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Investigation on enthalpies of combustion and heat capacities for 2-aminomethylpyridine derivatives

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Abstract

The molar energies of combustion ($\Delta_c U_m$) for 2-aminomethylpyridine (AMP), tert-butyl 2-[*N*-(tert-Y)-2-picolyamino]acetate (AMPY), *N*,*N*-dioctyl-2-(aminomethyl)pyridine (AMPO), and tert-butyl 2-(*N*-octyl-2-picolyamino)acetate (AMPA) were measured by an oxygen bomb calorimeter at 298.15 K, and the results are: − (3585.5 ± 2.4) kJ mol^{-1}, − (10145.0 ± 7.2) kJ mol^{-1}, − (14047.1 ± 18.8) kJ mol^{-1}, − (12141.3 + 12.3) kJ mol^{-1}, respectively. The standard molar enthalpies of combustion $\left(\Delta_c H_m^{\Theta}\right)$ and standard molar enthalpies of formation $\left(\Delta_f H_m^{\Theta}\right)$ for these compounds were derived from the experimental results and literature values. The values of $\Delta_f H_m^{\Theta}$ for AMP, AMPY, AMPO and AMPA are 88.4 ± 2.5 kJ mol^{-1}, − (930.4 ± 7.6)kJ mol^{-1}, − (304.8 ± 19.0)kJ mol^{-1} and − (569.9 ± 12.6) kJ mol^{-1}. The heat capacities at constant pressure for these compounds were measured with a DSC over the temperature range from 280 to 323 K, and the relationships between the heat capacity and temperature were obtained from measured results. The melting point and enthalpy of fusion for AMPY were also determined by DSC.

Keywords Energies of combustion · 2-Aminomethylpyridine derivatives · Standard molar enthalpies of combustion · Heat capacities at constant pressure

Introduction

Pyridine derivatives play important roles in the chemistry and biochemistry due to their unique biological activity and the diversity of the structure [1, 2]. These compounds have been widely used as medicines, pharmaceutical intermediates, and functional materials [3–7]; for example, vitamin B3 (niacin) is used as an antihyperlipidemic drug and an effective high-density lipoprotein cholesterol-raising agent for reducing the cardiovascular risks [4]; some pyridine derivatives have great potential for the development of value-added metallovitamins for therapeutic applications [8], and some corrosion-free pyridine derivatives exhibit improved stability on perovskite solar cells [9].

2-Aminomethylpyridine (AMP) derivatives are one of the important compounds containing the pyridine ring, which have attracted great interests of researchers due to the presence of the flexible alkylamine side chain. These derivatives are commonly used as synthetic intermediates to obtain novel compounds in chemical synthesis [10], and they are also served as ligands in coordination chemistry to interact with a number of metals ions [11, 12]. The metal complexes have great applications in the chromatographic separation of the metallic ions, catalysis, antibacterial and antifungal properties [11–13].

In order to achieve the separation of copper (II), nickel (II) and cobalt (II) in aqueous solution, 2-[*N*-(tert-butyloxycarbonylmethyl)-2-picolyamino]acetate (AMPY), *N*,*N*-dioctyl-2-(aminomethyl)pyridine (AMPO), and tert-butyl 2-(*N*-octyl-2-picolyamino)acetate (AMPA) were synthesized from 2-aminomethylpyridine (AMP) in our laboratory. The previous investigation shows that the compounds have excellent extraction ability and high selectivity activity toward copper (II) over nickel (II) and cobalt (II) in aqueous solution [14–16]. In particular, the AMPA has a good effect in deeply removing copper (II) from the nickel or cobalt electrolysis anolyte [15]. The

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thermodynamic properties of these derivatives can provide significant data, which have a theoretical instructive in the designing process of novel materials. However, to our knowledge, the fundamental thermodynamic properties of these 2-aminomethylpyridine derivatives have not been reported in the literature. Therefore, it is necessary to investigate the fundamental thermodynamic properties of these compounds.

