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3 Evaluation of a phase change solvent for CO<sub>2</sub> capture:  
4 Absorption and desorption tests

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8 **Abstract**

9 A blend of a tertiary amine (DEEA) and a diamine (MAPA) was studied in  
10 a screening apparatus for preliminary absorption tests. Two immiscible liquid  
11 phases were formed upon CO<sub>2</sub> loading and the system was shown to have large  
12 capacity for CO<sub>2</sub>. The two phases were analyzed individually for both amines  
13 and CO<sub>2</sub>. MAPA and water were found concentrated in the heavy CO<sub>2</sub> rich  
14 phase whereas the CO<sub>2</sub> lean phase contained mainly of DEEA. Volumetric phase  
15 ratio was measured as function of CO<sub>2</sub> loading and together with the individual  
16 phase compositions this forms part of a basis for an equilibrium model. The  
17 CO<sub>2</sub> rich phase was heated to desorption temperatures and shown to regenerate  
18 CO<sub>2</sub> at higher pressures than normally used for 30 wt.% MEA. The data enabled  
19 understanding the system behavior as MAPA is first loaded in the heavy phase  
20 and subsequently DEEA reacts with CO<sub>2</sub> and dissolves. The data show that  
21 the system has potential for significant reduction in regeneration heat through  
22 high cyclic capacity, very high CO<sub>2</sub> stripping pressures, and for operating where  
23 the heat of reaction lies between primary and tertiary amines. The tests also  
24 give good estimates for the absorption rate at higher loadings.

25 *Keywords:* Screening, phase change solvent, DEEA, MAPA, CO<sub>2</sub> Capture

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## 26 1. Introduction

27 Reduction of carbon dioxide emissions is still a very important topic and the  
28 research on reducing cost and energy demand for its capture has increased over  
29 the past years. Using energy efficiently, reducing energy waste, changing fuel  
30 sources to alternative sources poorer in carbon content and carbon capture and  
31 storage (CCS) are some ways of reducing the CO<sub>2</sub> emissions. Among the CCS  
32 technologies, chemical absorption using aqueous amine solutions with thermal  
33 regeneration of the solvent is the most developed and applied technology for CO<sub>2</sub>  
34 capture (Svendsen et al., 2011). The 30 mass % monoethanolamine (MEA)  
35 system is the benchmark solvent for this technology (Aroonwilas & Veawab,  
36 2009; Rey et al., 2013) and research on this solvent is still progressing (see, for  
37 example, Razi et al., 2013; Vevelstad et al., 2013).

38 Most of the energy required for CO<sub>2</sub> capture in amine scrubbing systems is  
39 used for regenerating the solvent (Aroonwilas & Veawab, 2007). Process modifi-  
40 cations are used in an attempt to reduce this demand. Several process modifica-  
41 tions were studied and proposed in the literature (Cousins et al., 2011a,b; Karimi  
42 et al., 2011; Oyenekan & Rochelle, 2007; Rochelle et al., 2011). Nevertheless,  
43 the development of new solvents or solvent blends is an important way of reduc-  
44 ing the energy demand in amine scrubbing plants. Apart from MEA, piperazine  
45 (PZ) and piperazine blends, AMP(2-amino-2-methyl-1-propanol) based systems,  
46 for instance with PZ, and amino acid based systems have been intensively stud-  
47 ied (Jockenhvel & Schneider, 2011; Kuettel et al., 2013). Dugas & Rochelle  
48 (2009), for instance, showed that the 8 m piperazine solution has 75% higher  
49 capacity than a 7 m MEA solution. Also, CO<sub>2</sub> reaction rates are 2-3 times faster  
50 on PZ solutions. Other examples are large scale tests with commercial solvents  
51 which are claimed to be better than aqueous solutions of MEA, as the Cansolv  
52 solvent and KS-1 from MHI, (see Endo et al., 2011; Just, 2013; Kamijo et al.,  
53 2013; Shaw, 2009).

54 A new breed of systems, the phase change solvents, has received much at-  
55 tention during the last 4-5 years. Precipitating systems with amino acid salts

56 (Ma'mun & Kim, 2013; Sanchez-Fernandez et al., 2013) and carbonated solu-  
57 tions (Moene et al., 2013) are claimed to be promising systems for CO<sub>2</sub> capture.  
58 IFPEN recently proposed the DMX<sup>TM</sup> process in which the solvent forms two  
59 immiscible liquid phases upon CO<sub>2</sub> loading (Aleixo et al., 2011; Raynal et al.,  
60 2011a,b). This process is able to operate with energy demands as low as 2.1  
61 GJ/ton of CO<sub>2</sub>. Other amine based solvents that form two phases before or  
62 after CO<sub>2</sub> loading were recently patented (Svendsen & Trollebø, 2013).

63 The development of a new solvent is not simple. A large set of experiments  
64 must be performed in order to characterize the system. Screening experiments  
65 is a fast way to identify potential solvents for CO<sub>2</sub> capture, e.g. Ma'mun et al.  
66 (2007) used a screening apparatus to evaluate the absorption rate of different  
67 amine based solvents for CO<sub>2</sub> capture. Aronu et al. (2009) modified the same  
68 apparatus to allow for desorption operation testing. The authors presented  
69 a comparison of absorption and stripping performance for some amine/amine  
70 blend solutions. Aronu et al. (2010) and Aronu et al. (2011) used the screen-  
71 ing apparatus to evaluate amino acid salts and amine/amine blend solvents,  
72 respectively.

73 In this work, a blend of 5M DEEA (Diethylethanolamine) and 2M MAPA  
74 (N-Methyl-1,3-diaminopropane) was tested in the screening apparatus. This  
75 mixture, as in the DMX<sup>TM</sup> solvent, forms two liquid phases upon CO<sub>2</sub> loading.  
76 By separating out the CO<sub>2</sub> rich phase, it is possible to send a smaller volume  
77 of solvent for regeneration, thereby, potentially reducing the process energy  
78 demand. Also, the CO<sub>2</sub>-rich phase can have a potential for easy stripping,  
79 thereby also reducing the stripping steam demand and possibly increasing the  
80 regeneration pressure. The amines here studied are potential solvents for CO<sub>2</sub>  
81 capture, and were also studied by other authors (Hartono et al., 2013; Monteiro  
82 et al., 2013b,a; Pinto et al., 2014; Voice et al., 2013). The screening apparatus  
83 used in Aronu et al. (2010) was used for absorption tests at different CO<sub>2</sub>  
84 partial pressures and temperatures. The system was characterized by individual  
85 analyses of the two liquid phases at equilibrium, and the phase ratio recorded.  
86 The CO<sub>2</sub>-rich phase formed upon CO<sub>2</sub> loading was separated after absorption

87 and used for desorption tests in a separate apparatus which is also shown in this  
88 work.

