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## Phase Behavior of CO<sub>2</sub> in Monoethylene Glycol between 263.15-343.15 K and 0.2-40.3 MPa

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Phase Behavior of CO<sub>2</sub> in Monoethylene Glycol  
between 263.15 – 343.15 K and 0.2– 40.3 MPa – An  
Experimental and Modeling Approach

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## **Abstract**

Solubility data for CO<sub>2</sub> in Monoethylene Glycol (MEG) are limited in the open literature, with most of the data limited to temperatures above 298.15 K through a small number of sources. This work focused on the solubility of CO<sub>2</sub> in pure MEG in a wider range of temperatures and pressures as experimental data in such conditions are extremely limited in the open literature. These results can be used to optimize EoS and increase prediction reliability due to the wider range. The solubility of CO<sub>2</sub> in MEG was measured between 263.15 – 343.15 K and 0.2– 40.3 MPa. The experimental results from this study are compared to available data from the open literature together with the CPA-SRK72 calculations. The data from this work together and open literature were used to calculate a Binary Interaction Parameter (BIP) of 0.053 between 263.15 – 398.15 K to correlate the experimental data. The experimental results showed an overall absolute average deviation of 4.81% from the calculated modelling results.

## **Keywords**

Carbon capture and storage (CCS), Enhanced Oil Recovery (EOR), Gas Hydrate Inhibitor Distribution, Carbon Dioxide, MEG

## Introduction

Global warming has become a key focus for governments and in turn for energy companies due to the evidence pointing to humans as one of the likely culprits. <sup>1</sup> Carbon Capture and Storage (CCS) has been identified as a possible solution to mitigate the effect of anthropogenic CO<sub>2</sub> emissions. It is predicted that CCS will account for 20% of the anthropological carbon emission reduction by 2050. <sup>2</sup> CCS consists of three components: Capture, Transport and Storage. <sup>3</sup>

CCS can also provide a solution for sustainable extraction of high acid gas reservoirs. The global population and economic growth, has been responsible for the ever-increasing energy requirements. As a results energy companies have moved to exploit natural gas reservoirs that were previously considered not economically viable. The oversupply from shale basins resulting in a sharp fall in the market value of hydrocarbon resources has recently reduced this demand. As drilling stalls and many hydraulic fracking companies declare bankruptcy, the surplus will likely decrease resulting in a slow resurgence of demand to accommodate the ever increasing population of the planet. <sup>4</sup> This will likely lead the petroleum industry to continue exploiting the more inaccessible locations.

Carbon dioxide (CO<sub>2</sub>) is usually present in natural gas accounting for less than 1% of the composition; however, some reservoirs such as Bujang, Noring and Inas, contain very high concentrations of CO<sub>2</sub>. Hence it is important to understand the phase behavior of such systems in order to facilitate the economic removal of these components. Natural gas also contains large amounts of water when produced, which can lead to issues such as hydrate formation, as well as the possibility of corrosion.

Majority of researchers believe that CO<sub>2</sub> is one of the major contributors to global warming. There has been a significant number of research work studying the storage of CO<sub>2</sub> in underground

reservoirs as well as in deep <sup>5-8</sup>. In order to ensure the economical and safe transport and storage of high CO<sub>2</sub> streams, the knowledge of thermophysical and phase behavior of the systems are essential. There has been a number of studies in this field assisting design engineers with equipment sizing as well as thermodynamic calculation requirements. <sup>9-13</sup>

MEG is often used at the well head to prevent hydrate formation; It is also occasionally used in glycol dehydration units, when there are resource restriction, to remove water from natural gas streams. <sup>14</sup> CO<sub>2</sub> is partially soluble in MEG, resulting in the reduction of dehydration efficiency; hence it is important to understand the phase behavior of CO<sub>2</sub> in MEG, ensuring sufficient information is available for the economic design and operation of process equipment. <sup>15</sup> MEG is the most popular hydrate inhibitor with operators around the world. It is water soluble and shifts the hydrate phase boundary to the left, resulting in lower hydrate dissociation temperatures and higher pressures, by reducing the water activity. The injection of hydrate inhibitors requires a variety of process equipment resulting in significant Capital Expenditure (CAPEX) as well as well as chemical expenditure overheads. Hence it is important for the operators to be able to accurately calculate their requirements, based on the system compositions, using their thermodynamic models. To reduce the risk of corrosion and prevent hydrate formation, CO<sub>2</sub> being transported must undergo a degree of dehydration, therefore, the knowledge of CO<sub>2</sub>-MEG phase behavior is vital for the economic design and operation of CO<sub>2</sub> and high CO<sub>2</sub> content gas systems. This study focused on the solubility of CO<sub>2</sub> in MEG due to the paucity of the published data, especially at lower temperatures. This wide range of data enabled the authors to further optimize the CPA-SRK72 calculations. To increase the reliability of thermodynamic models, it is essential to optimize the model. An efficient method is using Binary Interaction Parameters (BIPs). BIPs can be calculated using solubility data as a fast, low cost and efficient experimental approach. The

