Heat Capacity of Alkanolamines by Differential Scanning Calorimetry

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> Measurements of the heat capacities of the alkanolamines monoethanolamine, diethanolamine, diglycolamine, di-2-propanolamine, triethanolamine, *N*-methyldiethanolamine, 2-amino-2-methyl-1-propanol, and 2-piperidineethanol were performed from 30 to 80 °C with a differential scanning calorimeter (DSC). The heat capacity of liquid water has been measured to verify the validity of the C_p measurements. The measured C_p of each alkanolamine has been expressed as a function of temperature. The estimated uncertainty of the measured heat capacities including the effect of impurities in a substance with a purity of 95% is ±3%. The measured heat capacities are, in general, of sufficient accuracy for most engineeringdesign calculations.

Introduction

Solutions of alkanolamines are industrially important compounds used in the natural gas industry, oil refineries, petroleum chemical plants, and synthetic ammonia plants for the removal of acidic components such as CO2 and H2S from gas streams. A wide variety of alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA), diglycolamine (DGA), di-2-propanolamine (DIPA), triethanolamine (TEA), and N-methyldiethanolamine (MDEA) has been used industrially for a number of years (Kohl and Riesenfeld, 1985). A recent advancement in gas-treating technology is the application of sterically hindered amines which offer absorption capacity, absorption rate, selectivity, and degradation resistance advantages over conventional amines for acid gases removal (Sartori and Savage, 1983). Two sterically hindered amines of interest are 2-amino-2methyl-l-propanol (AMP) and 2-piperidineethanol (2-PE).

Heat-capacity data for alkanolamine solutions are required for the design of the heat-exchanger equipment used in gas-treating processes. Little information on the heat capacity of alkanolamines is available in the literature. For the heat capacity of alkanolamines, Riddick et al. (1986) reported the C_p values of MEA (data of Dow Chemical Co., 1954), DEA (data of Dow Chemical Co., 1954), and TEA (data of Mehl, 1934a and 1934b) at 30 °C. In a research report by Lee (1994), the C_p values of MEA, DEA, TEA, and MDEA for temperatures from 30 to 150 °C were also studied. Lide (1995) listed the C_p values of MEA at 25 °C, of DEA at 30 °C, and of TEA at 25 °C. For alkanolamine aqueous solutions, heat capacities of 23 wt % and 50 wt % MDEA aqueous solutions have been reported in the literature (Hayden et al., 1983). Also, the C_p values of MEA aqueous solutions (15, 30, and 50 wt %) (Union Carbide Chemical Co., 1957), DEA aqueous solutions (15, 30, and 50 wt %) (Union Carbide Chemical Co., 1957), and DGA aqueous solutions (50, 60, 70, 80, and 90 wt %) (Texaco Chemical Co., 1969) have been reported for temperature ranges from 10 to 110 °C (Kohl and Riesenfeld, 1985). At 25 °C, the heat capacities of CO₂-loaded, aqueous solutions of MEA, DEA, and MDEA and of aqueous MDEA + MEA and MDEA + DEA solutions were also studied (Weiland et al., 1997). Except for the C_p data of Hayden et al. (1983), most of the C_p data are presented in graphical form or as a single value at a particular temperature. Furthermore, the agreement of the C_p values of alkanolamines from different investigators is usually not very good; for example, for MEA at 30 °C, the literature C_p values range from 2.082 to 2.720 kJ·kg⁻¹·K⁻¹. Recently, the constant-pressure heat capacities of 21 hydrogen-containing, fluorinated propane and butane derivatives and one fluorinated ether with boiling points ranging from -34.6 °C to 76.7 °C have been measured to within $\pm 3\%$ accuracy by differential scanning calorimetry at 40 °C (Hwang et al., 1992). It is the purpose of this study to determine experimentally the heat capacities of alkanolamines by using a differential scanning calorimeter (DSC). The alkanolamines chosen for C_p measurements are MEA, DEA, DGA, DIPA, TEA, MDEA, AMP, and 2-PE. The temperature range will be from 30 to 80 °C.