## 89 2. The phase change solvent

90 As in the DMX<sup>TM</sup> process (Raynal et al., 2011a), the system studied here  
91 forms two phases upon CO<sub>2</sub> loading. The chemicals used on the DMX<sup>TM</sup> process  
92 are not given anywhere. In this work, an aqueous solution of a tertiary alka-  
93 nolamine (DEEA) and a diamine (MAPA) with a primary and a secondary  
94 amine group was studied. In Fig. 1 the chemical structure of the amines used  
95 in the solution is shown.

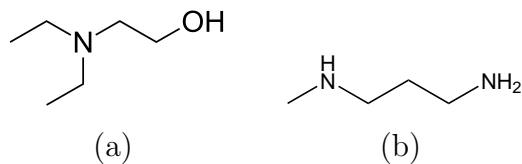


Figure 1: Chemical structure: (a) DEEA; (a) MAPA.

96 The system has an advantage of combining high absorption rate, provided  
97 by the diamine, and high capacity from the tertiary amine. Puxty et al. (2009)  
98 showed that DEEA has a significant absorption capacity while MAPA has a high  
99 initial absorption rate. Moreover, the energy required for regenerating a tertiary  
100 amine is lower than for primary and secondary amines (Kim & Svendsen, 2011).  
101 It is therefore a potential for lowering the total energy consumption of the overall  
102 process significantly.

103 A solution of 5M DEEA and 2M MAPA is a single phase solution. When the  
104 solution starts to absorb CO<sub>2</sub>, at some point, it becomes turbid which indicates  
105 a phase changing behaviour. After leaving the loaded solution to rest two clear  
106 liquid phases can be observed. This behaviour is shown in Fig. 2.

### 107 2.1. The phase change solvent capture process

108 The phase change solvent uses the advantage of the phase split to reduce  
109 the energy demand of the CO<sub>2</sub> capture process. The changes in the process

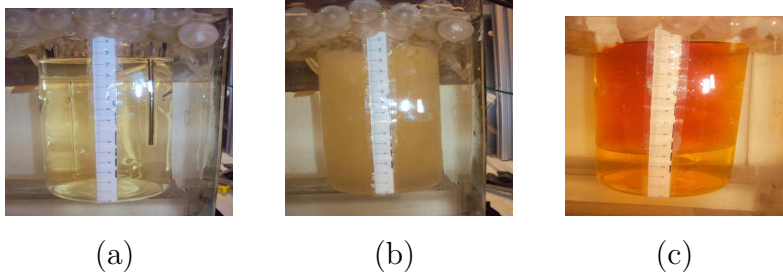


Figure 2: A 5M DEEA/2M MAPA solution: (a) Before, (b) During and (c) after CO<sub>2</sub> loading.

110 configuration, as shown in Fig. 3, are made not only to reduce the energy  
 111 demand, but also as a result of the characteristics of the system. The process  
 112 flow diagram presented in this work is very similar to the one presented in  
 113 Raynal et al. (2011b). However, the phase separator is placed before the cross  
 114 heat exchanger.

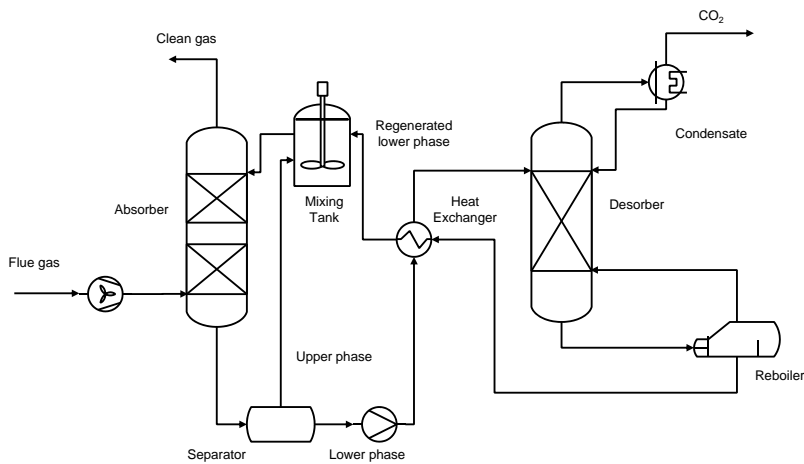


Figure 3: Capture process layout for the phase change solvent

### 115 3. Experiments

116 The apparatus' descriptions and their operational details are given in this  
 117 section.

118 *3.1. Screening apparatus*

119 The screening apparatus (Fig. 4) is designed to operate up to 80°C and at  
120 atmospheric conditions for rapid evaluation of absorption and stripping perfor-  
121 mance of solvents. As discussed in Aronu et al. (2009) and Ma'mun et al. (2007),  
122 the results from the screening apparatus are semi quantitative and should be  
123 understood only as an indication of a solvent's performance. The apparatus  
124 used in this work is the same as the one used in Aronu et al. (2010) with minor  
125 modifications. The reactor was changed to a glass volume graded vessel where  
126 it was possible to see the phase change formation and the CO<sub>2</sub> analyzer was  
127 also replaced by a Rosemount BINOS 100 IR CO<sub>2</sub>-analyzer.

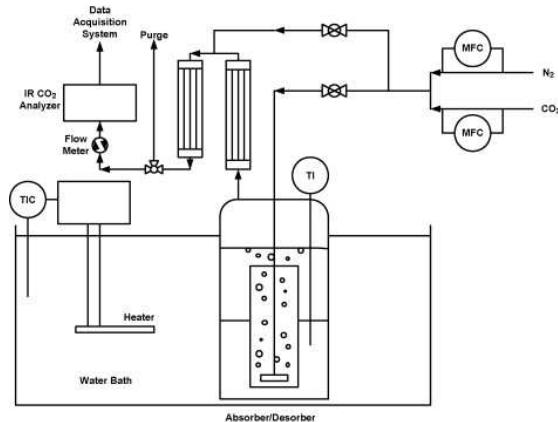


Figure 4: Simplified diagram of the screening apparatus (Aronu et al., 2010).

