Journal of Thermal Analysis and Calorimetry, Vol. 89 (2007) 1, 289–294

THERMODYNAMIC INVESTIGATION OF ROOM TEMPERATURE IONIC LIQUID
Heat capacity and thermodynamic functions of $BPBF_4$

Z. H. Zhang[1,2], *L. X. Sun*[1*], *Z. C. Tan*[1], *F. Xu*[1], *X. C. Lv*[1,2], *J. L. Zeng*[1,2] *and Y. Sawada*[3]

[1]Materials and Thermochemistry Laboratory, Dalian Institute of Chemical Physics, Chinese Academy of Sciences
Dalian 116023, P.R. China
[2]Graduate School of the Chinese Academy of Sciences, P.R. China
[3]Department of Nanochemistry, Faculty of Engineering, Tokyo Polytechnic University, 1583 Iiyama, Atsugi, Kanagawa 243-0297, Japan

The molar heat capacities of the room temperature ionic liquid 1-butylpyridinium tetrafluoroborate ($BPBF_4$) were measured by an adiabatic calorimeter in temperature range from 80 to 390 K. The dependence of the molar heat capacity on temperature is given as a function of the reduced temperature X by polynomial equations, $C_{p,m}$ [J K^{-1} mol^{-1}]=$181.43+51.297X-4.7816X^2-1.9734X^3+8.1048X^4+11.108X^5$ [$X=(T-135)/55$] for the solid phase (80–190 K), $C_{p,m}$ [J K^{-1} mol^{-1}]= $349.96+25.106X+9.1320X^2+19.368X^3+2.23X^4-8.8201X^5$ [$X=(T-225)/27$] for the glass state (198–252 K), and $C_{p,m}$ [J K^{-1} mol^{-1}]= $402.40+21.982X-3.0304X^2+3.6514X^3+3.4585X^4$ [$X=(T-338)/52$] for the liquid phase (286–390 K), respectively. According to the polynomial equations and thermodynamic relationship, the values of thermodynamic function of the $BPBF_4$ relative to 298.15 K were calculated in temperature range from 80 to 390 K with an interval of 5 K. The glass transition of $BPBF_4$ was observed at 194.09 K, the enthalpy and entropy of the glass transition were determined to be ΔH_g=2.157 kJ mol^{-1} and ΔS_g=11.12 J K^{-1} mol^{-1}, respectively. The result showed that the melting point of the $BPBF_4$ is 279.79 K, the enthalpy and entropy of phase transition were calculated to be ΔH_m=8.453 kJ mol^{-1} and ΔS_m=30.21 J K^{-1} mol^{-1}. Using oxygen-bomb combustion calorimeter, the molar enthalpy of combustion of $BPBF_4$ was determined to be $\Delta_c H_m^0=-5451\pm3$ kJ mol^{-1}. The standard molar enthalpy of formation of $BPBF_4$ was evaluated to be $\Delta_f H_m^0=-1356.3\pm0.8$ kJ mol^{-1} at T=298.150±0.001 K.

Keywords: *$BPBF_4$, combustion calorimetry, glass transition, heat capacity, room temperature ionic liquid, phase transition, standard enthalpy of formation*

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 liquid such as $[EtNH_3][NO_3]$, which has a melting point of 285 K, has been known since 1914 [4]. It is only very recently that these systems as a new kind of media for synthetic electrochemical and catalytic applications have been explored [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 polymer 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 and these data are very scarce [14].

As a continuation of previous investigations of RTILs [15–18], in the present study the molar heat capacities of RTIL, $BPBF_4$ were measured by an adiabatic calorimeter in temperature range from 80 to 390 K. Based on the measured heat capacity data and thermodynamic relationship, the values of thermodynamic function of the $BPBF_4$ relative to 298.15 K were calculated. The glass transition of $BPBF_4$ was observed at 176.24 K, the enthalpy and entropy of the transition were calculated. The melting point of the $BPBF_4$ is 279.79 K, the enthalpy and entropy of its phase transition were calculated, too. The molar enthalpies of combustion of $BPBF_4$ were determined by oxygen-bomb combustion calorimetry. The standard molar enthalpy of formation was further evaluated.

