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The design, construction, and testing of a new Knudsen effusion apparatus

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Abstract

A new Knudsen effusion apparatus, enabling the simultaneous operation of nine effusion cells at three different temperatures, is fully described. The performance of the new apparatus was checked by measuring the vapour pressures, between 0.1 Pa and 1 Pa, over *ca.* 20 K temperature intervals of benzoic acid, phenanthrene, anthracene, benzanthrone, and 1,3,5-triphenylbenzene. The derived standard molar enthalpies of sublimation are in excellent agreement with the mean of the literature values available for these five compounds and with the recommended values for four of them.

Keywords: Effusion apparatus; Knudsen effusion; Vapour pressures; Enthalpy of sublimation; Entropy of sublimation; Benzoic acid; Phenanthrene; Anthracene; Benzanthrone; 1,3,5-Triphenylbenzene

1. Introduction

The Knudsen effusion method [1–3] is one of the most widely used methods for measuring the vapour pressures of crystalline organic compounds for pressures less than 1 Pa. In a typical effusion experiment, the crystalline sample is placed at the bottom of a cylindrical cell kept at a constant temperature and the vapour (assumed to be in equilibrium with the crystalline phase) is allowed to effuse through a small orifice located at the top of the cell into an evacuated space. At the temperature T, the mass m of the sample sublimed from the effusion cell, during the time period t, is related to the vapour pressure of the crystalline compound by the following equation:

$$p = (m/A_o w_o t) \cdot (2\pi RT/M)^{1/2}, \tag{1}$$

where M is the molar mass of the effusing vapour, R is the gas constant, A_o is the area of the effusion orifice and w_o is the transmission probability factor which is usually calculated by means of equation (2) or of equation (3) where l is the length of the effusion orifice and r its radius:

$$w_o = \{1 + (3l/8r)\}^{-1}, \tag{2}$$

$$w_o = \{1 + (l/2r)\}^{-1}. \tag{3}$$

This method has been widely used by our Research Group for measuring the vapour pressures of several organic compounds using an effusion apparatus enabling the simultaneous operation of three effusion cells at each experimental temperature [4]. As each effusion cell has a different effusion orifice area, the obtained results may be checked for deviations from the equilibrium pressure. If the areas of the effusion orifices are not very different, the pressures calculated at each temperature for each effusion cell are usually equal to within experimental error. For some compounds, however, the calculated pressures systematically decrease with the increasing size of the effusion orifice indicating that the results may be affected by a low condensation coefficient value or by a self cooling effect [5,6]. In this case, according to the equation developed by

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Whitman [7] and Motzfeldt [8], the equilibrium pressure at each temperature may be derived by plotting p against (pw_oA_o), to obtain the intercepts of the derived straight lines at zero area as the equilibrium pressures.

The new apparatus presented in this work enables the simultaneous operation of nine effusion cells, which may be controlled at three different temperatures, during one effusion experiment. By keeping the same temperature for each group of three effusion cells with different orifice areas, deviation of results from the equilibrium pressures at three different temperatures may be checked, simultaneously. So in one experimental run the equilibrium pressures at three different temperatures are determined.

2. The experimental apparatus and procedure

Besides the possibility of the simultaneous operation of nine effusion cells instead of only three, the main differences between the new effusion apparatus and the previous one are related to the control and measurement of the effusion temperature. The previous thermostatic oil or water bath has been replaced by temperature controlled aluminium blocks enabling experimental measurements between ambient temperature and *ca.* 480 K. The temperatures are measured using platinum resistance probes instead of mercury thermometers. A schematic representation of the apparatus is presented in figure 1.

2.1. The pumping system

The main components of the pumping system are the rotary pump (Edwards model RV12) which is used for pre-evacuating the system and for backing the oil diffusion pump (Edwards cryo-cooled diffstack model CR160). The pumping system enables the achievement of a pressure lower then $5 \cdot 10^{-4}$ Pa in less than one minute and an ultimate pressure of $5 \cdot 10^{-5}$ Pa.

2.2. The sublimation chamber

Each effusion cell is contained in one of the three cylindrical holes inside the aluminium blocks. The three aluminium blocks are contained inside the sublimation chamber, represented in figure 2, consisting of a glass bell jar ($\phi_i = 296$ mm, $h = 360$ mm, $l = 5$ mm) with a cylindrical aluminium lid. Each block contains three cylindrical holes of dimensions similar to the effusion cells and is connected to a sliding aluminium platform by three ceramic elements. To prevent sample contamination of the pumps, the glass connection between the pumping system and the sublimation chamber includes a glass cold finger for liquid nitrogen connected to the lid of the sublimation chamber.

2.3. Temperature measurement and control

Each aluminium block may be heated to the desired temperature by two circular heating elements – fast response 115 Ω electrical resistances from Ari, model Aerorod BXX – connected in parallel to a power supply of 40 or 60 V, ac, depending on the controlled temperature. The temperature of each block is kept constant by a PID (proportional, integral and differential) controller receiving information of a Pt-100 sensor located near the heating element as shown in figure 3. The temperature of each block is measured by

