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***Vapour pressure measurements of hydroxyacetaldehyde and hydroxyacetone
in the temperature range (273 to 356) K.***

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Abstract

Vapour pressures of ethylene glycol, hydroxyacetaldehyde and hydroxyacetone were measured by using a standard closed system equipped with a vacuum line, built recently in our laboratory.

First, our vapour pressures measurements of ethylene glycol and their excellent agreement with the literature in the studied temperature range, i.e. (308 to 385) K, permitted us to validate our experimental set-up and procedure.

The data obtained in the temperature ranges (296 to 356) K and (273 to 304) K were very satisfactorily (Absolute Relative Deviations (ARD) < 2.5 %) fitted according to the Antoine's equation: $\log_{10} (P^{\circ}_{\text{hydroxyacetaldehyde}} / \text{Pa}) = 12.96 \pm 0.82 - (3657 \pm 238) / T/K$ and $\log_{10} (P^{\circ}_{\text{hydroxyacetone}} / \text{Pa}) = 10.13 \pm 0.06 - (2201 \pm 153) / T/K$. Then, the resulting vapour pressures of both hydroxyacetaldehyde and hydroxyacetone at 293 K were $P^{\circ} = (3.0 \pm 0.2)$ Pa and $P^{\circ} = (415 \pm 20)$ Pa for hydroxyacetaldehyde and hydroxyacetone, respectively. The quoted errors correspond to 2σ obtained from the least square fit analysis and the estimated systematic relative error of 5 %.

Finally, these experimental expressions of temperature dependences of P° permitted then to derive the enthalpy of vaporization for both compounds.

Keywords: vapour pressure, Antoine's equation, hydroxyacetone, hydroacetaldehyde, glycolaldehyde, ethylene glycol, enthalpy of vaporization.

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Introduction

Hydroxyacetone ($\text{HOCH}_2\text{C}(\text{O})\text{CH}_3$) and hydroxyacetaldehyde (HOCH_2CHO) are atmospheric pollutants of particular interest. Hydroxyacetone is produced in the atmosphere mainly from the OH-initiated oxidation of isoprene, the main non-methane hydrocarbon in the troposphere¹⁻³. Hydroxyacetaldehyde, usually named glycolaldehyde, is also formed *in situ* in the atmosphere from the oxidation of several volatile organic compounds (VOCs) including ethene, 2-methyl-3-buten-2-ol, and isoprene.⁴⁻⁷ Once produced in the atmosphere, these two hydroxyl carbonyls compounds have a potentially large atmospheric impact since they lead, in the upper troposphere, to HO_x radicals known to increase the oxidizing capacity of the atmosphere and therefore they can influence the ozone budget.⁸

So far, hydroxyacetaldehyde and hydroxyacetone have not been considered to be significant aerosol constituents since it is assumed that these compounds have relatively high vapour pressures. However, recent field observations indicate that they are important atmospheric aerosol constituents. For example, Matsunaga et al., 2005⁹ provide evidence that hydroxyacetone, hydroxyacetaldehyde (and methylglyoxal) contribute significantly to organic aerosols, (10 to 120) Tg annually. These findings have been confirmed, at least for hydroxyacetaldehyde, by Angove et al. 2006¹⁰ in smog chamber experiment. Note that, in this work, Angove et al. (2006)¹⁰ report an estimated value of 5.3 kPa for the equilibrium vapour pressure P° of hydroxyacetaldehyde at 298 K, while Butler et al. (2001)¹¹ report also an estimated value of 4 Pa at room temperature. These two estimated values differ by more than two orders of magnitude. The situation is even worst for hydroxyacetone for which, to our knowledge, only an estimated upper limit exists i.e. $P^\circ < 0.5$ kPa at room temperature.¹²

The precise knowledge of the equilibrium vapour pressure of a component is important to predict its partitioning between the gas and particle phases in the atmosphere¹³. In addition, the saturation vapour pressure P° can also be used to estimate Henry's Law constant that represents the partitioning between the gas and the aqueous phases at equilibrium. These partitionings are key physical properties to understand and predict the behaviour of a component in the environment.¹⁴⁻¹⁶

The main goal of this work is therefore to provide accurate experimental vapour pressures for both hydroxyacetone and hydroxyacetaldehyde. For this, our experimental set-up based on previous static devices¹⁷ was built and validated by vapour pressures measurements of ethylene glycol between (308 and 385) K.

