A NEW FACILITY ON ACCURATE VISCOSITY AND DENSITY MEASUREMENTS

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Abstract

A new facility for accurate measurement of viscosity and density of CO_2-rich mixtures relevant for CO_2 Capture and Storage (CCS) has been constructed. The facility includes a two-capillary viscometer with several novel solutions to enable high performance over a range between 213.15 K and 473.15 K (-60 and 200 °C) in temperature and up to 100 MPa (1000 bar) in pressure. There are currently very little data available on viscosity of liquid CO_2-rich mixtures relevant for CCS, needed for instance in simulations of injection processes and reservoirs. Integrated in the setup is also a densimeter which is controlled to the same temperatures and pressures. Apart from providing density data, this enables accurate conversion between kinematic and dynamic viscosity. The total uncertainty target of the facility is 0.1 % (95% confidence level), except close to the critical point.

Keywords: Thermophysical properties, viscosity, density, impure CO_2, CO_2 transport and storage.

1. Introduction

In CO_2 capture, transportation, and storage (CCS), accurate predictions of thermophysical properties are required to optimize design and operations with respect to safety and costs. Thermophysical properties such as viscosity and density are crucial factors to determine the key design and operational characteristics of CO_2 transportation, storage, and injection infrastructure such as pipeline diameter, the pump/compressor power consumption and performance of heat exchangers, [1-3].

Viscosity is a necessary parameter for simulating laminar reservoir flow, where the resistance or pressure drop is proportional to the viscosity. Reservoir injectivity index is inversely proportional to viscosity, and viscosity also strongly affects plume evolution and storage efficiency. Viscosity is necessary to predict convection and diffusion processes, and the sweep efficiency of CO_2 EOR depends strongly on the CO_2/reservoir fluid viscosity contrast. Viscosity also has impact on history-matching and seismic interpretation, long-term CO_2 migration, and potential for leakage[4-6].

Since CCS processes cover a large range of fluid conditions and involve multi-component mixtures, there is a cost trade-off between purification of the captured CO_2 streams and designing and operating a transport and storage systems to handle larger quantities of the involved impurities [7]. Impurities have significant impact on thermophysical properties, particularly close to the critical point [8, 9]. However, as of today, there are still large gaps in the published viscosity and density data for CO_2 with impurities at conditions relevant for CCS, which are crucial for building good reservoir models and simulation tools. In particular, CO_2 is typically compressed to a liquid or dense phase for efficient transportation [1]. This requires accurate measurement

data for viscosity of CO_2 mixtures at these conditions, which are currently missing to a large extent, and more experiments are needed to fill these gaps and develop more accurate reservoir models and simulation tools in vicinity of the critical point [3, 8] For liquid phase viscosity and relevant compositions, only two rather limited data sets from a single lab have been identified [10]. Little data are also available for the gas phase [8, 11]. The situation for density is better, but most binary systems still have important knowledge gaps or limited data. Lack of experimental data typically leads large uncertainties in model predictions. Uncertainties in these properties can lead to costly overdesign and/or risks of inefficient or unsafe transport, injection and storage [12]. Therefore, it is important to focus on gathering reliable data with well-defined uncertainty levels that cover the phase space of interest.

The aim of the ImpreCCS project is to reduce CCS cost and risk through improving thermo-physical knowledge of pure CO_2 and CO_2-rich mixtures with impurities and at conditions characteristic for CO_2 transport and storage by acquiring high quality data. ImpreCCS is coordinated by SINTEF Energy Research within the framework of Norwegian CCS Research Centre (NCCS), and Norwegian University of Science Technology (NTNU), NORCE, University of Western Australia are important research partners, and NIST is an associated partner.

In order to address the lack of experimental viscosity data, a new novel two-capillary viscometer has been designed and constructed to cover the temperature range between 213.15 K and 473.15 K and pressures up to 100 MPa. This facility, which is part of ECCSEL ERIC [13] is designed for pure and mixed liquid, supercritical or gaseous states at high accuracy. Density is measured at the same conditions as viscosity, using a commercial

vibrating tube densimeter, enabling both closures of data gaps in density and direct conversion between kinematic and dynamic viscosity. The total uncertainty target of the facility is generally 0.1 % (95% confidence level), but this is not feasible in close vicinity to the critical point.

In this work, a description of the new facility and its measurement principle is presented.

