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Vapor and Sublimation Pressures of Three Normal Alkanes: C_{20}, C_{24}, and C_{28}

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Vapor and sublimation pressures of three *n*-alkanes C_{20}, C_{24}, and C_{28} have been determined using a modified gas saturation method. The obtained pressure values range from 10^{-5} Pa to 5.5 Pa. From the temperature dependence of the vapor pressures, the molar enthalpies of vaporization and sublimation at the mean temperature of the experimental range were derived from the Clausius−Clapeyron equation. From these results, the standard enthalpies of vaporization and sublimation at $T = 298.15$ K were calculated.

Introduction

Vapor pressures of heavy *n*-alkanes are of practical importance in the petroleum industry for the characterization of heavy petroleum cut and synthetic fluids. These data are also needed for thermodynamic calculations and for estimating properties of other classes of compounds. Although vapor pressures of normal alkanes up to *n*-C_{92} are available in the literature,[1−9,21] sublimation pressures are practically inexistent.

In our previous works,[10,11] vapor pressures of some alkanes up to *n*-C_{30} were studied. Continuing this research line, the vapor and sublimation pressures of three normal alkanes, *n*-C_{20}, *n*-C_{24}, and *n*-C_{28}, have been measured using the gas saturation method. Enthalpy of vaporization ($\Delta_{vap}H$) and sublimation ($\Delta_{sub}H$) have also been determined from the vapor and sublimation pressures and compared with the existing literature data.

Experimental Section

Chemicals. The suppliers and the purities of the used materials are as follows: hexadecane, *n*-C_{16} (Janssen, 99 %); eicosane, *n*-C_{20}, and tetracosane, *n*-C_{24} (Aldrich, 99 %); octacosane, *n*-C_{28} (Acros Organics, 99 %).

Apparatus. The apparatus for the vapor pressure determination was based on the gas saturation method, also known as the transpiration method. The apparatus allows reliable measurements over a large pressure interval ranging from 10^{-5} Pa to 10^3 Pa. The detailed description of the saturation apparatus can be found elsewhere.[11,12] Therefore, we give only the most significant information and modifications made to improve the apparatus. The experimental apparatus, presented in Figure 1, was composed of two parts. The sampling part consisted of an equilibrium oven containing two saturators, which were constituted by stainless steel columns filled with a porous gas chromatography support, respectively, impregnated with the sample and the standard compound. The second part was a gas chromatograph equipped with a capillary column and a flame ionization detector (FID). When thermal equilibrium was reached, both compounds were simultaneously swept by the inert gas N_2 into the cold analysis column where they were trapped. To limit adsorption and desorption phenomena, the connection between the saturators and the gas chromatograph was modified. A silica capillary tube (T) was connected to the outlet tube of the saturators in one side, whereas the other end was penetrating inside the analysis column. Under these conditions, the carrier gas did not pass through the union tube of fused silica and desorbed only the compounds trapped in the analysis column. By heating the capillary column, the two compounds were eluted and detected by the FID. The present apparatus is totally automatic because all of the valves are controlled by the gas chromatograph output.

Table 1. Vapor and Sublimation Pressures of *n*-Alkanes, *n*-C_{20}, *n*-C_{24}, and *n*-C_{28}

T/K	P/Pa	T/K	P/Pa
n-C_{20} Solid		*n*-C_{20} Liquid	
302.37	0.00173	312.76	0.0113
304.59	0.00330	322.82	0.0345
306.79	0.00459	327.82	0.0601
307.86	0.00594	337.82	0.174
		342.89	0.293
		373.17	5.45
n-C_{24} Solid		*n*-C_{24} Liquid	
307.74	0.0000589	333.09	0.00458
312.83	0.000178	353.20	0.0407
317.93	0.000479	373.13	0.268
322.94	0.00123		
n-C_{28} Solid		*n*-C_{28} Liquid	
323.05	0.0000117	339.02	0.000358
326.15	0.0000237	352.98	0.00186
329.02	0.0000439	372.85	0.0176
		392.76	0.114
		412.72	0.606

Impregnation of the Support. The impregnation of the support by the sample or the standard compound was done by batch. The compound (0.5 g) was dissolved in an organic solvent (toluene). The chromatographic support (Gas Chrom P (147 to 175) μm) was added to the solution so as to obtain a "compound mass/support mass" ratio equal to 0.2. The cell containing the mixture was subjected to the action of an oscillating stirrer for 24 h before the solvent was totally evaporated using a rotary evaporator under vacuum. The dry support impregnated with the compound was finally introduced to the saturation column.