In the presented work, the energies of combustion for AMP and its derivatives at 298.15 K were determined by an oxygen bomb calorimeter. The standard molar enthalpies of combustion and formation for these compounds were derived from the corresponding energies of combustion. The heat capacities at constant pressure of these compounds were determined with a differential scanning calorimeter (PerkinElmer Diamond DSC) from 280 to 323 K, and the relationship between the heat capacities and temperature was established. The melting point and enthalpy of fusion for AMPY were also measured by the DSC. The molecular structures of these studied compounds are shown in Fig. 1.

Experimental

Materials

All reagents in presented study were used without additional purification. AMPO and AMPA were supplied by Yang et al. [15]. AMPY was supplied by Cheng et al. [16]. AMP, AMPO, and AMPA were in liquid phase, and AMPY was in solid phase at room temperature. Sapphire, a-Al_2O_3 and indium were provided by PerkinElmer (with a mass fraction purity of 0.999), and they were used as the standard reference materials in the DSC measurement. The information on the other chemical samples descriptions is listed in Table 1.

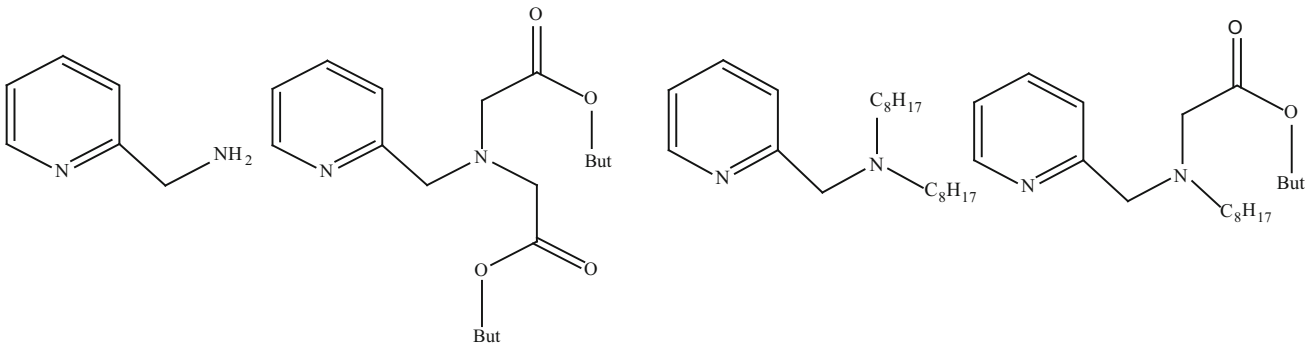
The relative atomic masses used in the calculations were recommended by the IUPAC Commission in 1999 [17], and the molar mass of AMPY, AMPO, and AMPA was 336.43, 332.57, and 334.54 g mol^{-1}, respectively.

Differential scanning calorimetry

The melting point, molar enthalpy of fusion for AMPY and the heat capacities at constant pressure for AMP, AMPY, AMPA, and AMPO were measured by a differential scanning calorimeter (PerkinElmer Diamond DSC). The heat capacities at constant pressure of samples were calculated according to the three-step method previously described in the literature [18], using sapphire as standard reference material. The masses of the samples about 5–10 mg were sealed in aluminum crucibles. The scanning range of temperature of samples was from 280 to 323 K with a heating rate of 5 K min^{-1} under a constant flow of nitrogen atmosphere (20 mL min^{-1}). Prior to the measurement, the temperature and sensitivity of the DSC were calibrated by the indium, hexatriacontane, and sapphire.