Experimental section

Chemicals

Ethylene glycol (> 99.5 %) and solid hydroxyacetaldehyde dimer (> 99 %) were purchased from Merck and Aldrich, respectively. Hydroxyacetone (> 95 %) was provided by Alfa Aesar. Before use, hydroxyacetaldehyde dimer was melted at temperatures ranging between (80 and 90) °C for more than 2 hrs in order to obtain hydroxyacetaldehyde monomer.¹⁸ Hydroxyacetaldehyde monomer was then kept as limpid liquid even when temperature decreased typically down to 293 K.

To eliminate the effect of gaseous impurities, all the compounds were further purified by freeze-pump-thaw cycles using liquid nitrogen/ethanol baths at different temperatures and a vacuum pump (< 0.02 Pa).

Apparatus and Procedure

The experimental setup, shown schematically in Figure 1, is a static vapour-liquid equilibrium apparatus. It consists of a Pyrex flask containing the solid or liquid sample, connected to the vacuum line with a glass/metal connection (Caburn MDC®). It includes also a thermostatic bath, temperature and pressure measurement devices and a vacuum system.

The pressure was measured in the equilibrium cell by a capacitance manometer (Edwards®, 622 Barocel, ranges 0 to 1000 Pa and 0 to 10000 Pa) coupled to a digital indicator (BOC Edwards®) giving uncertainties of $(0.1 + 0.15 P/P^*)$ Pa and $(1 + 0.15 P/P^*)$ Pa, where $P^* = 100$ Pa, for 0 to 1000 Pa and 0 to 10000 Pa ranges, respectively.

Except for the vacuum system, the entire device was placed in an oven maintained at a temperature slightly higher than that of the oil bath in which the Pyrex flask was immersed, in order to avoid any vapour condensation on the tube walls. Both bath and oven temperatures were measured with thermocouples K coupled to a laboratory thermometer (Allcat Instruments®) having an accuracy of ± 0.25 % of the reading values in °C. In addition, the thermocouples were calibrated by the manufacturer few months before the measurements and were checked by dipping them into an ice bath maintained to 273.15 K.

The vacuum system (not shown) is composed of a combination of turbo-molecular/vacuum pump (Edwards® High Vacuum, type EXC120/ type RV8) and a liquid nitrogen trap. Two control valves allow to either isolating the sample from the vacuum system (control valve 1) or isolating the equilibrium cell from the vacuum system, leading to the establishment of an equilibrium between the liquid and gas phases (control valve 2).

Results and discussion

Ethylene glycol

The vapour pressure of ethylene glycol was measured in the temperature range 308 – 385 K in order to validate the experimental set-up.

After degassing of ethylene glycol *in situ* in the pyrex flask, the measurements of the saturation vapour pressure were performed by first establishing a highly stable vacuum in the equilibrium cell. The control valve (2) was then rapidly closed allowing the establishment of equilibrium between the liquid phase and the gas phase while the pressure values in the equilibrium cell were monitored on a computer. Figure 2 illustrates a typical data plot obtained for a given temperature (here at 326.1 K).

As shown in Figure 2 (see solid line), the pressure increased quickly at the beginning of the experiment and after a time scale of about 15 min, it leveled off meaning that the liquid phase reached equilibrium with the gas phase. Nevertheless when this equilibrium was reached, the pressure continued to slowly increase linearly with time due to the presence of leaks (see dash-do-dot line in Figure 2). This leak is specific to equipment used in our system, i.e. vacuum fittings with viton seals (NW references, @Edwards). The pressure reading at a given experimental time was then corrected by subtracting the leak contribution as follows:

$$P_{\text{corrected}} = P - v \times t \quad (1)$$

where P is the reading pressure, v is the leak rate and t is the time from the beginning of experiment.

