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Vapor Pressures and Thermodynamics of Oxygen-Containing Polycyclic Aromatic Hydrocarbons Measured Using Knudsen Effusion

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Abstract

Polycyclic aromatic hydrocarbons (PAHs) and their oxygenated derivatives (OPAHs) are ubiquitous environmental pollutants resulting from the incomplete combustion of coal and fossil fuels. Their vapor pressures are key thermodynamic data essential for modeling fate and transport within the environment. The present study involved nine PAHs containing oxygen heteroatoms, including aldehyde, carboxyl and nitro groups, specifically: 2-nitrofluorene; 9-fluorene-carboxylic acid; 2-fluorene-carboxaldehyde; 2-anthracene-carboxylic acid; 9-anthracene-carboxylic acid; 9-anthraldehyde; 1-nitropyrene; 1-pyrene-carboxaldehyde and 1-bromo-2-naphthoic acid. The vapor pressures of these compounds, with molecular weights ranging from 194 to 251 grams per mole, were measured using the isothermal Knudsen effusion technique in the temperature range of 329 to 421. The corresponding enthalpies of sublimation, calculated via the Clausius-Clapeyron equation, are compared to parent, non-oxygenated PAH compound data to determine the effect of the addition of these oxygen-containing heteroatoms. As expected, the addition of –CHO, –COOH, and –NO₂ groups onto these PAHs increases the enthalpy of sublimation and decreases the vapor pressure as compared to the parent PAH; the position of substitution also plays a significant role in determining the vapor pressure of these OPAHs.

Keywords

vapor pressure; oxygenated polycyclic aromatic hydrocarbons; OPAH; Knudsen effusion; enthalpy of sublimation

Introduction

Oxygenated polycyclic aromatic hydrocarbons (OPAHs), like their parent counterparts, polycyclic aromatic hydrocarbons (PAHs), result from incomplete combustion associated with coal and other fossil fuels, wood, and municipal waste incineration [1]. PAHs and OPAHs were shown by Rogge et al. [2] to comprise between 3.1 to 8.6% of the total identifiable fine organic particulate matter emitted from the burning of no. 2 distillate fuel oil in an industrial scale boiler. In addition, they form through the photooxidation of PAHs through several mechanistic pathways [3]. PAHs and OPAHs cause a wide range of biological effects resulting from their ability to produce reactive oxygen species (ROS), ultimately responsible for proinflammatory responses in respiratory cells. These compounds can induce premature aging, carcinogenesis, chronic inflammatory processes and acute respiratory symptoms [4].

Several studies identified oxygenated PAHs in various environmental phases. For example, Allen et al. measured seven PAH ketones, four PAH diones, one PAH dicarboxylic acid anhydride and seven potential other OPAHs in the atmosphere in Boston, Massachusetts [1]. Kallio et al. [5] collected particulate PAHs and OPAHs using high volume air samplers in Helsinki, Finland, which they attributed to local incineration. In another study, PAHs and OPAHs were detected beside a roadway near Munich, Germany; PAHs and OPAHs are known to exist on diesel exhaust particles [4]. Liu et al. (measured PAHs and OPAHs in the atmosphere around a highly trafficked city center of Augsburg, Germany, concluding that the majority of 5 to 7 ring PAHs and 4 to 5 ring OPAHs existed as particulate matter, not in the ozone [6]. While these carcinogenic compounds are present in the environment in detectable quantities, few thermodynamic data are available in the literature to assist in the modeling of their fate and transport.

The ability of a compound to partition appreciably into the atmosphere is governed largely by its vapor pressure [7]. In order to describe a PAH's ability to exist in the vapor phase, basic thermodynamic data - including vapor pressures - are necessary, but for many compounds may remain unknown. Current methods used to predict vapor pressures of these compounds cannot be applied confidently to describe PAHs containing heteroatoms; this study aims to furnish the data necessary to permit such predictions, as well as show key trends among substituted compounds.

