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Physical Solubility and Diffusivity of CO_2 in Aqueous Diethanolamine Solutions

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The physical solubility and diffusivity of CO_2 in aqueous DEA solutions were measured directly in this work via the "protonation method". The concentration of DEA in the aqueous solutions was 10, 20, and 30 mass %. The measurements were done in the temperature range (25−60) °C at atmospheric pressure. Correlations for the solubility and diffusivity as a function of temperature were developed. The results were compared with the values calculated on the basis of the N_2O analogy. New correlations for the solubilities and diffusivities of CO_2 and N_2O in water were also developed.

1. Introduction

Aqueous alkanolamine solutions are commonly used in gas-treating processes to remove acid gases, like H_2S and CO_2, from natural and industrial gases. Monoethanolamine (MEA), diethanolamine (DEA), di-2-propoanolamine (DIPA), and *N*-methyldiethanolamine (MDEA) are some of the amines widely used for this purpose. In the design of a gas-treating process for the removal of these gases, it is essential to calculate the mass-transfer rates accurately. Apart from information needed on the mass transfer, gas−liquid contact area, and reaction kinetics, data on the fundamental physicochemical properties such as physical solubility and diffusivity are needed. The physical solubility is the free-gas solubility that dissolves in the solvent.

The reactivity of acid gases with alkanolamine solutions makes the direct measurements of the physicochemical properties impossible. This led Clarke,[1] and later many researchers in this field,[2−12] to estimate these properties from corresponding data of similar nonreacting gases. For CO_2, the analogy with N_2O, because of molecular similarities, is widely applied and has been referred to as the "N_2O analogy". Clarke assumed that the ratios of the solubilities and diffusivities of CO_2 and N_2O in water and in aqueous solutions of organic solvents are similar within 5% at the same temperature.

The relations that have been used to calculate the solubility and diffusivity of CO_2 in amine solutions on the basis of the N_2O analogy are

$$(H_{CO_2})_{amine} = (H_{CO_2}/H_{N_2O})_{water}(H_{N_2O})_{amine} \tag{1}$$

$$(D_{CO_2})_{amine} = (D_{CO_2}/D_{N_2O})_{water}(D_{N_2O})_{amine} \tag{2}$$

Table 1. Solubility of CO_2 in Water

	$H/(Pa{\cdot}m^3{\cdot}mol^{-1})$					
t/°C	ref 5	ref 18	ref 19	ref 10	ref 12	this work
15			2240.3			
18	2469.1					
19	2490.2					
20	2631.6		2589.9			
25	3003.5	2994	2984	2993		2980.1
30	3571.4		3394.4		3382	
35	3937.1		3809.8			
37						3897.1
38.4	4098.4					
40	4219.4		4249.6		4227	
40.4	4201.7					
45	4854.4		4689.3			
50	5154.6		5166.6		5136	
56						5823.3
60	6134.9					
70.5	7142.9					
77.2	7575.8					
82.2	8333.3					
87.1	9259.3					

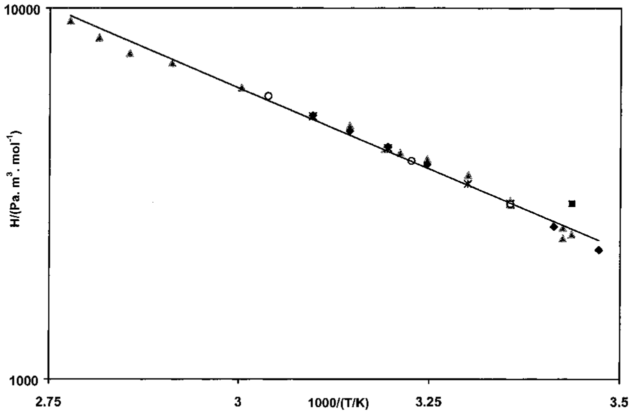

Figure 1. Solubility of CO_2 in water: ○, this work; ▲, Versteeg and van Swaaij;[5] ×, Saha et al.;[10] *, Li and Lai;[12] ■, Yasunishi and Yoshida;[18] ◆, *Int. Crit. Tables*;[19] —, fitted line.