The leak rate v was obtained from the slope of the linear part of the plot of P versus t (see dash-dot-dot line in Figure 2). The plot of the resulting corrected pressures (see solid line in Figure 2) reaches now a horizontal plateau that corresponds to the saturation vapour P° at a given temperature.

Similar experiments were repeated for temperatures ranging between 308 K and 385 K (see Table 1). The relative errors on the vapour pressure measurements correspond to $2\sigma + 5\%$, where σ is obtained from the least square linear fit analysis and 5 % is the estimated systematic relative error. The resulting relative errors varied between (5 and 7) %.

Since the plot of $\log_{10} P^\circ$ versus $1/T$ showed a linear behaviour (see Figure 3), the results were fitted according to a simplified Antoine's equation:

$$\log_{10}(P^\circ / Pa) = (12.02 \pm 0.72) - \frac{3257 \pm 204}{(T / K)}$$

where the quoted errors correspond to 2σ obtained from the least square linear fit analysis and the estimated systematic relative error of 5%.

The absolute relative deviation (ARD) was defined as follows:

$$ARD = \frac{1}{n} \sum_{i=0}^n \left[\frac{|P^0 - P_{calc}^0|}{P^0} \right]_i \quad (2)$$

where n is the total number of experimental data points and P_{calc}^0 is the calculated vapour pressure.

According to Eq. 2, the ARD value is less than 2%. It appears therefore that our data can be conveniently fitted according to the simplified Antoine's equation.

The vapour pressure of ethylene glycol derived from the experimental simplified Antoine's equation is $P^\circ = (8.02 \pm 0.41)$ Pa at 293 K.

The enthalpy of vaporization $\Delta_{vap}H$ is constant on the investigated temperature range. This later, derived from the slope of $\log_{10} P^\circ$ versus $1/T$, is then found to be (62.4 ± 4.0) kJ mol⁻¹.

Comparison with the literature

The equation derived from the literature¹⁹ in the same temperature range is:

$$\log_{10}(P_{lit}^\circ / Pa) = (12.22 \pm 0.69) - \frac{3325 \pm 193}{(T / K)}$$

The calculated literature values¹⁹ are summarized in Table 1 together with our experimental values (P°). Our extrapolated experimental value at 293 K ($P^\circ = 8.02 \pm 0.41$ Pa) is in good agreement with that of Stull et al. ($P_{lit}^\circ = 7.45 \pm 0.38$ Pa) at the same temperature. In the temperature range (308 to 385) K, the differences between the two set of data are at most 5 %, indicating that our experimental set up is suitable to measure equilibrium vapour pressure with good accuracy.

Similarly, our vaporization enthalpy of (62.4 ± 4.0) kJ mol⁻¹ is consistent with the literature values of 65.6 ± 0.3 kJ mol⁻¹²⁰, 67.6 kJ mol⁻¹²¹ and 61.9 kJ mol⁻¹.²²

Hydroxyacetone and hydroxyacetaldehyde

Once the set-up validated, saturation vapour pressures of hydroxyacetaldehyde and hydroxyacetone were measured in the temperature ranges (296 to 356) K and (273 to 304) K, respectively. The results are listed in Table 2. The relative uncertainties on the measurements varied between (5 and 13) % for hydroxyacetaldehyde and (5 and 7) % for hydroxyacetone.

The vapour pressures were fitted according to the Antoine's equation:

$$\log_{10}(P^0(\text{hydroxyacetaldehyde})/\text{Pa}) = (12.97 \pm 1.70) - \frac{3668 \pm 1101}{(T/K) + (0.45 \pm 48)}$$

$$\log_{10}(P^0(\text{hydroxyacetone})/\text{Pa}) = (10.12 \pm 2.88) - \frac{2193 \pm 1657}{(T/K) - (0.46 \pm 10)}$$

where the quoted uncertainties correspond to 1σ obtained from the least square fit analysis.