Combustion calorimetry

The energies of combustion for all compounds were measured at 298.15 K by an oxygen bomb calorimeter. The samples in a platinum crucible were placed inside the calorimeter bomb. The circuit was closed with Ni–Cr fuse wire about 10.0 cm, and the Ni–Cr fuse wire was contacted with the samples. The combustion bomb was filled with 10.0 mL of demineralized water and 2.5 MPa of high-purity oxygen. The combustion bomb was placed inside the calorimetric vessel, which was filled with 3000.0 mL of water. Taking into account the difficulty in igniting some liquid samples and the incompletely combustion of the samples, polyethylene bag and paraffin oil were served as the auxiliary combustion substance in some of the



AMP AMPY AMPO AMPA

Fig. 1 Molecular structures of 2-aminomethylpyridine (AMP), tert-butyl 2-[*N*-(tert-butyloxycarbonylmethyl)-2-picolyamino]acetate (AMPY), *N*,*N*-dioctyl-2-(aminomethyl)pyridine (AMPO), and tert-butyl 2-(*N*-octyl-2-picolyamino)acetate (AMPA)

Table 1 Information on the chemical sample descriptions

Sample	IUPAC name	Molecular mass	Source	Mass fraction purity
$C_7H_6O_2$	Benzoic acid	122.12	Aladdin industrial corporation	> 0.995
$C_{10}H_8$	Naphthalene	128.18	Aladdin industrial corporation	0.997
$C_{36}H_{74}$	Hexatriacontane	581.43	Aladdin industrial corporation	0.99(GC)
$C_6H_8N_2$	2-aminomethylpyridine	108.14	Aladdin industrial corporation	0.99

combustion experiments [19, 20]. In this work, we used benzoic acid as auxiliary substance to determine the energies of combustion.

The temperature of the calorimeter vessel was measured by a Pt resistance thermometer, and it was automatically recorded by HR-15 heat meter multi-function control box. The corrected temperature rise was calculated according to the literature [21].

After combustion experiment, the final products of combustion experiments in the calorimeter bomb comprise an aqueous solution of nitric acids and a gaseous phase. The inside wall of the calorimeter bomb was washed out with demineralized water. The aqueous phase was transferred to a conical flask and diluted to a volume of 100.0 cm^3. The carbon dioxide was removed by boiling the solutions. In order to obtain the amount of HNO_3 formed during the combustion reactions, the aqueous solution was titrated with 0.1 mol L^{-1} standard sodium hydroxide solution using phenolphthalein as indicator [22, 23].

The energy equivalent of calorimeter was determined from a series of independent combustion experiments of benzoic acid, and the literature value of massic energy of combustion for benzoic acid under the certified conditions was (26.434 ± 0.003) kJ g^{-1} [24]. The energy equivalent of the calorimeter was calculated according to Eq. (1)

$$\varepsilon = \frac{Qm + Gb + 5.983V}{\Delta T} \quad (1)$$

where ε/kJ K^{-1} is the energy equivalent of the calorimeter; Q/kJ g^{-1} is the combustion energy of benzoic acid; m/g is the mass of benzoic acid; G/8.87 J cm^{-1} refers to the combustion energy of Ni–Cr wire for ignition; b/cm is the length of actual Ni–Cr wire consumed; 5.983/J mL^{-1} is the formation enthalpy and solution enthalpy of nitric acid corresponding to 1 mL of 0.1 mol L^{-1} solution of NaOH; V/mL is the volume of consumed NaOH and ΔT/K is the corrected value of the temperature rise [25].

The steps of determining the energies of combustion for the other substances were same way as above-mentioned procedures.

Results and discussion

The melting point and enthalpy of fusion for AMPY

The obtained DSC curve over the temperature range from 270 to 343 K for AMPY is shown in Fig. 2. It can be seen that an endothermic peak occured in the temperature range from 320 to 335 K, and the onset (T_{on}) and maximum (T_{max}) temperatures are 330.7 and 333.8 K. The TG curve is given in Supplemental Figure. S1. It can be seen that no mass loss process happened in this temperature range, and this phenomenon shows that the endothermic peak is solid–liquid phase transition. The melting point and enthalpy of fusion determined by DSC are 330.7 K and 31.04 kJ mol^{-1} for AMPY.