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[*] Author for correspondence: lxsun@dicp.ac.cn

1388–6150/$20.00

Akadémiai Kiadó, Budapest, Hungary
Springer, Dordrecht, The Netherlands

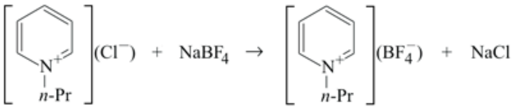
Experimental

Chemicals

Ethylacetate (AR) and 1-chlorobutane (AR) were purchased from Shenyang Chemical Agent Factory. Acetone, acetonitrile, pyridine and sodium tetrafluoroborate were of analytical grade and provided by Tianjing Chemical Agent Factory. The pyridine (AR) was vacuum distilled prior to use. The 39i benzoic acid was purchased from NIST.

Synthesis of BPBF$_4$

BPBF$_4$ was prepared through alkylation of 1-butylpyridinium chlorate (BPC) with sodium tetrafluoroborate (NaBF$_4$) in acetone as an inert solvent according to the following reaction scheme:



BPBF$_4$ were prepared according to literature procedures [19–25]. A 500 mL three-necked round-bottom flask was fitted with a water condenser. The pyridine and 1-chlorobutane were added into the reaction vessel with continuous magnetic stirring under N$_2$ atmosphere. The reaction mixture was heated under N$_2$ at 353.2 K for 96 h with stirring until two phases formed. The top phase, containing unreacted starting material, was decanted. The remainder was thorough rinsed with ethylacetate for twice. Any remaining ethylacetate was removed by heating the remainder to 343 K and stirring while on a vacuum box. The product is slightly yellow and may be crystalline at room temperature, depending on the amount of water present in that phase. The product was recrystallized twice in dry acetonitrile and dried under vacuum at 343 K for 12 h to yield the pure crystalline BPC. Equimolar sodium tetrafluoroborate was added to the solution of BPC in acetone at room temperature. After 72 h stirring, the resulting NaCl precipitate was then filtered through a Buchner funnel and the acetonitrile was removed by rotary evaporation. A colorless, clear liquid, the BPBF$_4$ was finally obtained. The chemical shifts for ^{1}H NMR spectrum (CDCl$_3$, TMS) of BPBF$_4$ appear as follows: δppm=8.040 (2H, pyridine), δppm=8.494 (1H, pyridine), δppm=8.841 (2H, pyridine), δppm=4.628 (2H, N–CH$_2$), δppm=0.826~1.982 (2H, –CH$_2$CH$_2$CH$_3$).

Heat-capacity measurement

Heat-capacity measurements were carried out in a high-precision automatic adiabatic calorimeter described in detail elsewhere [26, 27]. The principle of the calorimeter is based on the Nernst stepwise heating 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 distribution between the sample and the cell. The platinum resistance thermometer was inserted into the copper sheath, which was soldered in the middle of the sample cell. The heater wire was wound on the surface of the thermometer. The evacuated can was kept within ca. $1\cdot10^{-3}$ Pa during the heat-capacity measurements so as to eliminate the heat loss due to gas convection. Liquid nitrogen was used as the cooling medium. 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.

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 [28] 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 for water, ethanol and benzene determined in our laboratory were in excellent agreement with the data cited from literatures [29–32]. The mass used for the heat capacity measurement is 51.4809 g.

Combustion enthalpy measurement

Combustion calorimetric experiment was carried out in a precision oxygen-bomb calorimeter described in detail elsewhere [33]. 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 massic energy of combustion Δ_cU=–26434±3 J g^{-1} to be: ε_{calor}=13572.22±0.98 J K^{-1}. The uncertainty of the results was the standard deviation of the mean value of 10 combustion experiments.

The condition for determining the constant-volume combustion energy of $BPBF_4$ 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:

$$\Delta_c U\ [\mathrm{J\ mol^{-1}}]=(\varepsilon_{calor}\Delta T-Q_{Ni}-Q_{HNO_3})M/W \qquad (1)$$

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; W [g] the mass of the sample. The calculated results of the constant-volume combustion energy of $BPBF_4$ are listed in Table 1.

Table 1 The constant-volume combustion energy of $BPBF_4$

m/g	Q_{Ni}/J	Q_{HNO_3}/J	ΔT/K	$-\Delta_cE$/kJ mol^{-1}
2.5244	31.882	26.110	4.551	5451.7
1.8963	34.693	28.309	3.417	5446.8
1.9784	39.425	24.217	3.564	5445.6
1.7703	38.792	31.293	3.194	5452.3
1.7851	35.639	28.566	3.221	5453.6
2.1347	36.453	26.136	3.846	5446.9
average				5449±3

m [g] – mass of the sample, ΔT [K] – corrected temperature rise; $-\Delta_cE$ [kJ mol^{-1}], constant-volume combustion energy

Results and discussion

Molar heat capacity of $BPBF_4$

As a continuation of our previous investigations on thermodynamics for new materials significant in science and technology [34–37], the molar heat capacities of the $BPBF_4$ were determined by using the adiabatic calorimeter in temperature range from 80 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 for solid phase from 80 to 190 K, for glass state from 198 to 252 K and for liquid phase from 286 to 390 K, respectively.