The dearth of data on the vapor pressures of substituted PAHs, especially those with carboxyl, aldehyde and nitro groups, stems from the difficulty in performing such measurements. Common vapor pressure measurement devices often result in the degradation of high molecular weight compounds due to the high temperatures necessary to take such measurements. This difficulty is overcome through use of the Knudsen effusion technique, which enables the indirect measurements of vapor pressures of semi-volatile compounds, such as PAHs, at low to moderate temperatures.

Materials & Methods

The Knudsen effusion technique relies upon the measurement of the escape rate of molecules of the evaporating or subliming substance through a small orifice in an effusion cell without disrupting the equilibrium state of the vessel. The Knudsen effusion technique is well developed and widely applied [8, 9, 10, 11]. The Knudsen equation generally takes the form:

$$P^{\circ} = \frac{\omega}{tA_0W_0} \sqrt{\frac{2\pi RT}{M}} \quad [1]$$

where P° is the vapor pressure, ω is the mass loss, t the time, A_0 the orifice area, R the universal gas constant, T the absolute temperature, M the molecular weight of the effusing species and W_0 is the Clausing factor, accounting for the resistance of flow through the cell orifice. Tabulated values of the Clausing probability factor for cylindrical and rectangular orifices are available in the literature [12] or may be calculated as described previously [11]. The Clausing factors used in this research ranged from 0.96 to 0.98.

The primary instrumentation of our Knudsen effusion technique is the thermogravimetric apparatus (TGA), comprising a Cahn 2000 microbalance (ThermoCahn, Madison, WI, USA) with a sensitivity of 0.1 μ g and 100mg capacity in a high vacuum chamber with a suitable oven for heating. A sample cell is suspended on one arm of the microbalance in a wire holder such that it sits inside a blackened copper tube. An Omega CN 8201 Temperature Controller (Stamford, CT, USA) coupled with an aluminum block oven and

Omega resistance temperature detector comprises the temperature control system. A cold trap slightly downstream from the cell condenses the vaporized compounds, preventing them from contaminating the turbopump as well as maintaining a low backpressure in the thermogravimetric apparatus system. We heated the lines from the oven to the cold trap to prevent condensation of the effusing vapors on the sides of the vacuum enclosure or on the balance wire.

Temperature control and monitoring is of critical importance to the reliable measurement of vapor pressures; the vapor pressure of a given compound can vary by as much as an order of magnitude over the ambient temperature range [13]. The cell temperature is measured by an Omega type K thermocouple and read by an Omega DP41 temperature meter, accurate to $\pm 0.1\text{K}$, positioned directly above the cell opening, calibrated with a National Institute of Standards and Technology-traceable thermometer (Omega). The temperature and mass are recorded simultaneously, permitting average mass loss rates over extended periods of time.

We fabricate the effusion cells in our laboratory using a cylindrical mold designed by Oja [14]. Each cell is cleaned through heating in a propane flame to ensure any surface impurities are removed, and to darken its surfaces to improve heat transfer. The cell is sealed using a hand press to ensure the only leak in the cell is through the effusion hole. The effusion holes are made using a miniature drill press with an extremely small drill bit, resulting in holes with areas measuring approximately 0.004cm^3 to 0.006cm^3 , measured using an optical microscope.

To verify the experimental technique, we gathered data on fluorene, anthracene, and pyrene, spanning the molecular weights of 166 to 202 grams per mole, all with vapor pressures well established within the literature. These three compounds were used to calibrate the Knudsen effusion apparatus in the temperature range of 298–381 Kelvin and to verify estimates of the Clausius factor. We obtained good agreement with the literature values for these compounds [15].

The PAHs and OPAHs measured were all obtained at minimum purities of 95% from Tokyo Chemical International America (Portland, OR, USA). They were loaded into individual sample cells without further purification. Before data collection commences, we sublime a minimum of 5% (by mass) of each compound to ensure removal of any volatile impurities. This was observed by mass spectrometer to be sufficient for obtaining “pure” compound results [16]. We also halt data collection with more than 5% initial total weight percent remaining in case there are any nonvolatile impurities present. We run a minimum of two different sample cells for each compound to ensure reproducibility. In addition, the melting points were measured for each compound using the capillary melt technique and were well within literature reported values for the pure compounds.

