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MEASUREMENT OF ENTHALPIES OF VAPORIZATION OF VOLATILE HETEROCYCLIC COMPOUNDS BY DSC

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An experimental procedure is proposed for direct measurement of the heat involved in the vaporization of volatile heterocyclic compounds. This technique consists on the vaporization of the liquid substance by a sudden decrease of the pressure then, the direct register of heat flow as function of time by differential scanning calorimetry. This procedure allows quantifying the enthalpy of vaporization of compounds such as tetrahydropyran, 2-methoxy-tetrahydropyran, N-morpholine and N-methyl-morpholine. Enthalpies of vaporization were measured in isothermal mode at T=298.15 K and then compared with results from the literature, which currently are obtained by vapour pressure measurements.

Keywords: *differential scanning calorimetry, enthalpy of vaporization, 2-methoxy-tetrahydropyran, N-methyl-morpholine, N-morpholine, tetrahydropyran*

Introduction

Experimental evaluation of thermochemical properties of liquid organic compounds in the gas phase requires determination of the vaporization enthalpies. In the same way, the design of optimal industrial chemical process involving liquids frequently requires of this parameter. The currently utilized methods to quantify the change of enthalpy accompanying the transformation liquid–gas need of considerable time and are in the most part indirect [1, 2], based on the measurement of vapor pressures as a function of temperature. This is due to the fact that a high volatility and, in consequence high velocities of evaporation and other technical problems inherent to the liquid–gas change phase make difficult the calorimetric measurement of the enthalpy of vaporization of i.e. liquid hydrocarbons [3–5]. Furthermore, these calorimetric determinations are currently performed using a Calvet type microcalorimeter [3–5], which is not always available equipment in Thermochemistry research laboratories.

Differential scanning calorimetry has been a valuable technique for the study of the thermal behavior of organic and inorganic substances. Though, carrying out adequate modifications, a scanning calorimeter can also be developed as an instrument able to measure directly the enthalpy accompanying the mixing of liquids [6] and, the heat transfer involved in liquid–gas [7–10] and solid–gas [11–16] change of phase of organic substances.

In previous works the enthalpy of sublimation and vaporization of solid organic compounds has been measured by DSC, founding reliable results [15, 16]. On this line of research, in the current work we measure the enthalpy of vaporization of liquid organic compounds using our modified version of a differential scanning calorimeter DSC7. The measurement of the heat associated with the vaporization of volatile liquids present some experimental difficulties, being the most important the control of the mass loosing that occurs in the lapse of the thermal stabilization at the temperature of the sensor holders. Specific corrections for this cause are then carefully described and taken into account to calculate the molar enthalpy of vaporization of each liquid.

Chemical structures of the compounds involved in this study are shown in Fig. 1. Tetrahydropyran and 2-methoxy-tetrahydropyran, have been chosen for this series of experiments given our interest in the thermochemistry of pyranosides derivatives [17], while N-morpholine and N-methyl-morpholine were selected as key compounds in the development of a thermochemical study of heterocyclic compounds containing oxygen and nitrogen.

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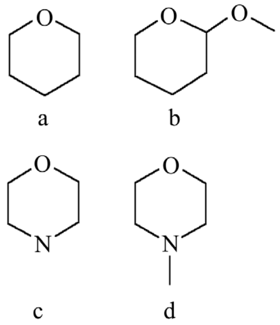

Fig. 1 Chemical structure of the studied compounds. a – tetrahydropyran, b – 2-methoxy-tetrahydropyran, c – N-morpholine, d – N-methyl-morpholine

Experimental procedure

The substances used in this work were commercial samples, the tetrahydropyran was a Fluka product, with a massic fraction purity of 99%. The 2-methoxy-tetrahydropyran, the N-morpholine and the N-methyl-morpholine were Aldrich products with a massic fraction purity of 98%, 99+% and 99%, respectively. All the substances were purified twice by fractional distillation under nitrogen atmosphere.

