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## Measurements of CO<sub>2</sub>-rich mixture properties: status and CCS needs

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### Abstract

In order to design and operate current and future CCS system and processes, accurate predictions of the behavior of the fluids involved is necessary. Currently, there are large knowledge gaps for fundamental properties needed to build models that can provide such predictions. Pure CO<sub>2</sub> is relatively well known, but in real processes there will be impurities that may have large impact. Hence, SINTEF Energy Research, Norwegian University of Science and Technology, and Ruhr-Universität Bochum are performing a project to close some of these knowledge gaps named CO<sub>2</sub>Mix. In the current paper, the need for new data will be explained, and some of the results produced so far in the project will be presented, including the establishment of highly accurate setups for the measurement of phase equilibria, density, and speed of sound, as well as new property data produced by these setups. Although the project has and will close important data gaps, more work is needed both with regards to the aforementioned target properties and other thermos-physical properties.

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## Nomenclature

$c$	Speed of sound
$p$	Pressure
VLE	Vapour-liquid equilibria
$S$	Entropy
$\mu$	Dynamic viscosity
$\nu$	Kinematic viscosity
$\rho$	Density



Fig. 1. Facilities of SINTEF ER for accurate phase equilibrium measurement using the analytical method developed in the CO<sub>2</sub>Mix project.

## 1. Introduction

For any process involving fluids, knowledge of relevant thermophysical properties is essential in order to design and operate it in an efficient manner and at the same time maintaining robustness and safety [1, 2]. This is the case for the various processes involved in CCS as well, and it is essential to optimize cost while ensuring safety in order to realize large-scale deployment.

For pure CO<sub>2</sub>, thermodynamic and transport properties are fairly accurately described by established models [3, 4]. However, in the CO<sub>2</sub> product of CCS capture processes impurities will be present [5] which will change the fluid properties. Even for small amounts of impurities, changes in for instance phase behavior and density can be fairly large [6], affecting processes both in capture, transport and storage of CO<sub>2</sub>-rich fluids [5]. Hence, accurate models are needed to predict properties of mixtures between CO<sub>2</sub> and the most relevant impurities. As shown by [5, 7-10], there are large gaps in available data for all thermodynamic and transport properties at conditions relevant for CCS. Thus, good models cannot be developed for all relevant situations, meaning that, in the best case, implementation of safe and robust processes will depend on large and expensive safety margins, for instance by excessive purification.

To cover some of these knowledge gaps, SINTEF Energy Research (SINTEF ER), Norwegian University of Science and Technology (NTNU), and Ruhr-Universität Bochum (RUB) have since 2010 conducted the BIGCCS

[11, 12] spin-off project CO<sub>2</sub>Mix. The project aim is to improve the thermodynamic-property data situation relevant for conditioning and transport in CCS, regarding phase equilibria, density, and sound speed by developing new infrastructure and provided highly accurate measurement data [6]. Measurements of vapor-liquid equilibria are performed by SINTEF Energy Research and NTNU, whereas density and speed of sound are measured by RUB.

In the current paper, Section 2 will in brief terms provide an overview of the current unsatisfactory state of thermophysical properties of fluids and conditions relevant for CCS, and its implications for the implementation of future CCS system, Section 3 will present an overview of the projects VLE measurements, Section 4 will discuss the density and speed of sound measurements, before the conclusions of Section 5.

## 2. Project motivation and data needs

In order to design any process system in an efficient and robust way, accurate predictions regarding the fluids involved are required, which again necessitates that their thermodynamic and transport properties are known within acceptable uncertainty limits. That these properties can change drastically with a relatively small amount of impurities, for instance present in the CO<sub>2</sub> product from capture processes, and that the impact of impurities in many cases currently is not satisfactory quantifiable, are facts that perhaps are not always realized.