Heat capacities for AMP and its derivatives

In order to check the accuracy of the method, the heat capacities at constant pressure ($C_{P\ exp}$) for a-Al_2O_3 from 280 to 350 K are measured by DSC. The experimental results and the literature values [26] are listed in Supplemental Table TS1. The relative deviations of experimental

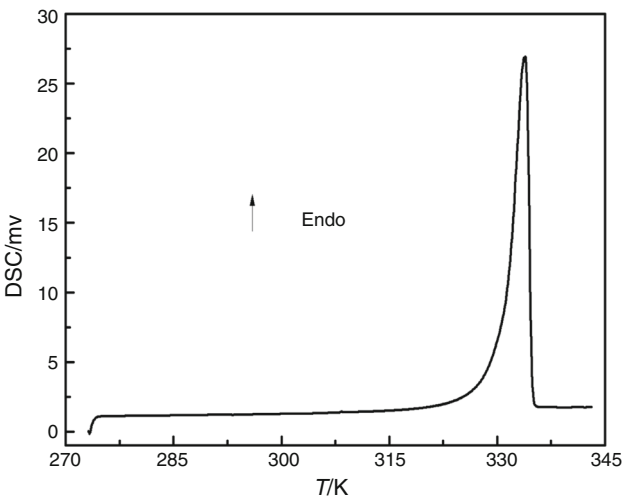


Fig. 2 Typical DSC curve for AMPY over the temperature range 270–343 K

results and the literature values are within 2%, which show the feasibility of the method and the reliability of the experimental results.

The values of heat capacities at constant pressure for AMP, AMPY, AMPO, and AMPA are given in Supplemental Tables TS2 to TS3. The experimental results of heat capacities for these compounds over the temperature range from 280 to 323 K are shown in Fig. 3. It can be seen that the heat capacities at constant pressure all increase smoothly with the increase in temperature, indicating there is no phase change or thermal anomaly occurred in this experimental temperature range. The values of heat capacities for AMPY increase sharply after 320 K, which can be ascribed to the temperature approaching the melting point. The experimental results of heat capacities at constant pressure for AMP, AMPO, and AMPA (from 280 to 323 K) and AMPY (from 280 to 310 K) are fitted as the following polynomial Eq. (2), the corresponding parameters and correlation coefficients are summarized in Table 2.

Table 2 Parameters of Eq. (2) for different compounds

Compounds	$a \times 10^{-3}$	$b \times 10^{-1}$	c	R^2
AMP	3.798	− 2.718	0.0517	0.9997
AMPY	3.859	− 2.604	0.0496	0.9832
AMPO	− 3.776	2.591	− 0.035	0.9998
AMPA	− 2.025	1.215	− 0.0097	0.9998

$$C_{p,m}/\mathrm{J\ K^{-1}\ mol^{-1}} = a + bT + cT^2 \quad (2)$$

The relative deviation of the experimental heat capacities and the fitted values using the parameters in Table 2 for AMP, AMPO, and AMPA from 280 to 323 K and AMPY from 280 to 310 K is shown in Supplemental Figure S2. The maximum value is 0.03. There is good agreement between the experimental and calculated results for heat capacities, indicating the polynomial Eq. (2) are applicable in above temperature range.