Results & Discussion

Data are analyzed using the well-known Clausius Clapeyron equation, under the assumption of constant enthalpy of sublimation over the temperature ranges measured. The vapor pressure of the pure compound, P° , is related to the enthalpy, $\Delta_{\text{sub}}H$, and entropy, $\Delta_{\text{sub}}S$, of sublimation via:

$$\ln P^\circ = -\Delta_{\text{sub}}H/RT + \Delta_{\text{sub}}S/R \quad [2]$$

Table 1 summarizes the compounds used in this investigation and the sublimation enthalpies and entropies obtained for each compound for their respective measured temperature ranges. A 95% confidence interval was calculated for each set of reported values via linear

regression. The last column in Table 1 is the vapor pressure, extrapolated using the Clausius-Clapeyron equation (equation 2, above) to ambient temperature, 298K to provide a rough sense of the relative volatilities of the compounds studied. Table 2 presents the raw vapor pressure data obtained using the Knudsen effusion technique; these data form the basis of the results in Table 1.

Figures 1 through 4 demonstrate the effect adding an oxygen-containing heteroatom to polycyclic aromatic hydrocarbons; in each case, the vapor pressure of the parent compound decreases and enthalpy of sublimation increases upon addition.

Figure 1 demonstrates the effect of adding both a brominated and oxygenated heteroatom on the thermodynamics of naphthalene. The vapor pressure of 1-bromo-2-naphthoic acid is more than six orders of magnitude less than that of pure naphthalene, whereas the addition of one bromine only decreases the vapor pressure by one order of magnitude [17, 18]. The enthalpy of sublimation of naphthalene, increased by 7.1kJ/mole with the addition of one bromine, and increases 35.7kJ/mole from naphthalene to 1-bromo-naphthoic acid. Figure 1 also presents the vapor pressure data on 1- and 2-naphthylacetic acid, where the vapor pressure decreases by five and six orders of magnitude respectively, over the parent compound, naphthalene [19]. The enthalpy of sublimation of 1-naphthylacetic acid is 53% higher than that for pure naphthalene; it is 70% higher for 2- naphthylacetic acid, alluding to the relative importance of the carbon position of the substituted heteroatom.

Figures 2 and 3 demonstrate the significant impact of a nitro group addition to fluorene and pyrene, respectively. The addition of a nitro group at the 2-carbon position of fluorene increases the enthalpy of sublimation from 88.1($\pm$ 1.9) kJ/mole to 114.2($\pm$ 3.0) kJ/mole. For pyrene, a nitro group substituted on the 1-carbon increases the enthalpy from 97.8($\pm$ 3.3) kJ/mole to 125.0($\pm$ 3.8) kJ/mole, a comparably similar increase. This trend was also noted in data measured by Ribeiro da Silva et al. [20] on the vapor pressures of 1-nitronaphthalene and 9-nitroanthracene; adding a nitro group to the former resulted in an increase of 21.8kJ/mole over naphthalene, while addition to the latter yielded an increase of only 16.9kJ/mole over anthracene. Hence, their results showed slightly lower enthalpy contributions than did ours. Adding the nitro group on the 9-carbon of anthracene produced the smallest effect on enthalpy of sublimation a 17% increase. However, the other compounds resulted in increases of 30%, 28%, and 30% for 2-nitrofluorene, 1-nitropyrene, and 1-nitronaphthalene, respectively.