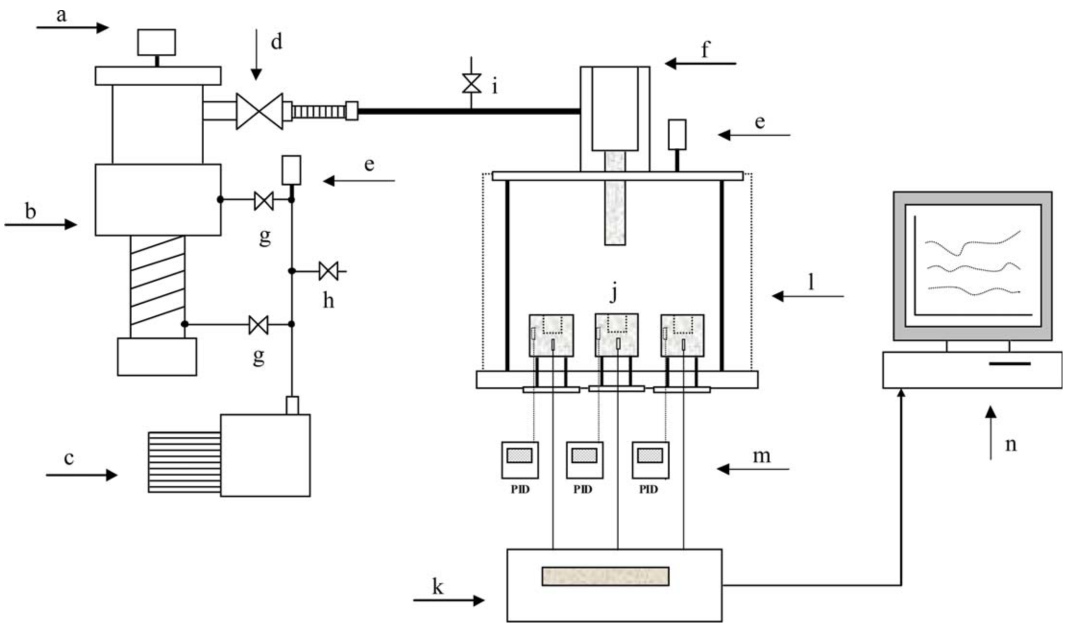

FIGURE 1. Schematic representation of the new effusion apparatus: a, inverted magnetron gauge Edwards AIM-S; b, oil diffusion pump Edwards cryo-cooled diffstak CR160; c, Rotary pump Edwards RV12; d, isolation valve Edwards IPV40 MKS; e, Pirani gauges Edwards APG-M; f, glass cold finger for liquid nitrogen; g, Speedivalves Edwards SP25K; h, air admittance valve AV10K; i, teflon greaseless gas admittance valve J. Young ALS1; j, aluminium blocks (ovens); k, data logger Agilent 34970A; l, glass bell jar; m, PID temperature controllers Omron E5CN; n, computer.

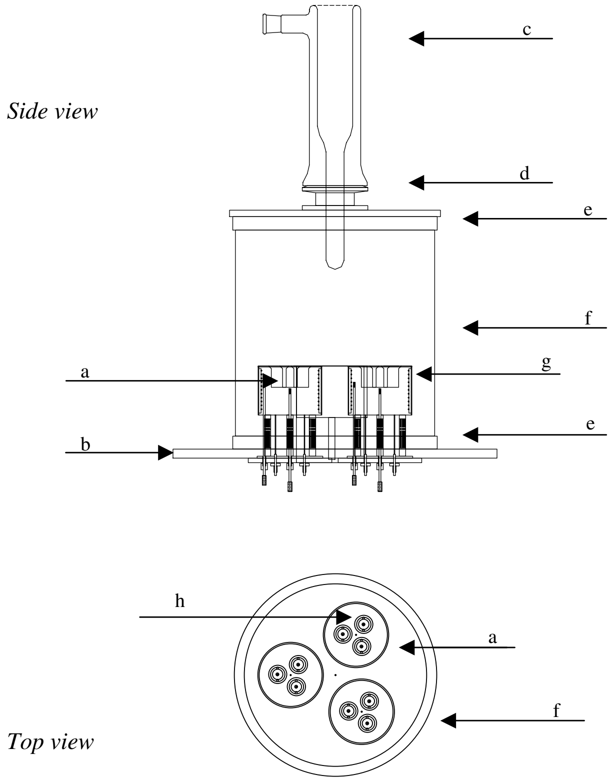

FIGURE 2. Side and top views of the vacuum chamber: a, aluminium blocks (ovens); b, sliding aluminium platform; c, glass cold finger for liquid nitrogen; d, glass-metal connection; e, neoprene seal; f, glass bell jar; g, effusion cells cavities; h, effusion cells.

a platinum resistance thermometer Pt-100 class 1/10. All the Pt-100 sensors were calibrated against a SPRT (25 Ω; Tinsley, 5187A) temperature probe, using an ASL bridge model F26 in accordance to ITS-90. Each sensor is located at the centre of the block near the basis of the holes containing the effusion cells. The signals of the thermometer sensors are received by an acquisition system, Agilent model 34970A, connected to a PC that continuously displays, with a resolution of 10^{-3} K, the temperature of the effusion cells which are assumed to be in thermal equilibrium with each aluminium block.

2.4. The effusion cells

The cylindrical effusion cells are made in aluminium. On the top of each cell an aluminium lid with a central hole of $\phi = 10$ mm is attached by means of a fine-pitched screw thread. The internal dimensions of the closed cells are diameter 20 mm and height 23 mm. The external dimensions are similar to the dimensions of the holes in the aluminium blocks: diameter 23 mm and height 27 mm. A thin platinum disk (diameter 21 mm and thickness 0.0125 mm) is mounted on each lid according to the scheme presented in figure 4. The disk is placed between a teflon washer and a brass washer which is pressured against the lid through a screw thread brass ring.

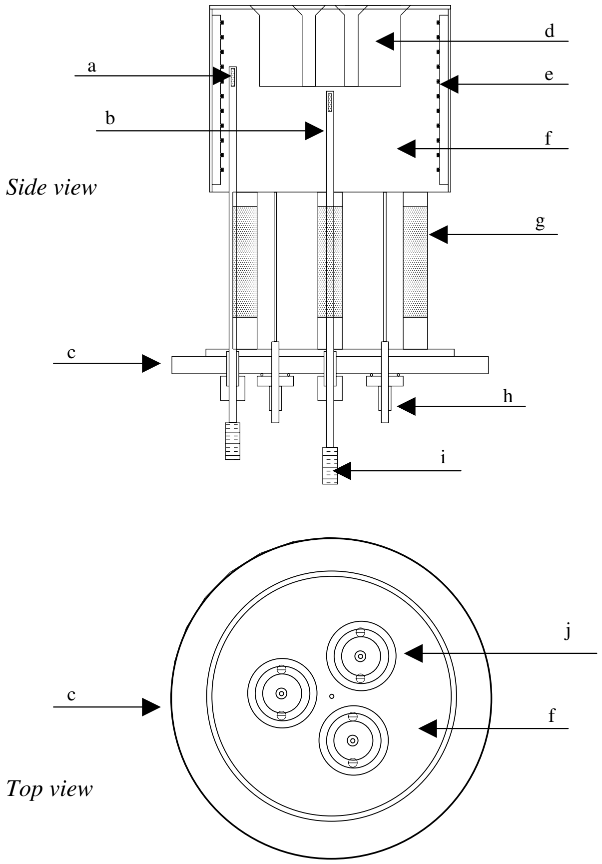

FIGURE 3. Side and top views of the aluminium blocks (ovens): a, platinum resistance thermometer, Pt100, connected to the PID controller; b, platinum resistance thermometer, Pt100, for the temperature measurement; c, aluminium base plate; d, cells cavities; e, circular heating elements; f, aluminium block; g, ceramic insulator; h, heating elements connections; i, thermometer connections; j, effusions cells.