128 A known volume and mass (around 750 ml) of the solvent was weighed into  
129 the reactor and a synthetic mixture of CO<sub>2</sub> and N<sub>2</sub>, with a total flow of 5  
130 Nl/min, was bubbled into the solvent. The CO<sub>2</sub> concentration (flow) in the  
131 inlet gas was adjusted to the desirable value by adjusting the mass flow of N<sub>2</sub>  
132 and CO<sub>2</sub> while keeping the total flow at 5 Nl/min. After bubbling through the  
133 solution, the gas stream was cooled on-line through two condensers placed on  
134 top of each other and the condensate was directly returned to the reactor. The  
135 dried gas was sent to the IR analyzer for CO<sub>2</sub> analysis.

136 The absorption tests were performed at 40, 60 and 80°C and from 1 to

137 20 kPa of CO<sub>2</sub> partial pressure in the inlet gas. An experiment was stopped  
138 when 95% of the concentration of the inlet gas was achieved in the outlet of the  
139 reactor. The mixture was then left to rest so that the phases could separate  
140 at the experimental temperature. The volumes of the lower and upper phases  
141 were recorded and sampled individually at the experimental temperature, after  
142 which they were separated and stored in different bottles for further analysis.

### 143 *3.2. High pressure desorption apparatus*

144 Stripping experiments were done on the lower phase (CO<sub>2</sub> rich phase) gener-  
145 ated in the screening apparatus. A different apparatus was used for this purpose.  
146 The apparatus consisted of a 150 ml stainless steel vessel immersed in an oil  
147 bath where the temperature was held constant by a Julabo 6 heating system. A  
148 thermocouple was placed inside the vessel and in contact with the liquid (Fig.  
149 5). The temperature of the liquid as well as the pressure of the system were  
150 read and recorded through a program coded in LabView software. The cylinder  
151 was evacuated to about 20 mbar and around 80 ml of a lower phase sample was  
152 sucked in. The oil bath was set to a desired temperature, and once the tempera-  
153 ture and pressure were stable, a point was recorded. Temperature and pressure  
154 were considered stable if in a 10 minutes window no variations occurred above a  
155 given limit ( $\pm 0.1^\circ\text{C}$  and  $\pm 1$  mbar). The temperature was then increased and  
156 the procedure was repeated until the pressure was close to 6 bar. This was due  
157 to the pressure transducer having an upper limit of 6 bar.

158 A total of eight samples from the screening tests were tested for high pressure  
159 desorption. Table 1 shows the samples selected for the high pressure desorption  
160 tests.

## 161 **4. Results**

### 162 *4.1. Screening calculations*

163 A Labview data acquisition software was used to record the measured vari-  
164 ables (temperatures, flows and CO<sub>2</sub> content in the outlet gas) every minute from

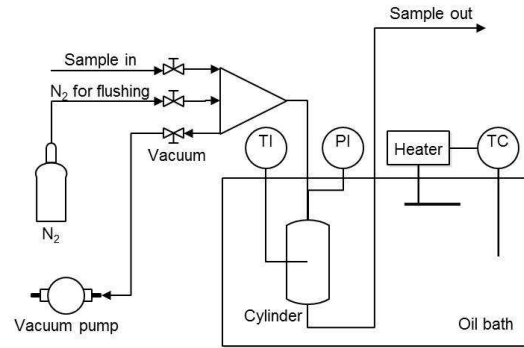


Figure 5: Simplified diagram of the high pressure desorption apparatus.

Table 1: Screening samples taken for high pressure desorption tests

Sample	Absorption Temperature [ $^{\circ}C$ ]	$P_{CO_2}$
1	40	6
2	40	8
3	40	10
4	40	13
5	60	8
6	60	10
7	60	13
8	80	8



165 the start of the experiment. The flows of dry N<sub>2</sub> and CO<sub>2</sub> in the inlet gas were  
 166 adjusted to the desirable concentration and were kept constant throughout the  
 167 experiment. The dry N<sub>2</sub> gas flow was considered to be the same in the inlet and  
 168 outlet since N<sub>2</sub> is an inert gas. The total molar flow could then be calculated  
 169 from the N<sub>2</sub> inlet flow and the CO<sub>2</sub> content given by the IR analyzer, as shown  
 170 by Eq. 1. The CO<sub>2</sub> mole fraction in the outlet gas stream was calculated by  
 171 Eq. 2. Finally, the amount of CO<sub>2</sub> absorbed is given by the difference between  
 172 amounts of CO<sub>2</sub> in the inlet and outlet, and can be calculated from the measured  
 173 variables according to Eq. 3.

$$Q_{total}^{out} [mol/min] = \frac{n_{N_2}^{in}}{x_{N_2}^{out}} = \frac{n_{N_2}^{in}}{(1 - x_{CO_2}^{out})} \quad (1)$$

$$x_{CO_2}^{out} = \frac{(CO_2 \text{ vol}\%)}{100} \quad (2)$$

$$Q_{CO_2} [mol/min] = n_{CO_2}^{in} - n_{CO_2}^{out} = n_{CO_2}^{in} - x_{CO_2}^{out} Q_{total}^{out} \quad (3)$$

174 The CO<sub>2</sub> absorption rate was calculated according to Eq. 4. Since the mass  
 175 ( $M^{sol}$ ) and the volume ( $V$ ) of the solvent in the reactor and the accumulated  
 176 amount of CO<sub>2</sub> ( $Q_{CO_2}^{Acc.}$ ) are known, the loading in moles of CO<sub>2</sub> per kilogram  
 177 of solvent could be calculated according to Eq. 5. It is important to point it  
 178 out that Eq. 5 computes the loading with respect to the total solvent mass. In  
 179 other words, this accounts for the weight of both the lower and upper phases.

$$r_{CO_2} \left[ \frac{mol}{kg.min} \right] = \frac{Q_{CO_2}}{M^{sol}} \quad (4)$$

$$\alpha \left[ \frac{mol \ CO_2}{kg \ solution} \right] = \frac{Q_{CO_2}^{Acc.}}{M^{sol}} \quad (5)$$

#### 180 4.2. Screening results

181 The absorption rates of CO<sub>2</sub> at 40, 60 and 80°C are shown in Fig. 6, 7 and  
 182 8, respectively. As reference a 5M MEA solution ( 30 % wt.) was tested at 40  
 183 °C and 10 kPa of CO<sub>2</sub> partial pressure.