The solubility is expressed in terms of H, the Henry's law constant. H and D of CO_2 and N_2O in water and H and D of N_2O in the amine are calculated at the particular temperature at which the solubility or the diffusivity of CO_2 in the amine is required to be estimated. To calculate the physical solubility of CO_2 in amines via the N_2O analogy requires three measurements; the physical solubility of CO_2 and N_2O in water, and the solubility of N_2O in the amine. For the diffusivity, six measurements are required; the physical solubility and the diffusivity of CO_2 and N_2O in water and the physical solubility and diffusivity of N_2O in the amine. Beside the efforts and cost to conduct the required measurements, in each measurement there is usually a certain experimental error, which can accumulate to give a high percentage of error in the calculated value.

Abu-Arabi et al.[13] developed a "protonation method" to measure directly the physical solubility and diffusivity of acid gases in alkanolamine solutions. The method is based on the protonation of the solutions with HCl before

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Table 2. Solubility of N_2O in Water

t/°C	H/(Pa·m³·mol⁻¹)													
	ref 5	ref 18	ref 9	ref 10	ref 12	ref 20	ref 14	ref 21	ref 22	ref 23	ref 6	ref 8	ref 4	ref 24
15			2897				3172				2992			
18.2	3344													
19	3484													
19.9	3333		2589.9											
20	3424		3321				3506				3482			
25	4073	4115	3911	4120			3982	4101	4179	4234	4169	4152	4118	4210
25.6	3774		3809.8											
29.9	4950													
30			4350		4406		4408							
35	5263		4710								5284			
39.9	5917		4249.6											
40	6136		5020		5725									
45	6993		4689.3											
49.6	7143		5166.6											
49.9	7407													
50			5371		7264	7254		7214	7260					
67	10309													
80	12821													
82.4	140845													

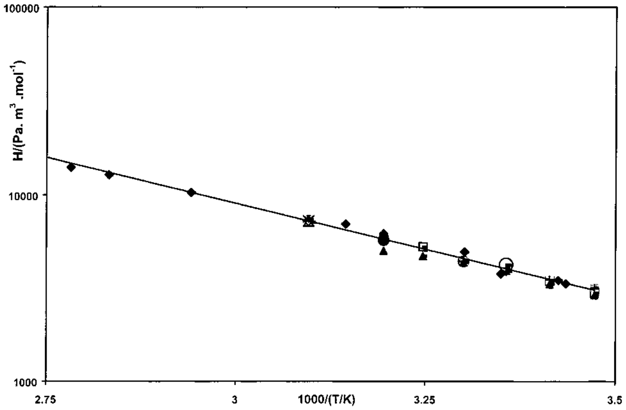

Figure 2. Solubility of N_2O in water: ◆, Sada et al.;[4] ▲, Versteeg and van Swaaij;[5] +, Haimour and Sandall;[6] ◇, Jossten and Danckwerts;[8] □, Saha et al.;[10] ●, Li and Lai;[12] ○, Haimour;[14] △, Yasunishi and Yoshida;[18] ×, *Int. Crit. Tables*;[19] ⊕, Xu et al.;[20] ○, Rinker et al.;[21] ■, Davis and Pogainis;[22] plus sign in a box, Browning and Weiland;[23] -, Markham and Kobe;[24] —, fitted line.