The calorimetric measurements of the enthalpies of vaporization were performed using the isothermal mode of the DSC7 calorimeter. For this type of determinations, modifications on the commercial apparatus have been carried out [15, 16]. The sensitive element of our device is a DSC7 calorimetric holder assembly, which was placed into a vacuum chamber. A heat exchanger, made in copper and aluminum, connected to a constant temperature bath allows the temperature regulation of the entire calorimetric holder. In the vacuum line a cold trap is assembled to condense most of the vaporized liquid, avoiding internal condensation in the rotary vacuum pump, which evacuates the vaporization system. The residual pressure inside of the chamber is monitored by a pressure gauge relayed to a Pirani gauge control. The calorimetric holder assembly is connected to the DSC7 control device using an electrical feedthrough. A set of three valves between the vaporization system and the rotary vane pump, allows the evacuation of the vaporization chamber and a control of the residual pressure. The calorimetric assembly, the vacuum chamber and all the accessories of the vaporization system are autonomous and, all other normal function of the DSC7 device can work independently of the described modifications. Currently the calorimetric holder assembly inside of the vacuum system is calibrated for energy and temperature using high-purity samples of indium and zinc at scanning rate between 1.0 K and 5.0 K min^{-1}.

The commercial pans for volatile samples (Perkin Elmer, 0219-0062) were utilized as vaporization cells, but previously were modified making a hole with a drill of diameter 0.3 mm. After drilling, the edge of the hole was sanded with a fine sanding film. This diameter of vaporization hole was chosen as most suitable because perfectly circular orifices of shorter diameter could not be done and, larger diameters caused a quick vaporization with a time of the experiment too short to reach a good register of the transferred heat, furthermore to an uncontrollable loss of mass previous to the calorimetric register.

From preliminary experiments, the working temperatures for the vaporization were established at 298.15 K. This set of experiments was also useful to verify the range of the calorimetric signal and the time for the total vaporization of each compound. This information and the mass of the sample are required for the isothermal operation of the DSC7 calorimeter.

In a typical vaporization experiment, an empty drilled pan for vaporization is weighed; then around 10 mg of the liquid compound are placed inside and the pan is sealed using a pan sealer (Perkin Elmer 219-0061). The pans and samples are quickly weighed on a Sartorius 4503 microbalance, sensitive to 1 μg (inaccuracy ±1 μg). The pan is then loaded in the sample holder and, an identical but empty vaporization pan is loaded in the reference holder and, the vacuum chamber is closed. Once inside of the calorimetric system, temperature control of the calorimetric holders maintains it at 298.15 K. Previous to the calorimetric register, some minutes are necessary for temperature and heat flux stabilizing and after this, data acquisition begins. 5 min are enough to get a good initial baseline, then the valve connected to the vacuum pump is opened, the pressure inside the vacuum chamber is downloaded quickly and the vaporization process of the liquid is carried out.

The speed of vaporization is controlled by regulation of the set of valves of the vacuum system, getting a pressure in the range of 2000 to 1500 Pa twenty seconds after the initial punching. Typical profiles of the registered curves are shown in Fig. 2 and are related to the diameter of the vaporization hole, the volatility and the initial mass of the substance.

Figure 3 shows a detailed description of the sharpened peak involving the vaporization process of 9.5 mg of tetrahydropyran. The evacuation of the vaporization chamber and, in consequence the change of phase beginnings at t_b. The amplitude of the vaporization signal for this liquid is more than 150 mW, and the energy involved in the process is around 3.2 J. Once the substance is evaporated, the calorimetric signal returns to the baseline and, at the time t_e, the vaporization process is ended. Below the peak of vaporization, there is a difference between the initial and final baseline. This dissimilarity is consequence of some asymmetry and different heat capacities of the full and empty sample pan; however, in Fig. 3, this difference is nearly imperceptible, given that in most of the experiments is less than 2 mW. In order to reduce any error due to this inequality, integration of the area under the peak was systematically performed in a straight line between the points t_b and t_e.

