Development of a CO2 Two-phase Flow Rig for

Flowmeters Calibration under CCS Conditions

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# Abstract

In order to calibrate and test flowmeters under CCS conditions, a dedicated gas-liquid CO2 two-phase flow rig has been developed. The CO2 flow rig offers a range of liquid, gaseous or gas-liquid two-phase flow conditions in horizontal and vertical pipelines. Fundamental design considerations, including the performance parameters, flow loop structure, working principle and traceability chain are presented. The design details of the weighing system are also included. The measures for maintaining multiphase CO2 flow conditions and controlling flow pattern stability are further discussed. Experimental results of calibrating a Coriolis flowmeter are finally introduced.

# 1. Introduction

Measurement and monitoring of CO2 flows across the entire Carbon Capture and Storage (CCS) chain are essential to ensure accurate accounting of captured CO2 and help prevent leaking during transportation to storage sites. The significant changes in physical properties of CO2 depending on its state (gas, liquid, two-phase or supercritical) mean that CO2 flows in CCS pipelines are complex by their nature. Meanwhile, impurities in a CO2 pipeline also make the flow more likely in the form of two-phase mixture. These characteristics of CO2 flows present a number of challenges for their accurate measurement. There are currently very few CO2 two-phase flow facilities that can be used under CCS conditions for flowmeter calibration. In order to calibrate and test flowmeters under CCS conditions, a dedicated gas-liquid CO2 two-phase flow rig has been developed as a result of a recently completed collaborative project funded by the UK CCS Research Centre.

# 2. Fundamental design considerations

*2.1 Performance parameters*

CO2 has a critical point at temperature of 31.06 ℃ and pressure of 73.8 bar (or 7.38 MPa) [1]. CO2 is a homogenous supercritical fluid when the temperature is higher than 31.06 ℃ and pressure greater than 73.8 bar. Above the critical pressure, but below the critical temperature, CO2 changes to a dense-phase liquid similar to the supercritical fluid at lower temperature. Above the saturated temperature, or below the saturated pressure, the liquid CO2 will change to gaseous fluid. The phase behaviour of CO2 is unstable due to phase-shifting effects of temperature, pressure and concentration.

For large-scale deployment of CCS technologies, pipelines are the preferred method of transporting CO2 between the capture and storage sites. For the transport of CO2 in large quantities, the two states of most interest are liquid and supercritical fluids [2]. The critical temperature of CO2 is very close to the ambient temperature of the pipeline. Sometimes CO2 is transported via pipelines near the saturated states of CO2. Gas-liquid two-phase CO2 flow conditions may appear in the pipeline as the ambient temperature rises or the pressure drops. Impurities in the CO2 will also make it more likely to exist in the form of gas-liquid two-phase flow.

In order to replicate such flow conditions, CO2 liquid flows are made available in the flow rig, and small fractions of gaseous CO2 can also be injected into the CO2 liquid so that gas-liquid two-phase flows can be created. Gaseous impurities such as air, O2, N2, Ar, propane can be injected into the pipeline to assess the effects of different impurities on flowmeter performance. The fluid in the test sections can be controlled to different phases, including CO2 liquid, gas, saturated gas-liquid mixture, and CO2 with impurities.

The typical operating conditions of CCS pipelines are in the range of -1~37 ℃ and 50~500 bar [1]. In order to make the flow conditions close to that in the actual CCS pipelines, the operating temperature range of the flow rig was set to 20~30 ℃. Under the saturated condition, the temperature and pressure are dependent on each other. The pressure range corresponding to this temperature range is 56.3~71.1 bar.

The flow velocity of the gas phase is maintained to 0.05~1 m/s (200~3600 kg/h) while the flow velocity of the liquid phase is 0.15~2.5 m/s (15~400 kg/h). Various flow regimes such as stratified, plug, slug and wave two-phase flows can be created in a horizontal pipeline [3]. In addition, bubble and slug flow patterns can also be simulated in a vertical pipeline.

*2.2 Flow loop and the working principle*

Closed-loop pipelines are designed so that the CO2 fluid can be reused, as illustrated in Figure 1. The phases of CO2 fluids in the flow rig include gas, liquid and gas-liquid phases as highlighted by blue, green and red colors, respectively, in Figure 1. CO2 is stored in a separator which also serves as a supply vessel. From the separator, the liquid CO2 flow is driven by a piston pump while the gas CO2 flow is driven by a compressor. Both flows are controlled separately and mixed at a downstream mixer. After the horizontal and vertical test sections, the gas-liquid two-phase CO2 either returns to the separator or diverted to a weighing vessel. Within this closed loop, the test sections can be used to provide single-phase gas or liquid flows or two-phase flows.

