Thermodynamic investigation of room temperature ionic liquid: The heat capacity and standard enthalpy of formation of EMIES

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Abstract

The molar heat capacities of the room temperature ionic liquid 1-ethyl-3-methylimidazolium ethyl sulfate (EMIES) were measured by an adiabatic calorimeter in temperature range from 78 to 390 K. The dependence of the molar heat capacity on temperature was given as a function of the reduced temperature X by polynomial equations, $C_{p,m}$ (J K^{-1} mol^{-1}) = 178.6 + 50.28X + 2.886X^2 − 1.362X^3 + 0.6616X^4 + 7.155X^5 [$X = (T − 132.5)/54.5$] for the solid phase (78–187 K) and $C_{p,m}$ (J K^{-1} mol^{-1}) = 376.2 + 25.94X − 3.397X^2 − 0.6407X^3 + 0.8091X^4 + 0.9869X^5 [$X = (T − 292.5)/97.5$] for the liquid phase (195–390 K), respectively. According to the polynomial equations and thermodynamic relationship, the values of thermodynamic function of the EMIES relative to 298.15 K were calculated in temperature range from 80 to 390 K with an interval of 5 K. The glass translation of EMIES was observed at 192.85 K. Using oxygen-bomb combustion calorimeter, the molar enthalpy of combustion of EMIES was determined to be $\Delta_c H_m^\circ = −5152.6 \pm 4.6$ kJ mol^{-1}. The standard molar enthalpy of formation of EMIES was evaluated to be $\Delta_f H_m^\circ = −579.13 \pm 0.51$ kJ mol^{-1} at $T = 298.150 \pm 0.001$ K.
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1. Introduction

Room temperature ionic liquids (RTILs) have emerged as a new kind of media alternative to the conventionally used organic solvents, which are generally volatile, flammable, and hazardous chemicals [1–3]. Even though ionic liquids such as [EtNH$_3$][NO$_3$], which have a melting point of 285 K, have been known since 1914 [4], it is only very recently that these systems have been explored as a new kind of media for synthetic electrochemical and catalytic applications [5–11]. Some of the properties that make the room temperature ionic liquids attractive media for various applications are the wide liquid range, non-volatility (negligible vapor pressure), non-flammable nature, less reactivity and the ability to dissolve a large variety of organic and inorganic substances including even the poly-mer materials in high concentration. Many of these properties have made the room temperature ionic liquids a nature-friendly "Green Solvent" [12]. The room temperature ionic liquids that are currently the focus of extensive investigation are generally a substituted imidazolium or a pyridinium salt. Even though the solvent properties of different halogenoaluminate (III) ionic liquids (ILs) were studied as early as in 1986 [13], very little is known about the various properties of the contemporary ionic liquids despite their extensive usage in synthetic applications. We refer, for instance, to that data of heat capacity, standard enthalpy of formation, thermodynamic functions and so on, which are paramount for the design of any technological processes, are even scarcer [14].

As a continuation of our previous investigations of RTILs [15–18], in the present study, the molar heat capacities of EMIES were measured by an adiabatic calorimeter in temperature range from 78 to 390 K. Based on the measured heat capacity data and thermodynamic relationship, the values of thermodynamic function of the EMIES relative to 298.15 K were calculated. A

glass translation of EMIES was observed at 192.85 K. Through oxygen-bomb combustion calorimetry, the molar enthalpies of combustion of EMIES were determined and the standard molar enthalpy of formation was further evaluated.

2. Experimental

2.1. Chemicals

Diethyl sulfate (Academy of Military Medical Sciences, normal mass fraction purity, AR) was used as supplied, 1-methylimidazole (ACROS, normal mass fraction purity, AR) was vacuum distilled prior to use. Toluene (Tianjing Chemical Agent Factory, normal mass fraction purity, AR) was AR reagent and was purified by standard method. The 39i benzoic acid was purchased from NIST.