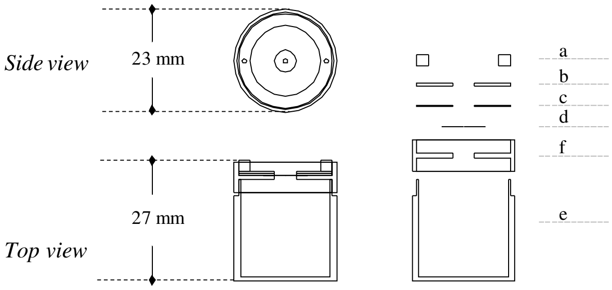

FIGURE 4. Side and top views of the effusion cell: a, brass ring; b, brass disk; c, teflon disk; d, Platinum foil; e and f, aluminium cell with aluminium lid.

2.5. Experimental procedure

The sample is compressed inside the cells by a brass piston in order to obtain a flat surface and to improve the thermal contact. The amount of sample used is the quantity necessary to obtain a disk of 3 to 5 mm height after the compression. The cells holding the sample are weighed, on an analytical balance (Mettler H54), with an accuracy of ±0.01 mg. After weighing the cells are lubricated with a thin layer of Apiezon L at the bottom and introduced inside the holes of the aluminium blocks. Although the cells fit the cylindrical holes very tightly, tests showed that inconsistent results were obtained when the cells were not lubricated. After mounting the bell jar and the aluminium lid, the blocks containing the cells were heated to the desired temperatures. The sublimation chamber is connected to the pumping system by means of a glass line containing the cold finger. After allowing for thermal stabilization of the cells, the sublimation chamber is connected to the pumping system using the isolation valve (figure 1, d). When the pressure is lower than 1 Pa, the cold finger is filled with liquid nitrogen and the effusion time period is considered to start. In less than one minute, after opening the gate valve, a pressure lower than $5 \cdot 10^{-4}$ Pa is obtained. When the chosen effusion time period (usually between 3 and 8 h, depending on the vapour pressure) is over, the isolation valve is closed and dry air is allowed to enter into the sublimation chamber, by opening the teflon valve (figure 1, i). After cooling to ambient temperature, the cells are carefully cleaned and weighed using the analytical balance.

3. Results

In order to test the quality of the results obtained with the new experimental apparatus, the vapour pressures of the following five compounds were measured over temperature intervals of *ca.* 20 K: benzoic acid and anthracene (recently recommended as primary standards for enthalpy of sublimation measurements [9]), phenanthrene and 1,3,5-triphenylbenzene (recently recommended as tertiary standards for enthalpy of sublimation measurements [9]), and benzanthrone for which we previously obtained results using different experimental apparatus.

Benzoic acid [65-85-0] (NBS Standard Reference Material 39i) was used without further purification. Anthracene [120-12-7] was obtained from Aldrich Chemical Co. with minimum mass fraction purity 0.99. The studied sample was purified by repeated sublimation under reduced pressure: G.C. analysis shows that the mass fraction purity was not less than 0.9999. Phenanthrene [85-01-8] was obtained from Aldrich Chemical Co with a minimum mass fraction purity 0.98, and further purified by zone refining and sublimed under reduced pressure: G.C. analysis shows that the mass fraction purity was higher than 0.998. The 1,3,5-triphenylbenzene [612-71-5], obtained from Aldrich Chemical Co. with a minimum mass fraction purity 0.97, was twice sublimed under reduced pressure: G.C. analysis shows that the mass fraction purity was higher than 0.997. Benzanthrone [82-05-3] was obtained from Fluka with a minimum mass fraction purity 0.98. The studied sample was purified by repeated crystallization from tetrachloroethane followed by sublimation at reduced pressure: G.C. analysis shows that the mass fraction purity was higher than 0.998.

Three series of effusion orifices were used. The areas and Clausing factors of the used effusion orifices, in platinum foil of 0.0125 mm thickness, are presented in table 1.

Table 2 presents, for each compound studied, the experimental results obtained from each effusion cell. Table 3 presents for each of the three groups of effusion cells used and for the global treatment of all the (p, T) points obtained for each compound studied (except for benzanthrone), the detailed parameters of the Clausius–Clapeyron equation, together with the calculated standard deviations and the standard molar enthalpies of sublimation at the mean temperature of the experiments $T = \langle T \rangle$. The equilibrium pressure at this temperature $p(\langle T \rangle)$ and the entropies of sublimation at equilibrium conditions, $\Delta_{\mathrm{cr}}^{\mathrm{g}} S_{\mathrm{m}}\{\langle T \rangle, p(\langle T \rangle)\} = \Delta_{\mathrm{cr}}^{\mathrm{g}} H_{\mathrm{m}}^{\circ}(\langle T \rangle)/\langle T \rangle$, are also presented. For benzanthrone, a systematic decrease of vapour pressure with increasing orifice size was observed. So the equilibrium vapour pressure at each temperature was calculated as the intercept of the plot of p_i against $(p_i w_o A_o)$ for each effusion temperature, where p_i represents the pressures calculated through the Clausius–Clapeyron equations presented in this table for each group of effusion cells. The so calculated equilibrium pressures are assumed to represent the vapour pressures that would be obtained using hypothetical effusion orifices of zero area.

The plots of $\ln p = f(1/T)$ for each compound studied are presented in figure 5.