For instance, phase behavior must be known in order to design transportation systems based on both pipelines and shipping. The gas-liquid phase boundary of pure CO<sub>2</sub> spanning between the critical point at 31 °C/ 74 bar and triple point at -57 °C/ 5.2 bar is centrally located with regards to conditions experienced during transport. Hence, unlike natural gas, there is lower pressure limit in pipeline transport in order to avoid two-phase flow, and care must be taken for instance when designing compression processes. As will be shown in Section 3, relatively small levels of impurities can change this phase behavior drastically. In order to avoid corrosion, it is also very important to predict the conditions where a water rich phase is formed. Unfortunately water solubility is known to be lower in the presence of other impurities such as methane, SO<sub>2</sub>, and NO<sub>x</sub> [13-15], and, like methane, CO<sub>2</sub> also forms hydrates at unfavorable conditions [16] that can block processes or wear down process equipment. Planned and or accidental depressurization may lead to strong cooling which can lead to e.g. the formation of dry ice or hydrates. In addition, water solubility is much lower in gas than in liquid phase [17].

Density is another thermodynamic property of obvious importance, which determines the dimensions of all equipment and in the end the storage capacity. Density is however also very important with respect to energy use in process equipment, and is also needed to high degree of accuracy for most mass flow measurement principles. Hence, to enable fiscal metering, necessary for a working CO<sub>2</sub> market, high fidelity models for CO<sub>2</sub> mixture density must be created. As seen in Fig. 2, according to the most accurate models today, only 5 % of N<sub>2</sub> may drastically alter the density of the fluid when getting close to the critical point. For instance at 40 °C, 9 degrees above the critical point, the reduction in density is up to 45 %. For other impurities the effect could be opposite [6].

Speed of sound is needed in the modeling of transient fluid dynamic phenomena, and is also useful for fitting equations of state, as in classical mechanics the speed of sound can be expressed through the relation  $c = \sqrt{(\partial p / \partial \rho)_S}$ , where the subscript *S* means that the derivative is performed with constant entropy.

Although not the focus of this project, there are a range of other properties that have to be known in order to design different processes in CCS. For instance, viscosity data are needed in order to calculate pressure drop in for instance pipes, wells, during injection, and in heat exchangers, and to calculate the power consumption of rotating machinery. Diffusion coefficient, another transport property, and the interfacial property surface tension are needed in a range of different fluid dynamic problems including reservoir modeling. The transport property thermal conductivity and the thermodynamic property heat capacity are needed for a range of heat transfer problems, especially relevant for CCS chains involving shipping and/or low-temperature CO<sub>2</sub> separation. As exemplified in Fig. 3, also these properties are strongly affected by impurities.