Previous studies from this laboratory indicated that for halogenated heteroatom substitution onto PAHs, the position of the halogen substituted on the parent molecule does not seem to play a large role in the vapor pressure behavior [16]. However, as we see here through the anthracenecarboxylic acid structural isomers (Figure 4), the position of the substituted group on the parent PAH is quite significant; the vapor pressure of 2-anthracenecarboxylic acid is almost a full order of magnitude less than that of 9-anthracenecarboxylic acid at ambient temperature. The differences in enthalpy are also significant; we report an enthalpy of sublimation for 2-anthracenecarboxylic acid of 134.8 $\pm$ 3.4 kJ/mole, whereas for 9-anthracenecarboxylic acid the $\Delta_{\text{sub}}H$ is 120.1 $\pm$ 3.8 kJ/mole. Additionally, as seen through the intercept of the Clausius-Clapeyron equation, the entropy of sublimation for 2-anthracenecarboxylic acid was calculated as 0.301 $\pm$ 0.008 kJ/mole-K, for 9-anthracenecarboxylic acid to be 0.278 $\pm$ 0.009 kJ/mol-K. Thus, we see a slightly larger impact on entropy of sublimation for the 2-anthracenecarboxylic acid. From these data, we note the significance of the substituent position of the carboxyl group on the parent PAH. Further investigations into this trend are warranted in order to establish whether molecular symmetry (i.e., the carboxyl group on 9-anthracenecarboxylic acid sits on a center carbon of the parent, whereas for 2-anthracenecarboxylic acid the carboxyl group sits on an end

carbon) is an important determinant in a compound's vapor pressure. Many questions remain as to the implications for potential hydrogen bonding and/or induced dipole moments within the PAH molecule and its oxygenated heteroatoms.

Also in Table 1, we see that the addition of a carboxyl group to fluorene at the 9-carbon position increases the enthalpy of sublimation by 21.9 kJ/mole, an increase of approximately 25%. We also see a vapor pressure depression of over four orders of magnitude, illustrated in Figure 2. We expect to see this larger increase in enthalpy of sublimation due to heteroatom substitution on a smaller compound, such as fluorene, than on anthracene.

Figure 4 presents the results of vapor pressure measurements on 9-anthraldehyde. The addition of the aldehyde group as seen in Figure 2, has a considerably larger impact, increasing the enthalpy of sublimation by 11.9 kJ/mole while decreasing the vapor pressure by almost two orders of magnitude at 298K. A similar impact is seen with 1-pyrenecarboxyaldehyde, where an aldehyde at the 1-carbon position increases the enthalpy of sublimation from 97.8 ± 3.3 kJ/mole to 110.4 ± 3.8 kJ/mole, an increase of almost 13%. Likewise, the vapor pressure is decreased by two orders of magnitude at 298K, demonstrated in Figure 3.

Conclusions

In summation, the addition of oxygen-containing heteroatoms to polycyclic aromatic compounds decreases the vapor pressure while increasing the enthalpy of sublimation. Our data show substantial increases in enthalpy with the addition of carboxyl and nitro groups to PAHs up to four rings in size, with a generally lower impact seen for the addition of an aldehyde group than for a carboxyl or nitro group. It is also evident from even this limited data that the position to which a heteroatom is substituted has a measurable impact on the vapor pressure of oxygenated polycyclic aromatics.

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Figure 1.

Vapor pressure of 1-bromo-2-naphthoic acid compared to parent and relevant polycyclic aromatic compounds, as measured by the Knudsen effusion technique plotted as the natural log of pressure ($\ln P^{\text{vap}}$ versus reciprocal of temperature (T); $\square$ naphthalene [17]; $\circ$ 2-bromonaphthalene [18]; $\diamond$ 1-bromo-2-naphthoic acid $\blacktriangle$ 1-naphthylacetic acid [19]; $\triangle$ 2-naphthylacetic acid [19].

Figure 2. Vapor pressures of oxygenated fluorene compared to parent PAH as measured by the Knudsen effusion technique; ν fluorene [15]; λ 2-fluorencarboxaldehyde; ν 9-fluorencarboxylic acid; σ 2-nitrofluorene

Figure 3. Vapor pressures of oxygenated pyrene compared to parent PAH as measured by the Knudsen effusion technique; ν pyrene[15]; λ 1-pyrenecarboxaldehyde; ν 1-nitropyrene

Figure 4.