Similarly to ethylene glycol, the data of $\log_{10} P^{\circ}$ versus $1/T$ can conveniently be fitted, as shown in Figure 4, with a two parameters Antoine's equation:

$$\log_{10}(P^0(\text{hydroxyacetaldehyde})/\text{Pa}) = (12.96 \pm 0.82) - \frac{3657 \pm 238}{(T/K)}$$

$$\log_{10}(P^0(\text{hydroxyacetone})/\text{Pa}) = (10.13 \pm 0.06) - \frac{2201 \pm 153}{(T/K)}$$

where the quoted errors correspond to 2σ obtained from the least square fit analysis and include a estimated systematic relative error of 5 %.

Again, the values of ARD determined according to eq. (2), found to be equal to 2.0 % and 2.5 % for hydroxyacetone and hydroxyacetaldehyde, respectively, confirm that the experimental values can be conveniently fitted according to the simplified Antoine's equation. These equations permit then to derive the vapour pressures of both hydroxyacetaldehyde and hydroxyacetone at 293 K, which were $P^{\circ} = (3.0 \pm 0.2)$ Pa and $P^{\circ} = (415 \pm 20)$ Pa for hydroxyacetaldehyde and hydroxyacetone, respectively.

Similarly to ethylene glycol, the enthalpies of vaporization of both compounds were derived from the Clausius-Clapeyron's equation: $\Delta_{vap}H$ (hydroxyacetaldehyde) = (70 ± 5) kJ mol⁻¹ $\Delta_{vap}H$ (hydroxyacetone) = (42 ± 3) kJ mol⁻¹.

Comparison with the literature

As noted in the introduction, the literature data concerning the vapour pressures of hydroxyacetaldehyde and hydroxyacetone are rather limited. For the determination of the millimeter- and sub-millimeter-wave spectrum of hydroxyacetaldehyde, Butler et al. (2001) estimated that the vapour pressure at room temperature was roughly 4 Pa¹¹ which is in good agreement with our determination of (3 ± 1) Pa at 293 K. On the contrary, Angove et al. (2006) reported an estimated value of about 5300 Pa at 298 K, a value three orders of magnitude higher than our determination, (4.9 ± 0.3) Pa at 298 K. Besides, in measurements of UV absorption cross-section, Magneron et al. (2005) used partial pressures as high as 66 Pa at 298 K, and 2100 Pa at 333 K²³. If this value obtained at 333 K is roughly consistent with

the vapor pressure of 980 Pa found in this work, their value reported at 298 K is approximately 20 times higher than our absolute determination. These discrepancies might be due to the presence of volatile impurities in hydroxyacetaldehyde in their study while they were removed, in the current work, after obtaining of hydroxyacetaldehyde monomers, by freeze-pump-thaw cycles.

Similarly, in experiments dedicated to Raman spectroscopy, the vapour pressure of hydroxyacetone was estimated to be less than 500 Pa at room temperature ¹². This reported upper limit is consistent with our determination of (415 ± 20) Pa at 293 K.

Conclusion

Our static device was tested to measure the vapour pressure of ethylene glycol and the obtained data shown an excellent agreement with those reported in the literature.

We have then measured the vapour pressures of hydroxyacetone and hydroxyacetaldehyde as a function of temperature. These data are the first direct measurements for these two compounds. They will be particularly valuable in laboratory studies where they are often necessary.

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Table 1: Experimental vapour pressures of ethylene glycol measured in this work and comparison with the literature. $100 \times \Delta P^\circ / P^\circ$ corresponds to the percentage deviation between experimental and literature values.