2. Experimental Methodology

Quantifying the resistance to flow through a capillary is one of the most common and oldest principles for viscosity measurements [14, 15]. In 2004 [16], Berg described a Quartz Capillary Flow Meter (QCFM) for the measurements of gas flow rates with an uncertainty of less than 0.03 % at the National Institute of Standards and Technology (NIST). Berg also introduced a hydrodynamic model based on the Hagen–Poiseuille equation with relevant correction factors. The idea, after several modifications [17-21], was extended from using the flow meter as a single viscometer for absolute measurements to introduction of a two-capillary viscometer based on ratio measurements at low densities in the temperature range from 200 to 400 K with uncertainty ranging between 0.024% and 0.077 %. However, measurement of flow rate is required independently. Using the viscosity of helium calculated *ab initio* [22] as reference viscosity is recommended for the ratio measurements due to the high accuracy of current molecular dynamics methods. Applying the underlying principles introduced by Berg [16] and May [20] to build a modified two capillary system to measure viscosity of pure and mixed gases at high accuracy and high density is the main objective of this research work.

2.1 Fluid Dynamic Model

The principle of the capillary viscometers is based on Hagen–Poiseuille equation. The volumetric flow rate Q for an incompressible and Newtonian fluid in laminar flow through a capillary tube with internal radius r and length L is determined from [23]:

$$Q = \frac{\pi r^4 \Delta P}{8\eta L} = \frac{\pi r^4 (P_1 - P_2)}{8\eta L},$$

Eq. 1

$$Q = \frac{\dot{n}}{\rho_m}.$$

Eq. 2

Here ΔP is the pressure drop, P_1 and P_2 are pressures at inlet and outlet of the capillary, respectively, η is the dynamic viscosity of the fluid, r is the inner diameter of the capillary, L is the capillary length, $\dot{n}$ is molar flow rate, and ρ_m is the molar density. However, for a compressible fluid at temperature T and pressure P at locations z along the tube, the relation between pressure drop and viscosity can be found using the following relation:

$$\int_{P_1,T}^{P_2,T} \frac{\rho_m(T,P)}{\eta(T,P)} dP = \int_0^L -\frac{8\dot{n}}{\pi r^4} dz$$

Eq. 3

As discussed above, ratio viscosity measurements are employed in this work, where the known viscosity of helium at low pressure and 25 °C is used as a reference

[18]. Helium should be measured just after or before the test gas, such that capillary geometry effects are canceled out. However, for liquids and fluids in dense phase the ideal gas law is no longer applicable. As will be further discussed below, we will in our case use two-capillary viscometers, where the upstream capillary is at test pressure and temperature, and the downstream reference capillary operates at a constant low pressure (around 0.1 MPa) and reference temperature 298.15 K. The following working equation can be defined to estimate the viscosity of the test fluid, $\eta_{P,T}^{fld}$:

$$\eta_{P,T}^{fld} = \eta_{0,298}^{He} \left(\frac{\eta_{0,T}^{He}}{\eta_{0,298}^{He}}\right)_{ab\ initio} \left(\frac{\eta_{P,T}^{He}}{\eta_{0,T}^{He}}\right)$$
$$\times \left(\frac{\eta_{0,298}^{fld}}{\eta_{0,298}^{He}}\right) \left(R_{T,298}^{fld,He}\right)_{P,0}$$

Eq. 4

This equation includes five factors:

1) $\eta_{0,298}^{He}$: viscosity of helium at zero density and 298.15 K which is calculated with an *ab initio* method from quantum mechanics and statistical mechanics, with uncertainty less than 0.01 %.

2) $\left(\frac{\eta_{0,T}^{He}}{\eta_{0,298}^{He}}\right)_{ab\ initio}$: is the temperature-dependent ratio for the viscosity of He at 298.15 K and desired temperature T which is calculated *ab initio* with uncertainty less than 0.01 % in the range 200 K < T < 400 K.

3) $\left(\frac{\eta_{P,T}^{He}}{\eta_{0,T}^{He}}\right)$: the temperature-dependent ratio for He viscosity at desired pressure P and the zero density. This could be obtained with the two-capillary viscometer by setting both baths at temperature T and operating the upstream capillary first at low pressure and then at high pressure P.

4) $\left(\frac{\eta_{0,298}^{fld}}{\eta_{0,298}^{He}}\right)$: a reference value for the viscosity ratio, measured at 298.15 K for both He and test fluid.