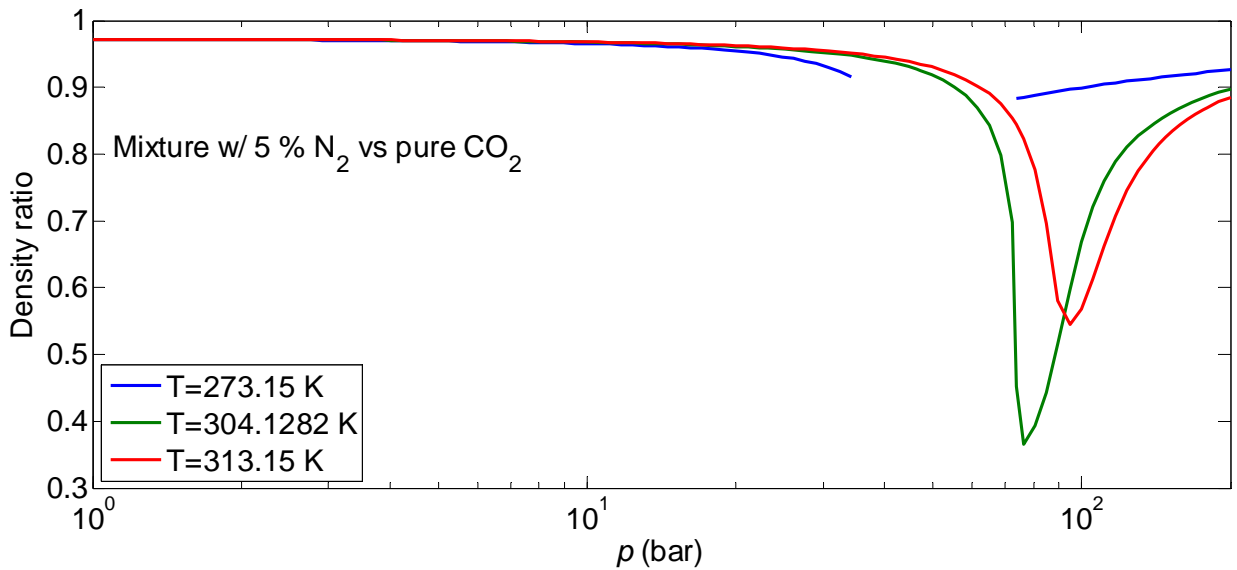


Fig. 2: Ratio between the density of CO<sub>2</sub> mixed with 5 mol % N<sub>2</sub> and pure CO<sub>2</sub> as a function of pressure for selected temperatures calculated using REFPROP v 9.1 using GERG 2008 EOS for mixture and Span-Wagner EOS for pure CO<sub>2</sub> [3, 18-20]. Curve of 0 °C is discontinuous since the viscosity is not defined in the two-phase region.