T / K	P°_{lit} ^(a) / Pa	P° ^(b) / Pa	$100 \times \Delta P^\circ / P^\circ$ ^(c)
307.7 (± 0.1)	26 (± 1)	28 (± 1)	0.81
312.6 (± 0.1)	39 (± 2)	41 (± 2)	1.30
316.5 (± 0.1)	52 (± 3)	55 (± 3)	2.52
318.8 (± 0.1)	62 (± 3)	61 (± 3)	5.12
322.1 (± 0.1)	79 (± 4)	77 (± 4)	5.38
326.1 (± 0.1)	124 (± 6)	124 (± 6)	0
328.2 (± 0.1)	128 (± 6)	137 \pm (23)	0.74
334.5 (± 0.2)	192 (± 10)	196 (± 13)	2.11
335.2 (± 0.2)	201 (± 10)	201 (± 14)	0.11
340.2 (± 0.2)	282 (± 14)	278 (± 19)	0.37
347.9 (± 0.2)	464 (± 23)	454 (± 32)	0.03
359.2 (± 0.2)	923 (± 46)	900 (± 58)	1.32
367.9 (± 0.2)	1528 (± 76)	1483 (± 91)	1.98
373.4 (± 0.2)	2086 (± 104)	1950 (± 131)	1.02
384.5 (± 0.2)	3772 (± 189)	3467 (± 236)	1.29

^(a) The quoted uncertainties correspond to 2σ level + 5% where σ is the standard deviation of the linear fit of the plot of $\text{Log}_{10} P_{lit}$ vs $1/T$ ¹⁹.

^(b) The quoted uncertainties correspond to 2σ level + 5%.

^(c) The deviation between the experimental and literature values is defined as follows:

$$\frac{\Delta P^\circ}{P^\circ} = \left(\frac{P^\circ - P^\circ_{lit}}{P^\circ} \right)$$

Table 2: Experimental vapour pressures of both hydroxyacetaldehyde and hydroxyacetone measured in this work. The quoted uncertainties correspond to 2s Level + 5%.

Hydroxyacetaldehyde			Hydroxyacetone		
T / K	P° / Pa	100×ΔP°/P° ^(a)	T / K	P° / Pa	100×ΔP°/P° ^(a)
295.85	3.7 (±0.2)	0.44	273.22	127 (±9)	3.26
305.10	9.0 (±0.5)	5.24	275.48	136 (±9)	4.73
305.51	9.6 (±1.2)	2.26	277.78	162 (±11)	2.59
319.50	33 (±3)	2.72	278.55	167 (±11)	4.44
319.84	33 (±3)	0.00	278.55	172 (±11)	1.55
322.38	37 (±3)	6.37	280.90	201 (±42)	0.48
323.64	46 (±3)	2.06	281.69	205 (±13)	3.52
327.73	62 (±5)	1.38	281.69	210 (±13)	1.47
333.04	98 (±10)	4.01	289.86	356 (±21)	1.74
339.32	154 (±9)	2.14	290.70	360 (±21)	2.25
340.91	170 (±10)	0.74	290.70	363 (±21)	1.23
341.95	182 (±12)	0.15	290.70	360 (±21)	2.25
342.82	198 (±15)	2.57	293.26	431 (±25)	0.84
346.83	253 (±18)	1.33	295.86	516 (±30)	3.83
355.98	486 (±26)	2.00	295.86	495 (±29)	0.10
			296.74	531 (±31)	1.90
			300.30	617 (±35)	3.04
			301.20	642 (±36)	4.05
			302.11	717 (±40)	2.03
			303.03	746 (±42)	1.06
			303.95	817 (±46)	4.96

^(a) The individual deviation $\Delta P^\circ/P^\circ$ is calculated as follows: $\Delta P^\circ/P^\circ = (P^\circ - P^\circ_{\text{Antoine}})/P^\circ$ where P°_{Antoine} is calculated from the simplified Antoine's equation and P° is the experimental value.