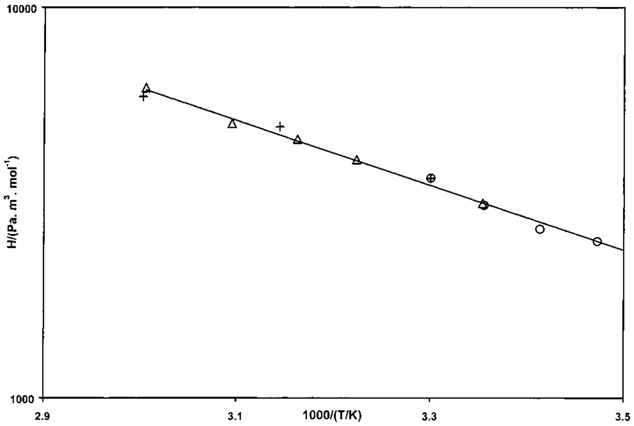

Figure 3. Solubility of CO_2 in protonated 10 mass % DEA solution: △, this work; ○, Haimour;[14] +, Littel et al.;[15] —, fitted line (this work).

contacting them with the acid gas. This way the reactivity between the acid gas and the alkanolamine solutions is eliminated. In their study, the physical solubilities of CO_2 and H_2S in water and in protonated aqueous DEA solutions were measured as a function of pressure at different constant temperatures.

In this study, the physical solubility and diffusivity of CO_2 in protonated aqueous DEA solutions (10, 20, and 30 mass % DEA) at various temperatures are presented. The results are compared with those calculated from the N_2O analogy.

2. Experimental Procedure

2.1. Solution Preparation. The protonation method was followed in this study to prepare protonated aqueous DEA solutions. Hydrochloric acid (12 M) was added to the solutions until the end point was reached. The prepared solutions were 10, 20, and 30 mass % DEA after protonation. Reagent DEA with a purity of 99%, supplied by Across, was used. The purity of the CO_2 gas was ≥99.8%.

2.2. Solubility. The apparatus and the experimental technique used to measure the physical solubility are similar to those used by Haimour and Sandall.[6] The principle of the method is to bring a known volume of gas with a known volume of liquid. The amount of CO_2 gas absorbed in the protonated DEA solutions is measured volumetrically after equilibrium is reached between the gas phase and the solution at constant temperature and pressure. The volume of the gas absorbed is equal to the volume of liquid minus the measured change in volume.

For each solubility measurement, the absorption flask was first purged with CO_2 gas saturated with vapors of the desired solution at constant temperature. A predetermined volume of degassed solution, which was kept at the same temperature of the experimental run, was injected into the absorption flask. The liquid was agitated with a magnetic stirrer until there was no further change in gas volume.

The whole apparatus was kept at constant temperature inside a temperature-controlled bath within ±0.5 K. The experimental error in the reported data of solubility is less than 2%.

The solubility is calculated in terms of the Henry's law constant as follows:

$$H_A = P_A/C_A^* \qquad (3)$$

where C_A^* is the equilibrium concentration of CO_2, which was calculated from the total moles of gas absorbed in a volume of absorbing liquid. The partial pressure of CO_2 in the absorption apparatus was calculated from Raoult's law

$$P_{CO_2} = P_{total} - x_{H_2O}P^v_{H_2O} - x_{amine}P^v_{amine} \qquad (4)$$

where P_{total} = total pressure (in this study it is atmospheric), x = mole fraction, and P^V = vapor pressure of water. The last term in eq 4 was very small for the temperature range covered in this study and was neglected. The vapor pressure of water was calculated from the

Table 3. Coefficients of Eq 6, $H/(\mathrm{Pa{\cdot}m^3{\cdot}mol^{-1}}) = A \exp(B/(T/\mathrm{K}))$

system	A	B	R^2
CO_2 + water	2 453 122	−1999	0.991
N_2O + water	8 210 416	−2271	0.988
CO_2 + 10 mass % DEA	1 868 183	−1901.1	0.996
CO_2 + 20 mass % DEA	1 108 919	−1695.4	0.997
CO_2 + 30 mass % DEA	654 525	−1494.7	0.997