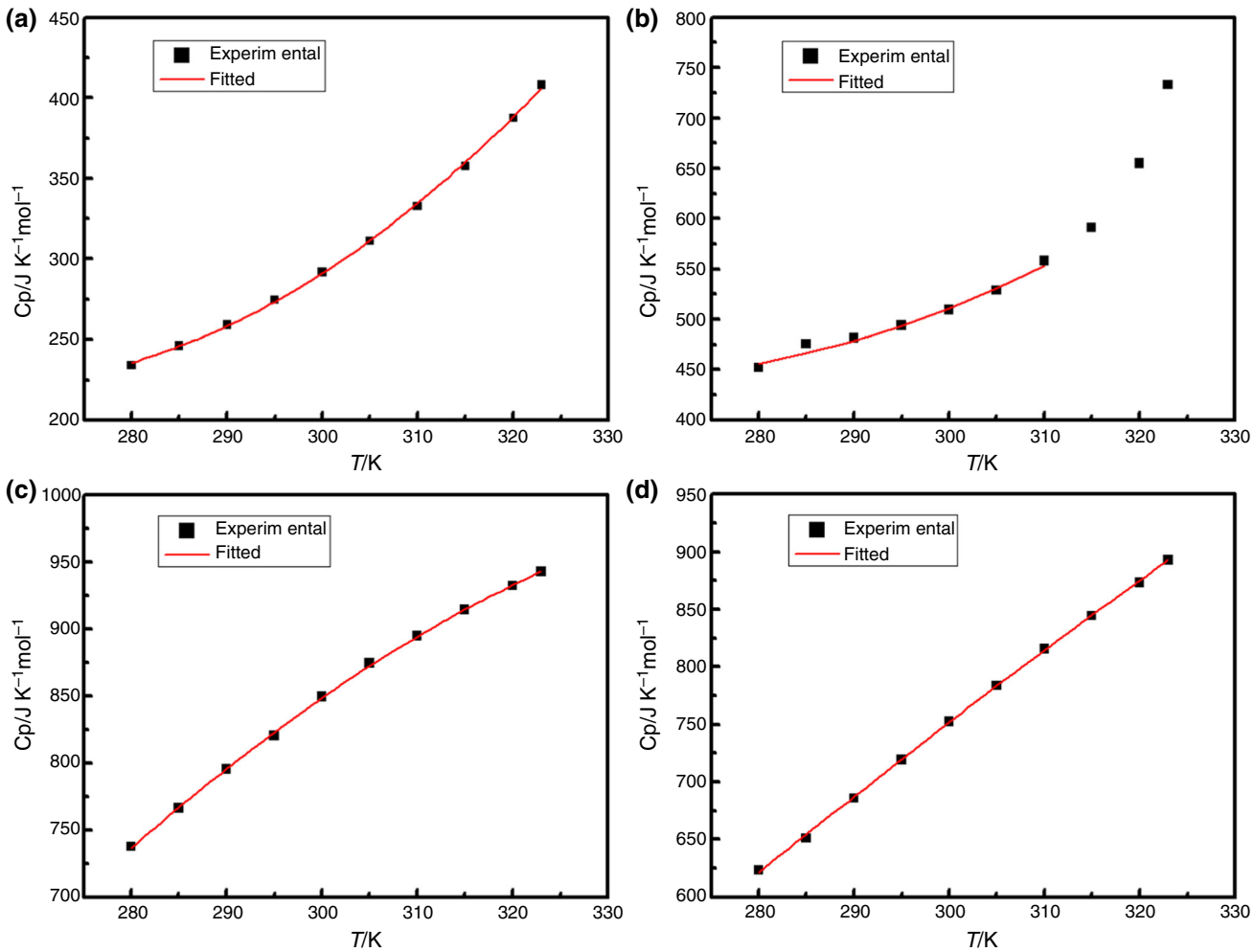


Fig. 3 Experimental and fitted data of the heat capacities **a** AMP, **b** AMPY, **c** AMPO, **d** AMPA

Combustion energies and standard molar enthalpies of formation for AMP and its derivatives

The experimental results for the determination of energy equivalent of the oxygen bomb calorimeter are given in Supplemental Table TS4. The energy equivalent value is calculated to be $\varepsilon(\text{calor}) = (14.592 \pm 0.011)$ kJ K^{-1}, where the uncertainty refers to the standard deviation of the mean.

The energies of combustion of the samples are calculated according to the formula:

$$m_s \Delta_c u_s = \varepsilon \Delta T - Q_{Ni-Cr} - Q_{HNO_3} - m_{fuel} \Delta_c u_{fuel} \quad (3)$$

where $\Delta_c u_s$ and $\Delta_c u_{fuel}$ denote the energies of combustion for the samples and auxiliary fuel; m_s and m_{fuel} are the mass of the sample and auxiliary fuel; Q_{Ni-Cr} is the heat of combustion wire; Q_{HNO_3} is the heat of acid containing nitrogen.

The molar energies of combustion for the compounds are corrected to the standard state by applying Washburn equation [27].