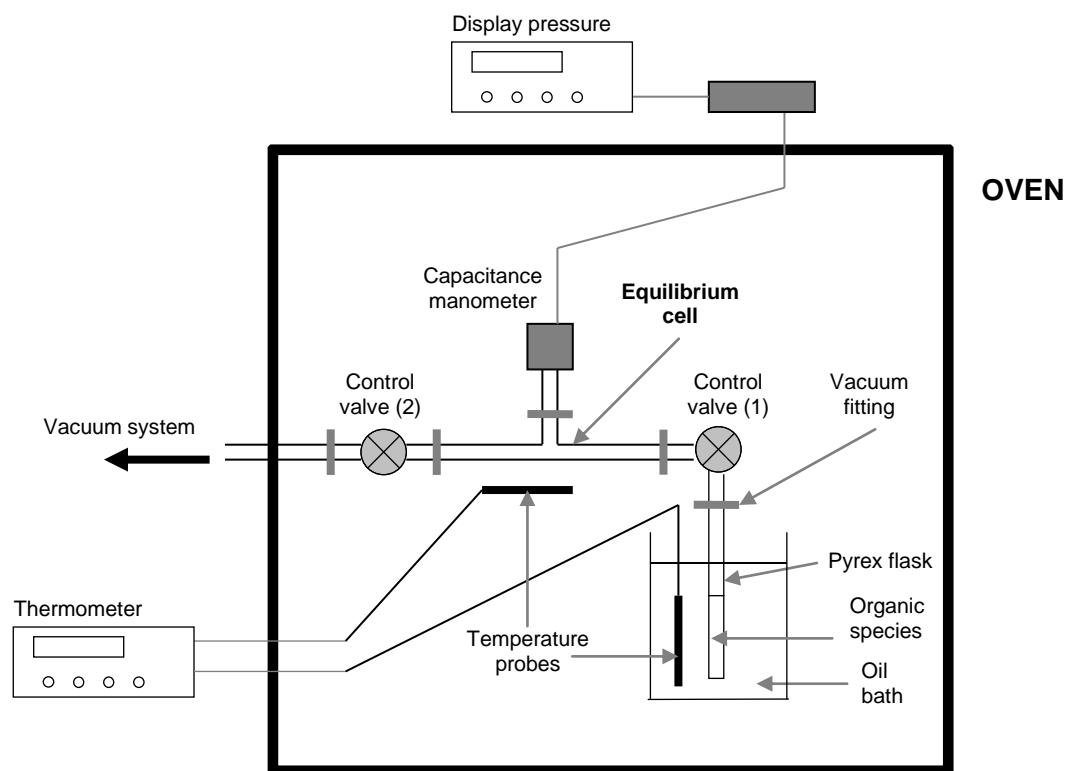


Figure 1: Schematic diagram of vapour pressure apparatus.