2.2. Synthesis of EMIES

1-Ethyl-3-methylimidazolium ethyl sulfate as a new ionic liquid was prepared through alkylation of 1-methylimidazole with diethyl sulfate in toluene as an inert solvent according to the following reaction scheme [19]:

$$H_3C-N\diagdown N + (C_2H_5)_2SO_4 \rightarrow H_3C-N\diagdown\oplus N-C_2H_5 + C_2H_5OSO_3^-$$
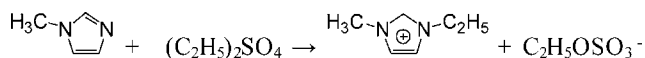
Diethyl sulfate was added dropwise at a rate to a solution of equimolar 1-methylimidazole in toluene cooled in an ice-bath to maintain the reaction temperature below 313.15 K. The IL product immediately formed and caused the initially clear solution to become opaque, followed by biphasic separation of the toluene solution and formation of a denser IL phase. After addition of the diethyl sulfate, the reaction mixture was stirred at room temperature for 2 h. The upper organic phase was decanted and the lower IL phase was washed with toluene thrice, dried with heating at 348.15 K under reduced pressure to remove residual organic solvents, and finally in vacuo to yield the resulting EMIES as a colorless ionic liquid. ^{1}H NMR and ^{13}C NMR spectra of the product showed the same with literature [18]. The electrochemical window of EMIES was measured by cyclic voltammetry at 303.15 K. The result showed reductive and oxidative limits of −1.40 and 2.40 V, respectively, relative to a Al/Al^{3+} reference, which gives an electrochemical potential window of 3.8 V which was the same with literature [19].

2.3. Heat capacity measurement

Heat capacity measurements were carried out in a high-precision automatic adiabatic calorimeter described in detail elsewhere [20,21]. The principle of the calorimeter is based on the Nernst stepwise method. The calorimeter mainly consists of a sample cell, an adiabatic (or inner) shield, a guard (outer) shield, a platinum resistance thermometer, an electric heater, two sets of chromel–copper thermocouples and a high vacuum can. The sample cell was made of gold-plated copper and had an inner volume of 48 cm^3. Eight gold-plated copper vanes of 0.2 mm thickness were put into the cell to promote heat distri-

bution between the sample and the cell. The platinum resistance thermometer was kept within ca. 1×10^{-3} Pa during the heat capacity measurements so as to eliminate the heat loss due to gas convection. Liquid nitrogen was used to detect the temperature difference between them was kept to be 0.5 mK during the whole experimental process.

One set of chromel–copper thermocouples was used to detect the temperature difference between the sample cell and the inner shield. Likewise, the other set of thermocouples was installed between the inner and outer shields. The temperature difference between them was kept to within 0.5 mK during the whole experimental process. The sample cell was heated by the standard discrete heating method. The temperature of the cell was alternatively measured by a platinum resistance thermometer which was made by the Instrument Manufactory of Yunnan, China, and calibrated at the National Institute of Metrology in terms of the IPTS-90. The temperature increment in a heating period was 2–4 K, and temperature drift was maintained at about 10^{-3} K min^{-1} in equilibrium period. All the data were automatically acquired through a data acquisition/switch unit (Model: 34970A, Aglient, USA) and processed by a computer.

The sample mass used for the heat capacity measurement was 49.3271 g. To verify the reliability of the adiabatic calorimeter, molar heat capacities for the reference standard material α-Al$_2$O$_3$, water, ethanol and benzene were measured. The deviations of our experimental results from the values reported by NIST [22] were within ±0.2% in the temperature range of 80–400 K for α-Al$_2$O$_3$. The enthalpy and entropy of the phase transitions determined in our laboratory are in excellent agreement with the data cited from literatures [23–26].

2.4. Combustion enthalpy measurement

Combustion calorimetric experiment was carried out in a precision oxygen-bomb calorimeter described in detail elsewhere [27,28]. The energy equivalent, ε_{calor}, of the calorimeter has been determined from 10 combustion experiments using about 0.7 g of NIST 39i benzoic acid with a certified mass energy of combustion under experimental conditions of $\Delta_c U = -26434 \pm 3$ J g^{-1} to be: $\varepsilon_{calor} = 13572.22 \pm 0.98$ J K^{-1}. The uncertainty of the results was the standard deviation of the mean value from the respective measurements.