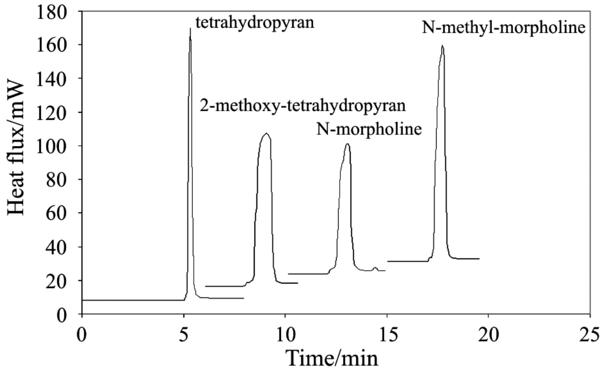

Fig. 2 Profile of the experimental vaporization curves

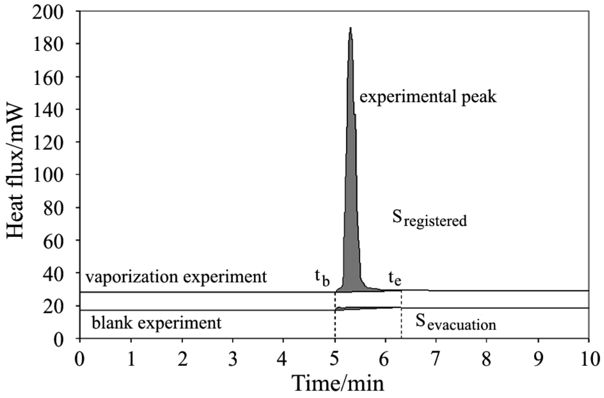

Fig. 3 Detailed vaporization curve and evacuation blank experiment to compare the respective areas

During the loading of the sample pan in the calorimeter and the lapse of thermal stabilization, a fraction of the liquid vaporizes before the calorimetric register. In consequence, an exact result of the enthalpy of vaporization requires calculate the mass lost in that interval and it was performed by independent experiments. In this set of experiments, a vaporization pan was also charged with masses of each liquid similar to those of the experiments of vaporization and was positioned in the sensor holder. The substance was stabilized for five minutes, as in a vaporization experiment, but after this lapse, the vacuum chamber was opened and immediately the sample was reweighed and the mass lost due to the loading and, thermal stabilization, was computed. Results of these experiments for each compound are reported in Table 1. There, the uncertainty associated to these results represents the standard deviation.

The area of the peak of vaporization was evaluated using the integration peak subroutine of the software of the DSC7 1020 controller, and represents the heat transferred per mass unit during the vaporization process but this peak includes also a small thermal effect due to the evacuation of the vacuum chamber. This effect was also quantified after each measurement vaporization experiment as follows. Once the vaporization experiment concluded, the vacuum inside the chamber is broken up, but only to be evacuated again, maintaining the same empty set of vaporization pans in the calorimetric holders. For this blank experiment data acquisition is restarted and, only the thermal effect due to evacuation is registered

Table 1 Mass lost during preparation and thermal stabilization of an experiment of vaporization

$m_{initial}$/mg	m_{final}/mg	m_{lost}/mg	m_{lost}/%	$m_{initial}$/mg	m_{final}/mg	m_{lost}/mg	m_{lost}/%
	tetrahydropyran				2-methoxy-tetrahydropyran		
6.163	5.589	0.574	9.314	11.600	11.480	0.120	1.034
5.589	5.109	0.480	8.588	11.480	11.378	0.102	0.889
5.109	4.609	0.500	9.787	11.378	11.242	0.136	1.195
4.609	4.114	0.495	10.740	11.242	11.127	0.115	1.023
4.114	3.666	0.448	10.890	11.127	11.020	0.107	0.962
3.666	3.223	0.443	12.084	11.020	10.892	0.128	1.162
	m_{lost}/%=10.23±1.25				m_{lost}/%=1.04±0.12		
	N-morpholine				N-methyl-morpholine		
13.011	12.959	0.052	0.400	7.724	7.601	0.123	1.62
12.959	12.907	0.052	0.401	4.601	4.474	0.127	2.84
12.907	12.844	0.063	0.488	4.474	4.352	0.122	2.80
12.844	12.783	0.061	0.475	4.352	4.222	0.130	3.08
12.783	12.723	0.060	0.469	4.222	4.090	0.132	3.23
				4.090	3.960	0.130	3.28
	m_{lost}/%=0.45±0.04				m_{lost}/%=2.81±0.62		

$m_{initial}$ is the mass of the sample measured previous to the preparation and stabilization experiment; m_{final} is the mass of the sample measured after the preparation and stabilization experiment; m_{lost} is the mass lost during the preparation and stabilization experiment and quantified. The small profile of this evacuation peak is shown in Fig. 3, where is also compared with the main vaporization peak. The energy comprised in each evacuation peak was between 30 and 250 mJ, representing 2.0 to 6.6% of the total energy of a vaporization experiment.