Experimental Section

Some chemicals are Riedel-de Haën reagent grade with the following purities: MEA, min. 99% (water max. 0.5%); DEA, min. 99% (water max. 0.3%); MDEA, 98.5% (water max. 0.5%); TEA, min. 99% (water max. 0.2%); AMP, min. 95%. Other chemicals are from Aldrich Chemical Co. with the following purities: DGA, min. 98%; DIPA, min. 95%; 2-PE, min. 95%. The estimated uncertainty of the measured heat capacities including the effect of impurities in a substance with a purity of 95% is $\pm 3\%$. The water is liquid chromatography grade from Alps Chem. Co.

The differential scanning calorimeter used for this study consists of a DSC-2010 and a thermal analysis controller from TA Instruments. The DSC operating range is from room temperature to 725 °C. Both the temperatures and the heat flow associated with transitions in materials can be easily and rapidly measured with the system. The DSC features a temperature of ± 0.1 °C repeatability. The temperature sensors are Chromel–alumel type and Platinel II type for the sample thermocouple and the control

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thermocouple, respectively. The calorimetric sensitivity is 1 μ W (rms). The constant calorimetric sensitivity is $\pm 2.5\%$ from -100 to 500 °C. The calorimetric precision is $\pm 1\%$, based on measurements of metal samples. The purge gas is nitrogen with a flow rate of 40 mL·min⁻¹. By using the sample-encapsulating press, the liquid sample was prepared within a hermetic sample pan. Hermetically sealed sample pans provide the following advantages: better thermal contact between sample, pan, and constantan disc, reduced thermal gradients in the sample, and an airtight seal that can resist higher internal pressures up to 3 atm. These hermetic pans are usually used for studies of volatile liquids, materials that sublime, aqueous solutions above 100 °C, and materials in a self-generated atmosphere. The internal volume of the hermetic pan is approximately around 10 mm³. The sample mass is in the range 15-20 mg.

To obtain accurate results, the calibrations have to be checked periodically. The DSC calibrations consist of the baseline slope, the cell constant, and the temperature calibrations. The baseline calibration involves heating an empty cell through the entire temperature range expected in subsequent experiments. Both the cell constant and the temperature calibrations are obtained from the run in which a calibration material (e.g., indium) is heated through its melting point. The heat of fusion and the melting point of indium are utilized to calibrate the cell constant and the temperature calibrations.

The specific heat of a substance can be determined conveniently and rapidly with a DSC by creating a baseline profile, a standard sample profile, and a sample profile. The thermograms were plotted using the data analysis program. The specific heat of the substance is determined by measuring the difference in *y*-axis displacement (calorimetric differential) between the sample and blank curves at any desired temperature. The heat capacities of the substances can be calculated as follows

$$C_{\rm p} = \left[\frac{60E}{H_{\rm r}}\right] \frac{\Delta H}{m} \tag{1}$$

where E is the cell calibration coefficient at the temperature of interest, $H_{\rm r}$ is the heating rate (in K·min⁻¹), ΔH is the difference in y-axis deflection between sample and blank curves at the temperature of interest (in mW), *m* is the sample mass (in mg), and C_p is the specific heat (in kJ·kg⁻¹·K⁻¹). The quantity $60E/\dot{H}_r$ is a constant under a given set of experimental conditions. It converts the measurement directly into units of specific heat in kJ·kg⁻¹· K^{-1} . The value of $60E/H_r$ was determined by running a standard sapphire (A12O3) under conditions identical to those of the unknown sample. Then we substituted the values of ΔH , *m*, and C_p for the sapphire into the above equation at the temperature of interest. The C_p values determined by Ginnings and Furukawa of the National Bureau of Standards on aluminum oxide in the form of synthetic sapphire (corundum) were used (Ditmars et al., 1982). The sapphire pieces passed a #10 sieve but were retained by a #40 sieve, and had 99.98-99.99% purity by weight.

During the run, a method was created that holds isothermally at the desired starting temperature (25 °C) for 10 min, heats at the heating rate 5 K·min⁻¹, and then holds at the limit temperature (82 °C) for 10 min. For each sample, the average number of runs is five. The accuracy of the C_p measurements is estimated to be ±2% on the basis of the measurement of the C_p of liquid water.