The standard molar sublimation enthalpies at the temperature 298.15 K were derived from the sublimation enthalpies calculated at the mean temperature $\langle T \rangle$ of the experiments, by the equation:

$$\Delta_{\mathrm{cr}}^{\mathrm{g}} H_{\mathrm{m}}^{\circ}(T = 298.15\ \mathrm{K}) = \Delta_{\mathrm{cr}}^{\mathrm{g}} H_{\mathrm{m}}^{\circ}(\langle T \rangle) + \Delta_{\mathrm{cr}}^{\mathrm{g}} C_{\mathrm{p,m}}^{\circ} \times (298.15\ \mathrm{K} - \langle T \rangle), \tag{4}$$

where $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{\mathrm{p,m}}^{\circ}$ represents the mean value of the difference between the heat capacities of, respectively, the gas and crystalline phases over the temperature interval 298.15 K and $\langle T \rangle$.

For benzoic acid, the mean value of $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{\mathrm{p,m}}^{\circ} = -44.4\ \mathrm{J} \cdot mol^{-1} \cdot \mathrm{K}^{-1}$ was calculated from the equation $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{\mathrm{p,m}}^{\circ} = (-0.121T - 7.2)\ \mathrm{J} \cdot \mathrm{mol}^{-1} \cdot \mathrm{K}^{-1}$, derived from the values of the heat capacity of the crystalline phase

TABLE 1
Areas and transmission probability factors of the effusion orifices

	Orifice number	A_0/mm^2	w_0
Small orifices	A1	0.502	0.988
	A2	0.499	0.988
	A3	0.497	0.988
Medium orifices	B4	0.774	0.991
	B5	0.783	0.991
	B6	0.773	0.991
Large orifices	C7	1.116	0.992
	C8	1.125	0.992
	C9	1.150	0.992

TABLE 2
Effusion results for the studied compounds

T/K	t/s	Orifices	m/mg			p/Pa		
			m_A	m_B	m_C	p_A	p_B	p_C
				Benzoic acid				
299.33	23525	B4–C7		6.75	9.55		0.134	0.131
301.04	24347	A1–B4–C7	5.44	8.32	11.64	0.162	0.160	0.155
303.16	24347	A2–B5–C8	6.79	10.56	15.88	0.203	0.201	0.210
305.24	23525	A2–B5–C8	8.53	13.19	19.71	0.265	0.261	0.271
307.13	14045	A1–B4–C7	6.39	9.68	13.87	0.332	0.326	0.323
309.25	14045	A2–B5–C8	7.89	12.21	18.12	0.414	0.408	0.420
311.30	14045	A3–B6–C9	9.92	15.08	22.45	0.525	0.512	0.524
313.20	23525	A3–B6–C9	20.30	30.60	46.29	0.643	0.622	0.647
315.27	11462	A2–B5–C8	12.56	19.35	28.92	0.815	0.799	0.830
317.32	11462	A3–B6–C9	16.19	24.39	34.68	1.05	1.02	1.01
				Phenanthrene				
313.46	23888	A2–B5–C8	4.82	7.55	10.95	0.124	0.123	0.124
315.48	23888	A3–B6–C9	6.03	9.09	13.52	0.156	0.151	0.155
317.41	23603	A1–B4–C7	7.35	11.42	15.83	0.191	0.193	0.185
319.47	23603	A2–B5–C8	9.10	14.07	20.46	0.239	0.235	0.238
321.48	23603	A3–B6–C9	11.18	17.21	25.11	0.296	0.292	0.293
323.16	16455	A1–B4–C7	9.36	14.44	20.29	0.352	0.352	0.343
325.46	16455	A2–B5–C8	11.91	18.01	26.53	0.453	0.436	0.446
327.47	16455	A3–B6–C9	14.15	22.10	32.51	0.543	0.544	0.550
329.47	7515	A3–B6–C9	7.95	12.55	18.17	0.670	0.678	0.675
331.42	7515	A2–B5–C8	9.93	15.43	21.91	0.834	0.825	0.814
333.16	7515	A1–B4	12.10	17.68		1.01	1.00	
				Anthracene				
340.41	21996	A1–B4–C7	4.96	7.70	10.90	0.143	0.144	0.141
342.25	21996	A2–B5–C8	5.91	9.33	12.98	0.172	0.173	0.167
344.06	21996	A3–B6	6.99	11.18		0.206	0.211	
346.40	14775	A2–B5–C8	6.04	9.34	13.23	0.262	0.263	0.258
348.15	17292	A3–B6–C9	8.13	12.61	19.02	0.306	0.304	0.308
350.15	17292	A2–B5–C8	9.87	15.52	22.24	0.371	0.375	0.376
352.39	17292	A1–B4–C7	12.26	19.14	27.28	0.459	0.464	0.458
354.06	12598	A3–B6–C9	10.55	16.41	24.10	0.550	0.548	0.554
356.20	12598	A2–B5–C8	13.06	20.36	28.64	0.679	0.673	0.658
358.22	11072	A1–B4–C7	13.91	21.36	30.75	0.821	0.815	0.810
360.38	12598	A1–C7	19.38		42.08	1.01		0.980
				Benzanthrone				
390.31	24264	A1–B4	6.16	9.32		0.152	0.149	
392.23	24264	B5–C8		11.31	16.02		0.179	0.177
396.35	24264	A3–B6–C9	10.46	16.26	24.03	0.263	0.262	0.260
394.57	19981	A1–C7	18.04		16.37	0.234		0.221
398.22	19981	A3–B6–C9	10.61	16.43	24.06	0.325	0.323	0.325
400.41	15838	B4–C7		15.88	22.18		0.394	0.381
402.19	15838	A2–B5–C8	12.20	18.42	26.51	0.471	0.453	0.453
404.34	15838	A3–B6–C9	14.62	22.58	32.86	0.570	0.564	0.565
406.35	10789	A3–B6–C9	11.96	18.47	26.90	0.686	0.679	0.680
408.35	10789	A2–B5–C8	14.17	21.90	31.66	0.810	0.796	0.800
410.23	10789	A1–B4–C7	16.87	25.88	36.38	0.962	0.954	0.926
				1,3,5-Triphenylbenzene				
407.36	29774	A3–B6–C9	7.74	12.23	17.53	0.140	0.141	0.139
409.30	29774	A1–B4–C7	9.67	14.35	20.52	0.173	0.166	0.164
411.30	24245	A1–B4–C7	9.78	14.70	20.87	0.215	0.210	0.206
413.23	24245	A2–B5–C8	11.55	17.80	25.64	0.256	0.251	0.251
415.35	24245	A3–B6–C9	14.17	22.04	32.01	0.317	0.316	0.316
416.86	10629	A1–C7	7.15		15.34	0.361		0.348
417.22	29774	A2–C8	20.37		46.26	0.370		0.371
419.24	10629	B5–C8		14.01	20.87		0.454	0.470
421.36	10629	A3–B6–C9	10.54	17.10	25.06	0.542	0.563	0.568
423.29	13248	A1–B4–C7	16.47	25.24	35.56	0.672	0.668	0.649
425.26	13248	A2–B5	19.34	29.95		0.796	0.784	