Results and discussion

The actual area due to the vaporization process $S_{\text{vaporization}}$ is calculated as the difference between the registered area in the measurement experiment $S_{\text{registered}}$ and the area of the evacuation peak registered in the blank experiment $S_{\text{evacuation}}$. In similar way, the actual mass of substance vaporized in the experiment $m_{\text{vaporized}}$, is known from the difference of the initial mass m_{initial}, measured in the balance and, the mass lost during preparation and thermal stabilization for the experiment m_{lost}. The vaporization heat in J g^{-1} was obtained using the relation:

$$Q_{\text{vap}}\,(\text{J g}^{-1}) = \frac{[S_{\text{registered}} - S_{\text{evacuation}}](\text{mW s})}{[m_{\text{initial}} - m_{\text{lost}}](\text{mg})} = \frac{S_{\text{vaporization}}\,(\text{mW s})}{m_{\text{vaporized}}\,(\text{mg})} \quad (1)$$

Table 2 shows the experimental values of all the quantities involved in Eq. (1), and the resulting enthalpies of vaporization for each studied compound. The mean value for the enthalpy of vaporization was obtained at the working temperature of 298.15 K with at least nine experiments. This quantity was calculated employing the molar mass of 116.158, 86.132, 87.120 and 101.147 g mol^{-1}, for the tetrahydropyran, the 2-methoxy-tetrahydropyran, the N-morpholine and the N-methyl-morpholine respectively, which were calculated from data of atomic masses recommended by the IUPAC [18].

Given that the pressure throughout the vaporization experiment does not remain constant, a factor of correction must be considered over the heat of vaporization given for Eq. (1) in order to obtain the actual value of enthalpy of vaporization. Morawetz [3] demonstrated that this correction could be quantified as:

$$\delta\Delta_{\text{vap}}H = RT(1 - P/P_0) \quad (2)$$

where P is the vaporization cell external pressure and P_0 is the saturation pressure of the liquid passing to the gaseous phase in the vaporization process.

To estimate the magnitude of this factor in our experiments, the progress of the pressure inside of the vacuum chamber for some vaporizations of the N-morpholine was followed. A slow evolution of the pressure around 1900 Pa begins 20 s after the initial punching, while the calorimetric signal has just surpassed the maximum of the vaporization peak, then the pressure continues dropping, being at the end of the vaporization process of around 530 Pa. Available literature data for the N-morpholine report a vapour pressure of 1239 Pa at 298.5 K [19], so the factor of correction calculated from Eq. (2) goes from –1.32 to 1.42 kJ mol^{-1}, values that are comparable to twice the standard deviation calculated from the nine experimental results of $\Delta_{\text{vap}}H_{\text{m}}$ of the N-morpholine reported in Table 2. In order to include a similar correction in all our results, the uncertainty associated with the average value of enthalpy of vaporization of each of the four liquids here studied is then twice the standard deviation.

Table 3 shows the enthalpies of vaporization, at the experimental temperature and at T=298.15 K, measured in this work and compared with the few reliable values available in the literature for the heterocyclic compounds here studied [19–22]. No calorimetric data of $\Delta_{\text{vap}}H_{\text{m}}$ have been reported for these substances [1, 2].

For the N-morpholine, vapour pressure measurements or thermogravimetry procedure have been employed in the study of the change of phase for this substance [19, 20]. In Table 3, from the reported results at the experimental temperature, the values of the enthalpy of vaporization at 298.15 K were calculated employing the Sidgwick correction, in the same way as is reported in [19]. The result of $\Delta_{\text{vap}}H_{\text{m}}$ (298.15 K)=(45.30±1.48) kJ mol^{-1} provided in the current work, presents an excellent agreement with the most recent value of (45.0±0.4) kJ mol^{-1} reported for this substance [19].