Table 4. Solubility of CO_2 in Protonated DEA Solutions

	$H/(\mathrm{Pa{\cdot}m^3{\cdot}mol^{-1}})$			
t/°C	this work[a]	ref 14	ref 15	this work[b]
		10 mass %		
15	2539	2530		
20	2841	2719.3		
25	3168	3129.8		3166
30	3520	3677.4	3671.8	
37	4056			4102
43	4557			4627
45	4732		4981.4	
50	5191			5066
60	6195		5943.4	6255
		20 mass %		
15	3078	2748		
20	3403	2857.2		
25	3750	3348.5		3739
30	4120	4271.7	3824.3	
37	4674			4669
44	5274			5333
45	5364		5118.4	
50	5825			5891
58	6613			6537
60	6820		5962.3	
		30 mass %		
15	3647	3051.5		
20	3985	3117.7		
25	4341	3778.4		4405
30	4716	5207.8	3989.3	
37	5239			5223
42	5691			5629
45	5951		5278.7	
50	6400			6413
59	7256			7342
60	7355		5981.2	

[a] Calculated from eq 8 and Table 3. [b] Experimental data.

following equation, which was taken from Al-Ghawas et al.:[9]

$$P^{\mathrm{v}}_{\mathrm{H_2O}}/\mathrm{bar} = 1.33567 \times 10^{6} \exp(-5243.04/(T/\mathrm{K})) \quad (5)$$

2.3. Diffusivity. The wetted sphere apparatus used in this study is described in detail by Al-Ghawas et al.[9] To carry out the experimental runs, the solution was degassed by boiling the liquid with heating under vacuum. At the same time, a gas saturated with vapors of the desired solution was passed through the system long enough to completely flush the system of any air and fill the system with the saturated gas being absorbed. After degassing and flushing, the solution was transferred to an overhead feed tank using pressurized nitrogen gas to prevent any contamination with atmospheric air. The temperature of the feed tank was held constant by a heating coil containing hot water. The solution and the gas passed through coils embedded in the water jacket surrounding the wetted-sphere absorber before entering the absorber.

Degassed solution flows by gravity through a rotameter and then enters the liquid distributor at the inlet of the absorber. Initially, the solution was pumped at a high flow rate to ensure complete wetting of the sphere surface; the flow rate was then reduced to a value such that the flow rate is in the range 1.5 mL/s to achieve laminar flow conditions.

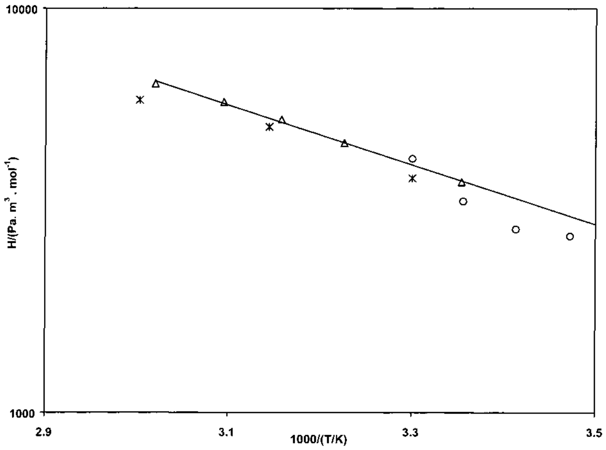

Figure 4. Solubility of CO_2 in protonated 20 mass % DEA solution: △, this work; ○, Haimour;[14] *, Littel et al.;[15] —, fitted line (this work).

The temperatures of the gas and liquid were measured to within ±0.2 K using a thermocouple located in the gas space surrounding the sphere and the liquid inlet to the sphere. The volumetric gas absorption rate was measured using a soap bubble meter. When the system reached steady state, the temperature, pressure, gas, and liquid flow rates were taken. Each run was repeated twice, and the average is reported. Experimental errors were less than 2%.

The diffusivities of CO_2 in aqueous DEA solutions were calculated from the series solution developed by Olbrich and Wild[34] as follows:

$$R_{\mathrm{A}} = LC^{*}_{\mathrm{A}}[1 - \sum \beta_i \exp(-\gamma_i \alpha)] \quad (6)$$

where

$$\alpha = 3.36\pi(2\pi g/3\nu)^{1/3} R_{\mathrm{s}}^{7/3} L^{-4/3} D \quad (7)$$

R_A is the rate of absorption, C^{*}_{A} is the equilibrium concentration, L is the liquid flow rate, g is the acceleration of gravity, R_s is the radius of the sphere, ν is the kinematics viscosity, D is the diffusion coefficient, and β_i and γ_i are constants. An iterative procedure is required for the calculation of diffusivity from eq 6.