TABLE 2 (*continued*)

T/K	t/s	Orifices	m/mg			p/Pa		
			m_{A}	m_{B}	m_{C}	p_{A}	p_{B}	p_{C}
427.36	13248	A3–B6–C9	23.28	36.99	54.71	0.966	0.984	1.00
429.29	16119	A1–B4–C7	34.57	54.97	79.43	1.17	1.20	1.20

Results related to the small (A1, A2, A3), medium (B4, B5, B6) and large (C7, C8, C9) effusion orifices are denoted, respectively, by the subscripts A, B and C.

TABLE 3
Experimental results for the studied compounds where a and b are from Clausius–Clapeyron equation $\ln(p/\mathrm{Pa}) = a - b \cdot (K/\mathrm{T})$, and $b = \Delta_{\mathrm{cr}}^{\mathrm{g}} H_{\mathrm{m}}^{\circ}(\langle T \rangle)/R$; $R = 8.3145\ \mathrm{J \cdot K^{-1} \cdot mol^{-1}}$

Effusion orifices	a	b	$\langle T \rangle$/K	$p(\langle T \rangle)$/Pa	$\Delta_{\mathrm{cr}}^{\mathrm{g}} H_{\mathrm{m}}^{\circ}(\langle T \rangle)$	$\Delta_{\mathrm{cr}}^{\mathrm{g}} S_{\mathrm{m}}\{\langle T \rangle, p(\langle T \rangle)\}$
					$\mathrm{kJ \cdot mol^{-1}}$	$\mathrm{J \cdot K^{-1} \cdot mol^{-1}}$
			Benzoic acid			
A	34.41 ± 0.27	10910 ± 84		0.331	90.7 ± 0.7	
B	33.73 ± 0.32	10706 ± 97		0.323	89.0 ± 0.8	
C	34.24 ± 0.33	10857 ± 102		0.329	90.3 ± 0.6	
Global	34.11 ± 0.19	10821 ± 58	307.12	0.325	90.0 ± 0.5	293 ± 2
			Phenanthrene			
A	33.04 ± 0.21	11013 ± 70		0.359	91.6 ± 0.6	
B	33.13 ± 0.23	11046 ± 75		0.355	91.8 ± 0.6	
C	32.83 ± 0.24	10948 ± 76		0.356	91.0 ± 0.6	
Global	33.03 ± 0.14	11011 ± 44	323.31	0.358	91.6 ± 0.4	283 ± 1
			Anthracene			
A	33.21 ± 0.19	11971 ± 68		0.385	99.5 ± 0.6	
B	32.68 ± 0.17	11785 ± 61		0.386	98.0 ± 0.5	
C	33.02 ± 0.23	11908 ± 80		0.381	99.0 ± 0.7	
Global	32.97 ± 0.12	11888 ± 42	350.40	0.384	98.8 ± 0.4	282 ± 1
			Benzanthrone			
A	36.13 ± 0.31	14835 ± 126		0.394	123.3 ± 1.0	
B	36.34 ± 0.24	14927 ± 95		0.386	124.1 ± 0.8	
C	36.42 ± 0.40	14962 ± 159		0.330	124.4 ± 1.3	
Mean	36.30 ± 0.19	14908 ± 75	400.27	0.387	123.9 ± 0.6	309 ± 2
Zero area	35.94 ± 0.19	14750 ± 75	400.27	0.402	122.6 ± 0.6	306 ± 2
			1,3,5-Triphenylbenzene			
A	39.10 ± 0.25	16722 ± 106		0.417	139.0 ± 0.9	
B	39.79 ± 0.29	17010 ± 121		0.418	141.4 ± 1.0	
C	40.32 ± 0.42	17236 ± 174		0.414	143.3 ± 1.5	
Global	39.72 ± 0.20	16983 ± 82	418.32	0.416	141.2 ± 0.7	338 ± 2

presented by Furukawa *et al.* [10] and from the values presented by Stull *et al.* [11] for the gaseous phase. For anthracene, the mean value $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{p,\mathrm{m}}^{\circ} = -27\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$ was estimated from the value $C_{p,\mathrm{m}}^{\circ}$ (cr, $T = 298.15$ K) $= 211.7\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, presented by Radomska and Radomski [12] and from the value $C_{p,\mathrm{m}}^{\circ}$ (g, $T = 298.15$ K) $= 185.0\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, presented by Kudchaker *et al.* [13]. For phenanthrene, the mean value $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{p,\mathrm{m}}^{\circ} = -34\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$ was estimated from the value $C_{p,\mathrm{m}}^{\circ}$ (cr, $T = 298.15$ K) $= 220.3\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, presented by Steele *et al.* [14] and from the value $C_{p,\mathrm{m}}^{\circ}$ (g, $T = 298.15$ K) $= 186.8\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, presented by Kudchaker *et al.* [13]. The mean value $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{p,\mathrm{m}}^{\circ} = -55\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$ was estimated for 1,3,5-triphenylbenzene using the value $C_{p,\mathrm{m}}^{\circ}$ (cr, $T = 298.15$ K) $= 361.0\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, presented by Lebedev *et al.* [15], in the equation $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{p,\mathrm{m}}^{\circ} = -\{0.75 + 0.15 C_{p,\mathrm{m}}^{\circ}(\mathrm{cr})\}$ proposed by Chickos *et al.* [16]. For benzanthrone, the mean value $\Delta_{\mathrm{cr}}^{\mathrm{g}} C_{p,\mathrm{m}}^{\circ} = -29\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$ was estimated from the value $C_{p,\mathrm{m}}^{\circ}$ (cr, $T = 298.15$ K) $= 260\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, calculated using group contribution values derived by Domalski and Hearing [17] and from the value $C_{p,\mathrm{m}}^{\circ}$ (g, $T = 298.15$ K) $= 231\ \mathrm{J \cdot mol^{-1} \cdot K^{-1}}$, derived by using group contribution values calculated by Benson [18].