5) $R_{T,298}^{fld,He}$: measurement of the temperature-dependent ratio of viscosity ratios which is determined from:

$$\left(R_{T,298}^{fld,He}\right)_{P,0} = \frac{\left(\frac{\eta_{P,T}^{fld}}{\eta_{P,T}^{He}}\right)}{\left(\frac{\eta_{0,298}^{fld}}{\eta_{0,298}^{He}}\right)}$$

Eq. 5

3. Experimental Infrastructure- Apparatus description

A simplified schematic of the viscometer is shown in Figure 1. Two-capillary viscometer principle is based on measuring the flow rate of the fluid flowing through the capillary tube and the pressure differential between capillary's ends. The new design is employing a new configuration, with a total of 2×2 coiled capillaries with different inner diameters to cover variation of fluid properties corresponding to the wide range of
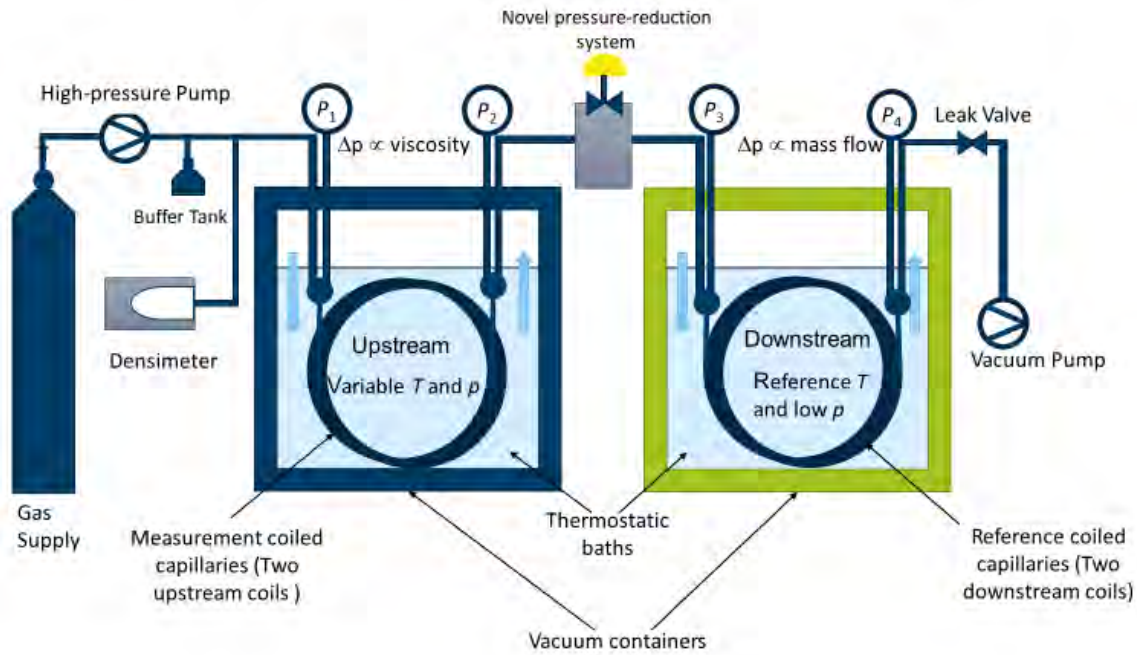
Figure 1: A simplified schematic of two- capillary viscometer.

temperatures and pressure for which it is specified. The pressure and temperature of the capillaries are precisely controlled. Their temperatures are controlled using thermostatic baths. Further, the containers with the capillaries are vacuum insulated to decrease heat loss to the surroundings and provide high temperature uniformity. Precise temperature control is important because the viscosity variations with temperature are significant. Since, the apparatus is comprised of different parts and complex processes, as illustrated in the 3-D sketch of Figure 2, the following section will discuss the main functions of the setup in more detail: 1) capillaries design and configuration, 2) high pressure gas source, 3) pressure and flow control, 4) Pressure measurements, 5) temperature measurements and control, 5) mass flow calibration, 6) density measurements.
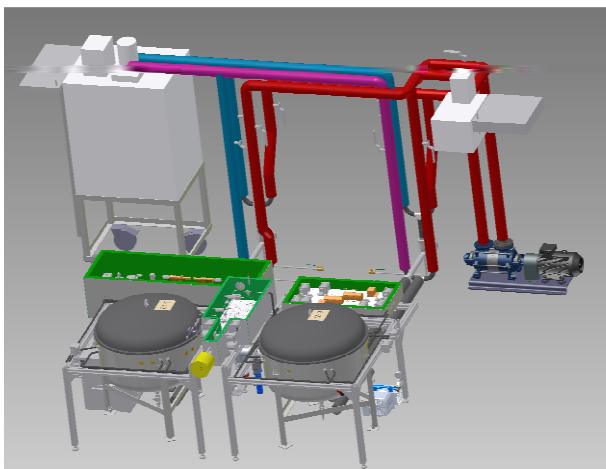

