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# Solubility of $H_2S$, $CO_2$, and Their Mixtures in an Aqueous Solution of 2-Piperidineethanol and Sulfolane

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The solubility of $H_2S$ and $CO_2$ in an aqueous solution containing 45 mass % 2-piperidineethanol, 40 mass % sulfolane, and 15 mass % water has been determined at 40 and 100 °C. Partial pressures of $H_2S$ ranged from 0.02 to 5550 kPa and partial pressures of $CO_2$ ranged from 0.002 to 18 900 kPa. Additional data for $CO_2$ were obtained at 25, 70, and 130 °C. Data were also obtained for mixtures of $H_2S$ and $CO_2$ in this solution at 40 and 100 °C.

The removal of the acid gases, $H_2S$ and $CO_2$, from refinery and natural gases is necessary to meet the specifications on the small amount of $H_2S$ and $CO_2$ remaining after treatment. Chemical solvents, usually consisting of aqueous solutions of an alkanolamine, have been in use for over 60 years. Physical solvents, usually an organic compound with an elevated boiling point, have been used for bulk removal of $CO_2$ from gas streams. Mixed solvents are an attempt to meld the advantages of chemical and physical solvents. Chemical solvents are able to reduce the $H_2S$ and $CO_2$ content to low levels, resulting in a sharp separation; however, the capacity is limited by the stoichiometry of the reaction of the acid gases with the alkanolamine base. Physical solvents are not limited, and their absorption capacity is approximately proportional to the partial pressure of the acid gas in the stream to be treated. Also, physical solvents are able to absorb mercaptans and other sulfur compounds, and less energy is required to regenerate physical solvents.

One of the earliest mixed solvents was Sulfinol (Dunn et al., 1964), which consisted of diisopropanolamine + sulfolane + water. Improvements in the Sulfinol process have been presented by Flynn et al. (1981), and new formulations replacing diisopropanolamine with methyldiethanolamine have been reported. Woertz (1972) investigated a number of mixed solvents in a screening study. The family of Ucarsols appeared in 1981, and a number of such mixed solvents have been described by Epps and Wimberly (1990). The Optisol mixed solvent has been presented by Byseda et al. (1985) and the Selefining mixed solvent has been described by Gazzi et al. (1986). Roberts and Mather (1988) presented solubility data for $H_2S$ and $CO_2$ in a mixed solvent consisting of 2-amino-2-methyl-1-propanol + sulfolane + water. Teng and Mather (1991) presented solubility data for mixtures of $H_2S$ and $CO_2$ in the same solvent. MacGregor and Mather (1991) reported solubility data for $H_2S$, $CO_2$, and their mixtures in a mixed solvent consisting of methyldiethanolamine + sulfolane + water.

This work was undertaken to provide solubility data for $H_2S$, $CO_2$, and their mixtures in a mixed solvent composed of 2-piperidineethanol + sulfolane + water over a range of temperatures and pressures.

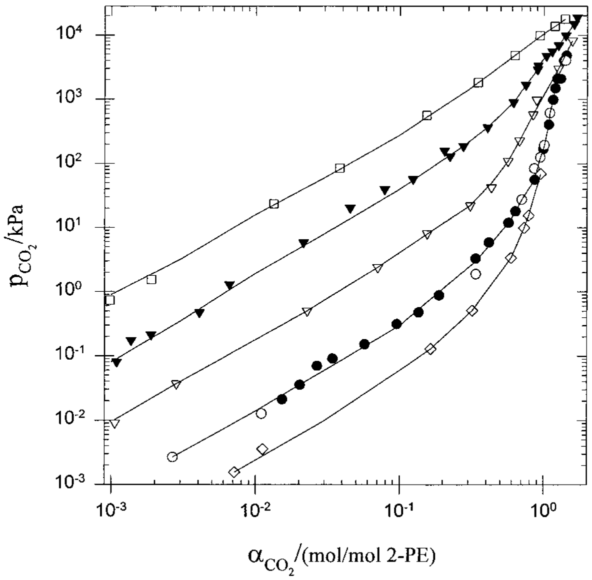

**Figure 1.** Solubility of $CO_2$ in a mixed solvent: ◇ 25 °C; ○, ●, 40 °C; ▽, 70 °C; ▼, 100 °C; □, 130 °C. Unfilled symbols indicate gas chromatographic analyses of the liquid phase; filled symbols indicate wet chemical analyses.