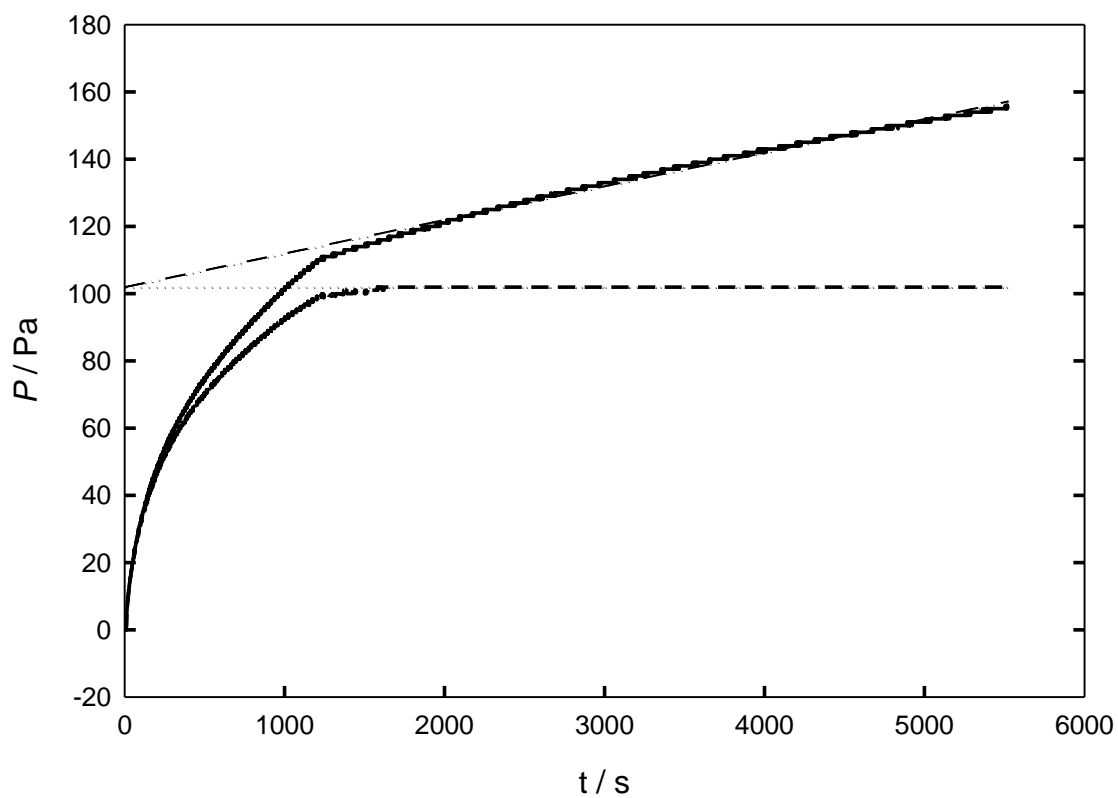


Figure 2: Plot of both P (solid line) and $P_{\text{corrected}}$ (short dash line) recorded as a function of time during an experiment performed on ethylene glycol at 326.1 K. The leak rate is determined from the slope of the linear asymptote (dash-dot-dot line).

