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THERMODYNAMIC INVESTIGATION OF SEVERAL NATURAL POLYOLS

Part III. Heat capacities and thermodynamic properties of erythritol

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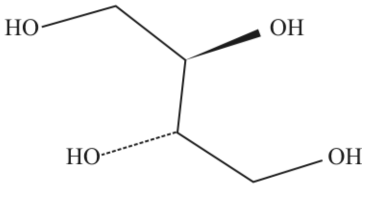
The low-temperature heat capacity $C_{p,m}$ of erythritol ($C_4H_{10}O_4$, CAS 149-32-6) was precisely measured in the temperature range from 80 to 410 K by means of a small sample automated adiabatic calorimeter. A solid–liquid phase transition was found at T=390.254 K from the experimental C_p–T curve. The molar enthalpy and entropy of this transition were determined to be 37.92±0.19 kJ mol^{-1} and 97.17±0.49 J K^{-1} mol^{-1}, respectively. The thermodynamic functions $[H_T–H_{298.15}]$ and $[S_T–S_{298.15}]$, were derived from the heat capacity data in the temperature range of 80 to 410 K with an interval of 5 K. The standard molar enthalpy of combustion and the standard molar enthalpy of formation of the compound have been determined: $\Delta_c H^0_m(C_4H_{10}O_4, cr)=–2102.90±1.56$ kJ mol^{-1} and $\Delta_f H^0_m(C_4H_{10}O_4, cr)=–900.29±0.84$ kJ mol^{-1}, by means of a precision oxygen-bomb combustion calorimeter at T=298.15 K. DSC and TG measurements were performed to study the thermostability of the compound. The results were in agreement with those obtained from heat capacity measurements.

Keywords: *DSC, erythritol, heat capacity, phase transition, standard molar enthalpy of formation, TG*

Introduction

Polyhydroxy compounds and their properties are important in many areas of chemistry and biochemistry. Their polar nature and ability to participate in hydrogen bonding give them a high affinity for water and other polar solvents. Erythritol [$(CH_2OH)(CHOH)_2(CH_2OH)$, CAS No.149-32-6], xylitol and sorbitol are important natural polyols in food and pharmaceutical applications, as they are increasingly used to provide sweetness to various products or replace sugar in confectionery [1–4]. Erythritol's molecular formula is $C_4H_{10}O_4$ with molar mass of 122.12 g mol^{-1} and structural formula as follows:



Compared with the sucrose, the erythritol has the characteristic sweet taste of sugars but the amount of energy (calories) in the products is reduced. Another important advantage is that it does not contribute to the development of dental caries. Moreover, it is suitable for diabetics, because it does not require insulin of glucose in their metabolism [3–5]. In industrial applications, the state and phase transition of the erythritol affects its molecular mobility and physicochemical properties [6–8]. The heat capacity and phase transition data of erythritol were reported in 1932 [9] and 1990 [10], respectively. Because it is very useful in food and pharmaceutical applications, in the present study, the heat capacity and phase transition were investigated in detail with precision adiabatic calorimetric and thermal analytic technique in a wide temperature range from 80 to 410 K.

Heat capacity is one of the most fundamental thermodynamic properties of substances and it closely related to other physical and chemical properties. Adiabatic calorimetry is one of the most accurate method for obtaining the heat capacity, melting point and enthalpy of fusion of substances [11–15]. In the present research, low temperature heat capacity measurements were carried out with an adiabatic calorimeter over the temperature range from 80 to 410 K, and thermodynamic properties such as molar enthalpies and entropies of phase transition were determined based on the heat capacity measurements. The thermodynamic functions, $[H_T–H_{298.15}]$ and $[S_T–S_{298.15}]$, were calculated from heat capacity data in the temperature range of 80–410 K.

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Experimental

Material

The Erythritol was purchased from Shandong Baolingbao Biotechnology Co. Ltd, in PR China with batch number 060715 and labeled purity >99.0% mass fraction.

Methods

Adiabatic calorimetry

Heat capacity measurements were carried out in a high-precision automated adiabatic calorimeter described in detail in literature [16, 17]. The calorimeter was established in Thermochemistry Laboratory of Dalian Institute of Chemical Physics, Chinese Academy of Sciences in PR China. It mainly consisted of a sample cell, a miniature platinum resistance thermometer, an electric heater, an inner and outer adiabatic shield, two sets of chromel-copel thermocouples and a high vacuum system. Its working temperature is from 80 to 580 K [18] with liquid nitrogen as cooling medium.