Concerning the N-methyl-morpholine the most accurate values of the enthalpy of vaporization probably are also those of [19, 20]. Correction in temperature to 298.15 K was done on those experimental results utilizing also the Sidgwick method, leading to the values of (36.3±0.34) and (39.6±0.26) kJ mol^{-1}. Our result of (38.16±1.06) kJ mol^{-1} is near to the higher of these values, which is also the most recently reported [19].

For the tetrahydropyran results of $\Delta_{\text{vap}}H_{\text{m}}(T)$ are between 35.0 and 33.2 kJ mol^{-1}, obtained by vapour pressure measurements of the pure compound [21] or from a mixture of the tetrahydropyran with chlorocyclohexane [22], respectively. Temperature correction on those experimental values was done by use of the equation:

$$\Delta_{\text{vap}}H_{\text{m}}(298.15\text{ K}) = \Delta_{\text{vap}}H_{\text{m}}(T) - \int_{298.15\text{ K}}^{T} \Delta_{\text{vap}}C_{\text{p,m}}\,\text{d}T \quad (3)$$

with a $\Delta_{\text{vap}}C_{\text{p,m}}$= –50.5 J K^{-1} mol^{-1}, calculated from $C_{\text{p,m}}$ data of the liquid phase [23] and the gas phase [24], giving values of enthalpy of vaporization at T=298.15 K of 35.8 and 34.0 kJ mol^{-1}. The result of (32.73±2.20) kJ mol^{-1} obtained in this work by DSC is

Table 2 Experimental data and resulting enthalpies of vaporization of the studied liquids

m_{initial}/ mg	m_{lost}/ mg	$m_{\text{vaporized}}$/ mg	$t_{\text{vaporization}}$/ s	$S_{\text{registered}}$/ mW s	$S_{\text{evacuation}}$/ mW s	$S_{\text{vaporization}}$/ mW s	Q_{vap}/ J g^{-1}	$\Delta_{\text{vap}}H_{\text{m}}$/ kJ mol^{-1}
				tetrahydropyran				
9.415	0.964	8.451	60	3242.9	31.3	3211.7	380.01	32.73
5.335	0.546	4.789	60	1808.4	32.5	1775.8	370.81	31.94
10.395	1.064	9.331	90	3381.9	73.9	3308.0	354.51	30.53
7.444	0.762	6.682	60	2623.5	44.6	2578.9	385.94	33.24
9.685	0.991	8.694	60	3437.4	38.7	3398.7	390.93	33.67
6.483	0.663	5.820	54	2358.7	53.6	2305.1	396.10	34.12
6.979	0.714	6.265	60	2458.6	53.2	2405.5	383.96	33.07
4.774	0.489	4.285	71	1734.2	76.5	1657.7	386.83	33.32
7.464	0.764	6.700	62	2631.2	46.2	2585.1	385.83	33.23
4.761	0.487	4.274	54	1598.0	39.4	1558.6	364.69	31.41
								32.73±2.20
				2-methoxy-tetrahydropyran				
12.255	0.128	12.127	131	4087.9	54.6	4033.3	332.59	38.63
9.399	0.098	9.301	180	3255.8	78.7	3177.1	341.59	39.68
9.886	0.103	9.783	150	3451.0	146.8	3304.2	337.75	39.23
9.736	0.102	9.634	216	3295.3	115.3	3180.0	330.07	38.34
10.515	0.110	10.405	216	3665.4	149.1	3516.3	337.93	39.25
11.326	0.118	11.208	156	3955.3	107.9	3847.4	343.28	39.87
8.847	0.092	8.755	234	3122.8	94.2	3028.6	345.94	40.18
4.054	0.042	4.012	154	1541.3	103.5	1347.8	335.96	39.02
11.382	0.119	11.263	195	3946.5	91.5	3855.0	342.27	39.76
								39.33±1.20
				N-morpholine				
5.162	0.023	5.139	148	2794.5	83.4	2711.2	527.59	45.96
6.014	0.027	5.987	149	3226.5	212.9	3013.6	503.36	43.85
12.226	0.055	12.171	227	6512.3	169.3	6343.0	521.16	45.40
10.079	0.045	10.034	186	5313.8	157.2	5156.7	513.94	44.77
8.798	0.040	8.758	196	4812.2	242.8	4569.5	521.72	45.45
7.286	0.033	7.253	184	3971.3	98.3	3873.0	533.97	46.52
10.175	0.046	10.129	197	5428.7	159.1	5269.6	520.23	45.32
13.585	0.061	13.524	217	7243.6	220.3	7023.3	519.33	45.24
5.626	0.025	5.601	242	3156.4	254.6	2901.8	518.12	45.14
								45.30±1.48
				N-methyl-morpholine				
8.998	0.236	8.762	190	3522.4	207.7	3314.7	378.29	38.26
7.773	0.204	7.569	119	3044.0	140.8	2903.2	383.55	38.79
7.095	0.186	6.909	112	2758.4	135.8	2622.6	379.59	38.39
7.468	0.196	7.272	168	2868.2	187.3	2680.9	368.64	37.29
5.779	0.151	5.628	121	2290.8	188.6	2102.2	373.55	37.78
6.467	0.169	6.298	137	2493.0	135.7	2357.3	374.32	37.86
5.056	0.132	4.924	170	2064.9	213.5	1851.5	376.04	38.04
5.887	0.154	5.733	107	2337.5	135.8	2201.8	384.06	38.85
8.003	0.210	7.793	182	3154.5	171.0	2983.5	382.83	38.72
5.104	0.134	4.970	119	2027.4	178.6	1848.9	371.98	37.62
								38.16±1.06