optimized model can then be used to make more reliable phase behavior predictions for multi-component systems. The measurement results from this work can be used to develop BIPs for various thermodynamic models, and can assist operators, consultants and designers in their operations.

Hayduk and Malik measured the solubility of CO<sub>2</sub> in MEG at 298.15 and atmospheric pressure<sup>16</sup>. The solubility of CO<sub>2</sub> in MEG, was measured and published by Jou et al. in a number of papers between 298.15 – 403.15 K and 0.03 – 21 MPa<sup>17-19</sup>. Zheng et al. reported the solubility of CO<sub>2</sub> in MEG at 323.15 – 398.15 K and 0.2 – 39.6 MPa<sup>20</sup>. Galvão et al. published a limited number of CO<sub>2</sub> in MEG solubility measurements in the range of 303.15 – 423.15 K and 0.3 – 6.3 MPa<sup>21</sup>. As illustrated above, the solubility of CO<sub>2</sub> in MEG has been of interest for over 40 years. It is important to note most of the publications mentioned have put great emphasis on elevated temperatures and moderate pressures. To ensure the reliability of thermodynamic model predictions, it is important for the models to be optimized using a wide range of temperatures and pressures. The focus of this study was to make measurements in a broad range of temperature and pressures to assist with thermodynamic model optimization.

## Materials and Method

Table 1 shows the chemical used in this study. The materials suppliers, component purities and analysis methods used to ensure purity have also been included.

Table 1 Details of the component, suppliers and purities of the components used in this study.

Chemical Name	Source	CAS Number	Mole Fraction Purity <sup>a</sup>	Certification	Analysis Method <sup>b</sup>
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MEG	Fisher Chemicals	107-21-1	0.9990	Fisher Chemicals	GC
CO <sub>2</sub>	BOC	124-38-9	0.9999	BOC Certified	GC

<sup>a</sup> No additional purification is carried out for all samples. <sup>b</sup> GC: Gas Chromatography

The experimental rig used in this work has been described in great details by the authors in previous publications.<sup>10,22,23</sup>

The 3D diagram of the high-pressure experimental setup used to complete the CO<sub>2</sub> solubility measurements in equilibrium with MEG at various pressures and isotherms is shown in Figure 1. The evacuated pressure cell was loaded from the top with the MEG. An injection pressure cell was used to bring the experimental cell to the desired pressure and maintain the pressure using CO<sub>2</sub> injection. The pressure cell was then disconnected. The cell was then rocked for one hour and the pressure was logged to ensure the system was at equilibrium. A sample of liquid was flashed at each pressure and the gas volume and mass of the liquid was measured and recorded using a VINCI Technology gas meter. The data was then used to calculate the solubility of CO<sub>2</sub> in MEG using Eq. (1). This process was repeated for all measurements made at various pressures and temperatures.

Equation (1) solubility of CO<sub>2</sub> in MEG.

$$x_i = \frac{[n_{CO_2}^v + n_{CO_2}^l] - n_{MEG}^v}{[n_{MEG}^l + n_{MEG}^v] + [n_{CO_2}^v + n_{CO_2}^l]} \quad (1)$$

The standard uncertainty of the high-pressure rocking cell transducer was  $u(P) = 0.04$  MPa and the standard uncertainty for the PRT temperature probe was  $u(T) = 0.05$  K. The analysis showed these had negligible effect on the overall standard uncertainty of the measurements.

