenser, and chiller. Test outcomes have indicated that the subsystem successfully met and surpassed the required test parameters, achieving a pressure of 200 bar(g) and temperatures up to 300°C. It also demonstrated capabilities to handle variable mass flow rates on demand, up to 20 kg/hr. However, the preheater demonstrated limited capacity, delivering maximum temperatures only for mass flow rates below 6 kg/hr (100 g/min). Other machinery parameters not discussed in the report but measured included vibration (less than 1.06 mm/s rms), noise (around 20 dB without venting), and leakages (none perceived). In general, the manufacturer and Cranfield University identified and agreed upon actions for the transfer of responsibility for the care, custody, and control of the mini plant.

5. Recommendations

The observations made during the commissioning of Subsystem 1 have highlighted several challenges that require careful consideration during the subsequent experimental activities:

The cylinder temperature and pressure experienced a significant drop during the test. This issue confirms the requirement of installing a manifold that enables connecting multiple cylinders to reduce the flow rate per cylinder, allowing sufficient time for thermal equilibrium to be reached. This feature for the mini plant's storage of CO_2 was considered in the conceptual design [1].

The limited heating capacity of the preheater restricts the achievable mass flow rate when the preheater is solely responsible for heating. To increase the mass flow rate, the installation of an additional heater is necessary to meet the temperature requirements.

There were noticeable temperature drops across the lines connecting the preheater and the pressure vessel, as well as the condenser and the pump. It is recommended to insulate these lines with suitable materials considering both temperature limits and allowable temperature drops.

Commissioning observations should be utilised as input in the design process and calibration of numerical models. For instance, predicting temperature drops in the connecting tubes will impact the selection of the heating source for the sCO_2 heater and its control in relation to the final layout of the rig loop.

The control strategy of Subsystem 1 will need to align with the operational requirements of the test apparatus from John Crane and the objectives of the test campaigns. Currently, both the pressure and flow control modes of Subsystem 1 appear suitable for integration, provided adjustments are made to the constants of the Proportional-Integral-Derivative control. In the case of choosing a control flow mode, a feature reproducing the functionality of a back pressure regulator downstream of the test cell is required.

Specific documentation for the operation and control of Subsystem 1 should be produced. The generic manufacturer manual only addresses operations for the commissioning phase, but additional graphical interfaces, control tuning, and instrument calibration procedures should be incorporated.

It is recommended to conduct additional runs of subsystem 1 to familiarise personnel and gain confidence in adjusting the controls. This will also aid in the early identification of challenges for the upcoming deliverables.

6. Key knowledge deliverable

To establish a sustainable and enduring test rig for future testing of DGS technologies linked to CCUS applications, the following considerations need to be considered:

1. The integration of the test apparatus and test loop can be achieved through either a control pressure or control flow mode, depending on the final configuration of the test apparatus. Depending on this choice, a feature resembling a back pressure regulator will be necessary for the control flow mode.

2. The final layout of the loop and the selection of materials for the tubing connecting the sCO_2 heater with the test apparatus will be significantly influenced by the temperature drops resulting from heat losses in the tubing.

3. The supply of CO_2 in the form of R744 liquid bottles should be designed to minimise interruptions in the flow supply and sudden drops in pressure and temperature in the cylinders.

After commissioning activities, the equipment comprising subsystem 1 of the test loop has met the acceptance criteria.

References

[1] DEPARTMENT FOR ENERGY SECURITY & NET ZERO. (2003, November). D3.1 Conceptual design of the rig [Key Knowledge Deliverable CCUS Innovation 2.0]. Project Report 2108.

[2] API Publication 700 (September 1981), “Checklist for Plant Completion”, Second Edition.

[3] Health and Safety Executive, “EH40/2005 Workplace exposure limits”. Published January 2020, ISBN 9780717667031

[4] R744 (CO2) Safety Data Sheet according to REACH regulation (EC) No 1907/2006, as amended by UK REACH Regulations SI 2019/758. Version 10.1 from A-Gas (UK) Ltd. https://www.agas.com/uk/products-and-services/refrigerants/r744-co2/ (Page visited September 2023)

[5] Health and Safety Executive (HSE) UK. (2012). Safety requirements for pressure testing: Guidance Note GS4

[6] Health and Safety Executive (HSE) UK. (2012). Pressure systems: A brief guide to safety. INDG261 (rev2) ISBN: 9780717665280

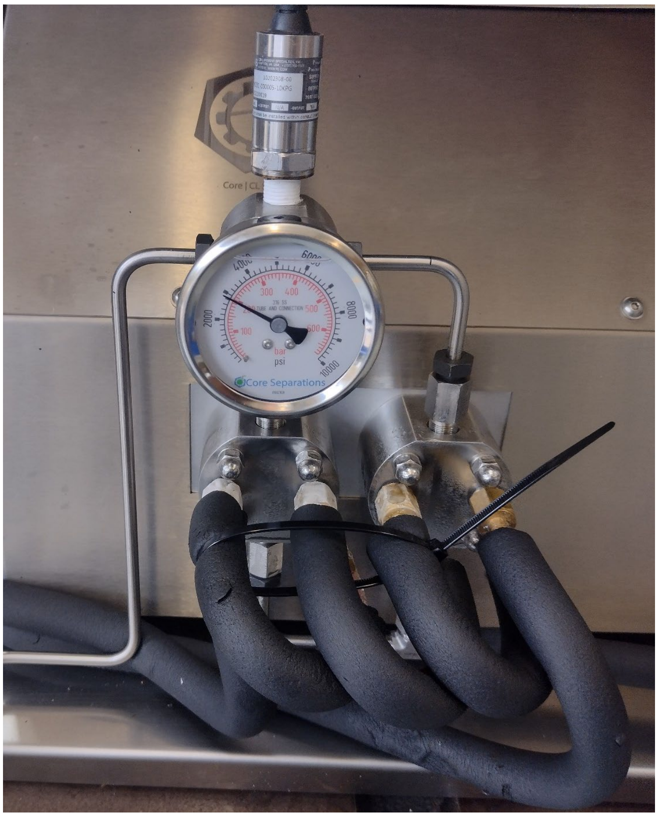
Appendix

Complementary pictures of the commissioning activity

Safe area for venting at B205a

Venting process outside building

Frozen nozzle during local venting (test)



Required delivery pressure of 200 bar(g)