The condition for determining the constant-volume combustion energy of EMIES was the same as that used in the calibration of the calorimeter with benzoic acid. The constant-volume combustion energy of the sample can be calculated from the following equation:

$$\Delta_c U \,(\text{J mol}^{-1}) = (\varepsilon_{calor}\Delta T - Q_{Ni} - Q_{HNO_3})\frac{M}{W} \tag{1}$$

in which ε_{calor} (J K^{-1}) was the energy equivalent of the oxygen-bomb calorimeter, ΔT (K) the corrected temperature rise, M (g mol^{-1}) the molar mass of the sample and W (g) was the mass of the sample. The calculated results of the constant-volume combustion energy of the sample were listed in Table 1.

W (g)	Q_{Ni} (J)	Q_{HNO_3} (J)	ΔT (K)	$-\Delta_c E$ (kJ mol^{-1})
2.0016	40.710	22.766	3.221	5143.26
1.0066	43.639	38.566	1.626	5151.47
1.0029	41.882	36.110	1.621	5155.51

W (g), the mass of the sample; ΔT (K), the corrected temperature rise; $-\Delta_c E$ (J g^{-1}), constant-volume combustion energy.

3. Results and discussion

3.1. Molar heat capacity of EMIES

The molar heat capacities of the EMIES were determined by using the adiabatic calorimeter in temperature range from 78 to 390 K. The results of the molar heat capacities are listed in Table 2 and shown in Fig. 1. No thermal anomaly was observed or no phase transition took place in temperature range from 78 to 187 K for solid phase and from 195 to 390 K for liquid phase, respectively.

In order to fit heat capacity data as function of temperature well, the temperature T was replaced by the reduced temperature X which was defined:

$$X = \frac{T - 0.5(T_{max} + T_{min})}{0.5(T_{max} - T_{min})} \quad (2)$$

where T is thermodynamic temperature and T_{max} and T_{min} the maximum and the minimum of the temperature in the experimental temperature range. Then, $-1 \leq X \leq 1$.

The values of molar heat capacities of the EMIES were fitted to the following polynomial expressions with least square method.

For the solid phase (78–187 K):

$$C_{p,m} \, (\text{J K}^{-1} \, \text{mol}^{-1}) = 178.6 + 50.28X + 2.886X^2$$
$$- 1.362X^3 + 0.6616X^4 + 7.155X^5 \quad (3)$$
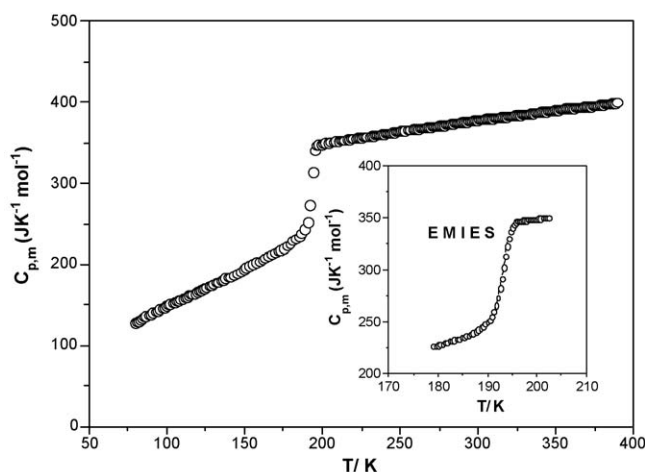
where reduced temperature, $X = (T - 132.5)/54.5$.

Fig. 1. Molar heat capacity of ionic liquid EMIES.

The above equation is valid from 78 to 187 K, with an uncertainty of ±0.2%. The correlation coefficient of the fitted curve, $R^2 = 0.9998$.

For the liquid phase (195–390 K):

$$C_{p,m} \, (\text{J K}^{-1} \, \text{mol}^{-1}) = 376.2 + 25.94X - 3.397X^2 - 0.6407X^3$$
$$+ 0.8091X^4 + 0.9869X^5 \quad (4)$$

where reduced temperature $X = (T - 292.5)/97.5$. The above equation is valid from 195 to 390 K with an uncertainty of ±0.2%. The correlation coefficient of the fitted curve, $R^2 = 0.9998$.