m_{initial}, mass of liquid measured in the balance; m_{lost}, mass lost in the lapse of preparation and thermal stabilization of the experiment; $m_{\text{vaporized}}$, mass vaporized during the calorimetric register; $t_{\text{vaporization}}$, time of vaporization; $S_{\text{registered}}$, area under the registered experimental peak; $S_{\text{evacuation}}$, area due to the evacuation of the vacuum chamber; $S_{\text{vaporization}}$, actual area due to the vaporization process; Q_{vap}, heat of vaporization; $\Delta_{\text{vap}}H_{\text{m}}$, molar enthalpy of vaporization

lower than those previously reported. The difference can be explained by the fact that this liquid is the most volatile of all here studied, then the loss of mass before the calorimetric vaporization register is difficult to control. The high vaporization speed of this liquid can have also as consequence, a no total transfer of heat from the sample to the sensor holder throughout all the vaporization process.

Finally, for the 2-methoxy-tetrahydropyran, no comparison of results is possible given that in our

Table 3 Comparison of results for the enthalpy of vaporization of the heterocyclic compounds studied

Reference	Method	Temperature/K	$\Delta_{vap}H_m(T)$/kJ mol^{-1}	$\Delta_{vap}H_m$(298.15 K)/kJ mol^{-1}
		N-morpholine		
19	vapour pressure measurements	288.50	45.55±0.36	44.97±0.36
20	thermogravimetry	328.00	44.30±0.44	46.12±0.44
this work	differential scanning calorimetry	298.15	45.30±1.48	45.30±1.48
		N-methyl-morpholine		
19	vapour pressure measurements	288.50	40.20±0.26	39.62±0.26
20	thermogravimetry	343.00	33.60±0.34	36.29±0.34
this work	differential scanning calorimetry	298.15	38.16±1.06	38.16±1.06
		tetrahydropyran		
21	vapour pressure measurements	280.70	34.95	35.8
22	vapour pressure measurements	350.00	33.20	34.0
this work	differential scanning calorimetry	298.15	32.73±2.20	32.73±2.20
		2-methoxy-tetrahydropyran		
this work	differential scanning calorimetry	298.15	39.33±1.20	39.33±1.20

knowledge no previous indirect or calorimetric measurements of the enthalpy of vaporization of this substance have been performed.

Conclusions

We conclude that the enthalpy change measurements of the vaporization process of volatile liquids at constant temperature, as is carried out in this work, produce results in very good agreement respect to those obtained by indirect techniques, but with the advantage that measurements are performed employing a calorimetric technique and the study is carried out faster than vapour pressure measurements. Nevertheless, an enhancement of the uncertainty interval associated with the average value of enthalpy is convenient, this in order to include any correction due to no total control of pressure throughout the vaporization process.

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