The measurements were conducted by means of the standard method of intermittently heating the sample and alternately measuring the temperature. The temperature difference between the sample and adiabatic shield was automatically kept to be about 10^{-3} K during the whole experiment. The temperature increment for a heating period was 2–4 K, and temperature drift was maintained about 10^{-4} K min^{-1} during each equilibrium period. The data were automatically collected through a $7^{1/2}$ Digit Nano Volt/Micro Ohm Meter (Model: 34420, Agilent USA) and processed on line by a personal computer according to the program developed in our thermochemistry laboratory [19].

The sample amount used for the heat capacity measurement is 4.76575 g, which is equivalent to 39.025 mmol based on its molar mass of 122.12 g mol^{-1}.

DSC and TG analysis

A differential scanning calorimeter (Model: DSC141, SETARAM, France) was used to perform the thermal analysis of erythritol under high purity nitrogen (99.999%) with a flow rate of 40 mL min^{-1} and heating rate of 10 K min^{-1}. The mass of the sample used in the experiment was 9.70 mg.

The TG measurements of the sample were carried out by a thermogravimetric analyzer (Model: DT-20B, Shimsdzu, Japan) under N_2 with a flow rate of 30 mL min^{-1} and heating rate of 10 K min^{-1}. The mass of the sample used in the experiment was 18.35 mg.

Oxygen-bomb combustion calorimetry

The constant-volume enthalpy of combustion of the sample was measured by means of a precision oxygen-bomb combustion calorimeter, which was an isoperibolic calorimeter with a static oxygen bomb. The calorimeter was set up in our thermochemistry laboratory and the structure and principle of the calorimeter have been described previously in detail [20–22].

The sample of (0.9 to 1.0) g was pressed into pellets and put in a small sample crucible of about 0.004 dm^3, which was suspended in the bomb with capacity about 0.3 dm^3, and burned under an oxygen pressure of 3.00 MPa ignited by a nickel fuse of about 16 cm and a small amount of standard benzoic acid sample. The purity of the oxygen used in the combustion was of research grade, mole fraction 0.99998. The real energy of combustion of the nickel fuse (Q_{Ni}) was calculated from the formula, Q_{Ni}/J=2.929 ΔL, in which ΔL/cm was the length of the combusted nickel wire. The energy of formation of the aqueous nitric acid produced by oxidation of a trace of nitrogen, which contained in the oxygen bomb and produced from the combustion reaction, was determined by the neutral titration with a 0.08684 mol dm^{-3} of sodium hydroxide solution by using the phenolphthalein as the indicator. The enthalpy of formation of the aqueous nitric acid in the oxygen bomb can be derived from the equation, Q_{HNO_3}/J=59.8 NV, in which N/mol dm^{-3} is the concentration of the sodium hydroxide solution and V/cm^3 is the volume of the consumed sodium hydroxide solution; based on the molar enthalpy of formation of HNO_3 (aq) from N_2 (g), O_2 (g) and H_2O (l), $\Delta_f H_m^0$=59.8 kJ mol^{-1} for 0.1 mol dm^{-3} of HNO_3 (aq) [23–25].

Results and discussion

Heat capacity

Experimental molar heat capacities of erythritol measured by the adiabatic calorimeter over the temperature range from 80 to 410 K are listed in Table 1 and plotted in Fig. 1. From the Fig. 1, a phase transition was observed in the range of 385 to 395 K with a peak temperature of 390.254 K.

The values of experimental heat capacities can be fitted to the following polynomial equations with least square method:

Table 1 Experimental molar heat capacity of erythritol (M=122.12 g mol^{-1})