## Experimental Section

The apparatus used for the solubility measurements was that used in our previous work (Jou et al., 1985). It consisted of a windowed Jerguson liquid level gauge with a 50-cm$^3$ tubular gas reservoir mounted on the top. The vapor phase was recirculated through the solvent by means of a magnetically driven piston pump. The gauge and pump were enclosed in an air bath controlled to within ±0.5 °C. The temperature inside the cell was measured with a calibrated iron−constantan thermocouple that extended through the cell wall into the liquid. The accuracy of the temperature measurements was ±0.1 °C. The pressure in the cell was measured by a calibrated Heise bourdon gauge that had an accuracy of 0.1% of full scale. The mixed solvent was prepared by mass: 45% 2-piperidineethanol, 40% sulfolane, and 15% water.

The 2-piperidineethanol was supplied by Exxon and had a purity of 96.7%. Although low, this purity is greater than

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**Table 1. Solubility of $CO_2$ in 45 Mass % 2-Piperidineethanol**

| *P* (kPa) | $p_{CO_2}$ (kPa) | $\alpha_{CO_2}$ (mol $CO_2$/mol 2-PE) | *P* (kPa) | $p_{CO_2}$ (kPa) | $\alpha_{CO_2}$ (mol $CO_2$/mol 2-PE) |
|---|---|---|---|---|---|
| | | 25 °C | | | |
| 140[a] | 69.2 | 0.951 | 150 | 0.513 | 0.320 |
| 200 | 15.5 | 0.787 | 200 | 0.129 | 0.165 |
| 170 | 9.95 | 0.731 | 150 | 0.00359 | 0.01119 |
| 150 | 3.38 | 0.592 | 150 | 0.00156 | 0.00714 |
| | | 40 °C | | | |
| *5600* | *5590* | *2.338* | *163* | *18.1* | *0.638* |
| *4830* | *4820* | *1.440* | *154* | *12.0* | *0.571* |
| *3990* | *3980* | *1.378* | *153* | *5.91* | *0.417* |
| 3990 | 3980 | 1.420 | *165* | *3.30* | *0.338* |
| *2110* | *2100* | *1.242* | 180 | 1.89 | 0.338 |
| *2100* | *2090* | *1.315* | *198* | *0.883* | *0.189* |
| *1490* | *1480* | *1.201* | *167* | *0.479* | *0.136* |
| *990* | *980.* | *1.162* | *146* | *0.316* | *0.0957* |
| 620[a] | 615. | 1.103 | *135* | *0.153* | *0.0572* |
| *600* | *407.* | *1.083* | *252* | *0.0917* | *0.0343* |
| 300 | 196. | 1.010 | *138* | *0.0704* | *0.0267* |
| *220* | *170.* | *0.994* | *153* | *0.0353* | *0.0203* |
| 130 | 126. | 0.945 | *150* | *0.0211* | *0.0153* |
| 203 | 84.8 | 0.862 | 150 | 0.0127 | 0.0110 |
| *236* | *56.3* | *0.864* | 150 | 0.00268 | 0.00266 |
| 165 | 27.6 | 0.705 | | | |
| | | 70 °C | | | |
| 8230[a] | 8200. | 1.576 | 200 | 22.2 | 0.309 |
| 3000 | 2980. | 1.263 | 200 | 8.12 | 0.156 |
| 1000 | 980. | 0.843 | 180 | 2.41 | 0.0705 |
| 600 | 580. | 0.892 | 150 | 0.506 | 0.0228 |
| 250 | 230. | 0.677 | 220 | 0.0367 | 0.00282 |
| 130 | 110. | 0.565 | 200 | 0.00913 | 0.00106 |
| 200 | 42.6 | 0.433 | | | |
| | | 100 °C | | | |
| *19000* | *18900.* | *1.704* | *270* | *189.* | *0.277* |
| *15000* | *14900.* | *1.622* | *280* | *160.* | *0.205* |
| *10000* | *9900.* | *1.419* | *137* | *57.2* | *0.124* |
| *7000* | *6920.* | *1.266* | *170* | *39.7* | *0.0786* |
| *5650* | *5570.* | *1.143* | *150* | *20.4* | *0.0452* |
| *4810* | *4730.* | *1.043* | *130* | *5.89* | *0.0215* |
| *3440* | *3360.* | *0.913* | *150* | *1.30* | *0.00660* |
| *3000* | *2910.* | *0.906* | *123* | *0.477* | *0.00407* |
| *1760* | *1680.* | *0.751* | *190* | *0.213* | *0.00188* |
| *980* | *896.* | *0.617* | *146* | *0.174* | *0.00137* |
| *450* | *370.* | *0.407* | *160* | *0.0809* | *0.00109* |
| *380* | *131.* | *0.224* | | | |
| | | 130 °C | | | |
| 18000 | 17700. | 1.405 | 730 | 560. | 0.154 |
| 14000 | 13700. | 1.190 | 400 | 84.9 | 0.0382 |
| 10000 | 9800. | 0.936 | 350 | 23.4 | 0.0133 |
| 5000 | 4800. | 0.628 | 340 | 1.53 | 0.00189 |
| 2000 | 1820. | 0.349 | 350 | 0.729 | 0.000979 |