In order to verify the accuracy of experimental device and procedures for the oxygen bomb calorimeter, naphthalene is used as a standard reference material in this experiment. The detailed results for a typical combustion experiment of naphthalene are shown in the second column of Table 3. The average value of the enthalpy of combustion for naphthalene is 5158.9 ± 1.7 kJ mol^{-1}. The molar enthalpy of combustion for naphthalene measured at 298.15 K is in good agreement with the reported literature value $\Delta_c H^{REF}$(− 5153.9 kJ mol^{-1}) [28]. The relative deviation is within 0.1%, indicating that the experimental method and the performance of instrument are reliable.

The combustion reaction of the samples can be written as the following equations: (4)–(7), corresponding to AMP, AMPY, AMPO and AMPA, respectively:

$$C_6H_8N_2(l) + 8O_2(g) = 6CO_2(g) + 4H_2O(l) + N_2(g) \quad (4)$$

$$C_{18}H_{28}N_2O_4(s) + 23O_2(g) = 18CO_2(g) + 14H_2O(l) + N_2(g) \quad (5)$$

$$C_{22}H_{40}N_2(l) + 32O_2(g) = 22CO_2(g) + 20H_2O(l) + N_2(g) \quad (6)$$

$$C_{20}H_{34}N_2O_2(s) + \frac{55}{2}O_2(g) = 20CO_2(g) + 17H_2O(l) + N_2(g) \quad (7)$$

The typical results of combustion experiments of AMP, AMPY, AMPO and AMPA are listed in Table 3. The individual values of the massic energies of combustion for each compound at 298.15 K are given in Table 4. The standard molar enthalpies of combustion for the compounds were derived from the respective combustion energies by Eqs. (8) and (9):

$$\Delta_c H_m^{\Theta} = \Delta_c U_m^{\Theta} + \Delta n RT \quad (8)$$

$$\Delta n = \sum n(\text{prod}) - \sum n(\text{react}) \quad (9)$$

where Δn is the total mole variation of the gas substances in the combustion reaction, R is the gas constant 8.314 J mol^{-1} K^{-1}; T = 298.15 K.

The standard molar formation enthalpies of the samples were obtained by Hess's law thermochemical cycle [25]

$$\Delta_f H_m^{\Theta} = x\Delta_f H_m^{\Theta}(CO_2, g) + y\Delta_f H_m^{\Theta}(H_2O, l) - \Delta_c H_m^{\Theta} \quad (10)$$

where x and y represent the amount of CO_2(g) and H_2O(l) in the combustion reaction.

The values of the energies of combustion $\Delta_c U_m$, standard molar energies of combustion $\Delta_c U_m^{\Theta}$, standard molar enthalpies of combustion $\Delta_c H_m^{\Theta}$, as well as the standard molar enthalpies of formation $\Delta_f H_m^{\Theta}$ for the four compounds at 298.15 K are shown in Table 5. The following auxiliary values for the standard molar enthalpies of formation of CO_2(g), − (393.51 ± 0.13) kJ mol^{-1} and

Table 3 Typical results of combustion experiments of these compounds at T = 298.15 K

	Naphthalene	AMP	AMPY	AMPO	AMPA
m_s/g	0.8733	0.1595	0.6231	0.1506	0.1576
m_{fuel}/g	0	0.5971	0	0.4665	0.7414
ΔT/K	2.413	1.452	1.297	1.286	1.742
ε(calor)/kJ K^{-1}	14.592	14.592	14.592	14.592	14.592
Q_{Ni-Cr}/kJ	0.0825	0.0985	0.0816	0.0798	0.0834
Q ($\varepsilon\Delta T$)/kJ	35.211	21.188	18.926	18.766	25.420
$Q_{(HNO3)}$/kJ	0.0126	0.0227	0.0365	0.0329	0.0245
− $\Delta_c u$/kJ g^{-1}	40.211	33.123	30.185	42.258	36.255

m_s, is the mass of burned compound in each experiment; m_{fuel}, is the mass of auxiliary fuel benzoic acid; ΔT, the corrected temperature rise; ε(calor), energy equivalent of the calorimeter; Q_{Ni-Cr}, ignition energy of Ni-Cr wire; $Q_{(HNO_3)}$, heat of acid containing nitrogen; $Q(\varepsilon\Delta T)$, total energy of the combustion process; − $\Delta_c u$, the massic energy of combustion for each compound.