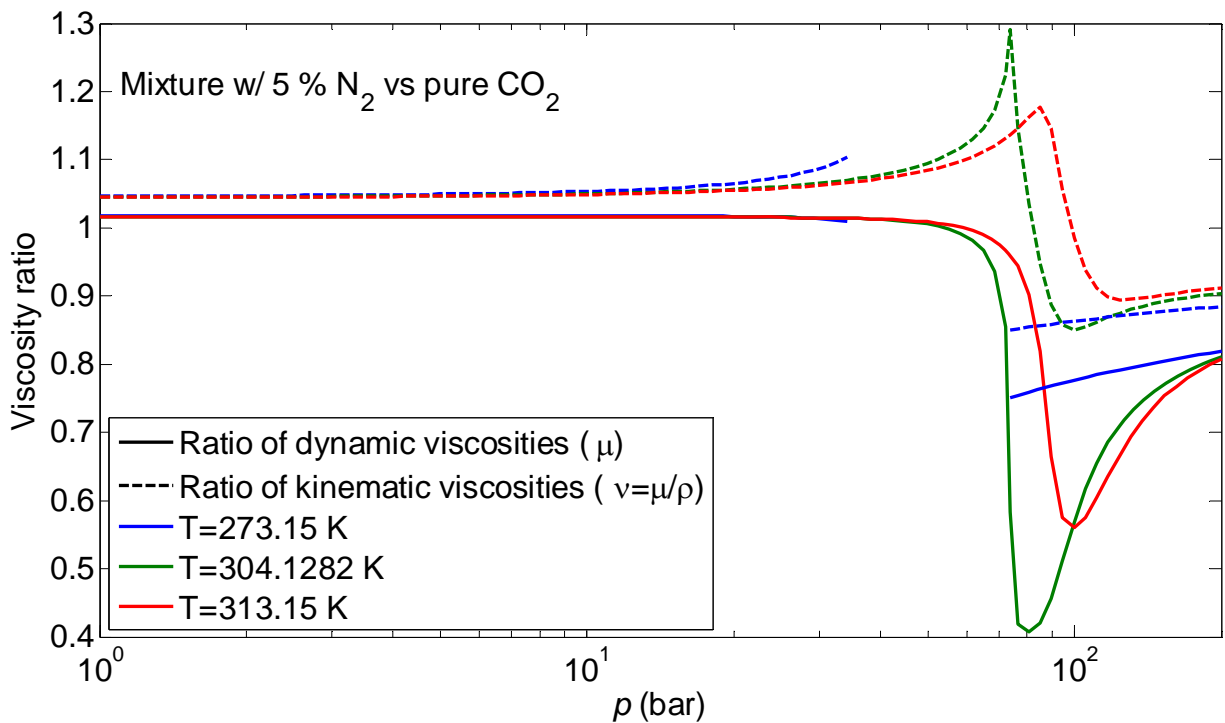


Fig. 3: Ratio between dynamic and kinematic viscosities of CO<sub>2</sub> mixed with 5 mol % N<sub>2</sub> and pure CO<sub>2</sub> as a function of pressure calculated using REFPROP v. 9.1 [19]. Curves of 0 °C are discontinuous since the viscosity is not defined in the two-phase region.

Although molecular modeling has come a long way for simple gases [21] like helium, but in general the accuracy is still questionable mixtures and conditions relevant for CCS. Hence, the best property predictions are currently still

dependent on empirical models. However, recent studies have shown that there are large gaps in the public available data suitable to fit such models [7-10]. For instance, for VLE, only a few systems like CO<sub>2</sub>-methane and CO<sub>2</sub>-N<sub>2</sub>, seem to be fairly well covered, but, as seen in for instance Refs. [9, 22] and Section 3, even here the available data could be so old and inconsistent that further studies are warranted. For transport properties the situation is much worse. For instance, there are no measurements available in the liquid phase except for some data on CO<sub>2</sub> mixtures containing water [5, 10].

On the modeling side, a fairly accurate reference model of state, GERG-2008, has now been established for natural gas [18]. Similar work has started for CO<sub>2</sub>-rich mixtures relevant for CCS [7, 8], but until the data situation is improved, these models do not have the required accuracy, as exemplified in Section 3. In order to tune thermodynamic models, measured data on binary mixtures are normally most useful, while multicomponent mixtures can be measured for model verification.

The effects of impurities discussed above have large impact on the design and economics of CCS systems in general and on transport systems in particular. A number of studies have been made on the cost of CO<sub>2</sub> transport, varying from a few euros to several tens of euros per tonne [23-30], and tools have been developed to estimate the future costs [31-33]. With the estimated annual needs for CO<sub>2</sub> capture numbering 6.3 gigatonne by 2050 in the 2 degree scenario of IEA [34], better estimates for the transportation costs are imperative. As discussed in [6], only 5 mol % of nitrogen could under certain conditions lead to a theoretical increase of the power consumption by a factor of 2.9. Hence, a significant part of the uncertainty in the cost picture is due to the uncertain property situation, and more data are needed in order to be able to make an optimized trade-off between level of purification and transportation costs, a fact which motivated this project.

### 3. Phase Equilibrium Measurements

In the CO<sub>2</sub>Mix project, a new facility, shown in Fig. 1, has been designed for highly accurate measurements of primarily vapor liquid equilibria (VLE). The setup is described in detail in Refs. [22, 35-37]. An analytical principle is employed, where the both the dew and bubble point composition at equilibrium conditions for a given temperature and pressure are measured. Due to the large span in pressures (< 200 bar) and temperatures ( -50 – 150 °C) of interest, and safety considerations relating to the gases to be studied, careful and custom design was required in order to reach the accuracy needed and at the same time maintain the safety and robustness of the setup. The setup has been verified and has so far been used to provide new phase equilibrium data for binary mixtures of CO<sub>2</sub> and N<sub>2</sub>