[a] Dew point (second liquid phase appears).

**Table 2. Solubility of $H_2S$ in 45 Mass % 2-Piperidineethanol**

| *P* (kPa) | $p_{H_2S}$ (kPa) | $\alpha_{H_2S}$ (mol $H_2S$/mole 2-PE) | *P* (kPa) | $p_{H_2S}$ (kPa) | $\alpha_{H_2S}$ (mol $H_2S$/mole 2-PE) |
|---|---|---|---|---|---|
| | | 40 °C | | | |
| *2420* | *2410* | *6.086* | *173* | *48.8* | *0.990* |
| *2180* | *2170* | *4.457* | *178* | *28.7* | *0.966* |
| *1740* | *1730* | *3.134* | *150* | *13.0* | *0.847* |
| *1410* | *1400* | *2.444* | *151* | *3.88* | *0.639* |
| *897* | *890* | *1.804* | *137* | *1.75* | *0.475* |
| *586* | *578* | *1.455* | *133* | *1.05* | *0.340* |
| *475* | *458* | *1.309* | *136* | *0.389* | *0.234* |
| *329* | *323* | *1.234* | *120* | *0.247* | *0.167* |
| *181* | *174* | *1.101* | *132* | *0.0791* | *0.073* |
| *118* | *111* | *1.060* | *125* | *0.0284* | *0.0358* |
| *147* | *67.9* | *1.049* | | | |
| | | 100 °C | | | |
| *5640* | *5550* | *3.936* | *534* | *454* | *0.974* |
| *4910* | *4820* | *3.001* | *473* | *393* | *0.973* |
| *4530* | *4460* | *2.915* | *334* | *254* | *0.792* |
| *4360* | *4270* | *2.649* | *217* | *137* | *0.658* |
| *3800* | *3720* | *2.403* | *460* | *104* | *0.597* |
| *3180* | *3090* | *2.152* | *133* | *52.3* | *0.381* |
| *3160* | *3080* | *2.070* | *157* | *31.8* | *0.267* |
| *2550* | *2470* | *1.825* | *504* | *18.8* | *0.184* |
| *1870* | *1790* | *1.515* | *562* | *4.45* | *0.0630* |
| *1350* | *1270* | *1.321* | *414* | *2.83* | *0.0413* |
| *1060* | *973* | *1.245* | *466* | *0.369* | *0.0105* |
| *860* | *790* | *1.130* | *128* | *0.254* | *0.00635* |