T/ K	$C_{p,m}$/ J K^{-1} mol^{-1}	T/ K	$C_{p,m}$/ J K^{-1} mol^{-1}	T/ K	$C_{p,m}$/ J K^{-1} mol^{-1}
79.518	39.11	211.524	108.5	353.745	178.1
82.281	41.44	214.559	109.9	357.045	180.0
84.955	43.40	217.636	111.1	360.331	181.5
87.502	45.31	220.690	112.7	363.602	181.7
89.945	47.08	223.726	114.9	366.925	185.1
92.297	48.70	226.779	116.8	370.301	186.9
94.608	50.49	229.812	117.7	373.671	188.9
96.870	51.87	232.914	118.6	377.030	191.4
99.094	53.39	236.069	119.9	380.379	194.2
102.159	55.24	239.167	121.2	383.708	200.5
105.529	57.39	242.772	123.4	386.784	262.9
108.345	58.99	246.401	124.1	388.913	807.4
111.178	60.57	249.467	125.6	389.759	3281
114.026	62.05	252.529	127.3	389.982	7468
116.895	63.47	255.581	128.8	390.080	12578
119.779	64.83	258.613	130.4	390.128	16556
122.649	66.53	261.700	132.3	390.157	21189
125.512	68.16	264.839	133.3	390.176	25983
128.394	69.44	267.955	134.9	390.190	28809
131.303	70.63	271.059	136.7	390.198	33672
134.199	71.98	274.143	138.5	390.207	37722
137.095	73.43	277.210	138.8	390.215	36041
140.015	74.68	280.274	140.2	390.219	37837
142.931	76.15	283.398	141.8	390.226	38215
145.849	77.64	286.563	144.2	390.229	38816
148.798	78.70	289.710	146.3	390.235	38262
152.581	79.99	292.834	148.1	390.235	40625
156.360	82.30	295.957	149.3	390.241	42544
159.317	83.72	299.078	150.2	390.242	41876
162.260	85.44	302.184	151.6	390.245	43066
165.172	87.20	305.344	153.1	390.247	47276
168.111	89.04	308.559	154.6	390.249	53135
171.078	90.74	311.759	156.2	390.252	56823
174.064	91.96	314.952	157.5	390.251	63604
177.069	93.26	318.129	159.5	390.254	73029
180.051	94.43	321.297	161.3	390.255	65628
183.056	95.62	324.455	162.8	390.256	64861
186.074	96.97	327.665	164.4	390.257	62048
189.076	98.39	330.919	166.6	390.267	35478
192.093	99.41	334.168	168.3	390.579	1788
195.128	100.9	337.404	169.8	392.541	321.5
198.149	102.1	340.629	171.7	395.698	321.4
201.147	103.2	343.852	173.1	399.066	321.8
204.829	104.7	347.124	174.6	402.424	322.1
208.524	106.9	350.440	175.8	405.777	322.4

Table 2 Thermodynamic parameters of erythritol

Thermodynamic properties		Melting point T_m/K	$\Delta_{fus}H_m$/ kJ mol^{-1}	$\Delta_{fus}S_m$/ J mol^{-1} K^{-1}
Present work	Adiabatic calorimetry	390.254	37.92±0.19	97.17±0.49
	DSC	389.60	34.90±1.05	87.82±2.63
Spaght, Thomas, *et al.*, [9]		381.6	42.36	111.0
Barone and Della Gatta, [10]		390.9	39.40	100.8

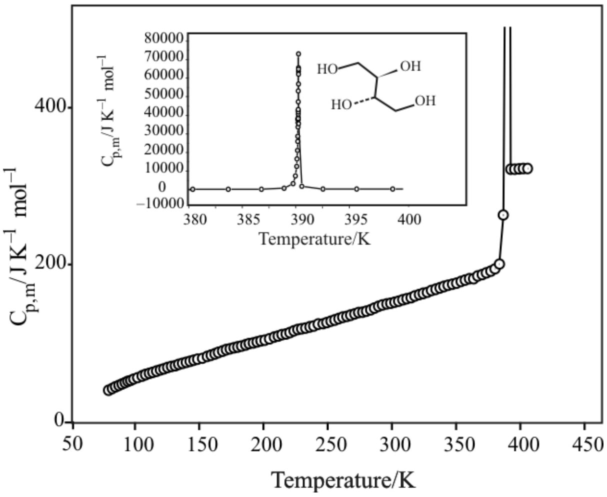

Fig. 1 Experimental molar heat capacity of erythritol as a function of temperature

For the solid phase over the temperature range 80 to 385 K:

$$C_{p,m}/\text{J K}^{-1}\text{ mol}^{-1}=118.22+72.424X+4.6835X^2-4.7788X^3-8.1937X^4+11.476X^5+4.48X^6 \quad (1)$$

where X is the reduced temperature $X=[T-(T_{max}+T_{min})/2]/[(T_{max}-T_{min})/2]$, T is the experimental temperature , thus, in the solid-state (80 to 385 K), $X=[(T/\text{K})-232.5]/152.5$, T_{max} is the upper limit (385 K) and T_{min} is the lower limit (80 K) in the above temperature region. The correlation coefficient of the fitting $R^2=0.9998$.