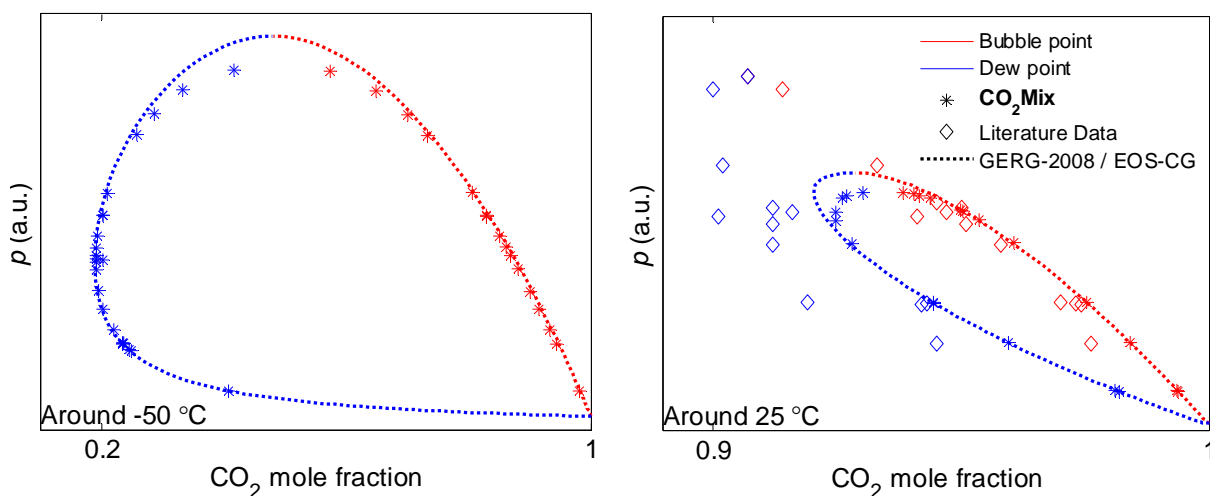


Fig. 4. CO<sub>2</sub>-N<sub>2</sub> phase equilibrium measurements compared with existing models [22, 37]. For this mixture, EOS-CG is identical to GERG-2008 [8, 18].

and CO<sub>2</sub> and O<sub>2</sub> [22, 37, 38]. Accuracy is ensured by using traceable and certified standards for temperature, pressure, and composition.

Two measured isotherms of the CO<sub>2</sub>-N<sub>2</sub> are shown in Fig. 4, detailed data will soon be published [38]. Both around 25 and around -50 °C, models based on existing literature data predict that the two-phase regions extend to significant higher pressure than our data suggest. Note that the two-phase pressure envelope around -50 extends almost to 200 bar, and the envelope around 25 °C covers a pressure range of approximately 20 bar, hence the shifts seen are highly relevant from an operating perspective. For the measurements around 25 °C, nearby literature data is included in Fig. 4, illustrating that even for nominally well covered systems there is a need to improve the data situation, especially for high CO<sub>2</sub>-concentrations / high temperatures and near critical conditions.

In Fig. 5 some measured isotherms of the CO<sub>2</sub>-O<sub>2</sub> binary system are shown [38]. For this system, the data situation is much more scarce than CO<sub>2</sub>-N<sub>2</sub>, most likely due to its lack of relevance for natural gas. Only four works are available on CO<sub>2</sub>-O<sub>2</sub> VLE measurements in the literature with reasonable data consistence, and latest of these sources was published in 1972 [39-42]. As an example, a single isotherm measured in CO<sub>2</sub>Mix is shown with related literature data in Fig. 6, indicating both the scatter of the literature data at this temperature, fidelity of the measurement around the critical point, and lack of model fit. The deviation between the model and the new data is in this case not only around the critical region, but also at lower temperatures. Some of the existing literature data seem to be inconsistent with the very accurate data available for pure CO<sub>2</sub>. The green marker in the plot indicates a supercritical data point, where two phases could not be separated neither visually or through sampling, illustrating the high resolution of the setup.

#### 4. Density and speed of sound measurements

Due to the nature of the CO<sub>2</sub> mixtures, existing methods and designs for the measurement of density and speed of sound had to be revisited, and also for these properties a new facility has been constructed, pictured to the left in Fig. 8. For density measurements, a single sinker setup based on the principles developed by Wagner et al. [43, 44], illustrated in Fig. 7 was employed. The buoyancy of the single sinker is measured using a magnetic coupling to an accurate weight comparator.