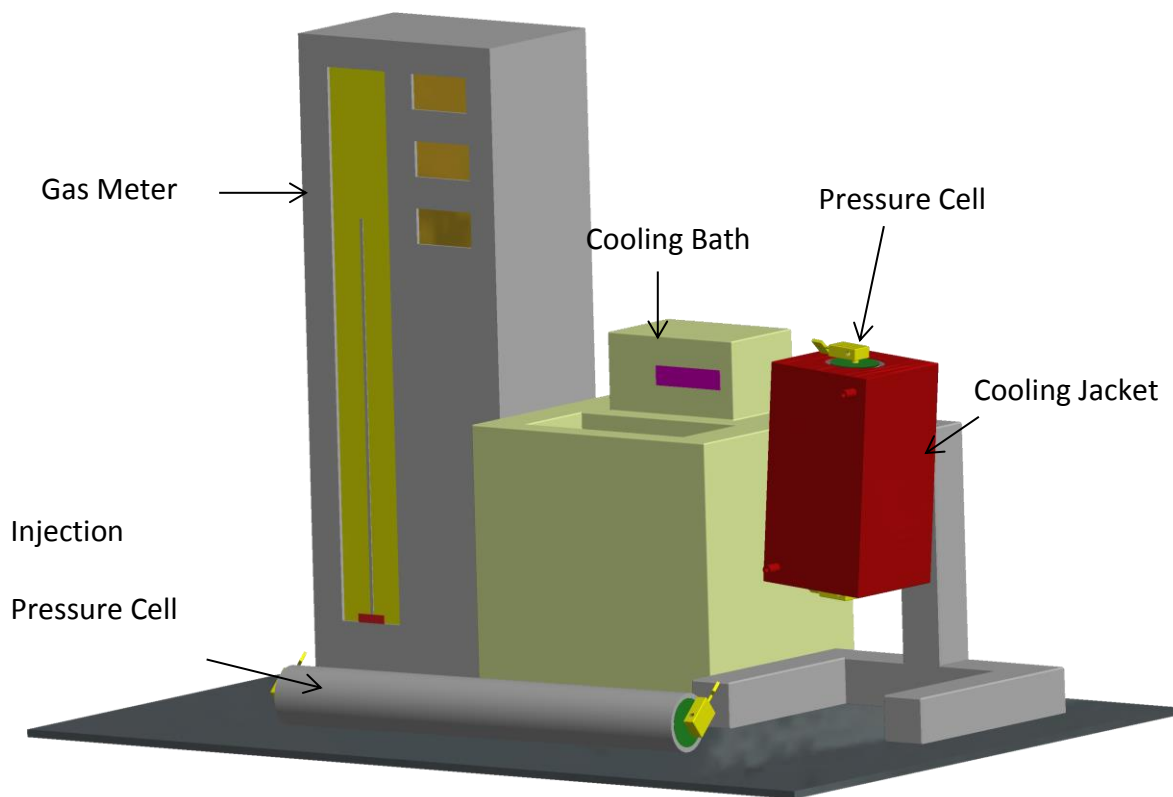


Figure 1. 3D schematic of the experimental setup used in this work.

### **Thermodynamic Modeling**

The thermodynamic methods used within this study have been repeatedly verified and are described in detail in a number of previous publications.<sup>10,22,23</sup> To summarize, the model is a combination of the Soave-Redlich-Kwong (SRK) combined with an association term. Its prediction ability is based on the uniformity of fugacity of each component throughout all the phases. The CPA-SRK72 (Cubic Plus Association) Equation of State (EoS) was used in this work to correlate the experimental results and demonstrate its capabilities.

Equation (2) expresses the CPA-SRK72 in terms of pressure with the sum of the SRK EoS and the contribution association term published by Michelsen and Hendriks<sup>24</sup>:



$$P = \frac{RT}{V_m - b} - \frac{\alpha(T)}{V_m(V_m + b)} - \frac{1}{2} \left( \frac{RT}{V_m} \right) \left( 1 + \frac{1}{V_m} \frac{\partial \ln g}{\partial \left( \frac{1}{V_m} \right)} \right) \sum_i x_i \sum_{A_i} (1 - X_{A_i}) \quad (2)$$

Where  $V_m$  is the molar volume,  $X_{A_i}$  is the fraction of A-sites of molecular  $i$  that are not bonded with other active sites and  $x_i$  is the mole fraction of the component  $i$ .

CPA parameters for MEG were taken from Derawi et al. <sup>25</sup>, and are reported in Table 2. The critical properties of carbon dioxide published by Poling et al <sup>26</sup> were used and shown in Table 3.

Table 2. CPA Pure Compound Parameters for MEG

	$a_0$ (bar L <sup>2</sup> mol <sup>-2</sup> )	$b$ (L/mol)	$c_1$	$\varepsilon$ (bar L mol <sup>-1</sup> )	$\beta$ (10 <sup>3</sup> )	Reference
MEG	10.819	0.05140	0.6744	197.52	14.1	Derawi et al. <sup>25</sup>

Table 3. Critical Properties of Carbon Dioxide <sup>26</sup>

$T_c$ / K	P / MPa	$\omega$ , acentric factor
304.12	7.374	0.225

The BIPs between CO<sub>2</sub> and MEG were adjusted using the solubility data reported by Jou et al. <sup>17</sup> and the new measured data through a Simplex algorithm using the Objective Function,  $OF$ , shown in Equation (3).