**Figure 1:** Schematic of the CO2 gas-liquid two-phase flow rig.

The flow rig includes two reference standards: a weighing system as the primary reference and two Coriolis Mass Flowmeters (CMFs) as the secondary reference. The output of the Meter Under Test (MUT) can be compared with either reference standard during its calibration or test.

The weighing system provides a gravimetric calibration approach and consists of a flow diverter, weighing vessel and electronic balance. The flow diverter is basically a pneumatic three-way ball valve and is used to realize a flying start-stop calibration method. In addition, a standing start-stop can also be achieved through the control system.

For the secondary reference, the single-phase liquid and gas CO2 flows are measured by two CMFs separately. These reference or master meters are KROHNE OPTMASS S15 and S08, respectively. With the weighing system working as the primary standard, the master meters are calibrated periodically using the gravimetric method to keep a low uncertainty.

*2.3 CO2 flow condition control*

The cross sectionally averaged flow velocities and the densities of single-phase gas and liquid basically determine the two-phase flow regimes. The pressure in the pipeline affects both the flow rate and the density and is generally related to the regulating valve opening in the pipeline. The pressure in the separator is roughly the same as that at the inlets of the compressor and the pump. The variation of the pressure in the separator is a key factor impacting the stability of system pressure when the states of the compressor, the pump and the regulating valves are relatively stable. The temperature determines the pressure in the separator as the CO2 fluid is in a saturated condition. The gas-liquid two-phase CO2 flow is actually a state of CO2 vapour and liquid coexisting near the saturated point. A large temperature difference between the gas and the liquid CO2 will create phase transition at the test sections downstream to the mixer. The real gas volume fraction will be different from that calculated through the mass flow rates measured by the master CMFs in the single-phase pipelines. Control of the temperature and pressure is necessary to maintain flow stability.

Air conditioning is used to maintain a stable ambient temperature in the lab. An insulation layer to the pipeline and vessels is also employed to reduce heat exchange between the fluid and the environment. The temperature of CO2 fluid in the separator can be increased by an electric heater wound on the outside surface of the vessel. The liquid CO2 can be chilled through a dedicated chiller using cooling water at the downstream of the pump. The chiller included in the piston compressor can also reduce the temperature of the CO2 gas. A refrigerator is responsible for controlling the temperature of cooling water to maintain the heat exchange efficiency. Regulating valves are applied to precisely control the flow rates of cooling water. Temperature transmitters, Programmable Logic Controller (PLC), heater and regulating valves make up a closed-loop feedback temperature control system.

The pressure in the separator, which is directly related to temperature, defines a minimum operating pressure for the rig. The pump and compressor can improve the pressure to different levels according to the requirement of each test. Two regulating valves are designed to control the pressure of single-phase gas CO2. The pressure of gas-liquid two-phase CO2 flows at the test sections can be adjusted by the back pressure regulator. The closed-loop feedback control system is composed of pressure transmitters, PLC and regulators to achieve accurate control of the pressure.

In order to eliminate the pressure pulsations generated by the piston pump and the piston compressor two buffers are used in the single-phase pipelines. For the liquid buffer, a 40 L pneumatic capsule inside the buffer vessel is filled with nitrogen gas to provide a damping action. For the gas buffer, a simple vertical vessel with a volume 40 L is used.

The volume flow rate of CO2 is proportional to the turn speeds of the pump and the compressor. However, the mass flow rate is also strongly related to the pressure in the pipeline due to the variation of CO2 density. The frequency of the piston movement will be very low and the pulsation of the pressure in the pipeline will be an issue for flow stability when the flow rate is near the low limit of the rig. In order to increase the turn speed and reduce the pulsation to improve flow stability, two bypass valves parallel to the pump and the compressor are used, respectively. The opening of the bypass valves will be set to a suitable value accordingly before the test, and then the working frequency, that is the turn speed of the pump, will be mainly used to regulate the flow rate. The master CMFs, PLC, frequency converter and the pump or the compressor form a closed-loop feedback flow rate control system.

A straight pipeline with a length of 50D is used at the upstream of the horizontal test section. The straight pipeline provides both space and time for a free development of the two-phase flow regime before the mixture flow reaches the test section. A sight glass, which can directly show the flow regimes in the pipeline, is used at the end of the straight pipeline. The length of the horizontal test section is 60D. The vertical pipeline is similar to the horizontal section with a straight pipe 20D downstream to an elbow. A sight glass and a 30D test section are also used for the vertical section.

*2.4 Safety design of the rig*

The primary risk is the leakage of CO2 from the pipeline, the vessels and other devices due to high pressure in the system. CO2 may gradually leak during the test. However, there might be a risk of sudden leakage due to a component failure.