Figure 1. DSC curves for heat capacity measurement on water.



Figure 2. Heat capacity of water.

Results and Discussion

The C_p measured with a DSC is derived from the heat flow measured for three measurements using eq 1. The $C_{\rm p}$ of liquid water is measured to justify the accuracy of the DSC. Osborne et al. (1939) measured the heat capacity of water with an uncertainty of ± 0.01 to $\pm 0.02\%$ at close intervals of temperature (1 K) by using a large adiabatic calorimeter. In this study, typical heat flow curves for water, that is, the baseline profile (empty pan), the standard sample profile (sapphire), and the sample profile (water) are shown in Figure 1. As shown in Figure 1, the three heat flow curves appear to be straight lines for temperatures from 30 to 82 °C. Consequently, the heat capacities of the sample (H₂O) are also in straight lines, as shown in Figure 2. Four test runs for the measurement of the C_p of water and the averaged values obtained for the C_p of water are presented in Table 1 and shown in Figure 2. The average percentage deviations between the four test runs and the literature values (Osborne et al., 1939) are -1.64%, -0.9%, 1.01%, and 1.62%. The average values given in the last column in Table 1 deviate by only 0.05% from the literature values (Osborne et al., 1939). Thus, the measured C_p values of liquid water for temper-

Table 1. Heat Capacities of Water

		heat capacity, $C_p/(kJ\cdot kg^{-1}\cdot K^{-1})$				
temp	Osborne		t	his stud	у	
(°C)	et al., 1939	run 1	run 2	run 3	run 4	ave value
30	4.1785	4.107	4.150	4.214	4.242	4.178
35	4.1782	4.109	4.149	4.217	4.244	4.180
40	4.1786	4.111	4.148	4.220	4.246	4.181
45	4.1795	4.112	4.148	4.222	4.248	4.183
50	4.1807	4.114	4.147	4.225	4.251	4.184
55	4.1824	4.116	4.147	4.227	4.253	4.186
60	4.1844	4.117	4.146	4.229	4.255	4.187
65	4.1868	4.119	4.146	4.231	4.256	4.188
70	4.1896	4.121	4.145	4.233	4.258	4.189
75	4.1928	4.123	4.145	4.235	4.260	4.191
80	4.1964	4.125	4.144	4.238	4.262	4.192
ave %	dev	-1.64%	-0.90%	1.01%	1.62%	0.05%

Table 2. Heat Capacities of MEA

		heat capacity,	$C_{\rm p}/({\rm kJ\cdot kg})$	$g^{-1} \cdot K^{-1}$)	
temp (°C)	est value	Riddick et al., 1986	Lee, 1994	Lide, 1995	this study
20	2.656 ^a				
25	2.670^{b}			3.201	
30		2.082	2.720		2.74
35			2.760		2.76
40			2.801		2.78
45			2.841		2.81
50	2.904^{b}		2.882		2.83
55			2.923		2.86
60			2.963		2.88
65			3.004		2.91
70			3.044		2.93
75			3.085		2.95
80			3.125		2.98

 a Estimated from Chueh and Swanson, 1973. b Estimated from Missenard, 1965.



Figure 3. Heat capacity of MEA.

atures from 30 to 80 °C are in good agreement with the C_p values of Osborne et al. (1939). On the basis of comparison with literature values for water, the accuracy of the C_p measurements is estimated to be $\pm 2\%$.