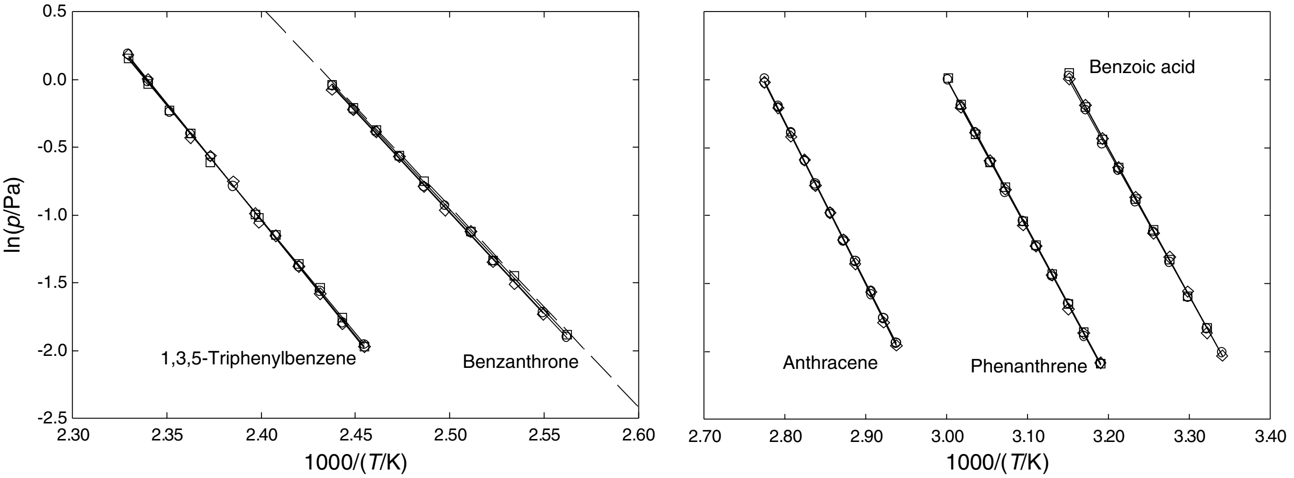

FIGURE 5. Plots of $\ln p$ against $1/T$ for the test compounds: □, orifices A; ○, orifices B; ◇, orifices C. The dashed line represents the linear regression on the equilibrium vapour pressures (zero area) for benzanthrone.

TABLE 4
The standard ($p^\circ = 0.1$ MPa) molar enthalpies, $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ$, entropies, $\Delta_{\rm cr}^{\rm g}S_{\rm m}^\circ$, and Gibbs energies, $\Delta_{\rm cr}^{\rm g}G_{\rm m}^\circ$, of sublimation at $T = 298.15$ K for the studied test substances

Compound	$\Delta_{\rm cr}^{\rm g}C_{p,{\rm m}}^\circ$ / $\rm J\cdot mol^{-1}\cdot K^{-1}$	$\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ$ / $\rm kJ\cdot mol^{-1}$	$\Delta_{\rm cr}^{\rm g}S_{\rm m}^\circ$ / $\rm J\cdot mol^{-1}\cdot K^{-1}$	$\Delta_{\rm cr}^{\rm g}G_{\rm m}^\circ$ / $\rm kJ\cdot mol^{-1}$
Benzoic acid	−44.4	90.4 ± 0.5	189 ± 2	34.0 ± 0.8
Phenanthrene	−34	92.5 ± 0.4	182 ± 1	38.2 ± 0.3
Anthracene	−27	100.2 ± 0.4	183 ± 1	45.6 ± 0.3
Benzanthrone	−29	125.6 ± 0.6	211 ± 2	62.7 ± 0.8
1,3,5-Triphenylbenzene	−55	147.8 ± 0.7	254 ± 2	72.1 ± 0.9

The above estimated values of $\Delta_{\rm cr}^{\rm g}C_{p,{\rm m}}^\circ$ are presented in table 4. This table also includes the calculated values, at $T = 298.15$ K, of the standard molar enthalpies of sublimation, the standard molar entropies of sublimation calculated by equation (5), where $p^0 = 10^5$ Pa, and the standard molar Gibbs energies of sublimation

$$\Delta_{\rm cr}^{\rm g}S_{\rm m}^\circ(T = 298.15\,{\rm K}) = \Delta_{\rm cr}^{\rm g}S_{\rm m}\{\langle T\rangle, p(\langle T\rangle)\} + \Delta_{\rm cr}^{\rm g}C_{p,{\rm m}}^\circ \ln(298.15\,{\rm K}/\langle T\rangle) - R\ln\{p^\circ/p(\langle T\rangle)\}. \quad (5)$$

4. Discussion

Table 5 presents 22 literature results for the enthalpy of sublimation of benzoic acid. Some values of the vapour pressures, calculated at the temperature limits of the experimental temperature range used in this work, are also presented. There is an excellent agreement between the mean of the literature results and the results obtained in the present work for both the standard enthalpy of sublimation at $T = 298.15$ K and the vapour pressures. In 1974, Cox [37] recommended for benzoic acid the value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ \times (298.15\,{\rm K}) = (89.7 \pm 0.5)\,{\rm kJ\cdot mol^{-1}}$ from the mean of selected published results. Sabbah *et al.* [9], in 1999, retained the value recommended by Cox. This value is similar, inside experimental uncertainties, to the presently obtained value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (90.4 \pm 0.5)\,{\rm kJ\cdot mol^{-1}}$.