In order to fit well, the temperature T was replaced by the reduced temperature X which was defined as:

$$X=[T-0.5(T_{max}+T_{min})]/[0.5(T_{max}-T_{min})] \qquad (2)$$

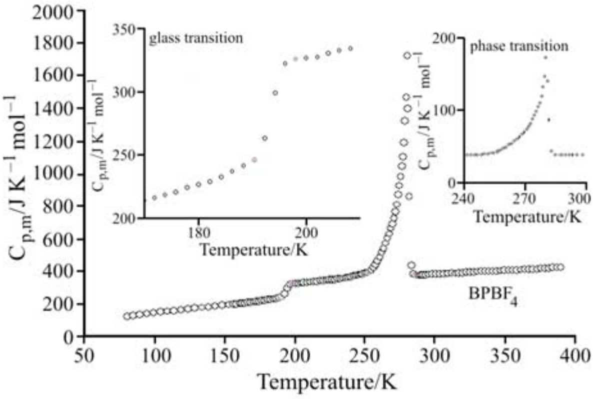


Fig. 1 Molar heat capacity of the ionic liquid $BPBF_4$

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

The values of molar heat capacities of the $BPBF_4$ were fitted to the following polynomial expressions with least square method.

For the solid phase (80–190 K):

$$C_{p,m}\ [\mathrm{J\ K^{-1}\ mol^{-1}}]=181.43+51.297X-4.7816X^2-1.9734X^3+8.1048X^4+11.108X^5 \qquad (3)$$

where reduced temperature, $X=(T-135)/55$.

The above equation is valid from 80 to 190 K, with an uncertainty of ±0.2%. The correlation coefficient of the fitted curve, R^2=0.9999.

For the glass state (198–252 K):

$$C_{p,m}\ [\mathrm{J\ K^{-1}\ mol^{-1}}]=349.96+25.106X+9.1320X^2+19.368X^3+2.23X^4-8.8201X^5 \qquad (4)$$

where reduced temperature $X=(T-225)/27$. The above equation is valid from 198 to 252 K with an uncertainty of ±0.2%. The correlation coefficient of the fitted curve, R^2=0.9979.

For the liquid phase (286–390 K):

$$C_{p,m}\ [\mathrm{J\ K^{-1}\ mol^{-1}}]=402.40+21.982X-3.0304X^2+3.6514X^3+3.4585X^4 \qquad (5)$$

where reduced temperature $X=(T-338)/52$. The above equation is valid from 286 to 390 K with an uncertainty of ±0.2%. The correlation coefficient of the fitted curve, R^2=0.9976.

Thermodynamic data of glass and phase transition [38]

It can be seen from Fig. 1 that the heat capacity jump, corresponding to the glass transition of the $BPBF_4$ [24], took place in the range from 190 to 198 K. The temperature of the glass transition, T_g, was determined to be 194.09 K. The molar enthalpy, ΔH_g, and entropy, ΔS_g, of the glass transition were derived by the following equation [39]:

Table 2 The values of molar heat capacity of ionic liquid $BPBF_4$

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}	T/K	C_p/ J K^{-1} mol^{-1}	T/K	C_p/ J K^{-1} mol^{-1}
79.71	124.31	175.85	221.16	231.98	357.25	272.41	811.47	316.31	392.68
83.81	129.18	177.95	224.21	233.85	359.62	273.38	872.53	318.89	393.08
88.77	135.13	180.04	226.66	235.69	362.15	274.31	927.10	321.49	394.85
93.64	140.68	182.12	229.01	237.54	364.49	275.21	981.56	324.12	397.04
98.56	146.12	184.19	232.88	239.37	367.00	276.07	1047.48	326.76	398.17
103.43	151.09	186.25	236.99	341.16	375.34	276.90	1104.49	329.40	399.75
108.25	156.03	188.29	242.01	242.91	377.10	277.68	1189.52	332.06	401.31
113.15	160.81	190.30	246.47	244.60	380.25	278.41	1324.19	334.74	401.10
118.09	165.40	192.26	263.47	246.26	382.82	279.10	1470.77	337.44	403.17
123.01	170.05	194.13	299.10	247.93	385.10	279.78	1722.79	340.22	402.56
127.90	174.78	195.94	322.01	249.55	389.40	280.47	1399.38	343.07	402.39
132.78	179.14	197.86	325.90	251.14	394.63	281.34	860.74	345.93	405.10
137.64	183.57	199.85	326.72	252.68	400.04	282.71	438.08	348.78	407.18
142.58	188.19	201.88	327.52	254.21	409.91	284.33	385.68	351.65	407.73
147.50	192.64	203.92	329.94	255.72	425.27	286.06	377.18	354.50	409.46
152.42	197.44	205.96	332.85	257.23	443.09	287.80	377.75	357.36	410.43
155.85	199.88	208.02	334.34	258.70	465.18	289.54	378.72	360.34	411.76
157.83	201.92	210.57	336.34	260.16	487.95	291.28	379.21	363.27	413.54
159.87	204.26	212.95	338.78	261.60	517.35	293.02	381.07	366.13	414.34
161.90	206.14	215.43	341.42	262.98	542.60	295.22	382.87	369.01	415.78
163.91	208.29	217.63	343.75	264.33	573.96	297.87	383.54	371.98	416.97
165.92	209.85	219.79	345.77	265.64	604.97	300.52	385.07	374.67	418.45
167.90	211.96	221.90	347.69	266.89	632.01	303.16	386.65	377.53	419.87
169.88	214.39	224.00	348.99	268.09	665.66	305.80	386.78	380.37	421.75
171.86	216.41	226.06	350.64	269.25	692.98	308.44	388.37	383.27	423.92
173.82	218.78	228.08	352.85	270.34	728.67	311.08	389.29	386.18	425.45
		230.06	354.80	271.42	767.44	313.70	389.04	389.03	427.76

$$\Delta H_g = \frac{Q - \int_{T_i}^{T_f} \overline{H}_{cell}\,dT - n\int_{T_i}^{T_g} C_{p,T_i}\,dT - n\int_{T_g}^{T_f} C_{p,T_f}\,dT}{n} \tag{6}$$

$$\Delta S_g = \Delta H_g / T_g \tag{7}$$

where T_i is slightly lower than T_g and T_f a little higher than T_g, $\overline{H}_{cell}$ is the heat capacity of the empty calorimeter, Q is the total energy introduced into the calorimeter during the course of glass transition, n is the mole number of the sample. The results of the calculation were ΔH_g=2.157 kJ mol^{-1} and ΔS_g=11.12 J K^{-1} mol^{-1}.

The molar heat capacity reached maximum in temperature from 252 to 286 K, as shown in Fig. 1, the phase transition took place in the temperature range. The phase transition temperature was determined to be T_m=279.79 K which was corresponding to the solid–liquid phase transition of $BPBF_4$ [24]. Using similar Eqs (6) and (7), the values of molar enthalpy, ΔH_m, and of molar entropy of fusion, ΔS_m, can be calculated. The results were ΔH_m=8.453 kJ mol^{-1} and ΔS_m=30.21 J K^{-1} mol^{-1}.

Thermodynamic functions of ionic liquid $BPBF_4$

Thermodynamic functions of $BPBF_4$ were calculated based on the empirical Eqs (3)–(5), and the following relationships of the thermodynamic functions:

$$H_T - H_{298.15} = \int_{298.15}^{T} C_{p,m}\,dT \tag{8}$$

$$S_T - S_{298.15} = \int_{298.15}^{T} \frac{C_{p,m}}{T}\,dT \tag{9}$$

The thermodynamic function data relative to the reference temperature (298.15 K) were calculated by Eqs (8) and (9) in the temperature range from 80 to 390 K with an interval of 5 K and are listed in Table 3.

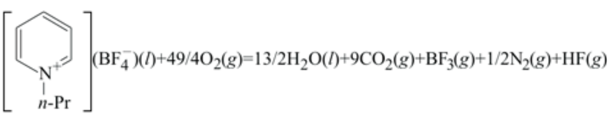
Table 3 The data of the thermodynamic functions of $BPBF_4$