The C_p values of eight alkanolamines were then measured for temperatures ranging from 30 to 80 °C. The measured C_p values of MEA along with the literature values are presented in Table 2 and are also shown in Figure 3. The C_p data of Riddick et al. (1986), Lee (1994), and Lide (1995) are also presented. For the purpose of

Table 5. Heat Capacities of Di

		heat capacity,	C _p /(kJ·kg	g ^{−1} •K ^{−1})	
temp (°C)	est value	Riddick et al., 1986	Lee, 1994	Lide, 1995	this study
20 25	2.388^{a} 2.393^{b}				
30		2.220	2.438	2.221	2.42
35			2.469		2.45
40			2.500		2.48
45			2.531		2.51
50			2.562		2.53
55			2.594		2.56
60			2.625		2.59
65			2.656		2.62
70			2.687		2.64
75			2.718		2.67
80			2.750		2.70

 a Estimated from Chueh and Swanson, 1973. b Estimated from Missenard, 1965.

comparison, C_p values for MEA are estimated from the group contribution methods of Missenard (1965) and Chueh and Swanson (1973). Due to the limited temperature range of functional group parameters, the calculated C_p from the group contribution method may only apply to a limited temperature range. For the C_p of MEA, there is an appreciable difference among different data sources, as shown in Figure 3. For 20-30 °C, the literature values for the C_p of MEA show quite inconsistent results; that is, the values of C_p vary from 2.082 kJ·kg⁻¹·K⁻¹ at 30 °C (Riddick et al., 1986) to 2.720 kJ·kg⁻¹·K⁻¹ at 30 °C (Lee, 1994) and to 3.201 kJ·kg⁻¹·K⁻¹ at 25 °C (Lide, 1995). The C_p value of Lide (1995) appears to be too large while the C_p value of Riddick et al. (1986) appears to be too small. For the temperatures 30 °C to 50 °C, the C_p values of MEA measured in this study are in good agreement with those of Lee (1994); the average deviation is only 0.9%. The temperature dependence of the C_p values of MEA obtained in this work is weaker than that of Lee (1994), as shown in Figure 3. The estimated values from both group contribution methods appear to be consistent with the results of both Lee (1994) and the present study. For temperatures from 20 °C to 45 °C, the values of the C_p of MEA obtained by Missenard (1965), Chueh and Swanson (1973), Lee (1994), and the present study are in reasonable agreement.

The measured C_p values of DEA are presented in Table 3 and shown in Figure 4. At 30 °C, the C_p values of DEA of both Riddick et al. (1986) (2.220 $kJ\cdot kg^{-1}\cdot K^{-1}$) and Lide (1995) (2.221 kJ·kg⁻¹·K⁻¹) are lower than that of Lee (1994) $(2.438 \text{ kJ} \cdot \text{kg}^{-1} \cdot \text{K}^{-1})$. The temperature dependence of the $C_{\rm p}$ of DEA obtained in this study is only slightly weaker than that of Lee (1994). The C_p values of DEA obtained in this study are in good agreement with those of Lee (1994); the average percentage deviation is 1.3%. As shown in Figure 4, the estimated values from both group contribution methods, Missenard (1965) at 25 °C and Chueh and Swanson (1973) at 20 °C, are in good agreement with those of the present study. The $C_{\rm p}$ values of Chueh and Swanson (1973) at 20 °C, Missenard (1965) at 25 °C, the data of this study, and Lee (1994) are recommended for the $C_{\rm p}$ values of DEA for temperatures ranging from 20 to 80 °C.

The results for the C_p values of DGA are presented in Table 4 and shown in Figure 5. There are no experimental data available for the C_p values of DGA. The estimated C_p values from the group contribution methods, Chueh and Swanson (1973) at 20 °C and Missenard (1965) at 25 and 50 °C (Missenard, 1965) are listed for comparison. As shown in Figure 5, at 50 °C the difference of the C_p value



Figure 4. Heat capacity of DEA.

Table 4. Heat Capacities of DGA and DIPA

	h	eat capacity, C	∽p /(kJ·kg ⁻¹ ·K	-1)
temp	D	GA	D	IPA
(°C)	est value	this study	est value	this study
20	2.454 ^a		2.481 ^a	
25	2.370^{b}		2.464^{b}	
30		2.48		
35		2.50		
40		2.52		
45		2.54		
50	2.527^{b}	2.55		2.78
55		2.57		2.81
60		2.59		2.84
65		2.61		2.87
70		2.62		2.90
75		2.64		2.93
80		2.66		2.96

^{*a*} Estimated from Chueh and Swanson, 1973. ^{*b*} Estimated from Missenard, 1965.