Figure 2: 3-D sketch of the facility

3.1 Capillaries Design and Configuration

The capillary tubes are the core components of the viscometer as measurements of the pressure drop across

the capillaries are used to calculate the viscosity. For this purpose, capillaries made of fused silica are used. The capillary tubes have an outer diameter of 1/32″ and are coated with polyimide for mechanical protection. One difference compared with previous two-capillary setups is the possibility to use in total 4 (2×2) different capillary configurations. There are two coiled capillaries at measurement conditions (upstream) and two coiled capillaries at reference condition (downstream), but only a single measurement capillary and a single reference capillary are used simultaneously. The two measurement capillaries, with an inner diameter of 75 and 200 μm, are at the measurement temperature and with the test fluid at the measurement pressure. The other two capillaries have an inner diameter of 250 and 500 μm and are used for measurements at reference temperature (298.15 K) and at low pressure, normally around atmospheric. By using different combinations of these four capillaries, viscosity can be measured for a wide range of temperatures, pressures and different fluids at flow rates and pressure drops that minimize the impact of the various uncertainty terms.

Table 1: Geometric parameters for capillary coils.

Capillary No.	Cap. 1	Cap. 2	Cap. 3	Cap. 4
Inner diameter (μm)	75	200	250	500
Outer diameter (μm)	794	794	794	794
No. of coils around stainless-steel grid	8.5	7.5	4.5	5.5
Capillary length (m):	13.225	11.671	7.009	8.563
Coil curvature radius (m), R_{curve}:	0.25	0.25	0.25	0.25

The capillary coil specifications are based on analysis of mass and volume flow, pressure drop, capillary diameter, temperature change, Dean number $De = Re \times \sqrt{r/R_{curve}}$ and Reynolds number $Re = 2M\dot{n}/$

$\pi r \eta(T, p)$), (where M is the molar mass) with respect to feasibility and uncertainty terms.
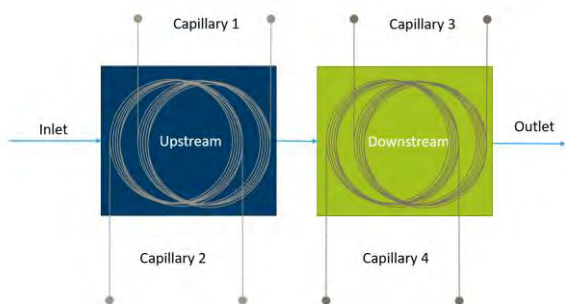

Figure 3: A Simplified schematic of 2x2 coiled capillaries with the two upstream coils and two reference coils.

In order to fit the capillaries to the other tubing of the viscometer, a metal sleeve was glued around each end of the capillaries using a special epoxy specified for the wide temperature range. The capillary mounts were tested with He and water at a wide range of pressures and temperatures before assembly. Each of these sleeves is fitted to a T-union, with the fluid inlet/outlet and pressure measurement system fitted to the other arms. The two capillaries of each container are mounted around a stainless-steel grid. Each grid with coiled capillaries in a horizontal orientation is immersed in a vacuum insulated thermostatic container in order to control their temperature. There are multiple high precision platinum resistance thermometers (PRTs) in the bath to allow temperature measurement and control and verify uniformity.

3.2 High pressure gas supply and upstream pressure control

The system is supplied with pure or mixed gas from gas cylinders. For investigation of mixtures, the composition must be known to high precision. Therefore, gravimetrically prepared mixed gases are provided from the CO₂Mix facility of SINTEF [24, 25]. The CO₂, CO₂ mixture, or He cylinder is connected to a high-pressure syringe pump (TOP-industry, PMHP model: 50-1000). The syringe pump pressurizes the system and keeps the upstream pressure P_1 constant during the measurements. The pump is connected to a buffer volume to a stabilize the upstream pressure. After the target upstream pressure has been reached, the fluid flows through measurement capillary. The pump is linked to the densimeter and thereby measurement of density at the same pressure as viscosity is enabled.

