Journal of Thermal Analysis and Calorimetry, Vol. 94 (2008) 1, 257–262

SOME THERMOPHYSICAL PROPERTIES OF SEVERAL SOLID ALDEHYDES

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The present study reports a differential scanning calorimetry (DSC) study of the solid aldehydes: 4-hydroxybenzaldehyde [123-08-0] **1**; 4-hydroxy-3-methoxybenzaldehyde (vanillin) [121-33-5] **2**; 3-ethoxy-4-hydroxybenzaldehyde (ethyl vanillin) [121-32-4] **3**; 3,4-dimethoxybenzaldehyde (veratraldehyde) [120-14-9] **4** and 4-methoxycinnamaldehyde [1963-36-6] **5**, in the temperature interval from T=268 K to their respective melting temperatures. Temperatures, enthalpies and entropies of fusion and the heat capacities of the compounds as a function of temperature are reported.

Keywords: *DSC, ethyl vanillin, fusion enthalpy, heat capacity, 4-hydroxybenzaldehyde, 4-methoxycinnamaldehyde, vanillin, veratraldehyde*

Introduction

Thermophysical data in the literature are still scarce and sometimes there are important discrepancies among the existing values. There are several compilations of critically evaluated calorimetrically measured heat capacities of organic molecules in the literature [1–4], but new data on heat capacity for important families of compounds are still needed [5], particularly for crystalline solids. In this regard, several determinations of heat capacities have been recently reported for polymers [6], biochemical [7], organic [8] and inorganic [9] pure compounds and mixtures [10].

There has been an effort to develop reliable and accurate group contribution schemes to improve the estimation and compensate for the scarcity of this data. The simplest schemes are based on first order additivity and only consider the constituent groups of the molecule [11]. Other methods use a second order additivity that takes into account nearest-neighbour interactions in the definition of structural units of molecules [12, 13]. These schemes normally neglect all next-to-nearest neighbour interactions due to the limited accuracy of the available experimental heat capacity data, and because differences among values of structural isomers are as large as the uncertainty of the methods. The main problems with the estimation routines are: (1) some adjusted group contribution values are based only in a limited number of experimental data; (2) fused ring, H-bond, *cis*, *ortho*, *diaxial*, etc. corrections are sometimes needed to improve the estimated value and some of these corrections are not very well established or are missing; (3) in the case of solid compounds, the existence of phase transitions or fusion in the temperature interval where these processes take place can result in a larger heat capacity than can be predicted by an estimation technique.

During the past few years, we have been involved in the experimental determination of enthalpies of fusion, heat capacities and the study of polymorphism of pure organic compounds [14–19]. The present work reports the temperature, enthalpy and entropy of fusion, and heat capacities of several solid aldehydes measured by differential scanning calorimetry (DSC). The target compounds (Fig. 1) are 4-hydroxybenzaldehyde [123-08-0] **1**; 4-hydroxy-3-methoxybenzaldehyde (vanillin) [121-33-5] **2**; 3-ethoxy-4-hydroxybenzaldehyde (ethyl vanillin) [121-32-4] **3**; 3,4-dimethoxybenzaldehyde (veratraldehyde) [120-14-9] **4** and 4-methoxycinnamaldehyde [1963-36-6] **5**.

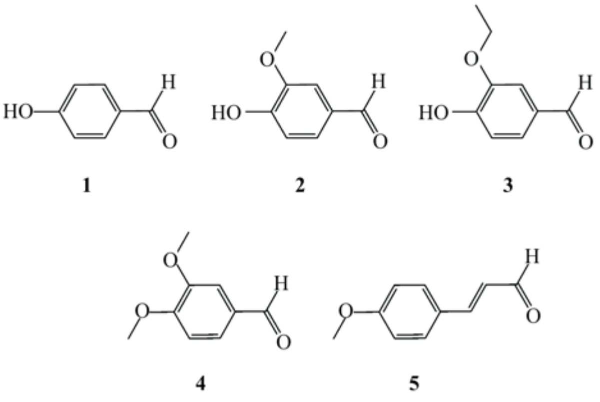

Fig. 1 Molecular formulas of the compounds studied

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1388–6150/$20.00

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Experimental

Materials

The compounds were all obtained from commercial vendors in high chemical purity (99+%). 4-Hydroxybenzaldehyde was carefully dried under vacuum at 50°C and the rest of the samples were dried at ambient temperature. Determination of purities, assessed by GC and DSC by the fractional fusion technique [20], indicated that the mole fraction of impurities in the compounds was less than 0.002, 0.0005, 0.001, 0.003 and 0.003 for 4-hydroxybenzaldehyde, 4-hydroxy-3-methoxybenzaldehyde, 3-ethoxy-4-hydroxybenzaldehyde, 3,4-dimethoxybenzaldehyde and 4-methoxycinnamaldehyde, respectively.