For the liquid phase over the temperature range 395 to 410 K:

$$C_{p,m}/\text{J K}^{-1}\text{ mol}^{-1}=322.1+0.7507X \quad (2)$$

where X is the reduced temperature, $X=[(T/\text{K})-402.5]/7.5$, T is the experimental temperature, 402.5 is obtained from polynomial $(T_{max}+T_{min})/2$, 7.5 is obtained from polynomial $(T_{max}-T_{min})/2$. T_{max} and T_{min} are the upper (410 K) and lower (395 K) limit temperature respectively. The correlation coefficient of the fitting $R^2=0.9985$.

The temperature, enthalpy and entropy of solid–liquid phase transition

The molar enthalpies and entropies of the solid–liquid phase transition $\Delta_{fus}H_m$ and $\Delta_{fus}S_m$ of the compound were derived according to the following Eqs (3) and (4):

$$\Delta_{fus}H_m=\frac{Q-n\int_{T_i}^{T_m}C_{p,m}(s)\mathrm{d}T-n\int_{T_m}^{T_f}C_{p,m}(l)\mathrm{d}T-\int_{T_i}^{T_f}H_0\mathrm{d}T}{n} \quad (3)$$

$$\Delta_{fus}S_m=\frac{\Delta_{fus}H_m}{T_m} \quad (4)$$

where T_i is a temperature slightly lower than the solid–liquid transition temperature, T_f a temperature slightly higher than the solid–liquid transition temperature, Q the total energy introduced into the sample cell from T_i to T_f, H_0 the heat capacity of the sample cell from T_i to T_f, $C_{p,m}(s)$ the heat capacity of the sample in solid phase from T_i to T_m, $C_{p,m}(l)$ the heat capacity of the sample in liquid phase from T_m to T_f and n is molar amount of the sample. The heat capacity polynomials mentioned above were used to calculate the smoothed heat capacities, and were numerically integrated to obtain the values of the standard thermodynamic functions above T=298.15 K. The calculated results are listed in Table 2.

Thermodynamic functions of the compound

The thermodynamic functions of the erythritol relative to the reference temperature 298.15 K were calculated in the temperature range 80–410 K with an interval of 5 K, using the polynomial equation of heat capacity and thermodynamic relationships as follows:

Before melting,

$$H_T-H_{298.15}=\int_{298.15}^{T}C_{p,m}(s)\mathrm{d}T \quad (5)$$

$$S_T-S_{298.15}=\int_{298.15}^{T}\frac{C_{p,m}(s)}{T}\mathrm{d}T \quad (6)$$

Table 3 Calculated thermodynamic functions of erythritol

T/ K	Cp,m/ J K^{-1} mol^{-1}	H_T–$H_{298.15}$/ kJ mol^{-1}	S_T–$S_{298.15}$/ J K^{-1} mol^{-1}
80	40.07	–21.28	–113.4
85	43.66	–21.07	–110.8
90	47.09	–20.84	–108.3
95	50.38	–20.60	–105.6
100	53.54	–20.34	–103.0
105	56.57	–20.06	–100.3
110	59.50	–19.77	–97.56
115	62.32	–19.47	–94.85
120	65.05	–19.15	–92.13
125	67.70	–18.82	–89.42
130	70.28	–18.47	–86.71
135	72.79	–18.12	–84.00
140	75.25	–17.75	–81.31
145	77.67	–17.36	–78.63
150	80.05	–16.97	–75.95
155	82.39	–16.56	–73.29
160	84.72	–16.15	–70.65
165	87.02	–15.72	–68.01
170	89.31	–15.27	–65.38
175	91.59	–14.82	–62.77
180	93.87	–14.36	–60.16
185	96.15	–13.88	–57.56
190	98.44	–13.40	–54.97
195	100.7	–12.90	–52.39
200	103.0	–12.39	–49.81
205	105.3	–11.87	–47.24
210	107.6	–11.34	–44.68
215	110.0	–10.79	–42.12
220	112.3	–10.24	–39.56
225	114.7	–9.669	–37.01
230	117.0	–9.090	–34.46
235	119.4	–8.499	–31.91
240	121.8	–7.896	–29.37
245	124.2	–7.281	–26.83
250	126.6	–6.654	–24.29
255	129.0	–6.015	–21.75
260	131.4	–5.364	–19.22
265	133.8	–4.701	–16.69
270	136.2	–4.026	–14.17
275	138.6	–3.339	–11.64
280	141.0	–2.639	–9.123
285	143.5	–1.928	–6.606
290	145.9	–1.205	–4.092
295	148.3	–0.469	–1.581
298.15	149.8	0.000	0.000
300	150.7	0.278	0.928
305	153.1	1.037	3.434
310	155.5	1.809	5.938
315	158.0	2.593	8.441
320	160.4	3.389	10.94
325	162.9	4.197	13.45
330	165.4	5.017	15.95
335	167.9	5.850	18.45
340	170.5	6.696	20.96
345	173.1	7.555	23.47
350	175.8	8.427	25.98
355	178.6	9.313	28.50
360	181.5	10.21	31.02
365	184.5	11.13	33.55
370	187.7	12.06	36.09
375	191.0	13.01	38.63
380	194.5	13.97	41.18
385	198.3	14.95	43.74
390	Phase change	/	/
395	321.3	49.21	130.5
400	321.9	50.81	137.3
405	322.4	52.41	144.0
410	322.9	54.02	150.6