Equation (3) Objective Function used to tune the BIPs between CO<sub>2</sub> and MEG

$$OF = \frac{1}{N} \sum_1^N \left| \frac{x_{\text{exp}} - x_{\text{cal}}}{x_{\text{exp}}} \right| \quad (3)$$

Where  $x$  is the solubility of CO<sub>2</sub> in MEG,  $N$  is the number of data points.

The MEG – CO<sub>2</sub> BIP values, was calculated to be 0.053 between 263.15 – 398.15 K from the measurements in this work together with data from Jou et al.<sup>18</sup> and Zheng et al.<sup>20</sup>. It is important to note that the BIP was calculated for this range by correlating the experimental results and its reliability has only been tested within this range.

## Results and Discussion

Table 4 shows the solubility of CO<sub>2</sub> in MEG at 7 different isotherms and various pressures where  $T$  is temperature in Kelvin,  $P$  is the pressure in MPa,  $x_1$  is the moles of CO<sub>2</sub> in the aqueous phase and  $u_r(x_1)$  is the relative standard uncertainty in moles. The phase behavior of these measurements showed a relative standard uncertainty of  $u_r(x_1) = 0.026$ . In order to calculate the standard uncertainty of the measurements the four main measurement variables combined with the repeatability analysis study published by the authors was considered and implemented in the calculations.<sup>22</sup> The variables considered were the volume of CO<sub>2</sub> measured using the gas meter, the mass of MEG, the standard uncertainty perpetrated by quantity of MEG in the atmospheric vapor phase and the mole fraction of CO<sub>2</sub> in the liquid phase using the CPA-SRK72 EoS. NIST reported the standard uncertainty of the CO<sub>2</sub> density data as 0.003%. It was determined that this had negligible effect on the overall standard uncertainty of the measurements. The apparatus standard uncertainties reported by the manufacturers were then used to calculate the standard uncertainty of each measurement. Figure 2 illustrates the solubility of CO<sub>2</sub> in MEG together with CPA-SRK72 model predictions for each isotherm. It is possible to determine the phase change in the system using the inflection point observed at each temperature. A liquid CO<sub>2</sub> phase cannot be

seen in the results at 323.15 and 343.15 K. The inflection point shows the CO<sub>2</sub> change of phase from gas, Vapor–Liquid–Equilibria (VLE) to liquid, Liquid-Liquid-Equilibria (LLE), at the specific pressure and temperature. The experimental results were used to determine the phase change point using the ‘break point’ method by fitting two linear equations to the experimental data and using simultaneous substitution to calculate the point of intersection. The intersection is used to estimate the pressure at which a secondary liquid phase becomes present as CO<sub>2</sub> phase moves towards its bubble point. It is important to note that the number of samples are mainly only sufficient to determine the solubility isotherm accurately and was only used to estimate the phase change pressures to help the readers.

Table 4. Experimental solubility of CO<sub>2</sub> (mole fraction) in MEG ( $x_1$ ) at five isotherms, T and various pressures, P<sup>a</sup>.

T/K	P/ MPa	$x_1$	Phase
263.15	0.16	0.0069	VLE
263.15	0.69	0.0310	VLE
263.15	0.83	0.0359	VLE
263.15	2.69	0.1301	LLE
263.15	3.71	0.1251	LLE
263.15	8.23	0.1308	LLE
263.15	12.27	0.1354	LLE
263.15	24.99	0.1435	LLE
273.15	0.48	0.0165	VLE
273.15	1.04	0.0369	VLE
273.15	1.74	0.0609	VLE
273.15	3.54	0.1199	VLE