184 The comparison between the DEEA/MAPA system and MEA is not straight  
185 forward. Several properties, for example, the viscosity, which is much higher in  
186 the DEEA/MAPA system, are different making the comparison difficult. Al-  
187 though the superficial gas velocity was the same for all experiments, the gas-  
188 liquid interfacial area could not be guaranteed to be the same, as explained  
189 before in Ma'mun et al. (2007). In Fig. 6, up to the region of 1.2 mol CO<sub>2</sub>/kg  
190 solution loading, it is possible to conclude that the DEEA/MAPA system shows  
191 a slightly higher absorption rate than the 5M MEA (indicating that it absorbs  
192 CO<sub>2</sub> faster than the 5M MEA) and it retains the absorption rate more constant  
193 than 5M MEA. However, when approaching the equilibrium (high loading re-  
194 gion) the MEA solution has a sharper fall towards zero absorption rate, while  
195 the DEEA/MAPA system presents a more drawn out tail type of ending. This  
196 is due to the saturation of the primary and secondary amine groups in MAPA  
197 while the tertiary amine (DEEA) is still absorbing but at lower absorption rates.

198 Increasing the temperature led to a small increase in the CO<sub>2</sub> absorption  
199 rate. Increasing the CO<sub>2</sub> partial pressure in the gas, also, increases the reaction  
200 rate, as shown in Fig. 9. This is already expected since the driving force is  
201 increase when the amount of CO<sub>2</sub> is increased in the gas. It should be noted,  
202 however, that the initial flat part of all the screening curves is not representative  
203 of the real absorption rates as in this region, all, or close to all, CO<sub>2</sub> in the gas  
204 is removed. However, in the tail end of the curves the measurements give good  
205 indication of the rate of absorption in the DEEA/MAPA system.

206 After an experiment was terminated (achieving 95% of the CO<sub>2</sub> concentra-  
207 tion in the inlet gas stream in the outlet gas stream), the solvent was left to  
208 separate at the experiments temperature, so the volume ratios could be regis-  
209 tered. The phase separation time was also recorded and it varied from 25-30  
210 minutes at 40°C to 3-5 minutes at 80°C.

211 Even though the rate measurements are only semi-quantitative, the screening  
212 experiments contribute with many other measurements which can be used in  
213 understanding and modeling of the system. The volume ratio ( $\phi$ ), defined as  
214 the ratio between the volumes of the lower and the upper phase, and the final

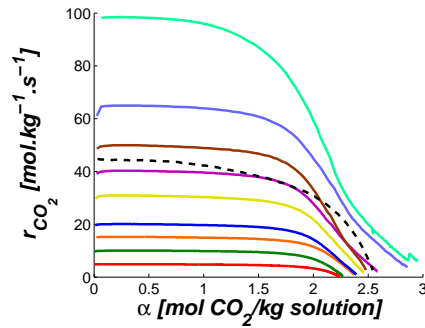


Figure 6: Screening tests performed at 40°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa  $P_{CO_2}$ . Dashed curve: 30 mass% MEA at 10 kPa  $P_{CO_2}$ .

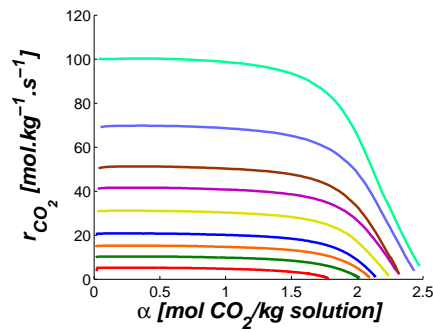


Figure 7: Screening tests performed at 60°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa  $P_{CO_2}$ .

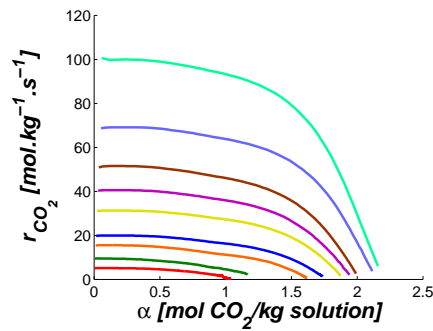


Figure 8: Screening tests performed at 80°C. Solid curves from bottom to top: 1, 2, 3, 4, 6, 8, 10, 13 and 20 kPa  $P_{CO_2}$ .

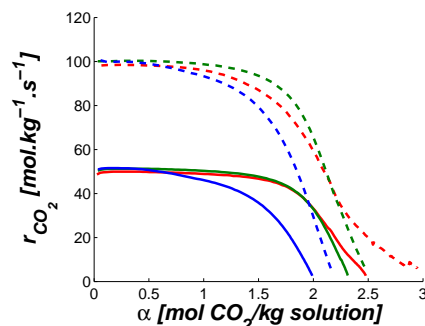


Figure 9: Screening tests performed at 10 kPa and: (—) 40°C, (—) 60°C and (—) 80°C. Screening tests performed at 20 kPa and: (- -) 40°C, (- -) 60°C and (- -) 80°C

215 loading ( $\alpha_{final}$ ), in mol CO<sub>2</sub>/ kg of solution, are given in Table A1 in the  
 216 appendix.

217 Both the upper and lower phases were analyzed for CO<sub>2</sub> and amine content.  
 218 LCMS analyses were used to quantify the ratio between the concentrations  
 219 of DEEA and MAPA while total alkalinity and CO<sub>2</sub> content analyses were  
 220 performed by the procedure described in Monteiro et al. (2013a). With the  
 221 concentration ratio, the total alkalinity and the CO<sub>2</sub> content, the individual  
 222 species concentrations were calculated for all samples and are shown in Table  
 223 A2 in the appendix.

224 From the analyses it was possible to identify a CO<sub>2</sub> rich phase (the lower  
 225 phase) and a CO<sub>2</sub> lean phase (the upper phase). The CO<sub>2</sub> rich phase was rich in  
 226 MAPA and H<sub>2</sub>O whereas the CO<sub>2</sub> lean phase was composed mainly of DEEA.  
 227 The upper phase would work as a buffer of DEEA, which would move to the  
 228 lower phase as more CO<sub>2</sub> is capture by the solvent.