The standards used for DSC calibration were hexafluorobenzene, 99.9% purity, supplied by Aldrich; benzoic acid, NIST standard reference sample 39j; high-purity indium (mass fraction>0.99999), tin and synthetic sapphire, supplied by Perkin-Elmer.

Calorimetry

A differential scanning calorimeter (Perkin Elmer, Pyris 1) equipped with an intra-cooler unit was used to monitor purity, to study the fusion process and the possible existence of phase transitions in the solid samples, and to measure the heat capacity of all compounds. Temperature and power scales were calibrated [21–23] at heating rates of 0.04 and 0.17 K s^{-1}. The temperature scale was calibrated by the melting temperature of the high-purity reference materials: hexafluorobenzene, benzoic acid, tin and indium [24]. The power scale was calibrated with high-purity indium [24].

Curves of samples hermetically sealed in aluminium pans were recorded in a nitrogen atmosphere. All the pans with the samples were weighed on a Mettler AT21 microbalance with a detection limit of $1\cdot10^{-6}$ g, before and after the experiments to confirm that no product had volatilized.

After calibration, several runs with high-purity benzoic acid and indium as reference materials [24] were performed under the same conditions as the experimental determinations. The accuracies associated with temperatures and enthalpies of fusion measurements were calculated as the percentage deviation of the experimental data with regard to the values given in [24]; in all the cases this was lower than 0.2 and 2.0% for temperature and enthalpy determinations, respectively [16].

For determination of temperature and enthalpy of fusion, a heating rate of 0.04 K s^{-1} was used. For each solid compound, five or six samples weighing 1–2 mg were recorded. A fresh sample was used for each run. All compounds showed thermal stability in the fusion process.

Different scans were performed on heating, cooling and second heating, to determine the possible existence of phase transitions or polymorphism in the samples over the temperature range from T=268 K to their respective melting temperature at heating rates of 0.04 and 0.17 K s^{-1}.

Heat capacities were determined by the 'scanning method' following the experimental methodology previously described [25–27] with synthetic sapphire (α-aluminum oxide) as reference material [24–26]. DSC is a commonly accepted method for the quantitative determination of heat capacities and it has been proven as a suitable technique to obtain reliable and accurate values [28, 29]. To check the experimental method, heat capacity experiments were made with benzoic acid and synthetic sapphire as reference materials [24] in the temperature intervals T=268–360 and 268–410 K, respectively [18]. The relative percentage error of our measurements in comparison with those reported in the literature is less than 2% [18].

The mass of sapphire used in each run was 0.030345 g. For heat capacity determinations, four to six fresh samples weighing 10–25 mg were scanned for each solid compound in the temperature range from 268 K to its melting temperature at 0.17 K s^{-1}. The complete temperature ranges for determination of the heat capacities were divided in intervals of approximately 40 K, overlapping by 5 K from one interval to another. The estimated uncertainty of the molar heat capacities is less than 2%. The experimental results of each compound were fit to a third order polynomial in temperature.

Results and discussion

The fusion temperatures and enthalpies and the calculated entropy of fusion of the compounds are given in Table 1. The uncertainty was taken as the standard deviation of the mean. The melting temperatures, T_{fus}, were taken as DSC onset temperatures. Also included in the last column of the table is the estimated total phase change entropy, $\Delta_{\mathrm{tpce}}S_{\mathrm{m}}$ [30]. This term includes the total phase change entropy associated in going from T=0 K to the liquid at $T=T_{\mathrm{fus}}$. For compounds without any other phase transitions, this entropy change is identical to the fusion entropy.

The temperature of fusion reported in the literature for 4-hydroxy-3-methoxybenzaldehyde, vanillin, is in the range 354.3–356 K [31, 32]. The value of 355.1±0.9 K, taken as the average of seven independent measurements [31, 32], is in perfect agreement with our experimental result 355.4±0.1 K. For the rest of the compounds studied, to our knowledge, there are no other calorimetric data for comparison of our results. No solid–solid phase transitions were observed over the temperature interval from T=268 K to the corresponding melting temperature for all the aldehydes studied.