3.2. Thermodynamic data of glass transition

It can be seen from Fig. 1 that the heat capacity jump, corresponding to the glass transition of the EMIES, took place in the range from 188 to 195 K. The temperature of the glass transition, T_g, was determined to be 192.85 K.

3.3. Thermodynamic functions of ionic liquid EMIES

Thermodynamic functions of EMIES were calculated based on the empirical Eqs. (3) and (4), and the relationships of the thermodynamic functions:

$$H_T - H_{298.15} = \int_{298.15}^{T} C_{p,m} \, dT \quad (5)$$
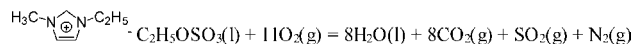
$$S_T - S_{298.15} = \int_{298.15}^{T} \frac{C_{p,m}}{T} \, dT \quad (6)$$

The thermodynamic function data relative to the reference temperature (298.15 K) were calculated by Eqs. (5) and (6) in the temperature range from 80 to 390 K with an interval of 5 K and listed in Table 3.

3.4. The standard molar enthalpy of formation for EMIES

The constant-volume combustion energy of EMIES was determined at $T = 298.150 \pm 0.001$ K, $\Delta_c U_m = -5150.1 \pm 4.6$ kJ mol^{-1}.

The standard molar enthalpy of combustion of EMIES, $\Delta_c H_m^\circ$, referred to the combustion enthalpy change of the following reaction at $P^\circ = 100$ kPa, based on the definition of the combustion enthalpy about the organic compound:

$$\text{H}_3\text{C-N} \overset{\frown}{\underset{\oplus}{\bigcirc}} \text{N-C}_2\text{H}_5 \cdot \text{C}_2\text{H}_5\text{OSO}_3(l) + 11\text{O}_2(g) = 8\text{H}_2\text{O}(l) + 8\text{CO}_2(g) + \text{SO}_2(g) + \text{N}_2(g)$$

The standard molar enthalpies of combustion can be derived from the constant-volume combustion energy by means of the following formula:

$$\Delta_c H_m^\circ = \Delta_c U_m + \Delta n RT, \quad (7)$$

in which $\Delta n = \Sigma n_i$ (products, g)–Σn_i (reactants, g), where Σn_i was the total molar amount of the gases in products or reactants.

Table 2
The values of molar heat capacity of ionic liquid EMIES ($M = 236.29 \, \text{g mol}^{-1}$)