3.3 Pressure and flow control

Since the pressure drop is proportional to the viscosity for laminar flow, a sophisticated pressure control system is employed, which can control the pressure at the desired values with high accuracy and stability. Four different critical pressure measurement systems are required. As discussed above, the pressure P_1 before the first two coils is controlled by the high-pressure syringe pump. The second critical pressure measurement is of P_2 at the outlet

of the measurement capillary, which together with P_1 provides the pressure drop through the measurement capillary and hence the viscosity of the fluid under test. The pressures before the reference capillary, P_3, is controlled by a novel pressure-reduction system. The pressure of the measurement capillary can be from close to ambient to 100 MPa and the pressure for the reference capillary should normally be below 0.2 MPa and must be stabilized to high relative precision. No control valve was identified that could operate to the precision required for this large span of temperature differences and flow rates. Hence, controlling the pressure drop between the upstream and reference capillaries has been a challenge requiring innovation. The chosen solution includes a cascade system of six shut-off valves, five capillary coils with high flow resistance, and a control valve as illustrated in Figure 4. The general idea behind the design is that depending on the upstream pressure, which is controlled by the pump, and the flow, the shut-off valves will be closed or opened, directing the flow through a selection of capillary coils providing a high scalability in flow resistance. These five capillaries are similar to the capillaries used for the main viscosity measurements, but with smaller outer diameter (363 μm) and lengths (up to 5 m). The inner diameters vary between 25 and 75 μm. Hence, the pressure is reduced to an acceptable value for the control valve to reduce the pressure further down to the specified value. The fourth pressure system is the pressure drops across the reference capillary, the difference between P_3 and P_4 in Figure 2, is regulated by a variable leak valve (VAT Series 590) at the outlet, connected to a vacuum pump downstream. The pressure drop through the reference coils is a function of the flow and should be maximum 0.2 MPa. This pressure drop corresponds to a mass flow which can be calculated through a fluid specific calibration, which will be further explained in mass flow calibration section.
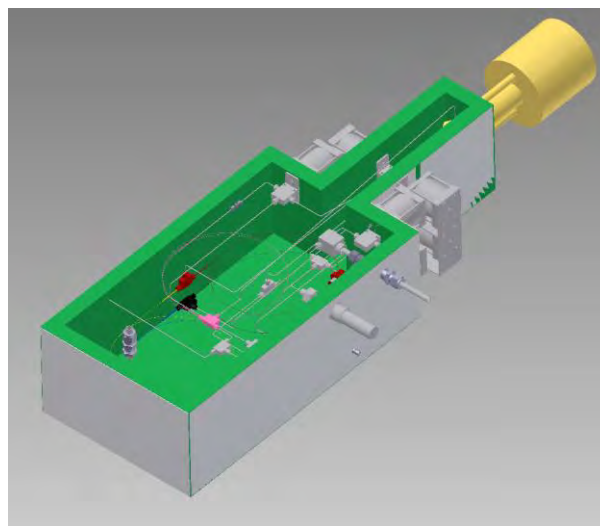

Figure 4: A 3-D sketch of the pressure control system between high- and low-pressure coils to reduce pressure to 0.2 MPa

3.4 Pressure measurements

A 3-D sketch of the pressure measurement system for P_1 and P_2 is shown in Figure 5. The pressure measurements

of P_1 and P_2 are performed using arrays of custom-made precision pressure transmitters with different full-scale pressures of 2.1, 6.9 and 13.8 MPa (Paroscientific) and 100 MPa (Keller) are used. This assortment in full-scale pressures allow for more precise low-pressure measurements, but also corrosion resistance must be taken into account, which is why arrays of two pressure transmitters with full-scale pressures of 6.9 MPa and 0.21 MPa (Paroscientific) are used for the pressure measurements of P_3 and P_4. In addition, there are two auxiliary pressure transmitters, one for measuring the pressure of high-pressure syringe pump and one transmitter to assess the pressure before the control valve used in the customized pressure-reduction system.
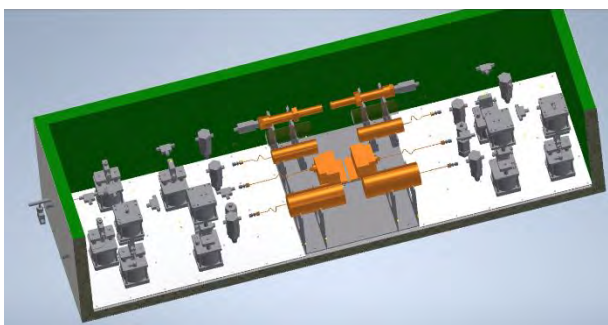