Table 4 The individual values of massic energies of combustion for naphthalene, AMP, AMPY, AMPO, and AMPA at $T = 298.15$ K

Naphthalene	AMP	AMPY	AMPO	AMPA
$\Delta_c u$/kJ g^{-1}				
40.211	33.123	30.185	42.258	36.255
40.188	33.145	30.143	42.168	36.303
40.213	33.130	30.155	42.327	36.277
40.205	33.147	30.137	42.252	36.351
40.225	33.187		42.236	36.277
40.223	33.148		42.188	
$\Delta_c u$/kJ g^{-1} (mean)				
40.211 ± 0.013	33.145 ± 0.022	30.155 ± 0.022	42.251 ± 0.065	36.292 ± 0.037

Table 5 Standard molar energies and enthalpies of combustion and the standard molar enthalpies of formation for these compounds at $T = 298.15$ K

Compound	$-\Delta_c U_m$ /kJ mol^{-1}	$-\Delta_c U_m^{\Theta}$ /kJ mol^{-1}	$-\Delta_c H_m^{\Theta}$ /kJ mol^{-1}	$\Delta_f H_m^{\Theta}$ /kJ mol^{-1}
Naphthalene	5154.2 ± 1.7	5154.0 ± 1.7	5158.9 ± 1.7	
AMP	3585.5 ± 2.4	3585.4 ± 2.4	3592.8 ± 2.4	88.4 ± 2.5
AMPY	10145.0 ± 7.2	10144.5 ± 7.2	10154.4 ± 7.2	− 930.4 ± 7.6
AMPO	14047.1 ± 18.8	14046.7 ± 18.8	14069.0 ± 18.8	− 304.8 ± 19.0
AMPA	12141.3 ± 12.3	12140.8 + 12.3	12159.4 ± 12.3	− 569.9 ± 12.6

H_2O(l), − (285.83 ± 0.04) kJ mol^{-1} at 298.15 K are chosen from CODATA [29]. The uncertainty of standard enthalpies of formation is calculated according to the literature [19]. The detailed results of all the combustion experiments are presented in Tables TS5 to TS9 in a supplementary material.

As can be seen from the results in Table 5, the standard molar enthalpy of combustion for AMPO is higher than other substances; in general, the enthalpies of combustion increase with carbon number and alkyl chain increasing. From the standard molar enthalpies of formation to analyze, it is observed that the enthalpy of formation for AMPY has a higher enthalpy, indicating that the bonding of branched alkanes and carbonyl group has more exothermic than no-branched alkanes and no-carbonyl group.

Conclusions

This study presented the experimentally determined thermodynamic properties of 2-aminomethylpyridine derivatives.

The melting point of AMPY was 330.7 K, and its enthalpy of fusion was 31.04 kJ mol^{-1}. The heat capacities at constant pressure for the compounds have been measured with DSC, and relationship between the heat capacities and temperature was established. Furthermore, the molar energies of combustion for these compounds were determined by the combustion calorimeter. The values of the energies of combustion were used to derive standard molar enthalpies of combustion and standard molar enthalpies of formation. The standard molar enthalpies of combustion for AMP, AMPY, AMPO, and AMPA were − (3592.8 ± 2.4), − (10154.4 ± 7.2), − (14069.0 ± 18.8), and − (12159.4 ± 12.3) kJ mol^{-1}, respectively. The standard molar enthalpies of formation for these compounds were 88.4 ± 2.5 kJ mol^{-1}, − (930.4 ± 7.6) kJ mol^{-1}, − (304.8 ± 19.0) kJ mol^{-1}, and − (569.9 ± 12.6) kJ mol^{-1}, respectively.

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