Vapor pressures of oxygenated anthracene compared to parent PAH as measured by the Knudsen effusion technique; ♦ anthracene[15]; ● 9-anthraldehyde; ■ 2-anthracenecarboxylic acid; ▲ 9-anthracenecarboxylic acid

Table 1

Compounds investigated and results obtained from vapor pressure measurements on oxygenated polycyclic aromatic hydrocarbons.

Compound	CAS Reg. No.	Molecular Weight g/mol	Melting Point K	Min. Purity %	Temp Range K	$\ln(P^{vap}/Pa) = a - b \cdot 10^3/(K/T)$	$\Delta_{sub} H$ kJ/mol	P^{vap} 298K Pa
naphthalene [17] $C_{10}H_8$	91-20-3	128.2	354	>99	258-314	31.97 ± 0.27	8.82 ± 0.074	73.3 ± 0.61 10.7
2-bromonaphthalene [18] $C_{10}H_7Br$	580-13-2	207.1	331	>99	280-328	32.27 ± 0.27	9.67 ± 0.082	80.4 ± 0.68 0.836
1-bromo-2-naphthoic acid $C_{11}H_7BrO_2$	20717-79-7	251.1	464	>98	340-401	32.21 ± 0.79	13.11 ± 0.32	109.0 ± 2.7 7.63E-06
fluorene [15] $C_{13}H_{10}$	86-73-7	166.2	388	97	298-324	32.85 ± 0.71	10.60 ± 0.23	88.1 ± 1.9 6.58E-02
2-fluoreneboxaldehyde $C_{14}H_{10}O$	30084-90-3	194.2	357	>95	338-356	33.27 ± 1.1	12.03 ± 0.41	100.0 ± 3.4 8.26E-04
9-fluoreneboxylic acid $C_{14}H_{10}O$	1989-33-9	210.2	503	>97	349-418	31.70 ± 1.3	13.25 ± 0.55	110.2 ± 4.6 2.86E-06
2-nitrofluorene $C_{13}H_9NO_2$	607-57-8	211.2	428	>99	349-384	35.89 ± 0.94	13.74 ± 0.36	114.2 ± 3.0 3.65E-05
anthracene [15] $C_{14}H_{10}$	120-12-7	178.2	491	99	322-348	32.59 ± 1.1	11.85 ± 0.40	98.5 ± 3.3 7.65E-04
9-anthraldehyde $C_{15}H_{10}O$	642-31-9	206.2	380	>99	329-363	32.05 ± 1.2	12.10 ± 0.47	100.6 ± 3.9 1.93E-04
2-anthraceneboxylic acid $C_{15}H_{10}O_2$	613-08-1	222.2	509	>98	401-421	36.16 ± 0.91	16.22 ± 0.41	134.8 ± 3.4 1.16E-08
9-anthraceneboxylic acid $C_{15}H_{10}O_2$	723-62-6	222.2	492	>97	385-420	33.42 ± 1.1	14.45 ± 0.46	120.1 ± 3.8 2.85E-07
pyrene [15] $C_{16}H_{10}$	129-00-0	202.3	423	99	322-381	31.94 ± 1.1	11.76 ± 0.40	97.8 ± 3.3 5.40E-04
1-pyreneboxaldehyde $C_{17}H_{10}O$	3029-19-4	230.3	462	>98	360-393	32.44 ± 1.2	13.28 ± 0.46	110.4 ± 3.8 5.43E-06
1-nitropyrene $C_{16}H_9NO_2$	5522-43-0	247.3	425	>98	379-408	36.18 ± 1.1	15.06 ± 0.46	125.2 ± 3.8 5.82E-07

Table 2

Raw vapor pressure data P^{vap} (in Pa) obtained for pure OPAHs as a function of temperature (T, in Kelvin) using the Knudsen effusion technique