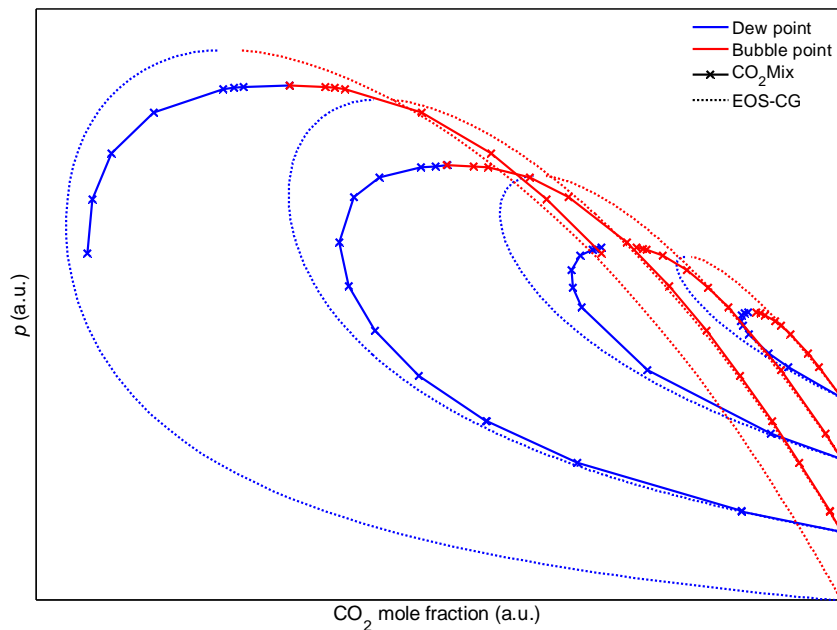


Fig. 5: New CO<sub>2</sub>-O<sub>2</sub> VLE measurements and comparison with the EOS-CG model [8, 38].

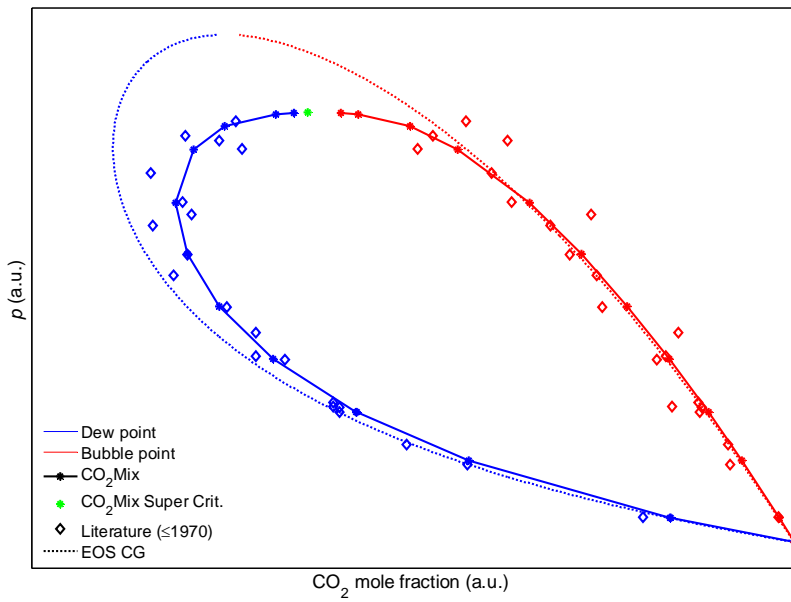


Fig. 6. Measured VLE isotherm of CO<sub>2</sub>-O<sub>2</sub> plotted with existing literature data and best available reference model (EOS-CG) [8, 38].