Figure 5. Heat capacity of DGA.

of DGA between this study (2.55 kJ·kg⁻¹·K⁻¹) and that of Missenard (2.527 kJ·kg⁻¹·K⁻¹) is only 1.0%. The estimated $C_{\rm p}$ from Chueh and Swanson (1973) at 20 °C for DGA appears to be in good agreement with the present study for 30–80 °C, as shown in Figure 5.



Figure 6. Heat capacity of DIPA.

Table 5. Heat Capacities of TEA

		heat capac	ity, C _p /(kJ·k	$g^{-1}\cdot K^{-1}$)	
temp (°C)	est value	Minadakis and Sabbah, 1982	Riddick et al., 1986	Lee, 1994	Lide, 1995	this study
20	2.291 ^a					
25	2.073 ^{<i>b</i>}	2.61			2.607	
30			2.078	2.365		2.43
35				2.382		2.44
40				2.398		2.45
45				2.415		2.46
50				2.432		2.47
55				2.448		2.48
60				2.465		2.49
65				2.482		2.50
70				2.498		2.51
75				2.515		2.52
80				2.532		2.53

 a Estimated from Chueh and Swanson, 1973. b Estimated from Missenard, 1965.

The results for the C_p of DIPA are also presented in Table 4 and shown in Figure 6. Since the melting point of DIPA is 45.5 °C, the C_p measurement for DIPA starts at 50 °C. The C_p values of DIPA estimated from group contribution methods, Chueh and Swanson (1973) at 20 °C and Missenard (1965) at 25 °C, can only be considered as hypothetical liquid state values. As mentioned earlier, the group contribution methods only apply to a limited temperature range. As shown in Figure 6, the C_p values of DIPA obtained in this study reasonably approach the C_p values at 25 and 20 °C, estimated from the group contribution methods (Missenard, 1965; Chueh and Swanson, 1973).

The C_p values of TEA obtained in this study along with the literature values are presented in Table 5 and shown in Figure 7. As shown in Figure 7, there is an appreciable difference for the C_p data among Minadakis and Sabbah (1982), Riddick et al. (1986), Lee (1994), and Lide (1995). The estimated values for the C_p of TEA from two methods, Chueh and Swanson (1973) at 20 °C (2.291 kJ·kg⁻¹·K⁻¹) and Missenard (1965) at 25 °C (2.073 kJ·kg⁻¹·K⁻¹), are also inconsistent. The C_p values of TEA obtained in this study are in good agreement with those of Lee (1994); the average percentage deviation is 1.3%. For the C_p of TEA, both group contribution methods (Missenard, 1965; Chueh and Swanson, 1973) yield poor results, as shown in Figure 7.



Figure 7. Heat capacity of TEA.

Table 6. Heat Capacities of MDEA

	heat ca	pacity, <i>C</i> _p /(kJ·k	$g^{-1} \cdot K^{-1}$)
temp (°C)	est value	Lee, 1994	this study
20	2.308 ^a		
25	2.103^{b}		
30		2.274	2.22
35		2.303	2.24
40		2.333	2.27
45		2.363	2.30
50		2.392	2.33
55		2.422	2.36
60		2.452	2.39
65		2.482	2.41
70		2.511	2.44
75		2.541	2.47
80		2.571	2.50

 a Estimated from Chueh and Swanson, 1973. b Estimated from Missenard, 1965.



Figure 8. Heat capacity of MDEA.

The measured C_p values of MDEA are presented in Table 6 and shown in Figure 8. The measured C_p values of MDEA are slightly smaller (about 2.7%) than those of Lee (1994). The C_p values obtained in this study appear to be more consistent with the predicted value from Missenard (1965) at 25 °C than those of Lee (1994). The C_p value estimated

Table 7. Heat Capacities of AMP and 2-PE

	$C_{\rm p}/({\rm kJ}\cdot{\rm kg}^{-1}\cdot{\rm K}^{-1})$			
temp	A	MP	2-	PE
(°C)	est value	this study	est value	this study
20	2.387 ^a		2.245 ^a	
25	2.541^{b}		2.237^{b}	
30		2.68		2.27
35		2.71		2.29
40		2.74		2.32
45		2.77		2.35
50	2.734^{b}	2.80		2.38
55		2.83		2.41
60		2.86		2.44
65		2.88		2.47
70		2.91		2.49
75		2.94		2.52
80		2.97		2.55

 a Estimated from Chueh and Swanson, 1973. b Estimated from Missenard, 1965.