3. Results and Discussion

3.1. Solubilities of CO_2 and N_2O in Water. These solubilities are needed to calculate the solubility of CO_2 in DEA solutions on the basis of the N_2O analogy. The solubility of CO_2 in water at several temperatures was measured to ensure the reliability of the measurements and the method of calculations followed in this study. The results from this study as well as the literature values are presented in Table 1 and plotted in Figure 1. The agreement is good between the results of this work and the literature values. The data for the solubility of N_2O were obtained from the open literature and are listed in Table 2 and plotted in Figure 2. The data collected on the solubilities of CO_2 and N_2O were fitted against temperature by an exponential form as

$$H/(\mathrm{Pa{\cdot}m^3{\cdot}mol^{-1}}) = A \exp(B/(T/\mathrm{K})) \quad (8)$$

where A and B are the constants to be evaluated. The values of A and B are given in Table 3 with the corre-

Table 5. Diffusivity of CO_2 in Water

	$10^5 D/(cm^2 \cdot s^{-1})$														
t/°C	ref 17	ref 25	ref 19	ref 26	ref 27	ref 28	ref 7	ref 29	ref 1	ref 30	ref 31	ref 10	ref 2	ref 33	this work
0	0.96														
6.5		1.15													
10			1.46												
15			1.6	1.39											
16	1.57														
18			1.71												
18.5					1.65										
19.5						1.68									
20			1.77	1.64										1.76	
25	1.74	1.85		1.94	1.95		1.98	1.87	2.05	2	1.87			1.94	1.88
30				2.29									2.12	2.2	
30.2												2.18			
34.7					2.41										
35		2.18											2.462		
40							2.8						2.775	2.93	
45.2					3.03										
52		3.61													3.45
54.9					3.68										
60														4.38	
65		4.3			4.4										
75.1					5.4										
80														6.58	
95														8.2	

Table 6. Diffusivity of N_2O in Water

	$10^5 D/(cm^2 \cdot s^{-1})$											
t/°C	ref 6	ref 5	ref 8	ref 26	ref 27	ref 4	ref 32	ref 7	ref 9	ref 10	ref 12	ref 33
15	1.39								1.285			
16.7				1.7								
18.1		1.47										
19		1.56										
19.9		1.48										
20	1.52	1.55			1.92		1.74		1.436			1.84
24.9				2.09								
25	1.86	1.84	1.92			1.78		1.69	1.569			1.88
29.9		2.27										
30									1.607		2.009	1.93
30.2										2.09		
30.8				2.35								
35	2.03	2.34							1.634		2.302	
39.9		2.35										
40		2.58						2.55	1.679		2.648	2.61
45		3.17										
49.7		2.85										
50									1.868			
60												4.51
67		5.33										
70		5.43										
80		6.32										6.5
95												7.3

sponding R^2 values. As shown in Figures 1 and 2, plotting the data as H versus $1/T$ on a semilog scale gives a straight line.

3.2. Solubility of CO_2 in Protonated DEA Solutions. The solubility of CO_2 was measured in protonated DEA solutions at temperatures ranging from (25 to 60) °C. The experimental data are presented in Table 4. The data were fitted to eq 8, and the values of the constants A and B are listed in Table 3. The data show good internal consistency, and the consistency across temperature and concentration also appears to be good. In Figures 3−5, the results of this study are compared with the solubility of CO_2 calculated from eq 1 on the basis of the N_2O analogy with H_{CO_2} and H_{N_2O} in water obtained from Table 3. The solubilities of N_2O in DEA solutions needed for the calculations are obtained from the data reported by Haimour[14] and Littel et al.[15] In Haimour's work, the concentrations of DEA in the solutions were (1.039, 2.077, and 3.116) mol/L. Converting these to mass percentage gives 10.75, 21.3, and 31.5, respectively, which are very close to the percentages used in this study, and the slight difference should not have much effect on the results. Little et al. represented their experimental data by a best-fit polynomial with the coefficients expressed in terms of temperature. The correlations were used to get the needed values of N_2O solubility in DEA solutions. The solubilities of CO_2 in DEA solutions calculated on the basis of the N_2O analogy from the two sources are listed in Table 4. The agreement is good with a maximum deviation of less than 10%. The deviation is expected knowing that the calculation of CO_2 solubility via the N_2O analogy requires, as mentioned previously, three measurements that can have accumulative experimental errors.

