METCCUS

REPORT

A4.1.1 - Protocol for the preparation of binary mixture of amine and CO_2

Ethanolamine (MEA), ACS reagent, ≥99.0%. CAS number: 141-43-5.
Reference number: 398136

This report was written as part of activity A4.1.1 of the *Metrology Support for Carbon Capture Utilisation and Storage* (MetCCUS) project. For more details about this project please visit https://metccus.eu/about-the-project/.

This report was written by:

Raffaella Romeo	INRiM	*r.romeo@inrim.it*
Simona Lago	INRiM	*s.lago@inrim.it*
David Vega-Maza	UVa	*david.vega@uva.es*
Alejandro Moreau Ortega	UVa	*alejandro.moreau@uva.es*

Contents

Introduction

With the aim to provide currently unavailable experimental measurements of the thermophysical properties of CO_2 mixtures which will be needed to implement the empirical models developed in Task 4.2, UVa and INRIM will measure the density and the speed of sound respectively of CO_2 + monoethanolamine between 20 °C and 120 °C and for pressures up to 12 MPa at the 3 different concentrations. The accuracy of the sample preparation will be determined in a comparison of the density measurements obtained by the two institutes at (30 and 120) °C at ambient pressure. To this end, a protocol for the preparation of the mixture is necessary.

Preparation of the binary mixture of amine and CO_2

UVa: Preparation using flowing amine and pure CO_2.

Protocol for filling the densimeter:

1. Set the thermostatic baths of the ISCO pumps B and A to 0 °C (CO_2 pump) and 20 – 40 °C (pure amine pump), respectively.

2. Fill ISCO pumps B and A with CO_2 and MEA or DEA, respectively.

3. Keep a constant pressure of 60 bar on both pumps using the constant pressure function.

4. The densimeter must be completely clean and under vacuum before filling. Keep it at a stable temperature of 20 °C.

5. Set the CO_2 and MEA or DEA flows (mL/min) with the pumps using the constant flow function. The flows are calculated according to the load you set by means of a material balance, as per one minute of operation.

Where (mL/min), is the CO_2 loading, standing for amount of substance (moles), for the MEA mole fraction, and for the molar density of CO_2 at temperature and pressure pump condition.

6. Maintain a back pressure of 60 bar on the densimeter using a back pressure regulator.

7. Fill the densimeter avoiding sudden pressure drops first with the amine. When it is completely full start flowing CO_2 at the desire rate.

8. Using the constant flow function, evacuate the mixture using the drain of the back pressure regulator to ensure that the loads are correct. This takes about 10 - 20 minutes.

9. Stop filling. Close the valve that connects the densimeter to the pumps.

10. Check the densimeter vibration period during the reaction time. It will be known that the reaction is finished when the vibration period is stable.

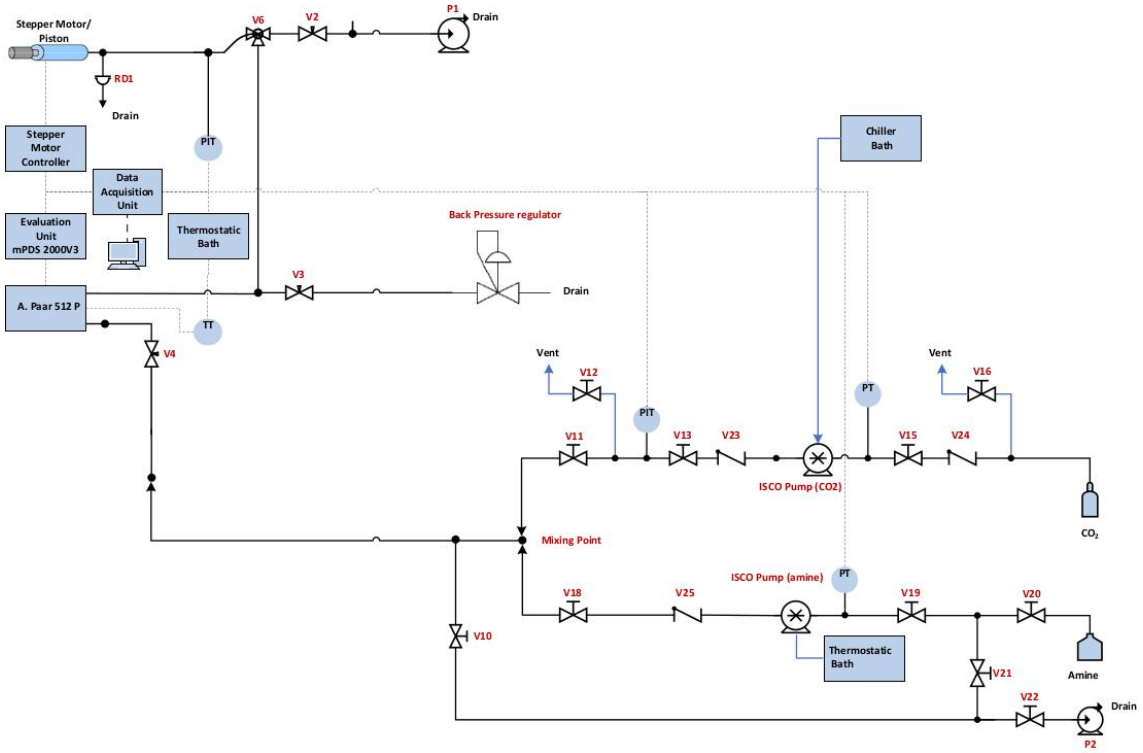

Fig. 1 - Schematic view of the densimeter.

INRIM: Gravimetric preparation.

The mixture with CO2 is prepared using a volumetric piston equipped with a pressure transducer to control the pressure and a valve to isolate the system.

1. Weight the empty piston with the pressure transducer and the valve.
2. Fill the piston with the MEA.
3. Compress the MEA up to reach the same pressure of the CO2 moles needed for the mixture.
4. Connect the piston to the CO2 cylinder.
5. Fill the piston with the amount of CO2 requested for the mixture and previously calculated.
6. Weight the system with the solution to check the mass content.
7. Set the control pressure of the system to ambient pressure.
8. Wait for the mixture stabilization.

Each weighing is performed recording the ambient parameters to determine air density for the buoyancy correction. The balance to be used has a full scale range of 50 kg with a resolution of 10 mg.

Risks

There is a lack of available data from the literature on the equations of state.

Mitigation of the risks

The participants will contact other experts from outside of the consortium to provide feedback on this topic.