The mean experimental values of the heat capacity measurements as a function of temperature for the target compounds are given in Table 2.

The heat capacities of all the compounds were fit to a third order polynomial in temperature. The coefficients of the polynomial are reported in Table 3. The range studied for each compound is given as the last column of this table.

The errors associated in fitting the experimental data using these correlation equations are less than 2 J K^{-1} mol^{-1} for all compounds studied. The standard deviation of all the data associated with multiple measurements is less than 1.5 J K^{-1} mol^{-1}. To our knowledge there are no other $C_{p,m}$ data for comparison with our results.

Estimations of heat capacity of solids are more problematic than their liquid counterparts. This is due in part to the lack of data but also due to the anisotropic nature of the solid state. As mentioned previ-

Table 1 Temperatures, enthalpies and entropies of fusion for the compounds studied

Compound	T_{fus}/K	$\Delta_{fus}H_m$/kJ mol^{-1}	$\Delta_{fus}S_m$/J mol^{-1} K^{-1} exp	$\Delta_{tpce}S_m$/J mol^{-1} K^{-1} calc[a]
4-Hydroxybenzaldehyde	390.8±0.1	21.6±0.1	55.4±0.1	53.8
4-Hydroxy-3-methoxybenzaldehyde	355.4±0.1	22.4±0.2	63.1±0.5	60.0
3-Ethoxy-4-hydroxybenzaldehyde	349.8±0.1	23.1±0.2	66.0±0.5	67.1
3,4-Dimethoxybenzaldehyde	317.0±0.1	20.3±0.1	64.1±0.1	65.8
4-Methoxycinnamaldehyde	332.7±0.2	19.0±0.1	57.2±0.4	69.0

[a] [30]

Table 2 Mean experimental $C_{p,m}$(cr) values

T/K	$C_{p,m}$(cr)/J K^{-1} mol^{-1}	T/K	$C_{p,m}$(cr)/J K^{-1} mol^{-1}	T/K	$C_{p,m}$(cr)/J K^{-1} mol^{-1}
		4-Hydroxybenzaldehyde			
268.15	136.0	300.15	150.5	340.15	170.6
270.15	136.9	305.15	153.4	345.15	171.9
275.15	139.3	310.15	156.0	350.15	174.3
280.15	141.7	315.15	158.5	355.15	176.4
285.15	143.8	320.15	161.0	360.15	179.2
290.15	146.1	325.15	162.8	365.15	182.6
295.15	148.4	330.15	164.9	370.15	186.3
298.15	149.9	335.15	167.5	375.15	190.2
		4-Hydroxy-3-methoxybenzaldehyde			
268.15	172.9	295.15	185.9	320.15	200.2
270.15	174.2	298.15	187.6	325.15	202.1
275.15	176.3	300.15	188.6	330.15	204.2
280.15	178.6	305.15	191.4	335.15	205.8
285.15	180.7	310.15	194.8	340.15	209.2
290.15	183.1	315.15	198.0		
		3-Ethoxy-4-hydroxybenzaldehyde			
268.15	195.2	295.15	209.9	320.15	226.2
270.15	196.1	298.15	212.2	325.15	231.7
275.15	198.5	300.15	213.3	330.15	234.1
280.15	201.2	305.15	216.3	335.15	237.6
285.15	203.5	310.15	219.6	340.15	241.9
290.15	206.8	315.15	222.4		
		3,4-Dimethoxybenzaldehyde			
268.15	193.8	280.15	201.3	295.15	210.5
270.15	194.3	285.15	204.7	298.15	213.5
275.15	198.2	290.15	207.4	300.15	215.5
		4-Methoxycinnamaldehyde			
268.15	199.3	285.15	209.5	300.15	218.9
270.15	200.5	290.15	212.5	305.15	223.1
275.15	203.4	295.15	215.5	310.15	227.4
280.15	206.4	298.15	217.4	315.15	233.2

ously, phase transitions in solids can affect their heat capacities near these transitions. Solids that form liquid crystals, for example, seem to have larger heat capacities in certain temperature regions and phase change entropies that appear attenuated in comparison to systems that melt directly to isotropic liquids [33]. Group values for estimating the heat capacity of crystalline solids have been reported but the estimations in many cases has been hampered by the lack of sufficient data.