273.15	7.30	0.1253	LLE
273.15	11.98	0.1303	LLE
273.15	17.73	0.1337	LLE
273.15	25.92	0.1350	LLE
283.15	0.59	0.0172	VLE
283.15	1.61	0.0474	VLE
283.15	3.52	0.0981	VLE
283.15	4.52	0.1226	VLE
283.15	16.57	0.1327	LLE
283.15	20.33	0.1339	LLE
297.75	0.49	0.0109	VLE
297.75	1.01	0.0193	VLE
297.75	6.46	0.1135	VLE
297.75	10.43	0.1202	LLE
297.75	17.84	0.1267	LLE
297.75	21.07	0.1308	LLE
298.15	0.85	0.0157	VLE
298.15	0.85	0.0175	VLE
298.15	1.85	0.0378	VLE
298.15	1.88	0.0393	VLE
298.15	0.85	0.0157	VLE
298.15	0.85	0.0175	VLE
298.15	1.85	0.0378	VLE
298.15	1.88	0.0393	VLE
298.15	2.54	0.0512	VLE
298.15	3.02	0.0578	VLE

298.15	3.85	0.0768	VLE
298.15	3.92	0.0793	VLE
298.15	5.41	0.1045	VLE
298.15	6.38	0.1175	VLE
298.15	6.45	0.1180	LLE
298.15	6.84	0.1213	LLE
298.15	7.25	0.1176	LLE
298.15	7.41	0.1160	LLE
298.15	8.09	0.1181	LLE
298.15	10.69	0.1196	LLE
298.15	11.20	0.1253	LLE
298.15	13.27	0.1256	LLE
298.15	13.27	0.1256	LLE
298.15	14.73	0.1261	LLE
298.15	14.80	0.1253	VLE
298.15	19.08	0.1313	VLE
298.15	19.39	0.1319	VLE
298.15	23.57	0.1332	VLE
323.15	0.43	0.0070	VLE
323.15	0.45	0.0069	VLE
323.15	1.50	0.0277	VLE
323.15	1.53	0.0229	VLE
323.15	2.54	0.0353	VLE
323.15	3.73	0.0475	VLE
323.15	3.74	0.0513	VLE
323.15	10.34	0.1011	VLE

323.15	10.40	0.0985	VLE
323.15	15.23	0.1150	VLE
323.15	15.28	0.1164	VLE
323.15	23.44	0.1281	VLE
323.15	23.57	0.1289	VLE
343.15	0.53	0.0052	VLE
343.15	1.36	0.0143	VLE
343.15	1.91	0.0173	VLE
343.15	3.59	0.0367	VLE
343.15	7.03	0.0702	VLE
343.15	8.14	0.0780	VLE
343.15	12.49	0.1050	VLE
343.15	18.14	0.1256	VLE
343.15	26.75	0.1406	VLE
343.15	36.625	0.1461	VLE
343.15	40.314	0.1485	VLE

<sup>a</sup> Standard uncertainties  $u$  are at  $u_r(x_1) = 0.026$ ,  $u(T) = 0.05$  K and  $u(P) = 0.04$ MPa