T (K)	C_p (J K^{-1} mol^{-1})	T (K)	C_p (J K^{-1} mol^{-1})	T (K)	C_p (J K^{-1} mol^{-1})
79.23	127.8	206.90	351.5	303.64	379.3
79.70	128.6	208.47	351.9	304.74	379.6
80.51	129.4	210.06	352.4	305.86	379.9
81.67	130.9	211.66	352.8	306.96	380.1
82.84	131.5	213.18	353.2	308.07	380.3
83.99	133.2	214.52	353.7	309.18	380.4
85.31	135.2	215.86	354.1	310.29	380.8
86.78	136.3	217.18	354.5	311.40	380.9
88.25	137.9	218.50	354.9	312.52	381.2
89.70	138.8	219.81	355.4	313.63	381.6
91.13	141.2	221.12	355.7	314.75	381.8
92.56	142.2	222.41	356.1	315.85	382.0
93.97	143.4	223.71	356.5	316.97	382.3
95.39	145.2	225.00	356.8	318.08	382.7
96.79	146.4	226.27	357.1	319.19	383.0
98.17	147.5	227.54	357.5	320.31	383.1
99.54	149.6	228.79	357.8	321.42	383.4
100.91	151.0	230.03	358.1	322.53	383.6
102.27	151.6	231.26	358.4	323.66	383.9
103.61	152.9	232.46	359.0	324.75	383.9
104.96	154.3	233.66	359.3	325.84	384.4
106.29	155.3	234.84	359.6	326.92	384.6
107.62	155.8	236.02	359.8	327.99	384.8
108.95	157.3	237.19	360.2	329.08	384.9
110.26	158.3	238.37	360.5	330.17	385.1
111.57	159.1	239.54	361.0	331.26	385.6
112.87	160.6	240.70	361.3	332.35	385.9
114.17	162.4	241.86	361.5	333.43	386.1
115.46	163.1	243.01	361.9	334.52	386.5
116.74	163.6	244.15	362.2	335.60	386.6
118.02	165.2	245.29	362.5	336.68	386.9
119.29	166.8	246.43	363.1	337.77	387.3
120.55	167.9	247.58	363.5	338.86	387.4
121.82	168.9	248.73	363.9	339.94	387.8
123.06	170.1	249.88	364.2	341.04	388.1
124.32	170.7	251.02	364.5	342.13	388.3
125.55	172.1	252.17	364.8	343.22	388.6
126.80	173.9	253.31	365.5	344.31	388.9
128.22	174.7	254.45	365.8	345.40	389.2
129.84	176.7	255.60	366.1	346.49	389.5
131.45	178.2	256.73	366.3	347.58	389.7
133.04	178.9	257.87	366.8	348.65	390.0
134.63	180.0	259.00	367.1	349.73	390.2
136.21	181.3	260.13	367.4	350.82	390.5
137.77	183.2	261.27	367.6	351.91	390.8
139.34	184.6	262.40	367.8	353.01	391.3
140.89	185.1	263.53	368.0	354.11	391.6
142.73	187.4	264.66	368.3	355.22	392.0
144.86	189.6	265.80	368.7	356.31	392.3
146.97	191.6	266.93	369.1	357.42	392.5
149.07	194.6	268.06	369.4	358.53	392.7
151.16	196.7	269.19	369.5	359.63	392.8
153.21	198.7	270.33	369.9	360.75	392.9
155.27	200.9	271.45	370.3	361.87	393.1
157.30	202.8	272.58	370.8	363.00	393.4
159.33	203.6	273.71	371.0	364.13	393.5
161.34	205.9	274.83	371.2	365.29	393.6
163.34	208.6	275.94	371.5	366.38	393.9
165.33	210.2	277.06	371.8	367.47	394.1
167.30	212.6	278.18	372.0	368.52	394.5
169.27	214.9	279.28	372.4	369.63	394.7
171.23	216.9	280.39	373.0	370.76	394.9
173.18	218.1	281.50	373.1	371.78	395.1
175.11	220.4	282.62	373.5	372.82	395.3

Table 2 (*Continued*)

T (K)	C_p (J K^{-1} mol^{-1})	T (K)	C_p (J K^{-1} mol^{-1})	T (K)	C_p (J K^{-1} mol^{-1})
177.05	223.2	283.72	373.8	373.90	395.6
178.98	226.0	284.83	374.2	374.98	395.8
180.91	229.3	285.95	374.5	376.02	396.2
182.83	231.6	287.05	374.7	377.08	396.5
184.74	234.3	288.15	375.0	378.13	396.8
186.65	238.0	289.26	375.4	379.52	397.1
188.56	243.9	290.37	375.6	380.42	397.3
190.42	251.8	291.47	376.0	381.38	397.5
192.11	273.4	292.58	376.1	382.35	397.8
193.57	313.7	293.69	376.5	383.43	398.0
194.94	340.7	294.79	376.8	384.34	398.2
196.36	346.8	295.90	377.0	385.25	398.5
197.81	347.7	297.00	377.4	386.14	398.8
199.27	348.4	298.11	378.0	387.09	399.1
200.76	349.4	299.22	378.2	388.01	399.5
202.25	349.9	300.32	378.6	388.98	399.7
203.79	350.5	301.43	378.9	389.95	399.9
205.33	350.9	302.54	379.1		