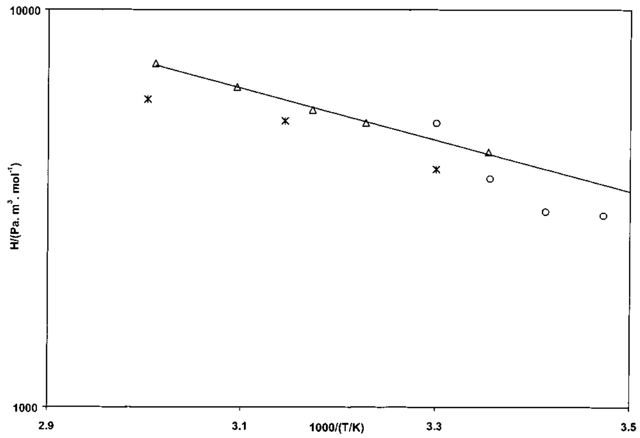

Figure 5. Solubility of CO_2 in protonated 30 mass % DEA solution: △, this work; ○, Haimour;[14] $*$, Littel et al.;[15] —, fitted line (this work).

3.3. Diffusivity of CO_2 and N_2O in Water. The results for CO_2 in water from this work and the literature values are presented in Table 5 and Figure 6. The agreement between the results of this work and the literature values

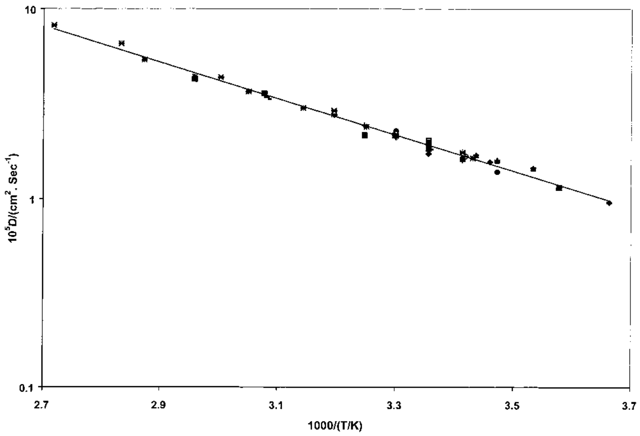

Figure 6. Diffusivity of CO_2 in water: -, this work; ■, Clarke;[1] ◆, Duda and Vrentas;[7] ∗, Saha et al.;[10] +, Li and Lai;[12] ◆, Tamann and Jessen;[17] ▲, *Int. Crit. Tables*;[19] □, Unver and Himmelblau;[25] ●, Davidson and Cullen;[26] ▽, Thomas and Adams;[27] ○, Nijsing et al.;[28] ⊕, Tang and Himmelblau;[29] ×, Vivian and King;[30] △, Scriven;[31] ▼, Tamimi et al.;[33] —, fitted line.