After melting,

$$H_T - H_{298.15} = \int_{298.15}^{T_i} C_{p,m}(s)\mathrm{d}T + \Delta_{fus}H_m + \int_{298.15}^{T} C_{p,m}(l)\mathrm{d}T \quad (7)$$

$$S_T - S_{298.15} = \int_{298.15}^{T} \left[\frac{C_{p,m}(s)}{T}\right]\mathrm{d}T + \frac{\Delta_{fus}H_m}{T_m} + \int_{298.15}^{T} \left[\frac{C_{p,m}(l)}{T}\right]\mathrm{d}T \quad (8)$$

where T_i is the temperature at which the solid–liquid phase transition started; T_f is the temperature at which the solid–liquid phase transition ended; $\Delta_{fus}H_m$ is the molar enthalpy of fusion; T_m is the temperature of solid–liquid phase transition.

The thermodynamic functions, H_T–$H_{298.15}$, S_T–$S_{298.15}$, are listed in Table 3.

The result of TG and DSC analysis

From the DSC curve in Fig. 2, a sharply endothermic peak corresponding to melting process was observed, with the melting temperature of 389.60 K and the enthalpy of 34.90±1.05 kJ mol^{-1}, which are slightly lower than the values (390.254 K, 37.92±0.19 kJ mol^{-1}) observed from the adiabatic ca-

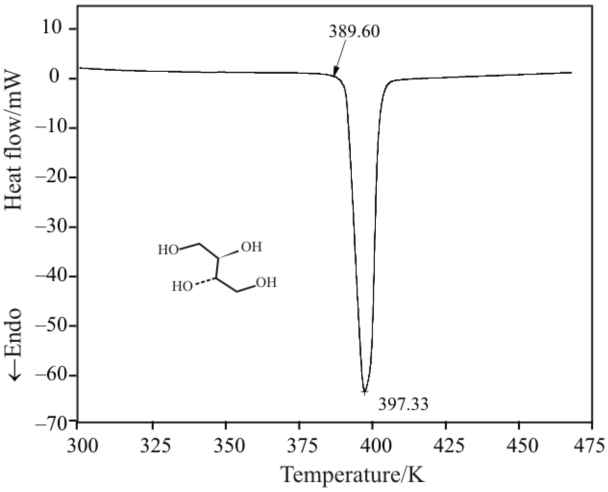

Fig. 2 DSC curve of erythritol under high purity nitrogen

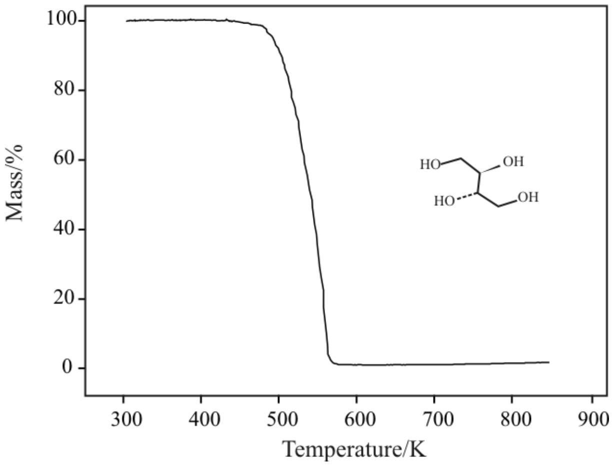

Fig. 3 TG curve of erythritol under high purity nitrogen

lorimetric measurements. The results were listed in Table 2, from which, it can be seen that the thermodynamic parameters obtained from adiabatic calorimetry and DSC in the present research are in accordance with each other and slightly lower than those reported in literature [9, 10].

From the TG curve in Fig. 3, it can be seen that the mass loss of the sample was completed in a single step. The sample keeps thermostable below 450 K. It begins to lose mass at 476.75 K, reaches the maximum rate of mass loss at 557.44 K and completely loses its mass when the temperature reaches 582.35 K.