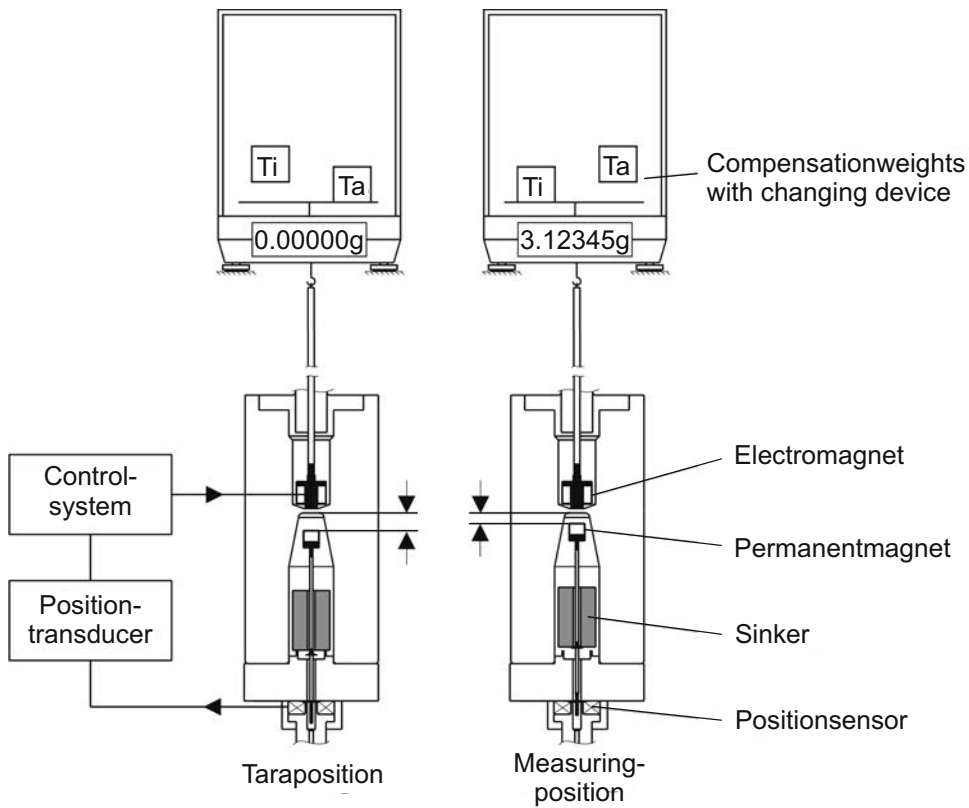


Fig. 7: Single sinker densimeter setup

The densimeter has now been verified and currently produces accurate data for mixtures. The instrument has been compared against a two-sinker setup, which has high accuracy, but has a narrower range, using CO<sub>2</sub>-Ar mixtures. An example is shown to the right in Fig. 8. The measurements of the two instruments have a similar deviation from GERG-2008 and a new reference equation of state developed for CCS fluids, EOS-CG [8].

Originally, the plan was to measure speed of sound of CO<sub>2</sub> mixtures using a pulse-echo method [6, 45]. Unfortunately, due to the high sound absorption and frequency used, the apparatus is not suitable for mixtures with CO<sub>2</sub>. Alternative methods are now pursued [46], but the pulse-echo instrument can be used for secondary mixtures relevant for CCS.

## 5. Conclusions and summary

In order to design and operate systems and processes for CCS, extensive new and accurate measurements of thermophysical properties are needed. For the large amount of CO<sub>2</sub> that should be captured, transported, and stored according to the IEA 2 degree scenario, the current uncertainty in CO<sub>2</sub> mixture properties will translate to huge costs.