Saturation Gas Flow Rate and Purge Time. A preheated nitrogen steam was passed through the saturators at constant

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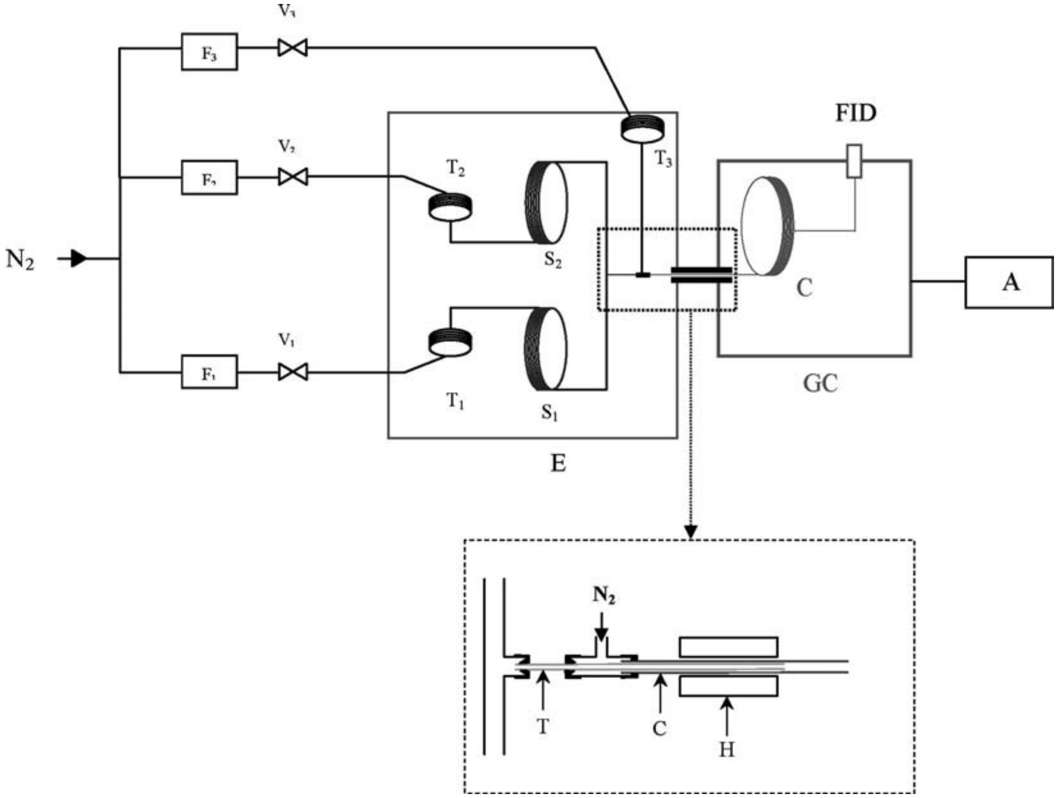

Figure 1. Saturation apparatus. A, chemstation acquisition from Agilent; C, analytical nonpolar capillary column (dimethylpolysiloxane), length: 10 m, film thickness of the stationary phase: 2.65 μm; F_1, F_2, F_3, mass flow meters from Bronkhorst, flow range (0 to 10) mL·min^{-1}, uncertainty 1 %; H, heated zone; S_1, S_2, saturation stainless steel column ($L = 2$ m; i.d. = 2.1 mm) containing Gas Chrom P support (particle diameter: (147 to 175) μm); T, capillary silica tube ($L = 25$ cm; i.d. = 0.32 mm); T_1, T_2, T_3, stainless steel tubing ($L = 3$ m; i.d. = 0.50 mm) for saturation and carrier gas preheating; V_1, V_2, V_3, electrovalves controlling gas flow.