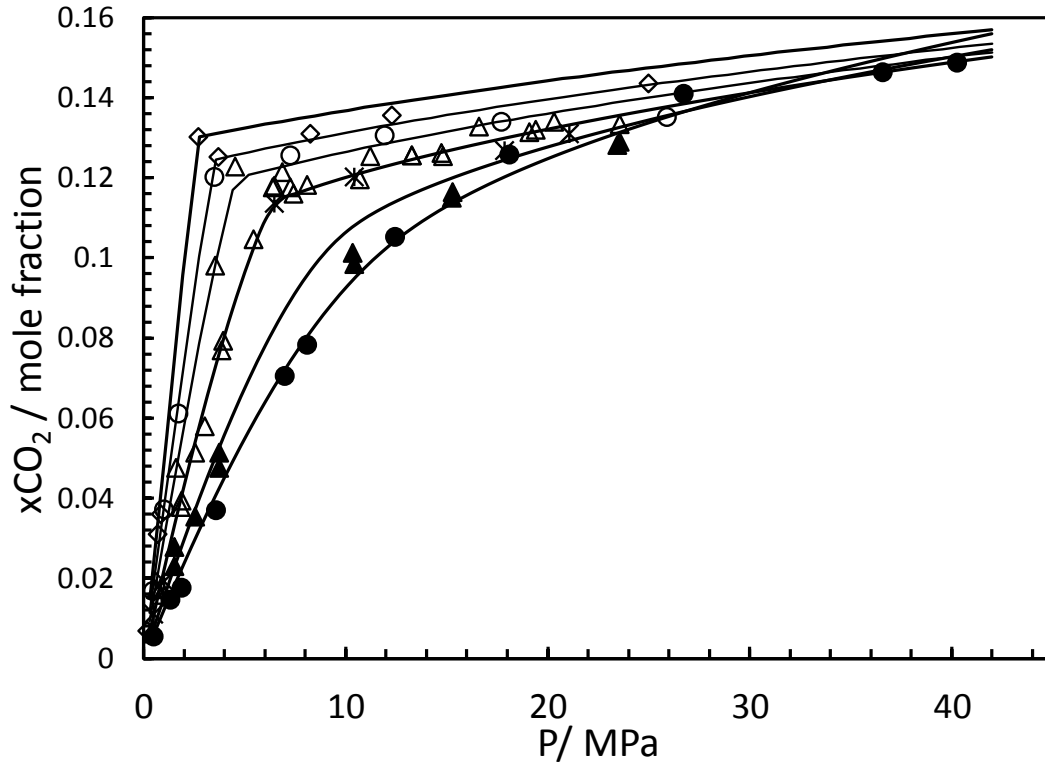


Figure 2. CO<sub>2</sub> solubility in pure MEG at 263.15 (◇), 273.15 K (○), 283.15 K (□), 297.75 K (\*), 298.15 K (△), 323.15 K (▲) and 343.15 K (●). Black Lines: CPA-SRK72-model  $k_{ij} = 0.053$ .

A cross tendency can be observed in the solubility of CO<sub>2</sub> in MEG, where solubility increases with temperature as opposed to decreasing. The sudo-critical effect is seen when the system is within the vapor-liquid-liquid region.

To ensure the reliability of the experimental equipment a number of measurements were carried out using the same temperatures as literature data. Figure 3 shows the solubility of CO<sub>2</sub> in MEG from this work and CPA-SRK72 model calculations together with data from Jou et al.<sup>18</sup> at 298.15 K. Using cubic spline interpolation trend equations were calculated from the Jou et al.<sup>18</sup> data. Using the trend equations, the expected solubility at the same point as this work was calculated. Comparing the solubility data showed an overall absolute average relative deviation of 5.43% between calculated Jou et al.<sup>18</sup> data and this work in the reported range. Figure 4 shows the

solubility of CO<sub>2</sub> in MEG at 323.15 K measured in this work and CPA-SRK72 correlations together with data from Jou et al.<sup>18</sup>, Galvao et al.<sup>21</sup> and Zheng et al.<sup>20</sup>. Using cubic spline interpolation, the data from this work and the data from Zheng et al.<sup>20</sup> demonstrated an overall absolute average relative deviation of 5.03 %. Figure 5 shows the solubility of CO<sub>2</sub> in MEG at 343.15 K measured in this work and CPA-SRK72 predictions together with data from Jou et al.<sup>18</sup>. The data from Jou et al.<sup>18</sup> showed a 4.28% relative overall average deviation from this work, within the reported range, calculated using cubic spline interpolation. The experimental results showed an overall absolute average deviation of 4.81% from the calculated modelling results.