Table 4 summarizes some group values of one estimation scheme that has been proposed to estimate heat capacities of solids. A complete list of group values for this scheme may be found in [11]. Several new group contribution values have been reported [15, 19]. Furthermore, several values previously calculated based on few experimental data have been further refined using new experimental heat capacity values reported [15, 19]. Application of the existing group value for an aldehyde, 84.5 J mol^{-1} K^{-1}, to the aldehydes of this study resulted in highly divergent results. Since this group value was previously based on only two experimental results, both from the same publication, a search was conducted for additional experimental data. During the course of evaluating a group value for the aldehydes of this study, the group value for acyclic ethers, previously 49.8 J mol^{-1} K^{-1}, also seemed excessive in comparison to the existing group value of a CH_2 group reported in Table 4. These group values for solid ethers and aldehydes were therefore evaluated in light of new and existing data that has since become available. The new group values for both ethers and aldehydes evaluated in this work are reported in Table 4 and a summary of the experimental and calculated heat capacities used to generate these new values is provided in Table 5. Previously, the group value for ethers was generated from 8 experimental values. Seven of those values in addition to new values have been used. One was an outlier and is not included in Table 5. The two values previously used for aldehydes were reported by the same group and also appeared out of line with the remaining data; they were not used in the correlations. The group values used in the estimations reported in Table 5 are provided in Table 4 and with the exception of the group values for crystalline ethers and aldehydes, are identical to the values reported previously [11]. The new group value for ethers appears more in line now with the group value for a CH_2 group. The new values were obtained by minimizing the function: $[(C_{p,m}(298.15\ K)_{exp}-C_{p,m}(298.15\ K)_{calc})/$

Table 3 Coefficients of the 3rd order polynomials

Compound	A*	B*	C*(10^2)	D*(10^5)	r^2	Temperature range/K
1	–440.4	1438.2	–1242.7	394.9	0.999	268–375
2	1073.2	–2917.7	3017.0	–984.9	0.996	268–344
3	642.0	–1426.2	1371.6	–376.2	0.998	268–342
4	–3414.4	11318.4	–11997.1	4306.3	0.998	268–300
5	–4929.5	15867.2	–16504.0	5784.0	0.999	268–318

*Parameters corresponding to an equation of the type: $C_{p,m}(cr)=A+B(T/298.15)+C(T/298.15)^2+D(T/298.15)^3$

Table 4 A summary of group values (Γ) used in evaluation of solid ethers and aldehydes*

Group		Γ_i/J mol^{-1} K^{-1}	Group		Γ_i/J mol^{-1} K^{-1}
Primary sp^3 carbon	$-CH_3$	36.6	Alcohol	$-OH$	23.5
Secondary sp^3 carbon	$-CH_2-$	26.9	Chloride	$-Cl$	28.7
Tertiary sp^2 carbon	$=CH-$	21.4	Fluoride	$-F$	[24.8]
Tertiary aromatic sp^2 carbon	$=CH-$	17.5	Nitro group	$-NO_2$	56.1
Quaternary aromatic carbon	$=CH-$	8.49	Nitrile	$-CN$	[42.3]
Cyclic secondary sp^3 carbon	$=CC$	24.6	Primary amine	$-NH_2$	21.6
Cyclic tertiary sp^3 carbon	$-CH_2-$	11.7	Secondary amine	$-NHC$	[–0.3]
Cyclic tertiary sp^2 carbon	$-CHC-$	15.9	Tertiary amine	$-NC_2$	31.5
Cyclic quaternary sp^2 carbon	$-CC_2-$	4.73	Tertiary sp^2 nitrogen	$=N-$	10.7
Aldehyde	$-CHO$	33.1	Cyclic secondary amine	$-NH-$	[23.9]
Ether	$-O-$	25.2	Secondary amine	$-CONHC$	44.4
Cyclic ether	$-O-$	9.71	Carboxylic acid	$-CO_2H$	53.1
			Ester	$-CO_2C$	45.2

*Values in brackets are still considered tentative

Table 5 A comparison of experimental and calculated heat capacities of crystalline aldehydes and ethers