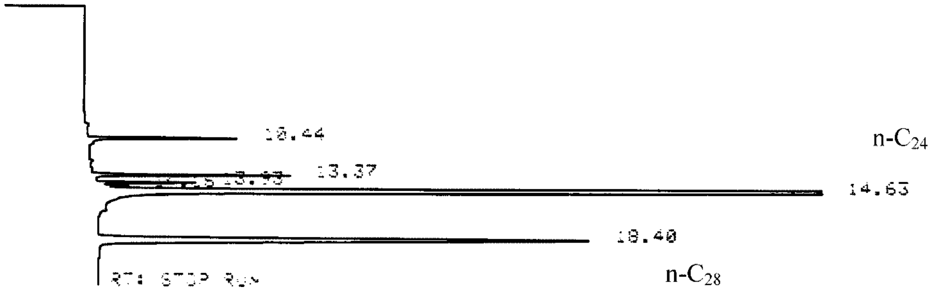

Figure 2. Chromatogram obtained at 372.85 K when studying n-C_{28} with n-C_{24} as reference compound (trapping time: 2 h).

temperature. The flow rates were measured with a relative uncertainty of 1 % using mass flow meters from Bronkhorst with a real-time monitoring system. The flow rates were optimized to reach the saturation equilibrium of the gas. Therefore, experiments were carried out using flow rates ranging from (3 to 8) mL·min^{-1} according to the equilibrium temperature and the volatility of the compounds. (In this field, the vapor pressures or the sublimation pressures are independent of the gas flow.) The same trap time was applied to both sample and reference compounds. It varies between 30 min and 10 h.

Vapor Pressure Determination. The vapor pressures were calculated using the following equation, which supposes ideal behavior of the vapor phase

$$\frac{P_1}{P_2} = k\frac{A_1}{A_2}\frac{M_2}{M_1}\frac{F_2}{F_1} \tag{1}$$

Subscripts $i = 1$ and $i = 2$ refer, respectively, to the standard and the sample, P_i is the vapor pressure, A_i is the chromatographic peak area, M_i is the molar mass, F_i is the saturation gas flow rate, and k is the relative mass response factor of the FID. For normal alkanes between n-C_{12} and n-C_{38}, the relative mass response factor, k, is equal to unity. It was verified experimentally in a previous paper.[11]

Figure 2 shows an example of a chromatogram obtained at 372.85 K when studying n-C_{28}. In this case, n-C_{24} was the reference compound. The small peaks appearing in the beginning of the chromatogram were due to the impurities contained in n-C_{24}.

Uncertainty of the Vapor and Sublimation Pressures. Knowing that the uncertainty of the flow rate of the saturation gas is 1 % and the relative uncertainty of the area ratio is 3 %,[11] we deduced from eq 1 the relative uncertainty of the pressure ratio, which is about 4 %. In the case of n-C_{20} and n-C_{24}, the reference compound (respectively, n-C_{16} and n-C_{20}) is known with good accuracy (1 % uncertainty). By a quadratic combination with the pressure ratio, the relative uncertainty of the vapor and sublimation pressures for these 2 alkanes is 5 %. In the case of n-C_{28}, the reference compound is n-C_{24}, presenting

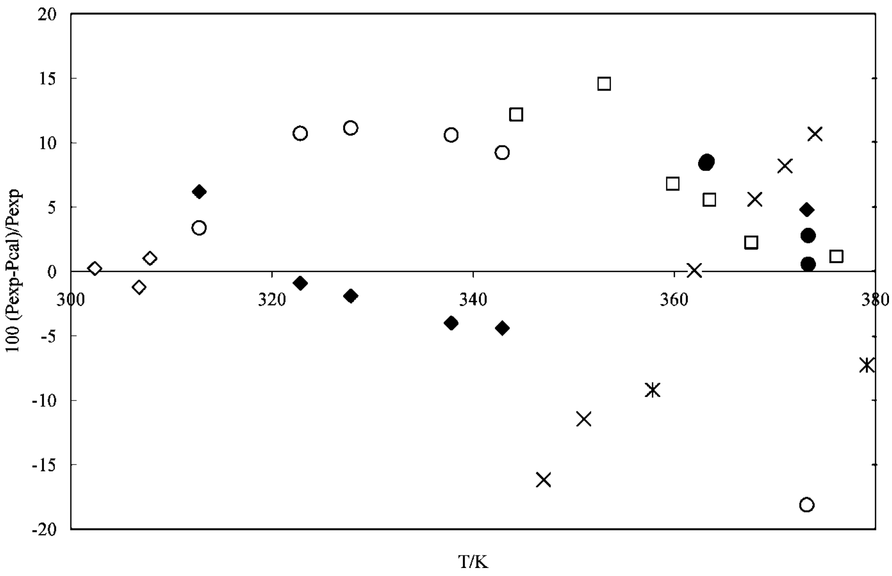

Figure 3. Relative deviation of the experimental vapor and sublimation pressures of n-C_{20} from values obtained with the Clausius−Clapeyron equation as a function of temperature T/K: ◇, solid phase (this work); ◆, liquid phase (this work); ●, Sasse et al.;[8] *, Grenier et al.;[7] ×, Piacente et al.;[5] ○, Ruzicka and Majer;[3] □, Macknick and Prausnitz.[6]