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}	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	124.31	–104.34	–259.00	225	349.96	–35.54	–33.36
85	130.96	–103.15	–252.36	230	355.04	–32.62	–28.27
90	136.89	–101.96	–246.42	235	361.47	–29.34	–21.84
95	142.35	–100.76	–240.96	240	369.79	–25.53	–13.52
100	147.52	–99.53	–235.79	245	380.14	–21.15	–3.17
105	152.52	–98.27	–230.79	279.79		phase transition	
110	157.43	–96.96	–225.88	290	379.16	–4.329	–4.15
115	162.30	–95.62	–221.01	295	381.70	–1.685	–1.62
120	167.14	–94.23	–216.17	298.15	383.32	0.00	0.00
125	171.95	–92.79	–211.36	300	384.27	0.99	0.95
130	176.72	–91.31	–206.59	305	386.85	3.70	3.53
135	181.42	–89.79	–201.89	310	389.40	6.42	6.08
140	186.04	–88.24	–197.27	315	391.89	9.16	8.57
145	190.59	–86.65	–192.72	320	394.32	11.89	11.00
150	195.08	–85.02	–188.24	325	396.67	14.63	13.34
155	199.56	–83.35	–183.75	330	398.93	17.36	15.61
160	204.13	–81.62	–179.19	335	401.12	20.08	17.79
165	208.91	–79.81	–174.40	340	403.24	22.81	19.91
170	214.11	–77.88	–169.21	345	405.31	25.54	21.99
175	219.97	–75.79	–163.35	350	407.36	28.28	24.04
180	226.81	–73.46	–156.50	355	409.42	31.05	26.10
185	235.05	–70.80	–148.26	360	411.54	33.86	28.22
190	245.18	–67.70	–138.14	365	413.75	36.73	30.43
194.09		glass transition		370	416.12	39.67	32.80
200	326.81	–48.92	–56.51	375	418.70	42.72	35.38
205	331.14	–46.40	–52.18	380	421.57	45.90	38.24
210	336.19	–43.68	–47.13	385	424.79	49.25	41.47
215	341.03	–40.96	–42.28	390	428.45	52.81	45.13
220	345.50	–38.27	–37.81				

The standard molar enthalpy of formation for $BPBF_4$

The constant-volume combustion energy of $BPBF_4$ was determined to be $\Delta_c U_m = -5449 \pm 3$ kJ mol^{-1}, at $T = 298.150 \pm 0.001$ K.

The standard molar enthalpy of combustion of $BPBF_4$, $\Delta_c H_m^0$, referred to the combustion enthalpy change of the following reaction at $P° = 100$ kPa, based on the definition of the combustion enthalpy about the organic compound [40]:



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

$$\Delta_c H_m^0 = \Delta_c U_m + \Delta n RT \tag{10}$$

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. The calculated standard molar enthalpy of combustion of $BPBF_4$ was: $\Delta_c H_m^0 = -5451 \pm 3$ kJ mol^{-1}.

The standard molar enthalpy of formation of the $BPBF_4$, $\Delta_f H_m^0$, was calculated by a designed Hess thermochemical cycle according to the reaction as follows:

$$\begin{aligned}\Delta_f H_m^\theta = {} & [9\Delta_{f,CO_2(g)} H_m^\theta + 13/2\Delta_{f,H_2O(l)} H_m^\theta + \\ & \Delta_{f,BF_3(g)} + \Delta_{f,HF(g)} H_m^\theta] - \Delta_c H_m^\theta\end{aligned} \tag{11}$$

In the above formula, the standard molar enthalpies of formation for $CO_2(g)$ and $H_2O(l)$, were recommended by CODATA [41, 42], $\Delta_f H_m^0(CO_2(g))=$

–(393.51±0.13) kJ mol^{-1}, $\Delta_\mathrm{f} H_\mathrm{m}^0$ (H_2O(*l*))= –(285.83±0.04) kJ mol^{-1}, and the standard molar enthalpies of formation for (BF_3(*g*)) and (HF(*g*)) were recommended, $\Delta_{\mathrm{f,BF_3(g)}} H_\mathrm{m}^0 = -1135.62$ kJ mol^{-1}, $\Delta_{\mathrm{f,HF(g)}} H_\mathrm{m}^0 = -272.55$ kJ mol^{-1} [43]. The above data were employed in the calculation of $\Delta_\mathrm{f} H_\mathrm{m}^0$ ($BPBF_4$(*l*)) values. The standard molar enthalpy of formation of the $BPBF_4$ can be derived to be: $\Delta_\mathrm{f} H_\mathrm{m}^0$ ($BPBF_4$(*l*))=–1356.3±0.8 kJ mol^{-1}.

Acknowledgements

This work was financially supported by the National Nature Science Foundation of China under the contract NSFC No. 20473091.

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Received: January 16, 2006
Accepted: February 2, 2006
OnlineFirst: June 27, 2006

DOI: 10.1007/s10973-006-7511-8