Figure 9. Heat capacity of AMP.

from Chueh and Swanson (1973) at 20 °C appears to be too large compared to the the C_p values of both Lee (1994) and the present study.

The heat capacities of AMP obtained in this study are presented in Table 7. As shown in Figure 9, at 50 °C the difference of the C_p values of AMP between this study (2.80 kJ·kg⁻¹·K⁻¹) and that of Missenard (2.734 kJ·kg⁻¹·K⁻¹) is 2.4%. As shown in Figure 9, at the temperatures 20 and 25 °C, both C_p estimation methods (Missenard, 1965; Chueh and Swanson, 1973) yield poor results compared to the measured C_p values.

The heat capacities of 2-PE obtained in this study are also presented in Table 7. As shown in Figure 10, the measured C_p values are in excellent agreement with the C_p values estimated from group contribution methods (Missenard, 1965; Chueh and Swanson, 1973).

In Figure 11, the values of C_p for eight alkanolamines are shown. Among the eight alkanolamines, MEA has the largest values of C_p and MDEA has the smallest values of C_p . AMP is observed to have the strongest temperature dependence of C_p while TEA has the weakest temperature dependence of C_p . The values of C_p of DEA and DGA cross each other in the neighborhood of 60 °C. At 40 °C, the values of C_p for amines are in the following order: MEA (2.78 kJ·kg⁻¹·K⁻¹) > AMP (2.74 kJ·kg⁻¹·K⁻¹) > DGA (2.52 kJ·kg⁻¹·K⁻¹) > DEA (2.48 kJ·kg⁻¹·K⁻¹) > TEA (2.45



Figure 10. Heat capacity of 2-PE.



Figure 11. Heat capacity of alkanolamines.

 $kJ{\cdot}kg^{-1}{\cdot}K^{-1})>2{\cdot}PE$ (2.32 $kJ{\cdot}kg^{-1}{\cdot}K^{-1})>MDEA$ (2.27 $kJ{\cdot}kg^{-1}{\cdot}K^{-1}).$

For the purpose of application, the measured C_p values of eight alkanolamines are expressed as a function of temperature as follows

$$C_{\rm p}/(\rm kJ\cdot \rm kg^{-1}\cdot \rm K^{-1}) = a + b(T/\rm K)$$
(2)

The parameters *a* and *b* of C_p for each amine are presented in Table 8. Using eq 2, the average deviation of the calculations is less than 1.0% due to the nearly linear dependence of C_p on temperature.

Conclusion

The heat capacities of eight alkanolamines (MEA, DEA, DGA, DIPA, TEA, MDEA, AMP, and 2-PE) were measured from 30 to 80 °C by using a differential scanning calorimeter (DSC). The C_p of liquid water was also measured to justify the accuracy of the DSC. The measured C_p of each

Table 8. Parameters of Heat Capacity of Amines

a ^a	$10^{3}b^{a}$
1.270 22	4.8364
0.747 14	5.5273
1.413 43	3.5273
0.841 10	6.0000
1.823 70	2.0000
0.501 73	5.6546
0.940 99	5.7455
0.540 71	5.6909
	<i>a^a</i> 1.270 22 0.747 14 1.413 43 0.841 10 1.823 70 0.501 73 0.940 99 0.540 71

^{*a*} $C_{\rm p}/({\rm kJ}\cdot{\rm kg}^{-1}\cdot{\rm K}^{-1}) = a + b$ (*T*/K).

alkanolamine was expressed as a function of temperature. The estimated uncertainty of the measured heat capacities including the effect of impurities in a substance with a purity of 95% was \pm 3%. The measured heat capacities of alkanolamines were, in general, of sufficient accuracy to be satisfactory for most engineering-design calculations.

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