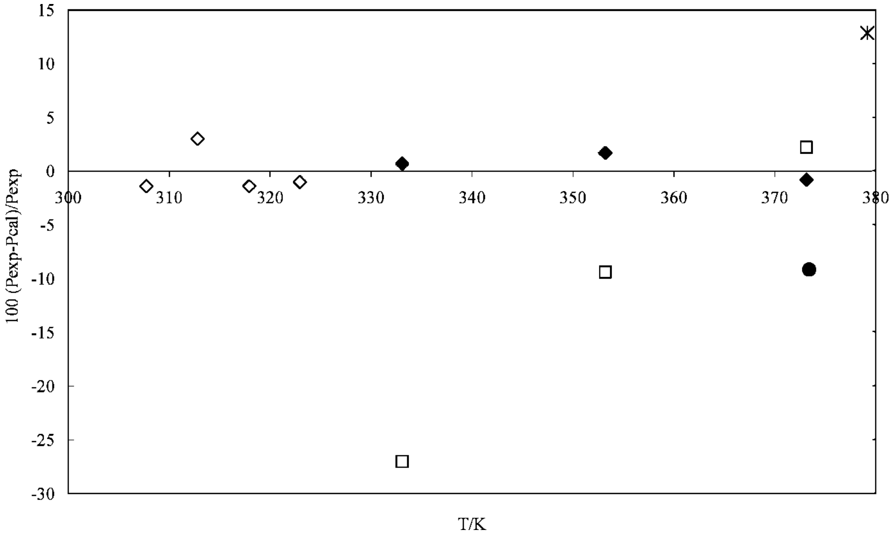

Figure 4. Relative deviation of the experimental vapor and sublimation pressures of n-C_{24} from values obtained with the Clausius−Clapeyron equation as a function of temperature T/K: ◇, solid phase (this work); ◆, liquid phase (this work); ●, Sasse et al.;[8] *, Grenier et al.;[7] □, Chickos and Hanshaw.[4]

Table 2. Clausius−Clapeyron Equation Parameters, *A* and *B*, Standard Deviation, *σ*, Mean Relative Deviation, *d*, and $B = \Delta_{vap}H\ (T_m)/R$ (or $B = \Delta_{sub}H\ (T_m)/R$) with $R = 8.314\ J \cdot K^{-1} \cdot mol^{-1}$[a,b]

alkanes	temperature range T K	T_m K	$A\ (\sigma_A)$	$B\ (\sigma_B)$	100d	$\Delta_{vap}H\ (T_m)\ (\sigma)$ kJ·mol^{-1}	$\Delta_{sub}H\ (T_m)\ (\sigma)$ kJ·mol^{-1}
n-C_{20} (solid)	302.37 to 307.86	305	62.37 (1.19)	20 784 (365)	0.83		172.8 (3.0)
n-C_{20} (liquid)	312.76 to 373.17	343	33.72 (0.37)	11 970 (130)	3.7	99.5 (1.1)	
n-C_{24} (solid)	307.74 to 322.94	315	54.74 (0.68)	19 840 (220)	1.8		164.9 (1.8)
n-C_{24} (liquid)	333.09 to 373.13	353	32.59 (0.16)	12 647 (56)	0.90	105.1 (0.5)	
n-C_{28} (solid)	323.05 to 329.02	326	61.54 (0.83)	23 550 (270)	0.58		195.8 (2.2)
n-C_{28} (liquid)	339.02 to 412.72	376	33.86 (0.35)	14 160 (140)	4.0	117.4 (1.2)	

[a] T_m: mean temperature of the experiments. [b] $d = (1/n) \sum (|P_{exptl} - P_{calcd}|)/(P_{exptl})$.

a relative uncertainty of 5 %. Therefore, the uncertainty of n-C_{28} is estimated to be 7 %. The uncertainty of the temperature is 0.02 K.