Compound	$C_{\mathrm{p,m}}(298.15\ \mathrm{K})/\mathrm{J\ mol^{-1}\ K^{-1}}$	
	exp.	calc
4-Hydroxybenzaldehyde[a]	149.9	143.6
4-Hydroxy-3-methoxybenzaldehyde[a]	187.6	196.4
3-Ethoxy-4-hydroxybenzaldehyde[a]	212.2	223.3
3,4-Dimethoxybenzaldehyde[a]	213.5	234.7
4-Methoxycinnamaldehyde[a]	217.4	191.6
2,4-Dichlorobenzaldehyde[b]	186.5	168.5
N,N-Dimethylaminobenzaldehyde[c]	234.0	224.8
4-Methoxybenzoic acid	207.5	201.9
p-Nitroethoxybenzene	246.0	231.8
Diphenyl ether	215.9	217.2
Di(*p*-methoxyphenyl)*trans*-1,4-cyclohexanedicarboxylate	478.6	500.0
[4-(Benzyloxy)phenyl]acetic acid[d]	340.0	315.1
4,4’-Diaminodiphenyl ether	278.2	242.4
N-(4-Methoxybenzylidene)-*p*-butylaniline	451.0	385.2
N-(2-Hydroxy-4-methoxy-benzylidene)-*p*-butylaniline	451.0	399.7
p-*n*-Hexyloxybenzylidene-*p*’-chlorobenzene	435.1	431.1
p-*n*-Ethoxybenzylidene-*p*’-butylaniline	429.3	412.1
p-*n*-Hexyloxybenzylidene-*p*’-toluidine	441.4	439.0
p-*n*-Hexyloxybenzylideneamino-*p*’-benzonitrile	432.6	444.7
N-*p*-*n*-Pentyloxybenzylidene-*p*’-*n*-butylaniline	478.0	492.8
N-*p*-*n*-Hexyloxybenzylidene-*p*’-*n*-butylaniline	512.1	519.7
4,4’-Dianilino-3,3’-diaminodiphenyl oxide	458.1	398.8
N,N’-*bis*(*m*-Methoxyphenyl)terephthalamide	458.1	473.3
N,N’-*bis*(*p*-Methoxyphenyl)terephthalamide	467.8	473.3
p-*n*-Hexyloxybenzylidieneamino-*p*’-fluorobenzene	442.7	427.2
Methyl-α-*D*-glucopyranose[e]	233.6	250.9
Methyl-β-*D*-glucopyranose[e]	270.4	250.9
Methyl-α-*D*-galactopyranose[e]	241.1	250.9
Methyl-β-*D*-galactopyranose[e]	225.2	250.9
Sucrose	425.1	413.0

[a]this work, [b][35], [c][36], [d][37], [e][38]

$C_{\mathrm{p,m}}(298.15\ \mathrm{K})_{\mathrm{exp}}]^2$. Unless otherwise noted, all heat capacities for the crystalline state reported in Table 5 were obtained from [34]. The average absolute error associated with the estimations reported in Table 5 is just under 6%.

Acknowledgements

We acknowledge the Spanish MCyT/DGI under project CTQ2006-12745 and CTQ2007-60895/BQU. Manuel Temprado thanks MEC/SEUI, AP2002-0603, Spain for financial support.

References

1 M. Zábranský, V. Růžička Jr. and E. S. Domalski, J. Phys. Chem. Ref. Data, 30 (2001) 1199.

2 M. Zábranský, V. Růžička Jr., V. Majer and E. S. Domalski, J. Phys. Chem. Ref. Data Monograph, 6 (1996).

3 M. Palczewska-Tulinska, D. Wyrzykowska-Stankiewicz, A. M. Szafranski and J. Cholinski, Solid and Liquid Heat Capacity Data Collection, Part 1: C_1-C_6, Dechema, Chem. Technik und Biotechnologie e.V., Frankfurt am Main 1997.

4 M. Palczewska-Tulinska, D. Wyrzykowska-Stankiewicz, A. M. Szafranski and J. Cholinski, Solid and Liquid Heat Capacity Data Collection, Part 1: C_6-C_{33}, Dechema,

Chem. Technik und Biotechnologie e.V., Frankfurt am Main, 1997.