Table 6 presents literature results for phenanthrene. The presently derived value for phenanthrene, $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ \times (298.15\,{\rm K}) = (92.5 \pm 0.4)\,{\rm kJ\cdot mol^{-1}}$, agrees within experimental uncertainties with both the mean of the literature results and the value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (91.3 \pm 1.1)\,{\rm kJ\cdot mol^{-1}}$ recommended by Peddley *et al.* [45] and retained by Sabbah *et al.* [9]. For anthracene, Kudchadker *et al.* [13] recommended the value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (100.9 \pm 2.8)\,{\rm kJ\cdot mol^{-1}}$ while Peddley *et al.* [45] recommended the value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (101.7 \pm 1.3)\,{\rm kJ\cdot mol^{-1}}$. The value derived in this work $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (100.4 \pm 0.4)\,{\rm kJ\cdot mol^{-1}}$ agrees with the mean of the literature results presented in table 7 and with the above recommended values. The value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (103.4 \pm 2.7)\,{\rm kJ\cdot mol^{-1}}$ recommended by Sabbah *et al.* [9] seems too high.

Table 8 presents the few available results for benzanthrone. The presently derived value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (125.6 \pm 0.6)\,{\rm kJ\cdot mol^{-1}}$ agrees with the mean of those results. The literature results for 1,3,5-triphenylbenzene are presented in table 9. The value derived in this work $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,K) = (147.8 \pm 0.7)\,{\rm kJ\cdot mol^{-1}}$ agrees within experimental uncertainties with the mean of the literature results and with the value $\Delta_{\rm cr}^{\rm g}H_{\rm m}^\circ(298.15\,{\rm K}) = (149.2 \pm$

TABLE 5
Literature values for benzoic acid

Temp. range/K	$\langle T\rangle$/K	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$ / $\mathrm{kJ\cdot mol^{-1}}$	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}$ (298.15 K)[a] / $\mathrm{kJ\cdot mol^{-1}}$	p(299.3 K)/Pa	p(317.3 K)/Pa	Year/method	Ref.
	298		88.3 ± 0.5			2001-Cal	[19]
307 to 354	331	90.5 ± 0.5	92.0 ± 0.5	0.111	0.874	1999-GS	[20]
304 to 317	310	90.5 ± 0.8	91.0 ± 0.8	0.128	1.01	1995-KE	[21]
304 to 317	310	89.4 ± 0.8	89.9 ± 0.8	0.136	1.04	1995-TE	[21]
279 to 295	287	89.9 ± 0.3	89.4 ± 0.3	0.135	1.05	1995-SR	[21]
307 to 314	310	88.7 ± 0.5	89.2 ± 0.5	0.129	0.976	1990-KE	[4]
	335	87.4 ± 0.3	89.1 ± 0.3			1988-DSC	[22]
275 to 322	298	95.1 ± 1.8	95.1 ± 1.8	0.116	1.01	1985-KE	[23]
293 to 319	306	90.8 ± 0.6	91.2 ± 0.6			1985-QR	[24]
320 to 370			88.9 ± 0.3			1982-Cal	[25]
316 to 391	353	89.44 ± 0.05	92.0 ± 0.2	0.128	0.991	1982-DM	[26]
293 to 313	303	90.3 ± 0.1	90.5 ± 0.1	0.126	0.990	1982-KE	[27]
296 to 317	308	90.0 ± 1.0	90.4 ± 1.0			1980-KE, TE	[28]
294 to 331	312	92.5 ± 0.4	93.1 ± 0.4	0.120	0.987	1975-TE	[29]
273 to 318	296	92.9 ± 0.2	92.8 ± 0.2			1974-MSKE	[30]
294 to 312	303	88.1 ± 0.2	88.3 ± 0.2			1973-TCM	[31]
338 to 384	361	86.1 ± 0.3	89.1 ± 0.3			1973-KE	[32]
	361	86.0 ± 0.4	89.0 ± 0.4			1973-Cal	[32]
281 to 323	302	88.3 ± 2.9	88.5 ± 2.9	0.101	0.752	1973-LE	[33]
290 to 316	303	86.7 ± 1.6	86.9 ± 1.6	0.192	1.37	1972-KE	[34]
	298	89.5 ± 0.2	89.5 ± 0.2			1972-Cal	[35]
291 to 307	299	90.9	90.9	0.108	0.861	1965-KE	[36]
			90.2 ± 1.9	0.13 ± 0.02	0.99 ± 0.15	Mean	
299.3 to 317.3	307.1	90.0 ± 0.5	90.4 ± 0.5	0.129	1.01	This work	

Cal, calorimetric; DM, diaphragm manometer; DSC, differential scanning calorimetry; GS, gas saturation; KE, Knudsen effusion; LE, Langmuir evaporation; MSKE, mass spectroscopy Knudsen effusion; QR, quartz resonator; SR, spinning rotor; TCM, thermal conductivity manometer; TE, torsion effusion.

[a] For temperatures $\langle T\rangle$ different from 298.15 K, the values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}$ (298.15 K) were calculated from the experimental values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$, presented in this table, through equation (4) using the value of $\Delta_{\mathrm{cr}}^{\mathrm{g}}C_{p,\mathrm{m}}^{\circ}$ presented in table 4.

TABLE 6
Literature values for phenanthrene

Temp. range/K	$\langle T\rangle$/K	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$ / $\mathrm{kJ\cdot mol^{-1}}$	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}$(298.15 K)[a] / $\mathrm{kJ\cdot mol^{-1}}$	p(313.5 K)/Pa	p(333.2 K)/Pa	Year/method	Ref.
310.6 to 333.2	321.9	91.8 ± 0.2	92.6 ± 0.2	0.120	0.962	2002-KE	[38]
303 to 333	318	95.0 ± 4.4	95.7 ± 4.4	0.129	1.11	1998- KE	[39]
			90.5			1998-CGC–DSC	[40]
313 to 453	383	88.9	91.8			1995-GS	[41]
	350	87.2 ± 1.1	89.0 ± 1.1			1988-DSC	[22]
283 to 323	303	95.0 ± 0.6	95.2 ± 0.6	0.105		1983-GS	[42]
315 to 335	325	90.5 ± 1.0	91.4 ± 1.0	0.117	0.908	1980-TE, KE	[43]
325 to 364	344	87.2	88.8		1.08	1979-GS	[44]
			90.9 ± 0.4			1972-Cal	[35]
			91.9 ± 2.6	0.12 ± 0.1	1.02 ± 0.10	Mean	
313.5 to 333.2	323	91.6 ± 0.4	92.5 ± 0.4	0.123	0.984	This work	

Cal, calorimetry; CGC–DSC, combined correlation gas chromatography–differential scanning calorimetry; DSC, differential scanning calorimetry; GS, gas saturation; KE, Knudsen effusion; TE, torsion effusion; SR, spinning rotor.