**Table 3. Solubility of Mixtures of $H_2S$ and $CO_2$ in 45 Mass % 2-Piperidineethanol at 40 °C**

| *P* (kPa) | $p_{H_2S}$ (kPa) | $p_{CO_2}$ (kPa) | $\alpha_{H_2S}$ | $\alpha_{CO_2}$ |
|---|---|---|---|---|
| 120. | 0.0508 | 0.0471 | 0.0573 | 0.0247 |
| 127. | 0.0746 | 0.0389 | 0.0804 | 0.0202 |
| 120. | 0.148 | 0.0718 | 0.0800 | 0.0330 |
| 152. | 0.157 | 0.321 | 0.0856 | 0.0974 |
| 121. | 0.169 | 0.0558 | 0.117 | 0.0267 |
| 134. | 0.231 | 0.0511 | 0.124 | 0.0171 |
| 158. | 0.275 | 1.90 | 0.0826 | 0.265 |
| 149. | 0.290 | 3.93 | 0.0791 | 0.365 |
| 171. | 0.466 | 12.2 | 0.0615 | 0.533 |
| 163. | 0.481 | 8.17 | 0.0755 | 0.496 |
| 155. | 0.486 | 87.3 | 0.0232 | 0.851 |
| 143. | 0.454 | 21.3 | 0.0626 | 0.615 |
| 219. | 1.37 | 211. | 0.0258 | 1.00 |
| 161. | 1.48 | 48.5 | 0.0647 | 0.787 |
| 171. | 1.58 | 0.225 | 0.417 | 0.0387 |
| 169. | 2.00 | 0.228 | 0.431 | 0.0427 |
| 150. | 2.64 | 0.252 | 0.463 | 0.0377 |
| 178. | 2.85 | 37.1 | 0.131 | 0.666 |
| 1010. | 3.07 | 994. | 0.0150 | 1.14 |
| 167. | 3.32 | 155. | 0.0552 | 0.858 |
| 763. | 3.83 | 753. | 0.0155 | 1.16 |
| 438. | 4.94 | 426. | 0.0429 | 1.05 |
| 340. | 5.19 | 329. | 0.0528 | 0.982 |
| 197. | 9.09 | 27.1 | 0.386 | 0.446 |
| 224. | 9.76 | 201. | 0.123 | 0.876 |
| 235. | 13.2 | 183. | 0.177 | 0.842 |
| 170. | 26.9 | 136. | 0.383 | 0.666 |
| 936. | 28.9 | 897. | 0.123 | 1.07 |
| 472. | 37.1 | 428. | 0.239 | 0.879 |
| 155. | 55.6 | 0.113 | 1.07 | 0.00097 |
| 214. | 87.5 | 5.33 | 1.10 | 0.0100 |
| 254. | 196. | 7.88 | 1.21 | 0.00661 |
| 212. | 205. | 0.632 | 1.19 | 0.000984 |
| 805. | 790. | 6.34 | 1.810 | 0.00250 |
| 200. | 3.91 | 1.43 | 0.555 | 0.131 |
| 200. | 25.0 | 9.36 | 0.842 | 0.119 |
| 623. | 316. | 293. | 1.16 | 0.405 |
| 507. | 30.0 | 3.18 | 0.935 | 0.0465 |
| 379. | 44.5 | 0.214 | 0.961 | 0.00107 |
| 423. | 322. | 83.3 | 1.29 | 0.145 |
| 399. | 337. | 54.0 | 1.20 | 0.0855 |
| 2190. | 2070. | 103. | 4.41 | 0.0596 |
| 3340. | 1890. | 1430. | 3.50 | 0.592 |