In the CO<sub>2</sub>Mix project, the three important properties, VLE, density, and speed of sound are addressed for a selected set of mixtures. From an operational point, measurements should be more accurate than the process data available in order further uncertainty in the process control, and the empirical models themselves can never be more accurate than the data on which they build. Hence, it has been a major goal of the project to provide as accurate data as possible. Due to the properties of CO<sub>2</sub> and typical impurities, design and construction of highly specialized and advanced setups have been required. New and very accurate property data have been and are being produced, of which some examples have been reported here. These data will soon be presented in the reference

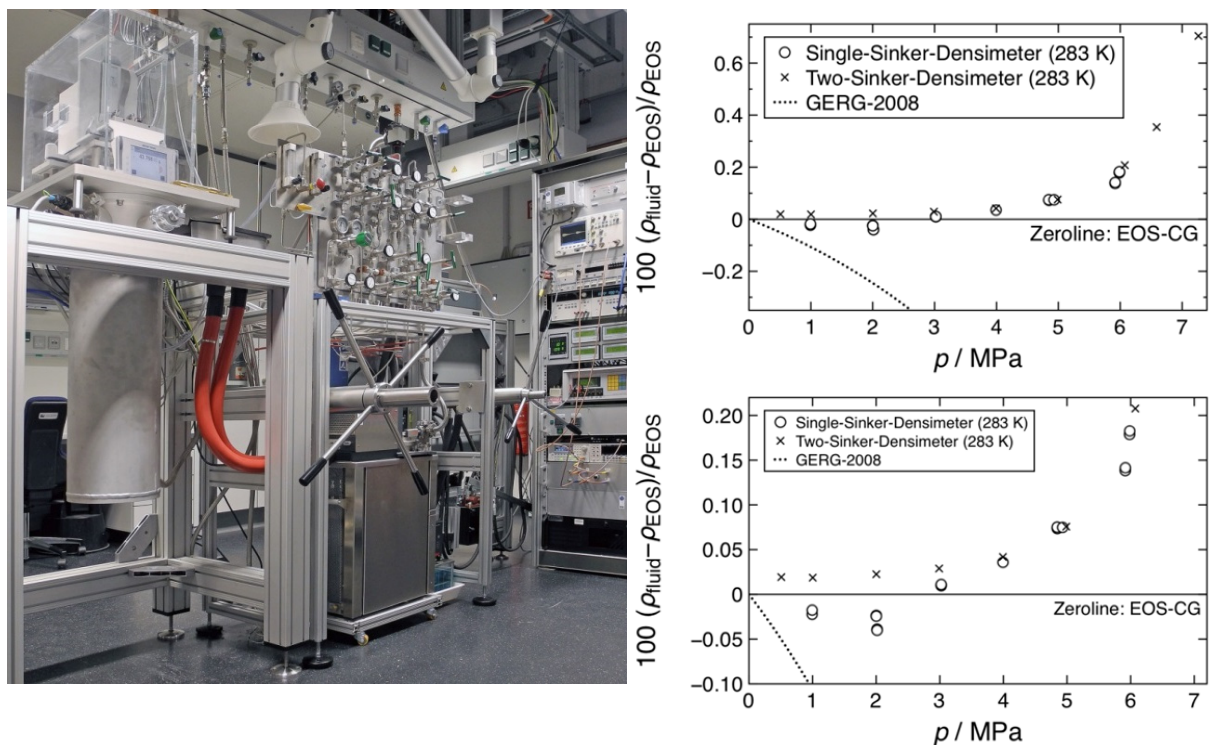


Fig. 8. Left: Density and speed of sound setup at RUB. Right (top): Comparison between single and two-sinker density measurements of a 25/75 of CO<sub>2</sub>-Ar. Right (bottom): Same results shown within a narrower density ratio range.



literature. Especially for phase equilibria measurements, relatively large deviations are found between the new data and established models, especially in the critical regions.

Although the project are closing important knowledge gaps, especially with regards to VLE and density, there should be continued focus on obtaining better data also for systems and conditions that cannot be covered within the CO<sub>2</sub>Mix project. Equally important, there is a large need to cover other properties, for instance transport properties such as viscosity and thermal conductivity and heat capacity. To cover all knowledge gaps, international cooperation is key, and hence the state of the art infrastructure established by SINTEF ER CO<sub>2</sub>Mix project is currently also involved in the IMPACTS project [47] and included in the ECCSEL network [48].

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