Figure 5: A 3-D sketch of the pressure measurement system-arrangement for P_1 and P_2

3.4 Temperature measurement and control

As mentioned, the experimental setup is designed for a process temperature range between 213.15 K and 473.15 K. The measurement capillaries are operating at a variable temperature and the temperature of the reference capillaries should normally be kept at reference temperature (298.15 K). However, as mentioned in Section 2.1, there is a need to keep both reference and measurement capillaries at the measurement temperature for full calibration with helium. The temperature of the measurement and reference capillaries are controlled by immersing them in circulating and thermo-stated liquids inside vacuum-insulated containers. Two Julabo thermostatic circulators with different temperature ranges are the main temperature providers to these two thermostated containers, The low-temperature circulator (FP89-HL) is employed between 213.15 K to 373K, using ethanol below its flash point temperature and water or silicon oil at the highest temperature. The high-temperature circulator (MA-12 w/ cooling water option) is used between 293.15 K to 423.15 K with water or silicon oil as working fluids.

The flow from the circulators are further temperature stabilized by using a small heater right before the inlet to the containers with the capillaries. The vacuum insulation, combined with careful design of the flow path in the setup, ensures high degree of thermal uniformity inside the tank.

The temperature is measured by several platinum resistance thermometers (PRTs). Two 25Ω Standard PRTs (SPRTs), calibrated with fixed point cells, are used for very high absolute accuracy. In addition, several PT100s are used to investigate uniformity and stability and enable control. In total, and based on experience from previous work [25], a thermal uniformity and accuracy of the order of 10 mK is expected.

3.5 Mass flow calibration

In order to calculate the viscosity, the mass flow of fluid must be known. In the two-capillary viscometer, reference capillary acts as a mass flow meter, if calibrated against the reference and test gas. For this purpose, a custom-made gravimetric setup is used. Mass flow is calibrated gravimetrically, by running a constant mass flow from a sphere into the reference capillary and measuring the flow duration and pressure before and after the reference coil during the flow period. In order to determine the mass of the gas that has flowed through the reference coil, the mass of the cylinder with content is carefully and very accurately determined before and after the flow experiment. The impact of various error sources, in particular varying buoyancy and nonideal repeatability /linearity of the scale, is reduced by applying the so-called ABBA scheme using a comparator [25-27]. The principle depends on comparing the object to be weighed (A) with a calibrated reference mass (B) by alternatingly placing the two objects on the comparator. In order to reduce impact of varying buoyancy, the objects should have similar shape and volume. In our case, object A is the sphere containing the test gas. B is a similarly the reference sphere which is empty. The mass of the two objects is made as equal as possible using calibrated weights, such that any impact of nonlinearity from the comparator is minimized. The weight of the gas that has left the sphere is hence found from the difference in mass of the calibrated weights used as well as the difference in comparator reading before and after the flow period.
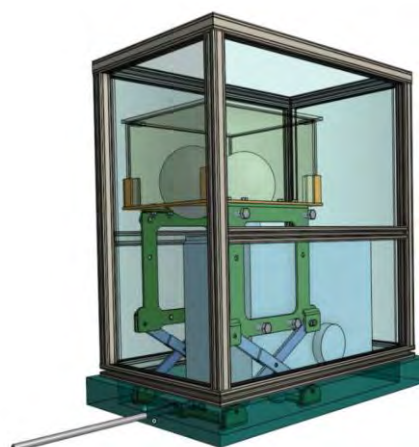

Figure 6: Mass flow calibration measurements.

3.6 Density Measurement

The density measuring cell DMA HPM (oscillating tube) manufactured by Anton Paar can be used within a temperature range of 263.15 K (223.15 K with lower

accuracy) to 473.15 K and a pressure up to 100 MPa. During a density measurement, the sample is introduced into the tube, which is excited to vibrate at its characteristic frequency. This frequency changes depending on the test fluid's density. By a precise determination of the frequency and a mathematical conversion, the density of the sample can be derived. The density meter will be calibrated as a function of pressure and temperature using fluids with density known to high accuracy.