Results and Discussion

The vapor and sublimation pressures of n-C_{20} were determined using n-C_{16} as the standard compound (Table 1). The values of the latter were taken from Ruzika and Mayer.[3] A deviation plot showing differences between the various literature sources and the fitted experimental results is represented in Figure 3. As can be observed, the present work is in good agreement with the data reported by Macknick and Prausnitz[6] and Sasse et al.,[8] and the deviation is below 10 %. (Our values are systematically low.) In the same way, the values reported by Grenier et al.[7] exhibit a difference of 7 % to 9 % from the present data. In this case, our values are systematically above

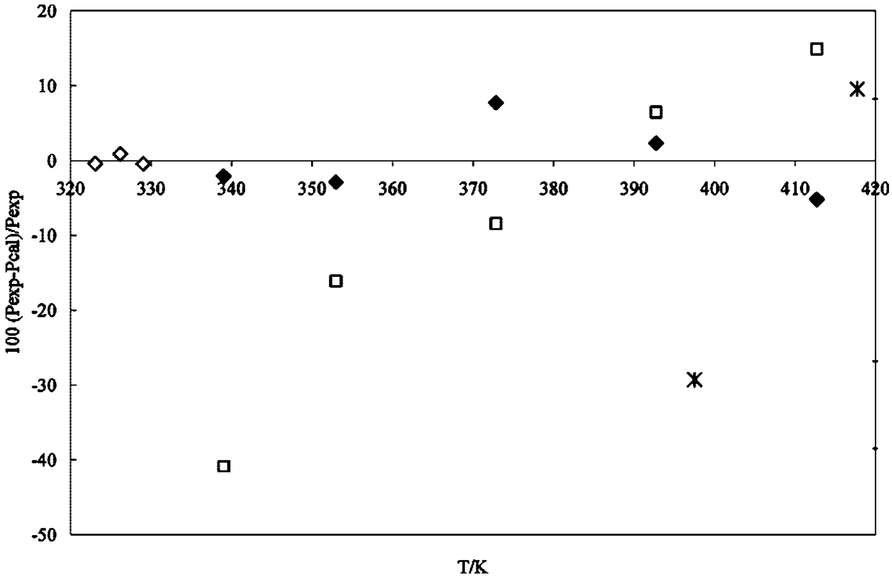

Figure 5. Relative deviation of the experimental vapor and sublimation pressures of *n*-C_{28} from values obtained with the Clausius−Clapeyron equation as a function of temperature *T*/K: ◇, solid phase (this work); ◆, liquid phase (this work); *, Grenier et al.;[7] □, Chickos and Hanshaw.[4]

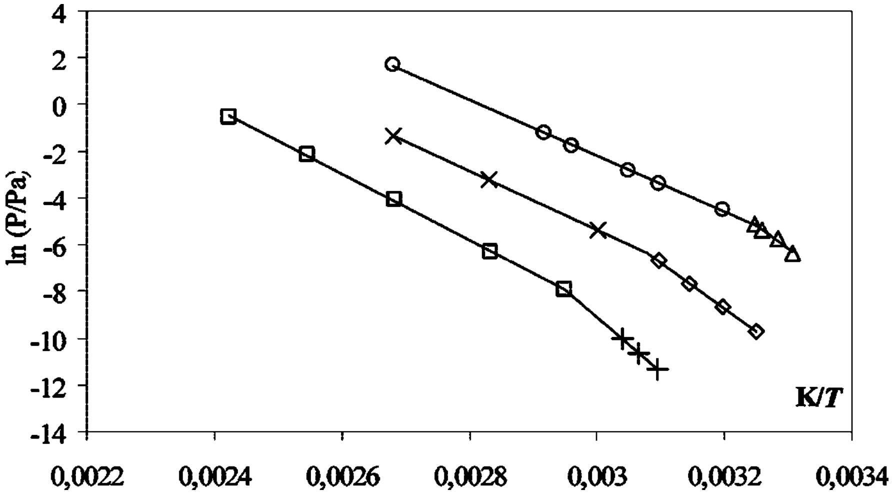

Figure 6. Clausius−Clapeyron representation (ln *P* versus 1/*T*) for *n*-C_{20} (○, liquid phase; △, solid phase), for *n*-C_{24} (×, liquid phase; ◇, solid phase), and for *n*-C_{28} (□, liquid phase; +, solid phase).

those of the authors. Except at (347 and 351) K, where the deviations are, respectively, 16 % and 11 %, the present study is in a good agreement with Piacente et al. data.[5] The comparison of the experimental vapor pressures with Ruzicka and Majer[3] recommended data shows good agreement except at 373 K, where the reported value is lower than the experimental measurement by 18 %; nevertheless, the agreement with the rest of the previously quoted literature values at 373 K is good.