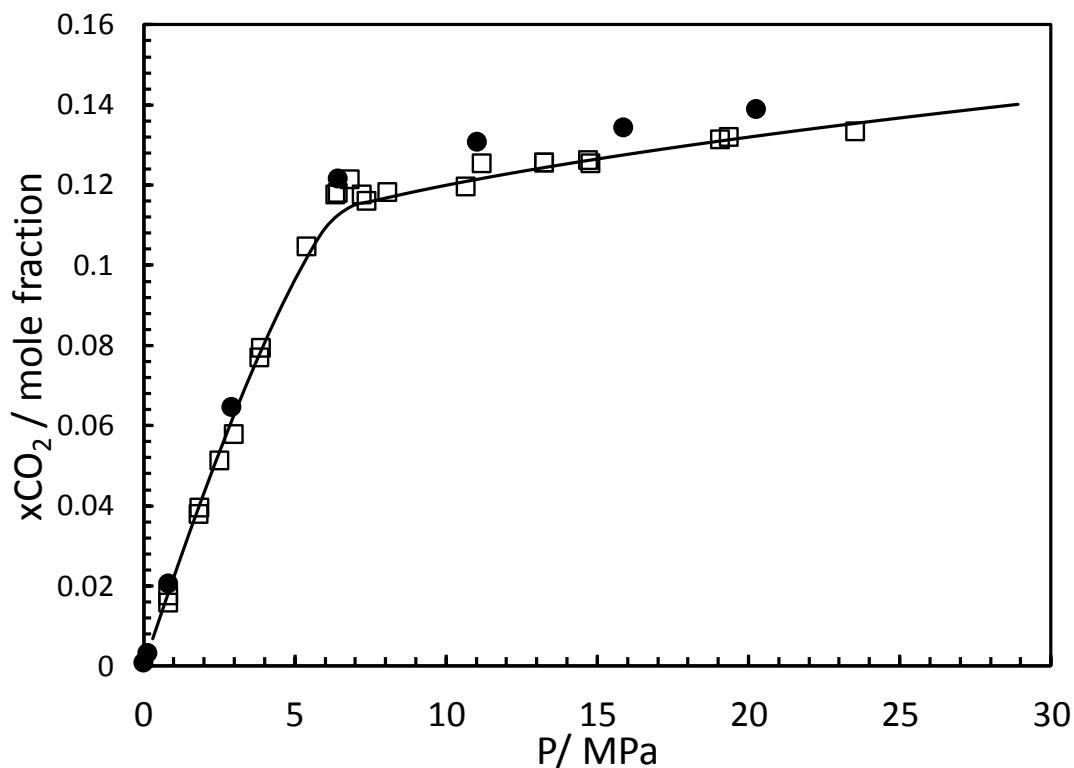


Figure 3. Solubility of CO<sub>2</sub> in Pure MEG at 298.15 K from this work (□) together with CPA-SRK72 model predictions (black line) and the data from Jou et al. (●)<sup>18</sup>.



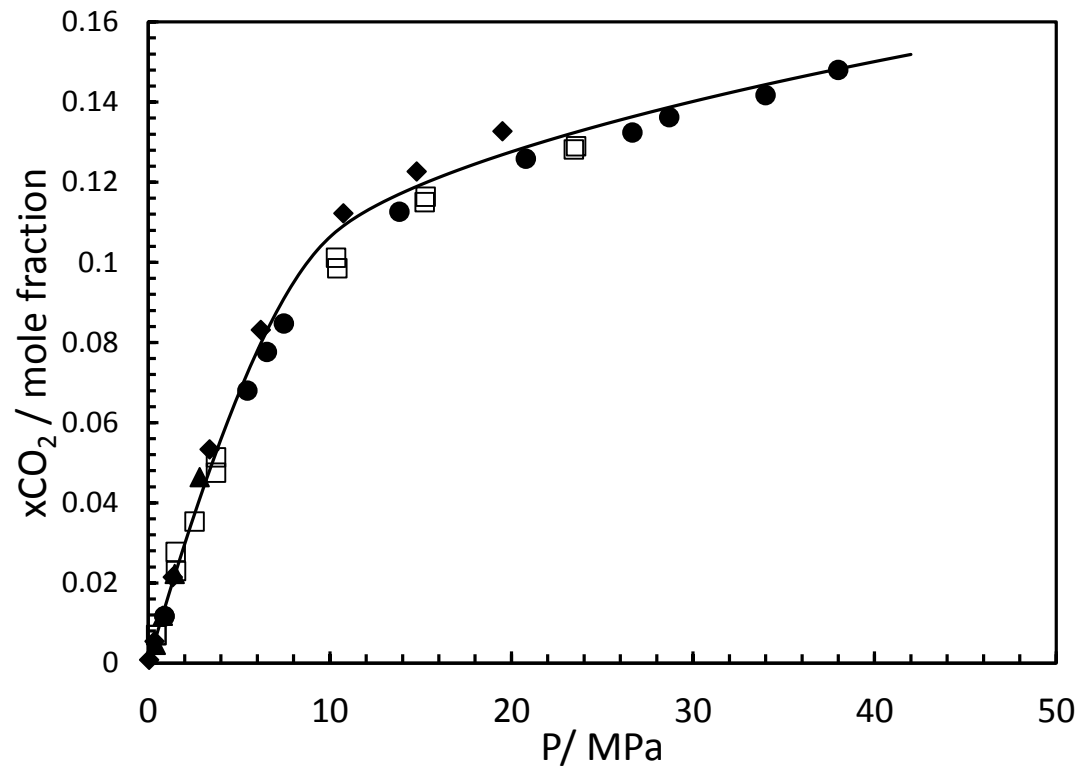


Figure 4. Shows the solubility of CO<sub>2</sub> in pure MEG from this work at 323.15 K (□). CPA-SRK72 model (black line), Zheng et al. (●)<sup>20</sup>, Jou et al. (◆)<sup>18</sup> and Galvao et al. (▲)<sup>21</sup>.