4. Experimental procedures

During ordinary viscosity measurements both the upstream and reference capillary are precisely controlled. The upstream capillary's set-point temperature is the measurement temperature T and the downstream capillary's set-point is the reference temperature 298.15 K. As mentioned above, helium should be measured just after or before the test gas and such that capillary geometry effects are canceled out. Then, either helium or test gas is flowed through the two capillaries while the pressure drops across them are measured. During a measurement, the pressures P_1, P_3, and P_4 are controlled to reach the desired measurement pressure (P_1) and flow and output pressure (P_3 and P_4), while the measurement of P_2 is used to estimate the viscosity. The is to a large degree automated using a LabVIEW control and data acquisition program. However, some manual operations are necessary.

Also, it is required that the measurement plan should be prepared in advance by modeling of the system using e.g. REFPROP, EOS-CG, in-house models or measurement data. This is required in order to estimate density, viscosity, and phase behavior. Measurement in the two-phase regions is meaningless, and solid-state regions must be avoided. In the pump and buffer tank, fluids of more than one component must be always kept in a single fluid phase during measurements in order to avoid changes in the composition of the fluid injected into the viscometer.

Further, an optimum flow must be determined to minimize uncertainty. The maximum flow rate is limited due to the need to be well within the laminar regime and to avoid a too large variation of fluid properties along the capillary. The minimum flow rate is limited by the accuracy of the pressure measurements.

In order to prepare for the measurements, the vacuum containers should be evacuated using vacuum pump (including a pre-vacuum pump and a turbo molecular pump for high vacuum pressure). The thermostatic systems are filled with the correct fluids and properly configured which are part of manual operations. Due to the long time required to heat and cool the containers, viscosity is measured isotherm per isotherm, from low temperatures to high temperatures. Then, after making sure the temperature of both containers are stable, automated measurements can be started. Desired pressures P_1, P_3, P_4 are regulated using the high pressure syringe pump pump, pressure reduction system, and leak valve. In order to improve measurement precision, the bias between inlet and outlet pressure transmitters is measured before and after each measurement point. For these complex purposes, different valve configurations are involved. Finally, for each fluid to be investigated, the mass flow is calibrated.

5. Estimation of uncertainty

The overall combined standard uncertainties for the viscosity measurements $u_c\,(\eta(T, p, x))$ is determined from:

$$U_c\left(\eta(T, p, x)\right) =$$

$$\sqrt{u\,(\eta)^2 + \left[\left(\frac{\partial\eta}{\partial T}\right)_{p,x} u(T)\right]^2 + \left[\left(\frac{\partial\eta}{\partial p}\right)_{T,x} u(p)\right]^2 + \left[\left(\frac{\partial\eta}{\partial x}\right)_{T,p} u(x)\right]^2}$$

Eq. 6

Here, $u\,(\eta)$, $u\,(T)$, $u\,(p)$ and $u\,(x)$ are the standard uncertainties in viscosity, temperature, pressure, and composition. One important contribution to the combined uncertainty is the viscosity measurements (first term of Eq. 6 $u(\eta)$), mainly related to the pressure drop along the capillaries. The facility has been designed for an overall combined expanded uncertainty ($k = 2$, with ~98% confidence) for the viscosity measurements, $U_c\,(\eta)$ is 0.1%, except close to the critical point. Complete uncertainty analysis will be provided with the experimental data in reference journal publications.

5. Conclusion

Acquiring high-quality data requires a well characterized experimental apparatus with a well-defined uncertainty level. A new ECCSEL ERIC facility is designed specifically for CCS to accurately measure viscosity and density of CO₂ and CO₂-rich mixtures. The apparatus includes a modified two-capillary viscometer with several novel solutions to enable high performance over a wide range in pressure and temperature. The experimental procedure relies on measuring the pressure drop through capillaries which is proportional to both the flow and the viscosity for laminar flow. Currently, the various systems of the facility have been assembled and are under final testing and calibration, and we hope to set up and run the measurements on the first fluids within 2021. For validation of the facility, the plan is to have pure CO₂ and N₂ among the first investigated fluids.

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