As for *n*-C_{24}, the used standard compound was the previously studied *n*-C_{20}. The data of the standard compound are from the present study and from the values published by Viton et al.[9] The vapor pressures of *n*-C_{24} were compared with literature data (Figure 4). There is good agreement with Sasse et al.[8] experimental point and with Grenier-Loustalot et al.[7] value. Two common points were found with Chickos and Hanshaw[4] values at (353.20 and 373.13) K. The relative deviations with the reported data are, respectively, 9 % and 2 %.

For *n*-C_{28} vapor pressure determinations, *n*-C_{24} was used as the standard compound (Table 1). The data of *n*-C_{24} were taken from Mokbel et al.[11] for the liquid phase and from the present study for the solid phase. As shown in Figure 5, the agreement with Chickos and Hanshaw[4] is good. Two common points were found with Grenier et al.[7] data, at (397.55 and 417.75) K. The relative deviation with the present study is, respectively, −29 % and 10 %. No literature data were found with which the solid phase of the studied alkanes could be compared. The pressure values below the temperature of fusion of *n*-C_{24} and *n*-C_{28} reported by Chickos and Hanshaw[4] were not taken into account in the present study because they are relative to subcooled liquid.

Another way to check the consistency of our data is to compare the enthalpies obtained in this work with the literature data. With this aim, the vapor and sublimation pressures were fitted to the Clausius−Clapeyron equation

$$\ln P/\text{Pa} = A - \frac{B}{T/\text{K}} \tag{2}$$

$$\text{with } B = \frac{\Delta_{\text{vap}}H}{R} \text{ or } B = \frac{\Delta_{\text{sub}}H}{R} \tag{3}$$

From the fits, the enthalpy of vaporization, $\Delta_{\text{vap}}H$, and the enthalpy of sublimation, $\Delta_{\text{sub}}H$, at the mean temperature of the

Table 3. Comparison of the Vaporization and Sublimation Enthalpies (in kJ·mol^{-1}) of Alkanes *n*-C_{20}, *n*-C_{24}, and *n*-C_{28} with Literature Data

alkanes	$\Delta_{vap}H$ (298.15 K)^a	$\Delta_{vap}H$ (298.15 K)	$\Delta_{sub}H$ (298.15 K)^a	$\Delta_{tr}H(T_t/K)^b$ (T_t/K)	$\Delta_{sub}H^a$ (298.15 K) + $\Delta_{tr}H$ $(T_t)^b$	$\Delta_{sub}H$ (298.15 K)
n-C_{20}		101.8^c				
		121.7^d	173.4		173	168.6^e
	107.5	101.8^{e,i}				170.5^i
		113.6^h				
		94.2^l				
n-C_{24}		118.8^d				
		121.9^e	166.6	31.3	198	205.6^e
		129.5^f		(321.3)		210.3^i
	116.7	127.4^g				
		125.6^i				
		115.5^j				
		116.1^j				
		133.5^l				
n-C_{28}		154.0^d				
		141.9^e	199.1	35.4	235	238.0^e
		157.6^g		(330.5)		250.8^i
	136.4	153.0^h				
		151.4^i				
		148.9^j				
		164.8^k				
		154.5^l				

[a] This work. [b] Transition enthalpy (kJ·mol^{-1}) at T_t (transition temperature) from refs 19 and 20. [c] Ref 3. [d] Ref 15. [e] Ref 4. [f] Ref 8. [g] Ref 7. [h] Ref 1. [i] Ref 16. [j] Ref 17. [k] Ref 5. [l] Ref 18.

experiments, T_m, were determined and are presented in Table 2 along with the calculated standard deviations. The plots of ln $P = f(1/T)$ for the solid and liquid phase obtained for each compound are presented in Figure 6. Sublimation and vaporization enthalpies at 298.15 K were derived from the sublimation and vaporization enthalpies calculated at the mean temperature, T_m, of the experiments using Chickos and Acree[13,14] equations

$$\Delta_{sub}H(298.15\ K)/J{\cdot}mol^{-1} = \Delta_{sub}H(T_m) + [0.75 + 0.15C_{ps}(298.15\ K)](T_m - 298.15\ K) \quad (4)$$

$$\Delta_{vap}H(298.15\ K)/J{\cdot}mol^{-1} = \Delta_{vap}H(T_m) + [10.25 + 0.26C_{pl}(298.15\ K)](T_m - 298.15\ K) \quad (5)$$

The heat capacities of the solid, C_{ps}, and liquid, C_{pl}, were calculated using a group additivity method developed by Chickos and Acree.[13,14] The calculated values of the standard enthalpies of sublimation and vaporization at $T = 298.15$ K are reported in Table 3 and compared with the literature data.