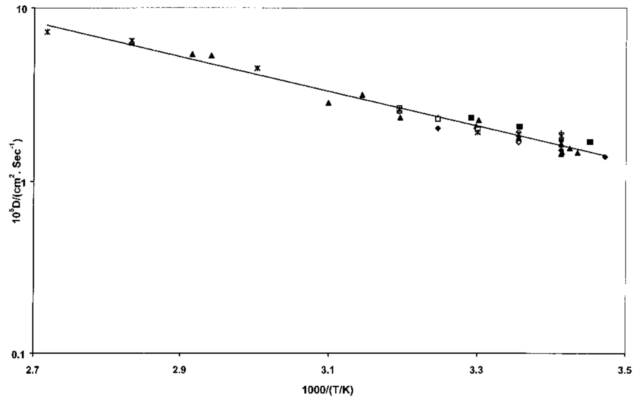

Figure 7. Diffusivity of N_2O in water: ▲, Sada et al.;[4] ◇, Versteeg and van Swaaij;[5] ◆, Haimour and Sandall;[6] ∗, Duda and Vrentas;[7] ○, Joosten and Danckwerts;[8] △, Saha et al.;[10] ■, Li and Lai;[12] □, Davidson and Cullen;[26] +, Thomas and Adams;[27] ●, Hufner;[32] ×, Tamimi et al.;[33] —, fitted line.

Table 7. Coefficients of Eq 9, $D/(10^{-5}\ cm^2{\cdot}s^{-1}) = A' \exp(B'/(T/K))$

system	A'	B'	R^2
CO_2 + water	3073.7	−2197.5	0.989
N_2O + water	4469.7	−2325.3	0.963
CO_2 + 10 mass % DEA	302.51	−1599.9	0.933
CO_2 + 20 mass % DEA	29.15	−937.2	0.988
CO_2 + 30 mass % DEA	28.6	−996.2	0.979

is good. Table 6 and Figure 7 present the diffusivity of N_2O in water as reported in the open literature. The diffusivity was well fitted by an exponential form as

$$D/\mathrm{cm}^2{\cdot}\mathrm{s}^{-1} = A' \exp(B'/(T/\mathrm{K})) \qquad (9)$$

where A' and B' are the constants of fitting. The values of these constants along with the corresponding R^2 values are given in Table 7.

3.4. Diffusivities of CO_2 and N_2O in Protonated DEA Solutions. The diffusivities of CO_2 in protonated DEA solutions determined in this work are given in Table 8 and plotted in Figures 8−10. The data show good internal consistency, and the consistency across temperature and concentration also appears to be good. The data were fitted to eq 9, and the values of the constants A' and B' are listed in Table 7. To calculate the diffusivity of CO_2 via the N_2O analogy (from eq 2), D_{CO_2} and D_{N_2O} are obtained from Table 7 and the reported diffusivities of N_2O in DEA solutions by Tamimi et al.[16] and Sada et al.[4] were used. The calculated values are listed in Table 8. The agreement is good mainly at low temperatures. The deviation at high temperatures can be due to the difficulty in keeping laminar flow and a continuous film on the sphere surface and to the fact that the calculations of CO_2 diffusivity in amines, as mentioned previously, require six measurements that can have accumulative experimental errors.

Table 8. Diffusivity of CO_2 in Protonated DEA Solutions

	$10^5D/(cm^2{\cdot}s^{-1})$			
t/°C	this work[a]	ref 16	ref 4	this work[b]
	10 mass %			
15	1.1699			
20	1.2862	1.2907		
25	1.4096	1.3676	1.6494	
30	1.5402	1.5685		1.6191
32	1.5945			1.6197
40	1.8232	1.7594		1.7491
42	1.8834			1.7825
50	2.1358	2.9428		2.0886
59	2.4428			2.5791
60	2.4784			
80	3.2538	3.9336		
95	3.9140	4.1688		
	20 mass %			
15	1.1255			
20	1.1898	1.0042		
25	1.2554	1.0272	1.4824	
29	1.3088			1.3273
30	1.3223	1.2320		
38	1.4318			1.4178
40	1.4596	1.6154		
40	1.4596			1.4459
50	1.6014	2.4270		
54	1.6592			1.6462
60	1.7472			1.7702
80	2.0492	3.4656		
95	2.2834	3.6760		
	30 mass %			
15	0.8996			
20	0.9543	0.5971		
25	1.0103	0.7235	1.2945	
30	1.0676	0.8102		1.0783
39	1.1738			1.1458
40	1.1859	1.2038		
47	1.2714			1.2947
50	1.3087	1.9012		1.3042
59	1.4228			1.4217
80	1.7009	2.8681		
95	1.9082	3.3902		

[a] Calculated from eq 9 and Table 7. [b] Experimental data.