2-fluorencarboxaldehyde			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
329.9	0.0408	345.2	0.216
330.1	0.0419	350.2	0.354
334.2	0.0713	353.2	0.471
337.8	0.0941	353.5	0.480
340.2	0.115	354.5	0.493
341.3	0.135		
9-fluorencarboxylic acid			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
349.1	0.0021	392.4	0.142
369.8	0.0146	392.8	0.133
370.5	0.0184	399.2	0.227
375.7	0.0271	402.7	0.303
375.9	0.0298	405.6	0.297
381.3	0.0433	405.7	0.333
381.6	0.0474	409.4	0.662
388.0	0.0861	418.4	1.17
2-nitrofluorene			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
349.1	0.0325	365.7	0.188
353.0	0.0509	365.8	0.181
353.1	0.0524	366.2	0.212
356.0	0.0661	367.4	0.226
357.3	0.0782	369.3	0.260
357.6	0.0775	372.0	0.334
357.6	0.0768	373.7	0.413
359.8	0.0933	375.4	0.517
359.9	0.0930	379.5	0.787
361.7	0.118	383.5	1.05
363.4	0.138		
2-anthracenecarboxylic acid			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
400.5	0.0128	424.7	0.235
401.4	0.0140	427.2	0.290
411.7	0.0701	429.9	0.366
415.3	0.0544	432.7	0.488

2-anthracenecarboxylic acid

T/K	P ^{vap} /Pa	T/K	P ^{vap} /Pa
416.7	0.129	437.5	0.717
420.6	0.174	442.2	1.05
421.3	0.161	446.4	1.45

9-anthracenecarboxylic acid

T/K	P ^{vap} /Pa	T/K	P ^{vap} /Pa
385.4	0.0233	402.7	0.0928
388.4	0.0323	403.8	0.156
390.2	0.0444	405.4	0.205
391.6	0.0489	407.1	0.220
394.4	0.0504	411.3	0.338
395.8	0.0663	415.8	0.514
400.0	0.0778	419.5	0.627

1-nitropyrene

T/K	P ^{vap} /Pa	T/K	P ^{vap} /Pa
378.5	0.0287	394.5	0.145
378.8	0.0290	396.6	0.175
380.7	0.0319	397.3	0.175
382.2	0.0399	398.9	0.224
384.9	0.0501	401.0	0.271
385.0	0.0502	401.9	0.277
385.1	0.0506	403.9	0.318
389.3	0.0841	405.4	0.350
392.5	0.120	407.9	0.460

9-anthraldehyde

T/K	P ^{vap} /Pa	T/K	P ^{vap} /Pa
328.5	0.00863	346.7	0.0534
330.7	0.00962	347.9	0.0613
332.4	0.0134	348.0	0.0704
338.6	0.0236	350.7	0.0828
340.4	0.0319	352.0	0.108
340.6	0.0341	355.1	0.132
342.5	0.0349	359.1	0.186
343.9	0.0465	363.2	0.279
344.6	0.0425		

1-bromo-2-naphthoic acid

T/K	P ^{vap} /Pa	T/K	P ^{vap} /Pa
340.4	0.0018	372.5	0.0462
355.5	0.0092	376.3	0.0729
355.9	0.0095	380.5	0.0923

1-bromo-2-napthoic acid			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
359.6	0.0142	385.0	0.157
361.4	0.0187	388.7	0.240
368.1	0.0322	392.7	0.332
368.5	0.0316	397.3	0.459
371.5	0.0480	401.2	0.562

1-pyrenecarboxaldehyde			
T/K	P^{vap}/Pa	T/K	P^{vap}/Pa
356.3	0.00810	380.1	0.0779
360.1	0.0119	380.3	0.0825
364.2	0.0164	381.1	0.0952
368.3	0.0271	388.1	0.166
372.3	0.0366	388.2	0.168
373.3	0.0479	389.0	0.177
376.0	0.0634	392.3	0.227
376.1	0.0584	393.2	0.287