*2.4.1 Pressure relief valves*

The set pressure of pressure relief valves is 7.8 MPa that is less than the maximum allowable working pressure of the pipeline and the vessel. The pressure in the pipeline or the vessel will quickly decrease once the pressure relief valve start to open and the fluid vents to outside.

Four pressure relief valves are used on the top of the separator, the weighing vessel, and the buffer vessel for the gas flow and at the horizontal pipeline to the downstream of the vertical test section. The CO2 liquid pump and the CO2 gas compressor both have their own pressure relief valve at the outlet to ensure their safety. There are some redundancies in these pressure relief valves, which enhances the safety operation of the whole system.

*2.4.2 Alarms and emergency response*

The control software of the CO2 flow rig can automatically give alarm using warning lights on the user interface and a detailed description for the alarm is also available if any process parameter goes over the preset limits. For example, the maximum working pressure is less than 7.2 MPa for most CO2 experimental tests. Consequently, if 7.3 MPa continues for more than 5 s in the pipeline or vessel, the software will trigger a yellow alarm of over pressure. Similarly, if 7.5 MPa continues for more than 3 s, it will trigger a red alarm. The control system will automatically run a series of operations to eliminate the risk if the red alarm is triggered.

The emergency response focuses on quickly reducing the pressure and temperature in the flow rig. In order to avoid the risk of over pressure, the gas CO2 compressor and the liquid CO2 pump will be switched off to stop further increasing the pressure in the rig. At the same time the venting valves in the horizontal pipeline, on the separator and the weighing vessel will be opened to release the CO2 to outside and reduce the pressure quickly. The electric heater will be turned off, and the opening of the chiller’s regulator valve will be quickly increased to 100% so as to reduce the temperature. The reduction of temperature can also help reduce the risk of over pressure.

# 3. Traceability chain

In the CCS chain, the accuracy of CO2 flow measurement should be better than 1%~1.5% as required by carbon management and trade [4,5]. It is of great interest to achieve lower uncertainty for flowmeter calibration. For gas flows, the flow rig uncertainty is targeted at 0.3% (*k*=2) so that flowmeters with 1% uncertainty can be calibrated. For liquid flows, the flow rig uncertainty is targeted at 0.16% (*k*=2) so that flowmeters with uncertainty of 0.5% can be calibrated. The two calibration references (primary weighing reference and secondary master meter reference) are designed for the flow rig (section 2.2). With the primary weighing reference, it is desirable to achieve even better uncertainty around 0.06%.

An electronic balance, supplied by Metter-Toledo, is used for the weighing system. The resolution in legal-for-trade application is 15 000 e with the value of e 20 g. The repeatability and the linearity are 1 g and 5 g, respectively. The resolution of display is 150 000 d and the division of reading is 2 g.

Considering the initial weight of CO2 in the weighing vessel before test, the actual quality injected into the 80 L vessel during one filling run is about 40 kg. Step-wise nonlinearity correction is used for reducing the error of the electronic balance. The interval of weights used for electronic balance calibration is 2 kg. The uncertainties of balance after nonlinearity correction *s*1 and *u*1 are shown in Table 1. The quantities of CO2 liquid and gas injected into the weighing vessel are respectively assumed to be 40 kg and 20 kg when calculating the relative uncertainties in Table 1. The filling time is typically 60 s.



**Figure 2:** Schematic of the weighing system.

Flexible tubes are used to reduce the effect of pipes on the reading of the electronic balance, as shown in Figure 2. However, high pressure in the pipe will make the flexible tube stiffer. Therefore, CO2 fluid in flexible tubes is vented out before and after each filling run. Venting of liquid CO2 in thebottom flexible tube through Valve SV3 before test will impact the zero point of the balance. A linear relationship between the drift value and the time after closing SV3 is observed. The drift of the zero point can, therefore, be corrected through a linear formula with time as an input variable. The uncertainties of zero point drift correction *s*3 and *u*3 are displayed in Table 1. Venting of gas CO2 in the top flexibletube through Valve SV6 after test will also impact the reading of the balance. The effect is a relatively constant value if the reading of the balance is collected at a fixed time point such as 1.5 min after closing Valve SV6.