The $\Delta_{vap}H$ (298.15 K) of the present work obtained for *n*-C_{20} is in good agreement with the different literature values (Chirico et al.,[1] Ruzicka and Majer,[3] Chickos and Hanshaw[4]) (relative deviation about 5 %), except for with the Piacente et al.[5] and Morgan and Kobayashi[18] values (relative deviation 12 %).

For *n*-C_{24}, $\Delta_{vap}H$ (298.15 K) of this study is in good accord with the values reported by Piacente et al.[15,17] (relative deviations are between 1 % to 2 %) but is in disagreement with the remaining authors (relative deviations of 7 % and 8 %, respectively, with Grenier et al.[7] and Sasse et al.[8] values and 13 % with Morgan and Kobayashi[18] enthalpy of vaporization). If the relative deviation between Chickos and Wilson[16] $\Delta_{vap}H$ (298.15 K) and the present study for *n*-C_{24} is 7 %, then the relative deviation drops to 4 % with the more recent value reported by Chickos and Hanshaw.[4] The $\Delta_{vap}H$ (298.15 K) obtained in this work for *n*-C_{28} presents relative deviations with the literature data ranging between 8 % and 17 % (Piacente et al.:[17,15,5] 8 %, 11 %, and 17 %), 10 % with the Grenier et al.[7] value, and 11 % and 13 % with, respectively, Chirico et al.[1] and Morgan and Kobayashi[18] enthalpy of vaporization. The relative deviations are 10 % and 4 % with, respectively, Chickos and Wilson[16] and Chickos and Hanshaw[4] values.

It is known that solid alkanes present different crystallographic structures according to the evenness and the length of the chain. For example, alkane *n*-C_{20} and *n*-C_{24} have triclinic structure, whereas *n*-C_{28} presents a monoclinic structure.[19,20] Moreover, in the case of *n*-C_{24} and *n*-C_{28}, solid−solid transition (order−disorder transition) is observed. [19] Chickos and Hanshaw[4] and Chickos and Wilson[16] deduced the enthalpy of sublimation by combining $\Delta_{vap}H$ (298.15 K), determined by gas chromatography correlation, with the total phase change fusion enthalpies adjusted to 298.15 K including the transition enthalpy. In column 5 of Table 3, the enthalpy of transition $\Delta_{tr}H$ (T_t/K) at the transition temperature, T_t, measured by Schaerer et al.[20] was added to the $\Delta_{sub}H$ (298.15 K) of the present work. The transition enthalpy was not adjusted to $T = 298.15$ K because the transition temperature is close to ambient temperature. In the case of *n*-C_{24}, the sublimation enthalpy deduced from the measured sublimation pressures comprised one point at $T = 322.94$ K, higher than the transition temperature, $T = 321.3$ K, by 1.6 K. This point formed a straight line with the remaining measurements in the solid phase. This observation speculates that the transition did not have a significant effect on the sublimation enthalpy.

The obtained values (column 6, Table 3) were compared with the literature values. The agreement between Chickos and Hanshaw[4] sublimation enthalpies of *n*-C_{20}, *n*-C_{24}, and *n*-C_{28} with the values obtained in the present study is good (relative deviation is comprised between 1 % and 4 %).

Conclusions

In this article, we presented sublimation and vaporization pressures of three *n*-alkanes determined by means of the gas saturation technique. The use of a reference compound allowed the automatic operation of the apparatus. The recent modifications of the apparatus carried out to limit adsorption phenomena have permitted the measurements of sublimation pressures with estimated good uncertainty between 4 % and 7 % depending on the associated uncertainty of the reference compound.

Although the explored domain of pressures is very low, between 10^{-5} Pa and 5 Pa, the obtained results are consistent and in good agreement with the available experimental data in the literature.

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