Table 3
The data of the thermodynamic functions of EMIES

T (K)	C_p (J K^{-1} mol^{-1})	$H_T-H_{298.15}$ (kJ mol^{-1})	$S_T-S_{298.15}$ (J K^{-1} mol^{-1})
80	128.6	−102.3	−248.8
85	134.6	−101.1	−243.2
90	139.9	−100.0	−237.9
95	144.8	−98.83	−232.9
100	149.4	−97.65	−228.1
105	153.9	−96.43	−223.6
110	158.3	−95.18	−219.1
115	162.7	−93.88	−214.7
120	167.1	−92.53	−210.4
125	171.6	−91.13	−206.0
130	176.2	−89.68	−201.5
135	180.8	−88.17	−197.0
140	185.5	−86.62	−192.3
145	190.2	−85.01	−187.5
150	194.9	−83.34	−182.6
155	199.8	−81.62	−177.5
160	204.7	−79.83	−172.4
165	209.9	−77.96	−167.3
170	215.3	−75.99	−162.3
175	221.1	−73.88	−157.4
180	227.6	−71.61	−152.8
185	234.9	−69.12	−148.6
190	243.3	−66.35	−145.0
192.85	Glass transition		
195	347.2	−44.87	−30.44
200	348.9	−42.80	−28.74
205	350.5	−40.73	−27.10
210	352.1	−38.64	−25.48
215	353.7	−36.54	−23.90
220	355.2	−34.43	−22.35
225	356.8	−32.31	−20.82
230	358.3	−30.17	−19.30
235	359.8	−28.03	−17.81
240	361.3	−25.87	−16.32
245	362.8	−23.70	−14.85
250	364.2	−21.52	−13.39
255	365.7	−19.33	−11.94
260	367.1	−17.13	−10.51

Table 3 (*Continued*)

T (K)	C_p (J K^{-1} mol^{-1})	$H_T-H_{298.15}$ (kJ mol^{-1})	$S_T-S_{298.15}$ (J K^{-1} mol^{-1})
265	368.5	−14.91	−9.08
270	370.0	−12.69	−7.67
275	371.3	−10.46	−6.27
280	372.7	−8.21	−4.89
285	374.1	−5.96	−3.52
290	375.4	−3.70	−2.16
295	376.8	−1.43	−0.83
298.15	377.6	0.00	0.00
300	378.1	0.84	0.48
305	379.4	3.12	1.78
310	380.7	5.42	3.06
315	381.9	7.72	4.31
320	383.1	10.02	5.55
325	384.4	12.33	6.77
330	385.6	14.65	7.97
335	386.8	16.98	9.15
340	387.9	19.31	10.32
345	389.1	21.66	11.47
350	390.3	24.01	12.62
355	391.4	26.37	13.76
360	392.6	28.74	14.91
365	393.7	31.13	16.06
370	394.9	33.53	17.22
375	396.1	35.95	18.41
380	397.3	38.39	19.64
385	398.5	40.85	20.90
390	399.8	43.34	22.23

The calculated standard molar enthalpy of combustion of EMIES was: $\Delta_c H_m^\circ = -5152.6 \pm 4.6 \text{ kJ mol}^{-1}$.

The standard molar enthalpy of formation of the EMIES, $\Delta_f H_m^\circ$, was calculated by a designed Hess thermochemical cycle according to the reaction as follows:

$$\Delta_f H_m^\circ = [8\Delta_{f,CO_2(g)} H_m^\circ + 8\Delta_{f,H_2O(l)} H_m^\circ + \Delta_{f,SO_2(g)} H_m^\circ]$$
$$- \Delta_c H_m^\circ \tag{8}$$

In the above formula, the standard molar enthalpies of formation for CO_2 (g) and H_2O (l), were recommended by CODATA [29,30], $\Delta_f H_m^{\circ}$ (CO_2, g) $= -393.51 \pm 0.13\,kJ\,mol^{-1}$, $\Delta_f H_m^{\circ}$ (H_2O, l) $= -285.83 \pm 0.04\,kJ\,mol^{-1}$, and the standard molar enthalpy of formation for (SO_2, l) was recommended, $\Delta_{f,SO_2(g)} H_m^{\circ} = -296.97\,kJ\,mol^{-1}$ [31], were employed in the calculation of $\Delta_f H_m^{\circ}$ (EMIES, l) values. The standard molar enthalpy of formation of the EMIES can be derived, based on these values, to be: $\Delta_f H_m^{\circ}$ (EMIES, l) $= -579.13 \pm 0.51\,kJ\,mol^{-1}$.

Acknowledgements

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