[a] For temperatures $\langle T\rangle$ different from 298.15 K, the values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}$(298.15 K) were calculated from the experimental values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$, presented in this table, through equation (4) using the values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}C_{p,\mathrm{m}}^{\circ}$ presented in table 4.

1.6) $\mathrm{kJ\cdot mol^{-1}}$ recommended by Sabbah *et al.* [9], although these two values are somewhat higher than the presently derived one.

Considering the results obtained and the above comments, we conclude that the new effusion apparatus is suitable for the accurate determination of vapour pressures of

TABLE 7
Literature values for anthracene

Temp. range/K	$\langle T\rangle$/K	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$ / $\mathrm{kJ\cdot mol^{-1}}$	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$[a] / $\mathrm{kJ\cdot mol^{-1}}$	$p(340.1\ \mathrm{K})$/Pa	$p(360.4\ \mathrm{K})$	Year/method	Ref.
423 to 488	455.5	94.5	98.7			1999-MEM	[46]
			99.4			1998-CGC–DSC	[40]
318 to 363	340.5	100.0 ± 2.8	101.1 ± 2.8	0.126	0.921	1998-KE	[39]
313 to 453	383	99.7	102.0			1995-GS	[41]
318 to 373	346	98.7	100.0			1986-GS	[47]
313 to 363	338	102.6	103.7	0.132	1.02	1986-GS	[48]
353 to 399	376	94.6	96.7	0.160	1.05	1983-GS	[42]
337 to 361	351.3	100.6 ± 1.0	102.0 ± 1.0	0.128	0.953	1980-TE	[43]
337 to 361	351.3	99.9 ± 1.0	101.3 ± 1.0	0.130	0.954	1980-KE	[43]
358 to 393	376	94.8	96.9	0.161	1.06	1979-GS	[44]
328 to 372	350	97.3 ± 1.7	98.7 ± 1.7	0.128	0.900	1976-KE	[49]
353 to 432	392	101.0 ± 0.5	103.5 ± 0.5			1973-KE	[32]
	392	99.7 ± 0.8	102.2 ± 0.8			1973-Cal	[32]
			100.5 ± 2.3	0.13 ± 0.01	0.97 ± 0.06	Mean	
340.1 to 360.4	350.4	99.0 ± 0.4	100.4 ± 0.4	0.137	0.988	This work	

Cal, calorimetry; CGC–DSC, combined correlation gas chromatography–differential scanning calorimetry; GS, gas saturation; KE, Knudsen effusion; MEM, modified entrainment method; TE, torsion effusion.

[a] For temperatures $\langle T\rangle$ different from 298.15 K, the values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$ were calculated from the experimental values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$, presented in this table, through equation (4) using the value of $\Delta_{\mathrm{cr}}^{\mathrm{g}}C_{p,\mathrm{m}}^{\circ}$ presented in table 4.

TABLE 8
Literature values for benzanthrone

Temp. range/K	$\langle T\rangle$/K	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$ / $\mathrm{kJ\cdot mol^{-1}}$	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$[a] / $\mathrm{kJ\cdot mol^{-1}}$	$p(390.3\ \mathrm{K})$/Pa	$p(410.2\ \mathrm{K})$/Pa	Year/method	Ref.
391 to 410	401	123.0 ± 0.5	126.0 ± 0.5	0.144	0.909	2003-KE	[50]
373 to 393	383	125.5 ± 2.1	128.0 ± 2.1			1999-QR	[51]
389 to 409	399	121.6 ± 0.6	124.5 ± 0.6	0.161	0.994	1999-KE	[51]
353 to 388	370	119.7 ± 5.4	121.8 ± 5.4			1984-QR	[52]
			125.1 ± 2.6	0.152	0.952	Mean	
390.3 to 410.2	400.3	122.6 ± 0.6	125.6 ± 0.6	0.157	0.982	This work	

KE, Knudsen effusion; QR, quartz resonator.

[a] The values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$ were calculated from the experimental values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$, presented in this table, through equation (4) using the value of $\Delta_{\mathrm{cr}}^{\mathrm{g}}C_{p,\mathrm{m}}^{\circ}$ presented in table 4.

TABLE 9
Literature values for 1,3,5-triphenylbenzene

Temp. range/K	$\langle T\rangle$/K	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$ / $\mathrm{kJ\cdot mol^{-1}}$	$\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$[a] / $\mathrm{kJ\cdot mol^{-1}}$	$p(407.4\ \mathrm{K})$/Pa	$p(429.3\ \mathrm{K})$/Pa	Year/method	Ref.
			150.9			1998-CGC–DSC	[40]
364 to 388	376	145.6 ± 0.9	149.9 ± 0.9			1997-T	[53]
410 to 444	427	142.0 ± 1.5	149.1 ± 1.5	0.101	0.855	1974-KE	[54]
410 to 444	427	143.1 ± 0.6	150.2 ± 0.6			1974-Cal	[54]
370 to 448	409	142.6	148.7	0.083	0.709	1967-KE	[55]
363 to 408	386	143.5	148.3	0.130	1.13	1958-KE	[56]
			149.5 ± 1.0	0.105	0.898	Mean	
407.4 to 429.3	418.3	141.2 ± 0.7	147.8 ± 0.7	0.140	1.17	This work	

Cal, calorimetry; CGC–DSC, combined correlation gas chromatography–differential scanning calorimetry; KE, Knudsen effusion; T, transpiration.

[a] For temperatures $\langle T\rangle$ different from 298.15 K, the values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(298.15\ \mathrm{K})$ were calculated from the experimental values of $\Delta_{\mathrm{cr}}^{\mathrm{g}}H_{\mathrm{m}}^{\circ}(\langle T\rangle)$, presented in this table, through equation (4) using the value of $\Delta_{\mathrm{cr}}^{\mathrm{g}}C_{p,\mathrm{m}}^{\circ}$ presented in table 4.

organic crystalline compounds and for the subsequent determination of their enthalpies of sublimation.

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