that commercially available. Our earlier work on the physicochemical properties (Xu et al., 1992) used 2-PE from Aldrich with a purity of 95%. The sulfolane was obtained from Aldrich Chemical and had a purity of 99%. Double-distilled water was used to prepare the mixed solvent. After the solvent had been introduced into the evacuated cell, $CO_2$, $H_2S$ or both were added. Sometimes nitrogen was added to maintain the total pressure above 200 kPa. The gas was recirculated and bubbled through the solvent in the cell for at least 8 h to allow the system to reach equilibrium. The vapor phase was analyzed by means of a Hewlett-Packard model 5710A chromatograph equipped with a 3-m long, 3.175-mm o.d. column packed with Chromosorb 104. The response factors for the three gases were $CO_2$, 1.00; $H_2S$, 1.01; $N_2$, 1.17. An amount of liquid phase was withdrawn into 1 mol/L NaOH solution for analysis. The $CO_2$ content in the liquid was determined by mixing an aliquot of the solution with an excess of 0.1 mol/L $BaCl_2$ solution to form a $BaCO_3$ precipitate. The precipitate was filtered, washed, and titrated with standardized 0.1 mol/L HCl solution using methyl orange–

**Table 4. Solubility of Mixtures of $H_2S$ and $CO_2$ in 45 Mass % 2-Piperidineethanol at 100 °C**

| *P* (kPa) | $p_{H_2S}$ (kPa) | $p_{CO_2}$ (kPa) | $\alpha_{H_2S}$ | $\alpha_{CO_2}$ |
|---|---|---|---|---|
| 3110. | 3030. | 2.97 | 2.038 | 0.00040 |
| 4370. | 2630. | 1650. | 1.715 | 0.237 |
| 5930. | 2590. | 3240. | 1.670 | 0.338 |
| 6340. | 2100. | 4120. | 1.396 | 0.506 |
| 3180. | 1880. | 1210. | 1.466 | 0.164 |
| 4230. | 1070. | 2900. | 1.034 | 0.481 |
| 2230. | 953. | 985. | 1.145 | 0.210 |
| 1400. | 903. | 145. | 1.238 | 0.286 |
| 5640. | 901. | 4560. | 0.762 | 0.663 |
| 5870. | 609. | 5150. | 0.557 | 0.857 |
| 894 | 448. | 365. | 0.912 | 0.121 |
| 640. | 165. | 66.0 | 0.708 | 0.0557 |
| 1460. | 111. | 1260. | 0.245 | 0.517 |
| 2250. | 59.8 | 2100. | 0.130 | 0.725 |
| 3890. | 58.5 | 3730. | 0.0953 | 0.870 |
| 4620. | 53.0 | 4460. | 0.0764 | 1.006 |
| 515. | 27.2 | 2.86 | 0.239 | 0.00478 |
| 969 | 17.8 | 867. | 0.0631 | 0.533 |
| 479 | 8.44 | 3.54 | 0.0930 | 0.00901 |
| 2240. | 6.81 | 2130. | 0.0165 | 0.770 |
| 573. | 5.72 | 478. | 0.0305 | 0.459 |
| 503. | 5.04 | 132. | 0.0437 | 0.212 |
| 465. | 4.95 | 0.499 | 0.0679 | 0.00137 |
| 463 | 3.20 | 0.467 | 0.0501 | 0.00155 |
| 431. | 3.18 | 10.5 | 0.0443 | 0.0314 |
| 440. | 1.47 | 0.141 | 0.0298 | 0.00068 |
| 610. | 1.25 | 3.56 | 0.0255 | 0.0137 |
| 562. | 0.861 | 2.23 | 0.0210 | 0.00887 |
| 383. | 0.609 | 0.761 | 0.0163 | 0.00422 |
| 452. | 0.521 | 0.419 | 0.0164 | 0.00237 |

xylene cyanol indicator. The $H_2S$ content in the liquid was determined by reacting an aliquot of the solution with an excess of acidic 0.1 mol/L $I_2$ solution. The unreacted $I_2$ was back-titrated with 0.1 mol/L sodium thiosulfate solution using starch indicator. When the gas pressure was high, a 40-cm$^3$ metal sampling bomb containing 1 mol/L NaOH solution was used for sampling the liquid phase. For pressures lower than 1000 kPa, a 50-cm$^3$ Erlenmeyer flask fitted with a rubber septum was used to collect the samples of the liquid phase.