229 The concentration ratios between DEEA and MAPA (mole/mole) in the  
 230 lower phase given by the LCMS are shown in Fig. 10. Apart from some points  
 231 at low CO<sub>2</sub> concentration at 80°C, the concentration of DEEA in the lower phase  
 232 increases more or less linearly with the increase of CO<sub>2</sub> in the system and the  
 233 slope of this linear tendency decreases with the increase of temperature. The 2-3  
 234 points at low CO<sub>2</sub> loading at 80°C are believed not to be outliers, but a result

235 of the increased miscibility between the two phases at higher temperatures.  
 236 There was, however, no second experiment performed at 80°C for checking  
 237 reproducibility. As temperature increases, the solubility of DEEA in the lower  
 238 phase goes up and this is reflected in the increased DEEA concentration seen in  
 239 these points. This is also shown in Fig. 11 where the volume ratio ( $\phi$ ), between  
 240 the lower and upper phase is given as function of CO<sub>2</sub> partial pressure and  
 241 temperature. At 40°C the lower/upper ratio increases with loading in an almost  
 242 linear fashion. At 60°C the ratio increases rapidly at low partial pressures, and  
 243 loadings, whereas it levels off at higher loadings. At 80°C this tendency is even  
 244 clearer as the lower/upper ratio is very low at low loading, indicating better  
 245 miscibility. This supports the previously discussed results shown in Fig. 10 at  
 246 80°C. Although the measurements show clear trends for the volume ratios of the  
 247 phases, the uncertainty in the measurements is relatively high and estimated to  
 248  $\pm 0.03$ - $0.05$  in volume ratio.

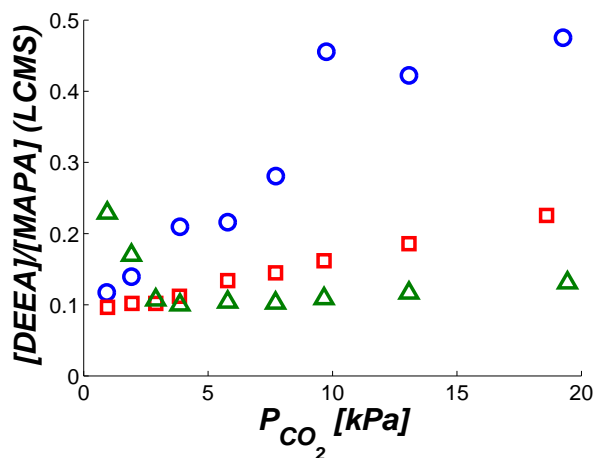


Figure 10: Concentration ratio (mole DEEA/ mole MAPA) from LCMS in the lower phase.  
 Experiments performed at: (○) 40, (□) 60 and (△) 80°C.

249 The mole fractions of the species are shown in Fig. 12, 13 and 14 for the ex-  
 250 periments performed at 40, 60 and 80°C respectively. As previously mentioned,  
 251 it is possible to identify a CO<sub>2</sub> rich (lower) and lean (upper) phase. The DEEA

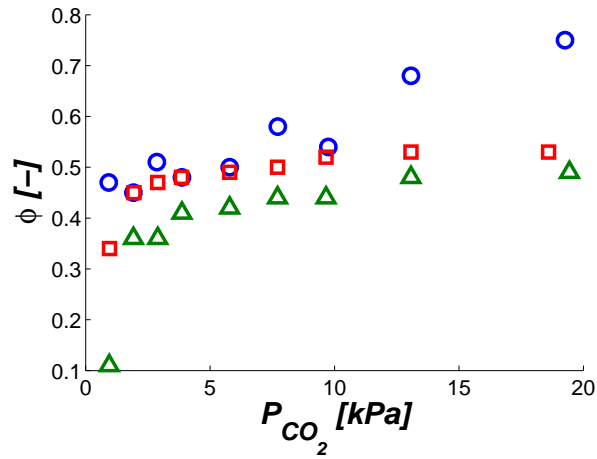


Figure 11: Volume distribution. Experiments performed at: (○) 40, (□) 60 and (△) 80°C.

252 is mainly concentrated in the upper phase. However, as more CO<sub>2</sub> is added to  
 253 the system, the DEEA tends to migrate to the lower phase and, therefore, its  
 254 concentration is reduced in the upper phase.

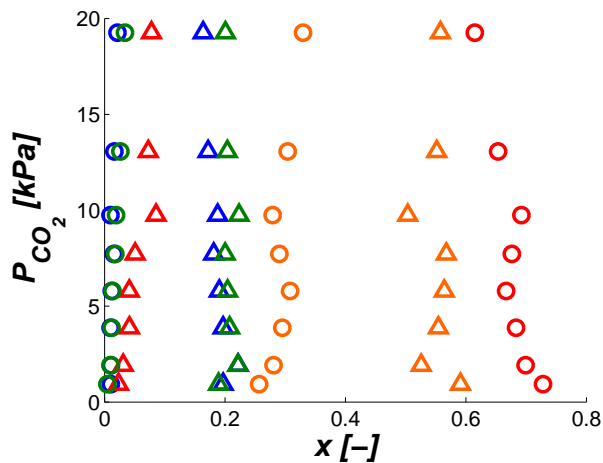


Figure 12: : Mole fraction distribution for experiments taken at 40°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO<sub>2</sub> and (○) H<sub>2</sub>O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO<sub>2</sub> and (△) H<sub>2</sub>O.

255 Fig. 15 shows the CO<sub>2</sub> content (from titration) per kg solution for the lower

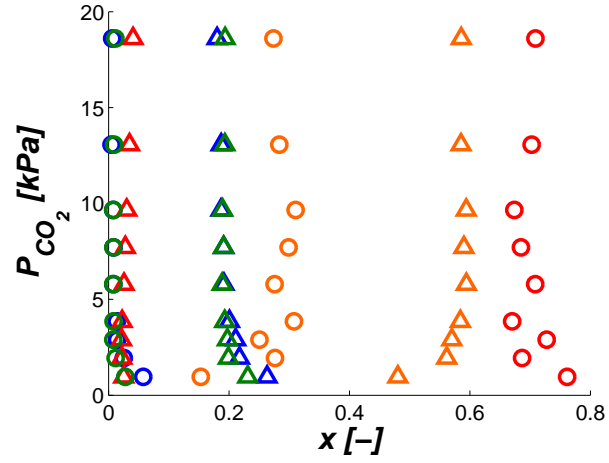


Figure 13: Mole fraction distribution for experiments taken at 60°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO<sub>2</sub> and (○) H<sub>2</sub>O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO<sub>2</sub> and (△) H<sub>2</sub>O.