Constant-volume combustion energy, standard molar enthalpy of combustion, and standard enthalpy of formation

The constant-volume combustion energy of the organic compound can be calculated from the equation:

$$\Delta_c U/\text{J mol}^{-1}=(\varepsilon_{\text{calor}}\Delta T-Q_{\text{Ni}}-Q_{\text{HNO}_3})M/W$$

in which $\varepsilon_{\text{calor}}$/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 results calculated by the equation were listed in Table 4.

The standard molar enthalpy of combustion of the organic compound, $\Delta_c H_m^0$, referred to the combustion enthalpy change of the following reaction at T=298.15 K and P^0=100 kPa based on the definition of the combustion enthalpy of the organic compound:

$$C_4H_{10}O_4(cr)+9/2O_2(g)=4CO_2(g)+5H_2O(l) \quad (9)$$

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

$$\Delta_c H_m^0=\Delta_c U_m+\Delta nRT$$

$$\Delta n=\sum n_i\ (\text{products, g})-\sum n_i\ (\text{reactants, g})$$

Table 4 Experimental results of constant-volume combustion energy for erythritol

No.	Sample mass m/ g	Heat value of nickel wire Q_{Ni}/J	Heat value of nitric acid Q_{HNO_3}/J	Corrected temperature rise, ΔT/K	Combustion energies, $-\Delta U_c$/kJ mol^{-1}
1	0.97812	41.2961	41.438	1.1527	2096.03
2	0.98072	37.4886	30.9487	1.1559	2098.07
3	0.98311	38.0744	25.4444	1.1632	2106.78
4	0.98961	39.5388	46.9942	1.1712	2104.51
5	0.99186	37.4886	30.9487	1.1709	2101.48
6	0.99497	34.267	47.7731	1.1778	2105.71
7	0.99854	37.4886	30.9487	1.1774	2099.02
$Avg\cdot\Delta U_c=(\overline{\Delta U_c}\pm\sigma^a)=(-2101.66\pm1.56)$					

[a] $\sigma=\sqrt{\sum_{i=1}^{n}(x_i-\bar{x})^2/n(n-1)}$, in which n is the experimental number (n=7); x_i, a single value of combustion energies; $\bar{x}$, the mean value of combustion energies

where $\sum n_{\mathrm{i}}$ was the total molar amount of the gases in products or reactants. The calculated standard molar enthalpy of combustion of the organic compound was:

$$\Delta_{\mathrm{c}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr}) = -2102.90\pm1.56\ \mathrm{kJ\ mol^{-1}}$$

The standard molar enthalpy of formation of the organic compound, $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}$, was calculated by a designed Hess thermochemical cycle according to the reaction (9) as follows:

$$\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr}) = [4\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{CO_2,g}) + 5\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{H_2O,l})] - \Delta_{\mathrm{c}} H_{\mathrm{m}}^{0}(\mathrm{C_5H_{12}O_5,cr})$$

In the above formula, the standard molar enthalpies of formation of CO_2(g) and H_2O(l), recommended by CODATA [22, 23], $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{CO_2,g}) = -393.51\pm0.13$ kJ mol^{-1}, $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{H_2O,l}) = -285.83\pm0.04$ kJ mol^{-1}, were employed in the calculation of $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr})$ values. Based on these values and the standard molar enthalpy of combustion, the standard molar enthalpy of formation of xylitol can be calculated to be: $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr}) = -900.29\pm0.84$ kJ mol^{-1}.

Conclusions

In this work, the low-temperature heat capacity $C_{\mathrm{p,m}}$ of erythritol was measured in the temperature range from 80 to 410 K using automated adiabatic calorimetry. The temperature, the molar enthalpy, and the entropy of fusion were 390.254 K, 37.92±0.19 kJ mol^{-1} and 97.17±0.49 J K^{-1} mol^{-1}, respectively. The thermostability of the compound was investigated by DSC and TG techniques. The standard molar enthalpy of combustion and the standard molar enthalpy of formation of the compound were: $\Delta_{\mathrm{c}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr}) = -2102.90\pm1.56$ kJ mol^{-1} and $\Delta_{\mathrm{f}} H_{\mathrm{m}}^{0}(\mathrm{C_4H_{10}O_4,cr}) = -900.29\pm0.84$ kJ mol^{-1}, by means of a precision oxygen-bomb combustion calorimeter at T=298.15 K.

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