In the more recent experiments for the solubility of $CO_2$ in the solvent, a gas chromatographic technique was used to analyze the liquid phase. The detector and injection port were set at 250 °C. The 3 m × 3.175 mm o.d. column of Chromosorb 104 was set at 120 °C for 4 min and then heated at a rate of 32°/min to 250 °C until the sulfolane eluted. For a fresh solution the retention times were 1.4 min for water, 19.9 min for 2-piperidineethanol, and 27.3 min for sulfolane. Their average area ratios (1% uncertainty) were water:2PE:sulfolane = 17.8:39.5:42.7. These ratios served as a standard for the mixture composition in a loaded solution. If the ratios deviated by more than 1% in any component, the composition of the solution must be adjusted or the solution replaced. Nitrogen and carbon dioxide appeared before the water peak and were well-separated. A 4-$\mu$L sample of the liquid was injected when the system pressure was less than 1000 kPa or the loading was less than 0.5 mol $CO_2$/mol 2-PE. Otherwise, the liquid sample was withdrawn into a 40-mL sample bomb containing 10−20 mL of 50 mass % diglycolamine, which elutes in the chromatograph at 14.0 min. Calibration curves were prepared with 10 $\mu$L to 5 mL of $CO_2$ and 1−100% 2-PE in water. A graph of log area versus log moles had a slope of unity for water and for $CO_2$, but it was concave downward for 2-PE. The solubility of carbon dioxide was determined from the calibration curves. The uncertainty in the liquid-phase analysis with both types of measurement is about ±4% at a given partial pressure of $CO_2$. The advantage of the gas chromatographic method is the much shorter time required for an analysis.

**Table 5. Conditions for Appearance of a Second Liquid Phase**

| *t* (°C) | 25 | 40 | 50 | 60 | 70 | 80 | 90 |
|---|---|---|---|---|---|---|---|
| *P* (kPa) | 140 | 620 | 2000 | 4620 | 8230 | 13050 | 20090 |
| $p_{CO_2}$ (kPa) | 69.2 | 615 | 1980 | 4590 | 8180 | 12900 | 19800 |
| $\alpha_{CO_2}$ (mol/mol 2PE) | 0.951 | 1.103 | 1.295 | 1.420 | 1.576 | 1.704 | 1.809 |

**Table 6. Enthalpy of Solution of $CO_2$ in the Mixed Solvent**

| $\alpha_{CO_2}$ (mol $CO_2$/mol 2-PE) | 0.001 | 0.01 | 0.1 | 0.3 | 0.5 | 0.8 | 1.0 | 1.2 |
|---|---|---|---|---|---|---|---|---|
| $-\Delta H_{diff}$ (kJ/mol) | 83 | 83 | 83 | 80 | 75 | 59 | 47 | 33 |

## Results and Discussion

The solubility of $CO_2$ in the mixed solvent (45 mass % 2-piperidineethanol, 40 mass % sulfolane, 15 mass % water) was determined at temperatures of (25, 40, 70, 100, and 130) °C over a range of partial pressures of $CO_2$ from 0.002 kPa to 18 900 kPa. The data are presented in Table 1. Values in italics were determined in 1981 using wet chemical analysis. The other values were determined in 1993 mainly using chromatographic analysis. The data are presented in Figure 1. The solubility of $H_2S$ in the mixed solvent was determined at 40 and 100 °C over a range of partial pressures of $H_2S$ from 0.02 to 5550 kPa. The results are presented in Table 2. Data for mixtures of $H_2S$ and $CO_2$ in the mixed solvent are reported in Tables 3 and 4.

As noted in Tables 1 and 2, at elevated pressures a second liquid phase formed. Because of the presence of the physical solvent, values of $\alpha_{CO2}$ can exceed unity. The conditions for appearance of a second liquid phase are reported in Table 5. The phase that appears is almost pure sulfolane.

The application of the Gibbs−Helmholtz equation

$$\left[\frac{\partial \ln p_{CO_2}}{\partial(1/T)}\right]_x = \frac{\Delta H_{diff}}{R}$$

to the solubility data allows the calculation of the enthalpy of solution of the $CO_2$. The values are presented in Table 6.

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