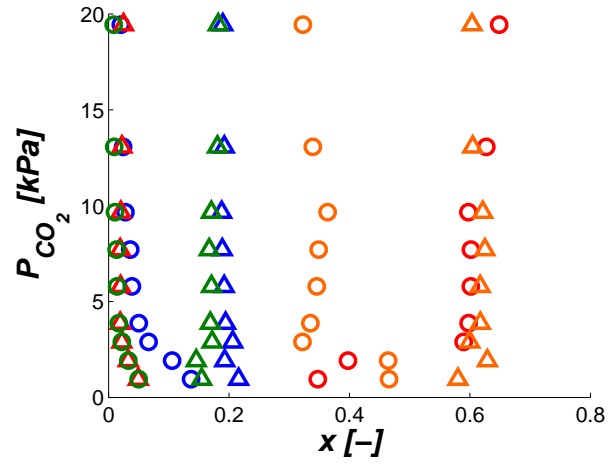


Figure 14: Mole fraction distribution for experiments taken at 80°C. Symbols for the upper phase: (○) MAPA, (○) DEEA, (○) CO<sub>2</sub> and (○) H<sub>2</sub>O. Symbols for the lower phase: (△) MAPA, (△) DEEA, (△) CO<sub>2</sub> and (△) H<sub>2</sub>O.

256 and upper phases as function of the absorption  $\text{CO}_2$  partial pressure. The  $\text{CO}_2$   
 257 content is nearly constant as function of the absorption  $\text{CO}_2$  partial pressure,  
 258 except for the 2-3 first points. As more  $\text{CO}_2$  is added to the system, more DEEA  
 259 migrates to the lower phase, thus, keeping the ratio mole  $\text{CO}_2/\text{kg}$  solution close  
 260 to constant.

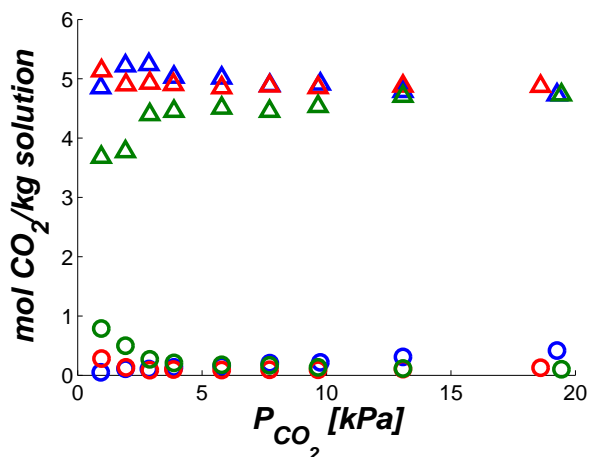


Figure 15:  $\text{CO}_2$  content in the lower and upper phases as function of absorption  $\text{CO}_2$  partial pressure. Upper phase: ( $\circ$ ) 40, ( $\circ$ ) 60 and ( $\circ$ ) 80°C. Lower phase: ( $\Delta$ ) 40, ( $\Delta$ ) 60 and ( $\Delta$ ) 80°C.

261 After separating the phases, the density of each phase was measured at 25°C  
 262 for all experiments. Apart from the measurements at 80°C and low  $\text{CO}_2$  partial  
 263 pressures, it seems that there is no significant variation in the sample densities.  
 264 Fig. 16 shows the densities for the upper and lower phases as function of the  
 265 experimental  $\text{CO}_2$  partial pressure and temperature. The density data for low  
 266 loadings at 80°C again show the increased miscibility at higher temperatures.  
 267 The data are given in Table A4 in the appendix.

#### 268 4.3. High pressure desorption tests

269 The lower phase solutions from the various screening tests were tested for  
 270 stripping performance as described in section 3.2. The lower phase of the DEEA



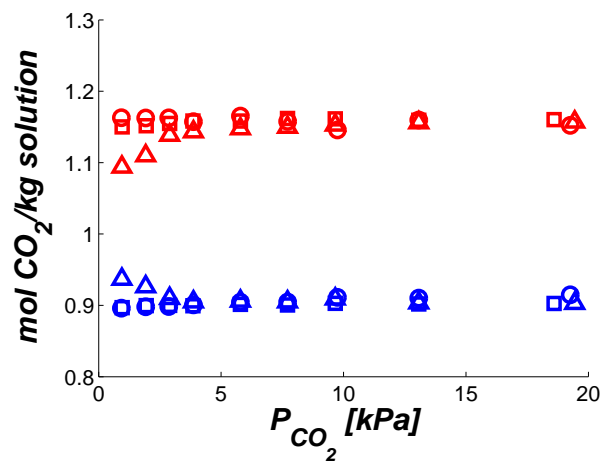


Figure 16: Densities of the phases at 25°C. Upper phase from experiments performed at: (○) 40, (□) 60 and (△) 80°C. Lower phase from experiments performed at: (○) 40, (□) 60 and (△) 80°C.

271 /MAPA system showed a high potential for generating CO<sub>2</sub> at elevated pres-  
 272 sures.

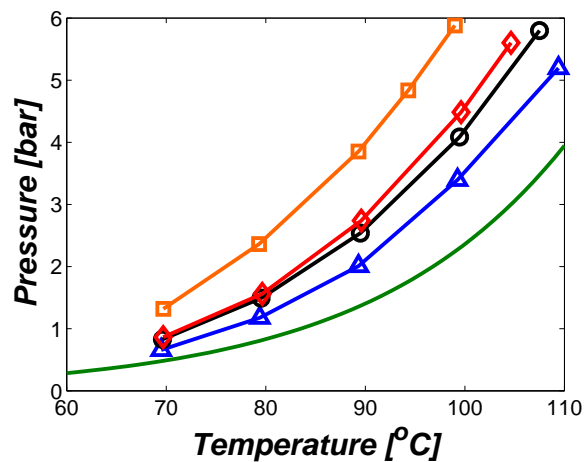


Figure 17: Total pressure from lower phase samples with absorption taken at 40°C from the screening apparatus.  $P_{CO_2}$ : (△) 6 kPa, (○) 8 kPa, (◇) 10 kPa and (□) 13 kPa. (—) MEA at loading 0.5 mol CO<sub>2</sub>/mol MEA (model from Hessen et al. (2010)).