5 M. Zábranský and V. Růžička, Fluid Phase Equilib., 194–197 (2002) 817.

6 E. Marti, E. Kaisersberger and E. Moukhina, J. Therm. Anal. Cal., 85 (2006) 505.

7 V. A. Drebushchak, Yu. A. Kovalevskaya, I. E. Paukov and E. V. Boldyreva, J. Therm. Anal. Cal., 85 (2006) 485.

8 B. Tong, Z. C. Tan, X. C. Lv, L. X. Sun, F. Xu, Q. Shi and Y. S. Li, J. Therm. Anal. Cal., 90 (2007) 217.

9 V. V. Novikov and A. V. Matovnikov, J. Therm. Anal. Cal., 88 (2007) 597.

10 Z. Nan and Z. C. Tan, J. Therm. Anal. Cal., 87 (2007) 539.

11 J. S. Chickos, D. G. Hesse and J. F. Liebman, J. Struct. Chem., 4 (1993) 261.

12 V. Růžička Jr. and E. S. Domalski, J. Phys. Chem. Ref. Data, 22 (1993) 597.

13 V. Růžička Jr. and E. S. Domalski, J. Phys. Chem. Ref. Data, 22 (1993) 619.

14 P. Jiménez, M. V. Roux, J. Z. Dávalos and M. Temprado, Thermochim. Acta, 394 (2002) 25.

15 M. V. Roux, M. Temprado, P. Jiménez, R. Guzmán-Mejía, E. Juaristi and J. S. Chickos, Thermochim. Acta, 406 (2003) 9.

16 M. V. Roux, M. Temprado, P. Jiménez, J. Z. Dávalos, C. Foces-Foces, M. V. García and M. I. Redondo, Thermochim. Acta, 404 (2003) 235.

17 M. V. Roux, M. Temprado, P. Jiménez, C. Foces-Foces, M. V. García and M. I. Redondo, Thermochim. Acta, 420 (2004) 59.

18 M. Temprado, M. V. Roux, P. Jiménez, R. Guamán-Mejía and E. Juaristi, Thermochim. Acta, 441 (2006) 20.

19 M. Temprado, M. V. Roux, A. R. Parameswar, A. V. Demchenko, J. S. Chickos and J. F. Liebman, J. Therm. Anal. Cal., 91 (2007) 471.

20 E. E. Marti, Thermochim. Acta, 5 (1973) 173.

21 E. Gmelin and S. M. Sarge, Thermochim. Acta, 347 (2000) 9.

22 S. M. Sarge, E. Gmelin, G. W. H. Höhne, H. K. Cammenga, W. Hemminger and W. Eysel, Thermochim. Acta, 247 (1994) 129.

23 GEFTA (Gesellschaft für Thermische Analyse, Germany), E. Gmelin and S. M. Sarge, Pure Appl. Chem., 67 (1995) 1789.

24 R. Sabbah, A. Xu-wu, J. S. Chickos, M. L. Planas Leitão, M. V. Roux and L. A. Torres, Thermochim. Acta, 331 (1999) 93.

25 S. C. Mraw and D. F. Naas, J. Chem. Thermodyn., 11 (1979) 567.

26 M. J. O'Neill, Anal. Chem., 38 (1966) 1331.

27 J. E. Callanan and S. A. Sullivan, Rev. Sci. Instrum., 57 (1986) 2584.

28 J. Pak, W. Qiu, M. Pyda, E. Nowak-Pyda and B. Wunderlich, J. Therm. Anal. Cal., 82 (2005) 565.

29 S. Rudtsch, Thermochim. Acta, 382 (2002) 17.

30 J. S. Chickos and W. E. Acree, Thermochim. Acta, 395 (2003) 59.

31 J. Bebie, J. Am. Pharm. Assoc., 19 (1930) 371.

32 V. V. Serpinski, S. A Voitkevich and N. Yu. Lyuboshich, Tr. Vses. Nauchno-Issled. Inst. Sint. Nat. Dushistykh Veshchestv., 4 (1958) 125.

33 W. Acree, Jr. and J. S. Chickos, J. Phys. Chem. Ref. Data, 35 (2006) 1051.

34 E. S. Domalski and E. D. Hearing, J. Phys. Chem. Ref. Data, 25 (1996) 1.

35 S.-X. Wang, Z.-C. Tan, Y.-Y. Di, F. Xu, H.-T. Zhang, L.-X. Sun and T. Zhang, J. Chem. Thermodyn., 36 (2004) 393.

36 S. H. Meng, P. Liang, Z.-C. Tan, Y. J. Song, L. Lja and L. Wang, Thermochim. Acta, 342 (1999) 47.

37 S. V. Kurkov, G. L. Perlovich and W. Zielenkiewicz, J. Therm. Anal. Cal., 83 (2006) 549.

38 A. R. Aguilar and E. O. Guareno, J. Chem. Thermodyn., 32 (2000) 767.

Received: November 19, 2007
Accepted: February 28, 2008
OnlineFirst: June 25, 2008

DOI: 10.1007/s10973-007-8883-0