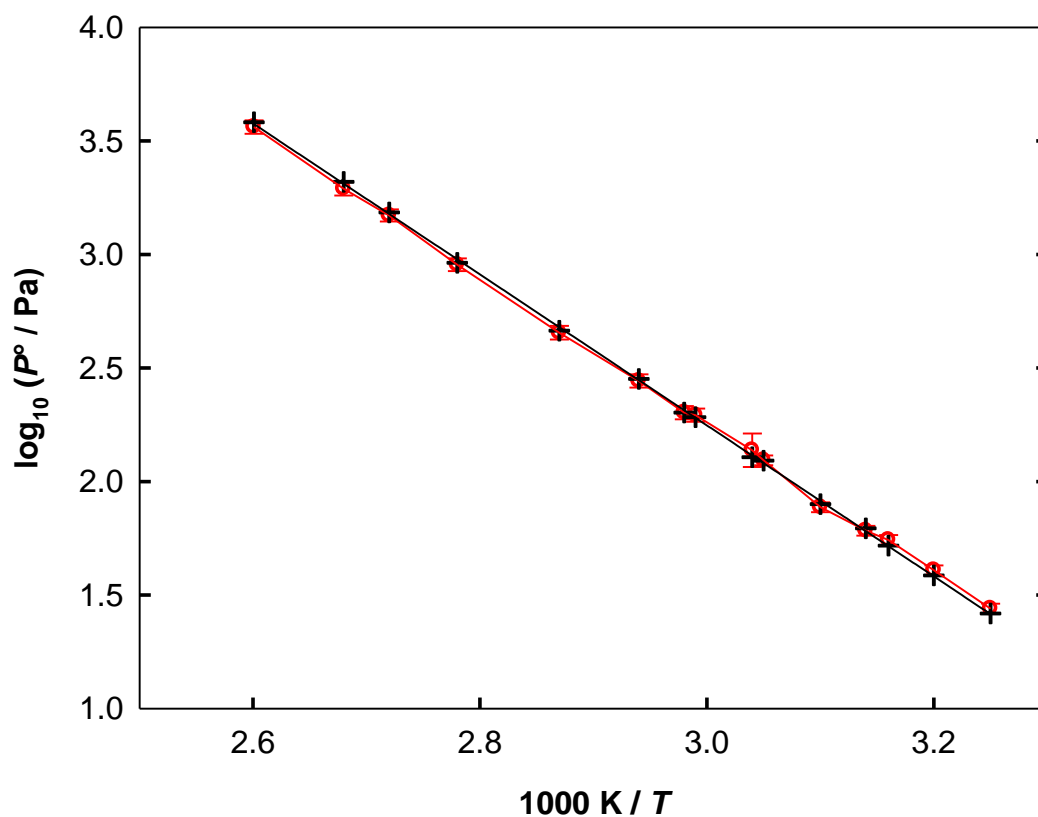


Figure 3: Comparison for vapour pressures of ethylene glycol measured from this study (red circles) and those from literature ¹⁹ (black cross) and the resulting fits using the simplified Antoine's equation (see solid lines). The quoted uncertainties on experimental data correspond to 2σ Level +5% (see text).

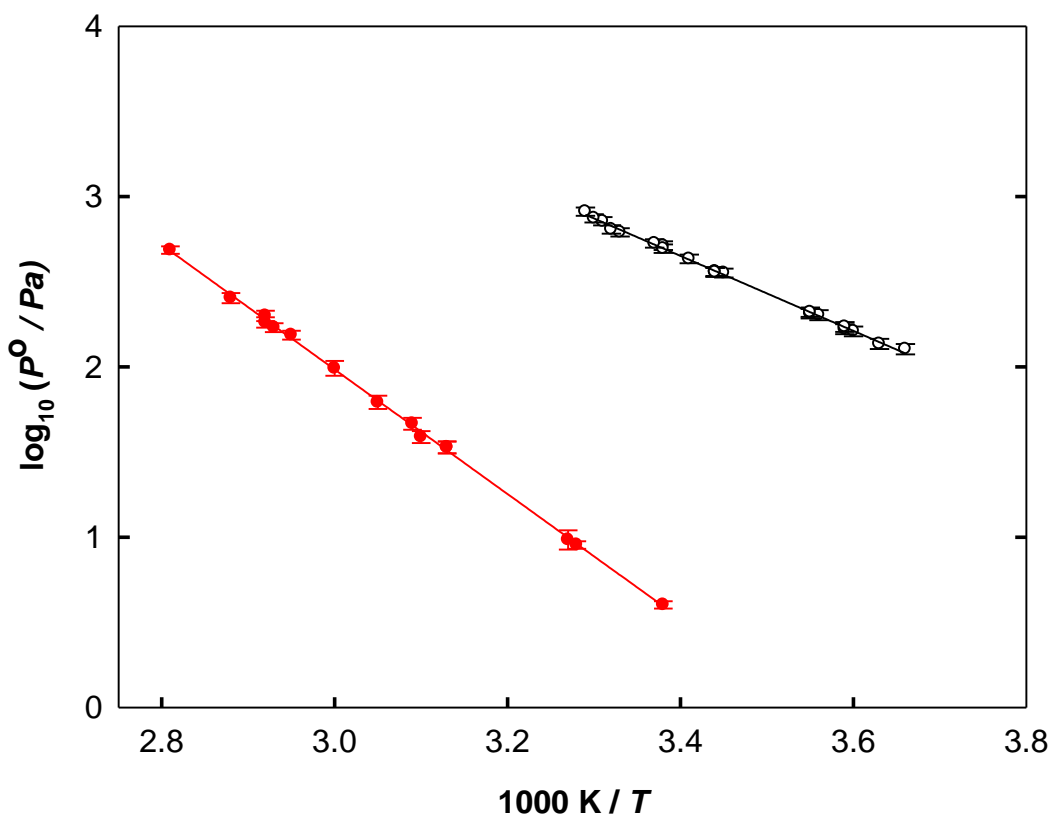


Figure 4: Plot of the $\log_{10}(P^\circ / \text{Pa})$ as a function of $1000/T$ for (●) hydroxyacetaldehyde and (○) hydroxyacetone. The solid lines correspond to the fit using the simplified Antoine's equation. The quoted uncertainties on experimental data correspond to 2σ Level +5% (see text).