**Table 1:** CO2 mass flow rate uncertainty of the weighing system

|  |  |  |
| --- | --- | --- |
| **Uncertainty items** | **Gas /%** | **Liquid /%** |
| **(20 kg)** | **(40 kg)** |
| Electronic balance (Type A) *s*1 | 0.00900 | 0.00900 |
| Electronic balance (Type B) *u*1 | 0.00670 | 0.00670 |
| Top tube venting *s*2 | 0.00750 | 0.00375 |
| Top tube venting *u*2 | 0.00184 | 0.00092 |
| Zero drift of electronic balance *s*3 | 0.01072 | 0.00536 |
| Zero drift of electronic balance *u*3 | 0.00566 | 0.00283 |
| Mass venting from top tube *u*4 | 0.00058 | 0.00029 |
| Chamber effect *u*5 | 0.00420 | 0.00502 |
| Weights *u*F1 | 0.00075 | 0.00075 |
| Weights *u*F2 | 0.00020 | 0.00020 |
| Weights *u*F3 | 0.00004 | 0.00004 |
| Flow diverter *u*6 | 0.0124 | 0.0124 |
| Flow diverter *s*4 | 0.0020 | 0.0020 |
| Flow diverter *s*5 | 0.0005 | 0.0005 |
| Standing start-and-stop method  overall uncertainty *U*S  (*k*=2) | 0.038 | 0.029 |
| Flying start-and-stop method  overall uncertainty *U*F (*k*=2) | 0.045 | 0.038 |

The influence of the top flexible tube venting can be associated with uncertainties *s*2 and *u*2 (Table 1). The mass of CO2 venting out through Valve SV6 can be calculated using the volume of the tube and the density of the CO2 which can be obtained according to the pressure and temperature in the tube. The uncertainty of mass calculation *u*4 is mainly from the error of the density caused by the inaccuracy of the pressure, temperature and the state equation of CO2 properties. The effect of piping as a chamber has to be considered because the pressure and temperature at the beginning and at the end of the test are usually different. Some CO2 fluid passes through the MUT, but may stay in the pipeline and does not flow into the weighing vessel. The mass variation of the CO2 in the pipeline from the MUT to the diverter is counted through the volume of the pipe and the density difference of the CO2 which is related to the pressure and temperature at the beginning and at the end of the test. The uncertainty of the chamber effect correction *u*5 is similar to that of the CO2 mass venting out from the top flexible tube *u*4.

The buoyancy correction for CO2 flow rig with a closed-loop pipeline and an enclosed weighing vessel is different from that for an open weighing tank. The buoyancy acting on the enclosed vessel is a constant since the volume change of the CO2 fluid inside the vessel does not affect the volume of the air occupied by the vessel. The quantity of the buoyancy is eliminated through zero setting at the beginning of the test. The incremental reading of the scale is related to the mass of the CO2 injected into the vessel during the test. The effect of the buoyancy acting on the weights during the calibration of the electronic balance has been included in the uncertainty *u*1. There is no buoyancy correction factor for such an enclosed weighing vessel.

In Table 1, *u*F1,*u*F2 and *u*F3 are respectively the uncertainties of 20 kg, 10 kg and 2 kg F2 class weights. The uncertainties of the flow diverter were tested by the method of transition time difference, as *u*6, *s*3 and *s*4 shown in Table 1. A highly accurate timer was realized by software using the CPU frequency and a counter. The actual accuracy is better than 20 *u*s so that the uncertainty induced by the timer can be ignored. The overall standard uncertainties of the standing and flying start-and-stop methods are calculated through formulas (1) and (2), respectively. The expanded overall uncertainties with 95% confidence probability are shown in Table 1.

. (1)

. (2)

# 4. Calibration of a Coriolis mass flowmeter

A vertically installed DN 15 CMF has been tested through the standard meter method under single-phase gas, single-phase liquid and gas-liquid two-phase flow conditions. The flowmeter has achieved errors within ±0.15% and ±0.25%, respectively, for single-phase liquid CO2 from 250 kg/h to 3600 kg/h or single-phase gaseous CO2 from 120 kg/h to 400 kg/h. The results have also shown that the CMF has greater but reproducible mass flow errors under two-phase flow conditions for a liquid flow rate of CO2 from 250 kg/h to 3200 kg/h and a gas volume fraction from 0 to 92%.

# 5. Conclusions

A dedicated flow test facility has been developed for CO2 flowmeter calibration and evaluation under CCS conditions. This facility is capable of providing single-phase liquid, gas and gas-liquid two-phase CO2 flows in one-inch bore, horizontal and vertical pipelines with pressures up to 72 bar. The precision weighing system as an integral part of the facility provides an uncertainty of 0.06% (*k*=2) for CO2 liquid flows. The reference CMFs equipped on the facility offer uncertainties of 0.16% (*k*=2) for CO2 liquid flows and 0.3% (*k*=2) for CO2 gas flows. Different two-phase flow regimes such as stratified, bubbly, plug and slug flows can be created. Impurity gases can also be injected into the test section to assess their impact on the performance of CO2 flowmeters.

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