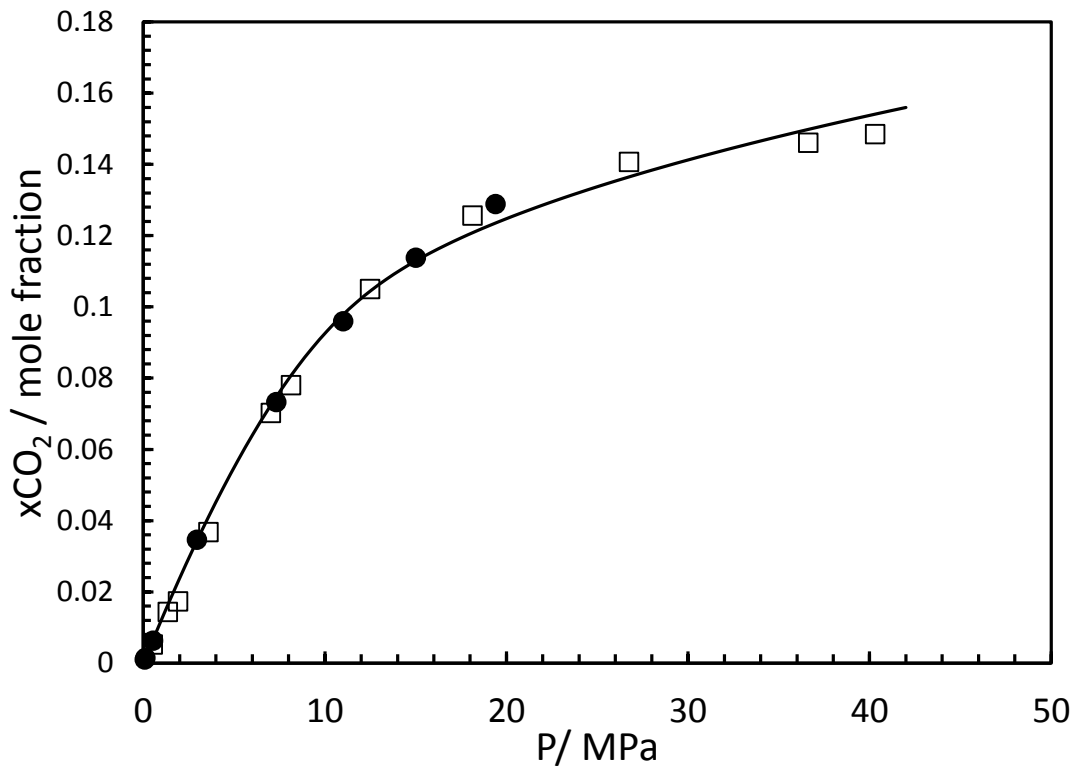


Figure 5. Solubility of CO<sub>2</sub> in pure MEG from this work at 343.15 K (□). CPA-SRK72 model (black line) and Jou et al. (●)<sup>18</sup>.

## Conclusion

After concluding a thorough literature survey, a clear gap in the CO<sub>2</sub> solubility data in MEG became apparent. The available data focused on temperatures above 298.15 K, which significantly reduces the reliability of model predictions for lower temperatures, if the data had been used for tuning the models. Furthermore, the limitation of published data significantly reduces the reliability of thermodynamic models optimized using the data due to the narrow number of sources. The focus of this study was to increase the reliability of the CPA-SRK72 predictions as well as provide solubility data required to calculate BIPs in various models. Hence the solubility of CO<sub>2</sub> was measured at 6 different isotherms and a wide range of pressures.

The CPA-SRK72 correlations closely agreed with the measurements in this study, showing an absolute average deviation of 5.13%. The measurements at 298.15 K were in good agreement with the work of Jou et al.<sup>18</sup> demonstrating a relative absolute average deviation of 5.4% from this work within the range published. The solubility measurements for CO<sub>2</sub> in MEG at 323.15 K were compared with the work of Jou et al.<sup>18</sup>, Zheng et al.<sup>20</sup> and Galvao et al.<sup>21</sup>. This work showed an overall absolute average deviation of 5.0% from Zheng et al.<sup>20</sup> data. The CO<sub>2</sub> in MEG solubility measurements at 343.15 demonstrated an absolute average deviation of 4.3% over the range. The measurements in this work showed an overall absolute average deviation of 4.81% from the calculated modelling results using the new BIP. It is important to note, it is highly recommended to independently verify the modelling results to ensure reliability.

## AUTHOR INFORMATION

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"Supporting Information Available: This shows the detailed material balance formulation used to determine the solubility of CO<sub>2</sub> in MEG as well as the formulations and assumptions used to determine the uncertainty of the measurements.

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