273 In Fig. 17 are shown the vapor pressures of the lower phase, from absorption  
274 tests at 40°C and different CO<sub>2</sub> partial pressures, as a function of temperature.  
275 As expected, the total pressure increases with the sample CO<sub>2</sub> loading, reflected  
276 in the partial pressure at which it has been generated. It should be kept in  
277 mind that the solutions were generated at 95% of the given pressure. As can  
278 be seen the pressures that can be generated are significantly higher than for a  
279 representative 30 mass% MEA solution with loading 0.5 mole CO<sub>2</sub>/mole MEA.  
280 The high pressures that can be obtained can be exploited in two ways. One  
281 way is to use a normal regeneration temperature of about 120°C and produce  
282 CO<sub>2</sub> at elevated pressure, possibly at 6-8 bar. This method will save electrical  
283 energy for the recompression of CO<sub>2</sub> to transportation pressure (around 110  
284 Bar). In a coal based power station typically recompression energy will be 25-  
285 30% of the total energy demand and thus significant savings can be achieved. A  
286 second option is to operate the regenerator at lower temperatures than normal,  
287 e.g. below 100°C. By doing this the stripping pressure will not be increased  
288 compared to the normal situation but the quality of the heat to be supplied will  
289 be reduced. In some cases this may be a better option. A lower regeneration  
290 temperature will also positively affect the solvent degradation processes as these  
291 will be slowed down. The reason for the ease of stripping from this system is a  
292 result of the behavior of the blended system. As observed, the diamine MAPA  
293 is very rapidly loaded. This is seen from the flat part of the screening curves  
294 in Fig. 6-8. When MAPA is almost fully loaded, DEEA starts loading up  
295 and is transferred to the heavy bottom phase. When stripping it is basically  
296 DEEA that strips and the loading of MAPA remains nearly unchanged. This  
297 mechanism has another advantage. As shown by Arshad et al. (2013), the heat  
298 of absorption goes significantly down when MAPA is almost fully saturated  
299 and DEEA is being loaded. The heat of absorption drops from about 85-90  
300 kJ/mole CO<sub>2</sub> in the range where MAPA loads to about 60 kJ/mole CO<sub>2</sub> when  
301 DEEA predominantly loads. The operational cycle in this system will be in  
302 this intermediate range where the heat of absorption and desorption will be  
303 determined mainly by DEEA, but also to some extent by MAPA. All the high

304 pressure desorption test results are displayed in Table A3.

## 305 **5. Conclusions**

306 Preliminary absorption tests made on a phase change solvent, composed  
307 of an aqueous mixture of 5M DEEA and 2M MAPA, were performed. The  
308 solvent showed a great absorption capacity. Upon CO<sub>2</sub> loading, the solvent  
309 splits in two immiscible phases. From the analyses it was possible to see that  
310 the CO<sub>2</sub> rich phase was rich in MAPA and H<sub>2</sub>O whereas the CO<sub>2</sub> poor phase  
311 was mainly composed of DEEA. As more CO<sub>2</sub> was added to the system, more  
312 DEEA is transferred to the lower phase. The volume of the lower phase was  
313 also increased by adding more CO<sub>2</sub> to the system. The phase separation was  
314 accelerated by increasing the temperature.

315 Desorption tests made on the CO<sub>2</sub> rich phase from the screening tests showed  
316 that the DEEA/MAPA system can produce CO<sub>2</sub> at elevated pressures with the  
317 solvent regeneration performed at lower temperatures compared to the tradi-  
318 tional 30 mass% MEA process.

319 The new, biphasic system will thus have three advantages. The cyclic capac-  
320 ity is high thereby reducing the sensible heat demand; the lower phase shows a  
321 significant potential for increased CO<sub>2</sub> pressure during stripping thus enabling  
322 regeneration at elevated pressure or lower temperature, and finally the operation  
323 will take place in a domain where mainly loading and stripping of the tertiary  
324 amine takes place, thereby lowering the heat of absorption.

325 The data generated are of great value for further modeling purposes. Nonethe-  
326 less, more experiments need to be carried out to confirm the systems potential  
327 as a solvent for CO<sub>2</sub> capture.

## 328 **Acknowledgements**

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331 acknowledged

332 **Appendix A: Experimental data**

Table A1: DEEA/MAPA system volume ratio and final loading at different temperatures and  $P_{\text{CO}_2}$ 

40 °C			60 °C			80 °C		
$P_{\text{CO}_2}$ [kPa]	$\phi$	$\alpha_{final}$ [mol/kg]	$P_{\text{CO}_2}$ [kPa]	$\phi$	$\alpha_{final}$ [mol/kg]	$P_{\text{CO}_2}$ [kPa]	$\phi$	$\alpha_{final}$ [mol/kg]
0.93	0.47	2.2347	0.96	0.34	1.7786	0.95	0.11	1.0368
1.92	0.45	2.2636	1.94	0.45	2.0180	1.92	0.36	1.1682
2.86	0.51	2.3675	2.90	0.47	2.0958	2.90	0.36	1.6157
3.87	0.48	2.3906	3.85	0.48	2.1380	3.87	0.41	1.7372
5.79	0.50	2.4595	5.79	0.49	2.2407	5.79	0.42	1.8720
7.72	0.58	2.5849	7.71	0.50	2.3207	7.71	0.44	1.9409
9.75	0.54	2.4792	9.66	0.52	2.3141	9.66	0.44	1.9901
13.07	0.68	2.8579	13.07	0.53	2.4329	13.07	0.48	2.1142
19.26	0.75	2.9505	18.6	0.53	2.4753	19.44	0.49	2.1587

Table A2: Individual species concentration (mol/l) for the upper and lower phases after phase separation.