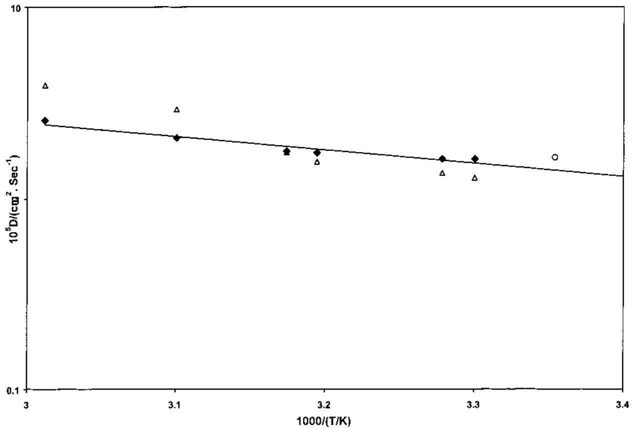

Figure 8. Diffusivity of CO_2 in protonated 10 mass % DEA solution: ◆, this work; ○, Sada et al.;[4] △, Tamimi et al.;[16] —, fitted line (this work).

4. Conclusions

The physical solubilities and diffusivities of CO_2 in aqueous DEA solutions are measured directly via the

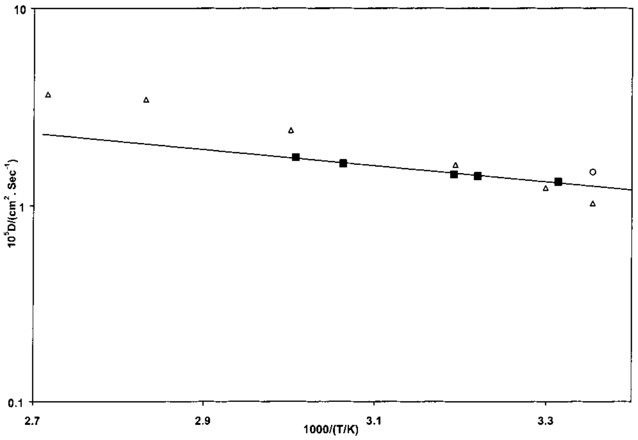

Figure 9. Diffusivity of CO_2 in protonated 20 mass % DEA solution: ■, this work; ○, Sada et al.;[4] △, Tamimi et al.;[16] —, fitted line (this work).

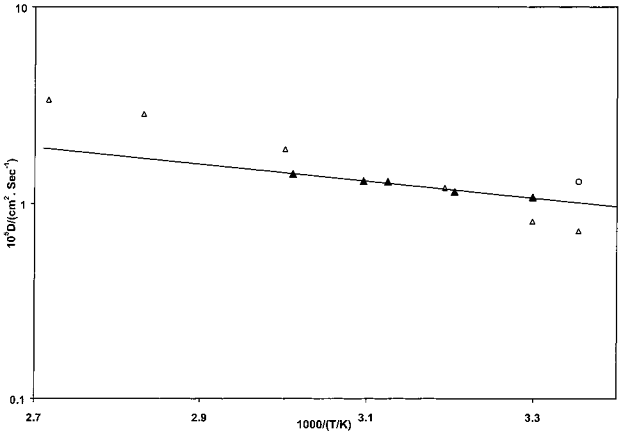

Figure 10. Diffusivity of CO_2 in protonated 20 mass % DEA solution: ▲, this work; ○, Sada et al.;[4] △, Tamimi et al.;[16] —, fitted line (this work).

“protonation method”. The results are compared with those calculated from the “N_2O analogy”. New correlations are developed for the solubilities and diffusivities of CO_2 in water and in aqueous DEA solutions, and of N_2O in water.

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