Absorption taken at 40°C									
$P_{\text{CO}_2}$ [kPa]	0.93	1.92	2.86	3.87	5.79	7.72	9.75	13.07	19.26
Upper phase									
MAPA	0.1036	0.1073	N/A	0.1043	0.126	0.1631	0.1016	0.1747	0.2421
DEEA	7.1625	7.1071	N/A	7.0916	7.0686	7.0616	7.1797	7.0264	6.9115
CO <sub>2</sub>	0.0460	0.1000	N/A	0.1211	0.1412	0.1861	0.1988	0.2826	0.3826
H <sub>2</sub> O	2.5246	2.8536	N/A	3.0625	3.2645	3.0329	2.895	3.2692	3.7069
Lower phase									
MAPA	5.8666	6.0861	N/A	5.5173	5.4462	5.1229	4.7332	4.6847	4.4476
DEEA	0.6882	0.8489	N/A	1.1560	1.1761	1.4384	2.1558	1.9786	2.1137
CO <sub>2</sub>	5.6418	6.0682	N/A	5.8192	5.8410	5.6590	5.6292	5.5524	5.4492
H <sub>2</sub> O	17.6096	14.418	N/A	15.5282	16.1239	16.0315	12.6823	15.0301	15.1468
Absorption taken at 60°C									
$P_{\text{CO}_2}$ [kPa]	0.96	1.94	2.9	3.85	5.79	7.71	9.66	13.07	18.6
Upper phase	0.5265	0.2572	0.1404	0.1430	0.0810	0.0862	0.0825	0.0508	0.0613
MAPA	6.9469	7.0057	7.1656	7.0374	7.1722	7.1121	7.1069	7.1783	7.1859

DEEA	0.2530	0.1200	0.0786	0.0887	0.0800	0.0833	0.0836	0.0938	0.1156
CO <sub>2</sub>	1.3974	2.8197	2.4677	3.2325	2.7909	3.1043	3.2767	2.8973	2.7762
H <sub>2</sub> O	0.5265	0.2572	0.1404	0.143	0.0810	0.0862	0.0825	0.0508	0.0613
Lower phase									
MAPA	6.7278	6.1633	6.0820	5.9125	5.7070	5.6784	5.5565	5.4773	5.2666
DEEA	0.6480	0.6275	0.6202	0.6617	0.7641	0.8228	0.8992	1.0175	1.1877
CO <sub>2</sub>	5.9074	5.6423	5.6908	5.6793	5.6169	5.6718	5.6340	5.6551	5.6545
H <sub>2</sub> O	12.282	15.9186	16.4308	17.1997	17.6921	17.5203	7.6773	17.1371	17.1123
Absorption taken at 80°C									
<i>P</i> <sub>CO<sub>2</sub></sub> [kPa]	0.95	1.92	2.90	3.87	5.79	7.71	9.66	13.07	19.44
Upper phase									
MAPA	2.0265	1.4879	0.7399	0.5586	0.4338	0.4045	0.3218	0.2660	0.2178
DEEA	5.1342	5.6052	6.5639	6.6581	6.7498	6.7594	6.8310	6.8973	6.9735
CO <sub>2</sub>	0.7380	0.4630	0.2445	0.1890	0.1599	0.1516	0.1229	0.1047	0.0915
H <sub>2</sub> O	6.8850	6.5461	3.5844	3.7324	3.8779	3.9188	4.1590	3.7296	3.4692
Lower phase									
MAPA	5.6119	5.5516	5.9994	5.8536	5.818	5.7878	5.7497	5.7860	5.6992
DEEA	1.2844	0.9419	0.6417	0.5854	0.6042	0.5925	0.6253	0.6744	0.7453
CO <sub>2</sub>	4.0244	4.1857	5.0056	5.0913	5.1720	5.1214	5.2283	5.4405	5.4730

H2O	15.0900	18.0978	17.4517	18.5929	18.6896	19.1558	19.0301	18.1897	18.1393
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Table A3: High pressure desorption data for the screening lower phase samples.

Screening experiment	Temperature [ $^{\circ}C$ ]	Pressure [mbar]
40 $^{\circ}C$ and 6 kPa	69.50	656.479
	79.41	1174.313
	89.32	2007.091
	99.27	3394.332
	109.41	5193.126
40 $^{\circ}C$ and 8 kPa	69.62	826.588
	79.53	1490.750
	89.51	2537.697
	99.47	4084.989
	107.50	5797.586
40 $^{\circ}C$ and 10 kPa	69.70	863.062
	79.65	1559.486
	89.60	2738.535
	99.62	4484.843
	104.63	5602.400
40 $^{\circ}C$ and 13 kPa	69.71	1318.472
	79.33	2361.130
	89.32	3850.758
	94.32	4834.645
	99.00	5879.321
60 $^{\circ}C$ and 8 kPa	69.63	376.760
	79.57	655.999
	89.51	1119.848
	99.50	1940.140
	109.50	3195.233
	119.60	5105.616

60°C and 10 kPa	69.47	411.752
	79.51	669.675
	89.46	1201.867
	99.46	2056.919
	109.47	3365.524
	119.57	5383.384
60°C and 13 kPa	69.30	437.794
	79.33	776.192
	89.13	1363.450
	99.25	2243.332
	109.01	3648.222
	119.24	5606.304
80°C and 8 kPa	69.64	209.694
	74.60	270.101
	79.44	344.823
	89.40	589.248
	99.37	990.095
	109.42	1641.672
	119.50	2697.620

Table A4: Densities of the loaded and unloaded phases at 25°C.

Temperature [°C]	$P_{\text{CO}_2}$	$\rho_{\text{upper}}$	$\rho_{\text{lower}}$
40	0.93	0.8960	1.1630
	1.92	0.8981	1.1625
	2.86	0.8984	1.1627
	3.87	0.9007	1.1574
	5.79	0.9044	1.1652
	7.72	0.9047	1.1577
	9.75	0.9112	1.1458

	13.07	0.9101	1.1597
	19.26	0.9149	1.1522
	0.96	0.8968	1.1500
	1.94	0.8997	1.1516
	2.90	0.9000	1.1550
	3.85	0.8994	1.1582
60	5.79	0.9014	1.1582
	7.71	0.9006	1.1619
	9.66	0.9028	1.1613
	13.07	0.9020	1.1594
	18.60	0.9026	1.1603
	0.95	0.9367	1.0939
	1.92	0.9262	1.1097
	2.90	0.9097	1.1384
	3.87	0.9050	1.1433
80	5.79	0.9061	1.1476
	7.71	0.9050	1.1498
	9.66	0.9092	1.1527
	13.07	0.9035	1.1559
	19.44	0.9029	1.1571

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