Density, Viscosity, and Refractive Index in the Range (283.15 to 353.15) K and Vapor Pressure of α-Pinene, *d*-Limonene, (±)-Linalool, and Citral Over the Pressure Range 1.0 kPa Atmospheric Pressure

René A. Clará, Ana C. Gómez Marigliano,* and Horacio N. Sólimo

Departamento de Física - Facultad de Ciencias Exactas y Tecnología, Universidad Nacional de Tucumán,
Avenida Independencia 1800, 4000 San Miguel de Tucumán, Argentina

Measurements of density, viscosity, and refractive index in the $T = (283.15 \text{ to } 353.15)$ K temperature range at atmospheric pressure and vapor pressure over the $P = (1.0 \text{ to } 96.7, \text{ or } 96.0, \text{ or } 95.2, \text{ or } 50)$ kPa pressure range for α-pinene, *d*-limonene, (±)-linalool, and citral, respectively, were made. The temperature dependence of these properties was fitted to empirical relations. The molar enthalpy of vaporization for these chemicals was calculated plotting the logarithm of the vapor pressure against the reciprocal temperature and using the Clausius−Clapeyron equation. The enantiomer ratios of α-pinene, *d*-limonene, and (±)-linalool were also obtained.

Introduction

α-Pinene, *d*-limonene, (±)-linalool, and citral are natural compounds found in essential oils of citrus fruits, flowers, and spice plants that have many technological uses.[1−7] However, only few experimental results are found in the literature on specific properties over a wide temperature range, although many works can be found where one or more of these compounds have been selected for a given study.[8−29]

In this paper, we report density ρ, viscosity η, and refractive index n_D for the sodium D-line measurements in the range $T = (283.15 \text{ to } 353.15)$ K at atmospheric pressure for α-pinene, *d*-limonene, (±)-linalool, and citral. These data are presented together with vapor pressure P data over the $P = (1.0 \text{ to } 95.2, \text{ or } 96.0, \text{ or } 96.7)$ kPa pressure range for α-pinene, *d*-limonene, and (±)-linalool, respectively. However, for citral vapor pressure, measurements were made over the $P = (1.0 \text{ to } 50.0)$ kPa pressure range only because this chemical suffers decomposition at high pressures, which is evidenced by a yellow−brownish coloration. From the experimental results, empiric equations for these properties as a function of the temperature are developed. We further calculate the molar enthalpy of vaporization for each compound from the dependence of the logarithm of the vapor pressure with the reciprocal temperature.

Experimental Section

Chemicals. α-Pinene was supplied by Carlo Erba, while *R*-(+)-limonene (also called *d*-limonene), (±)-linalool, and citral (*cis* + *trans*) were supplied by Fluka. These chemicals were fractionally distilled at reduced pressure under a nitrogen stream to avoid decomposition, collecting only middle fractions. Gas chromatographic analysis of the purified compounds showed that the mass fraction purities are 0.988, 0.975, 0.983, and 0.968 for α-pinene, *d*-limonene, (±)-linalool, and citral, respectively. Enantiomer ratios of the chiral compounds were also obtained by gas chromatographic analysis (see Appendix A, in Supporting Information).

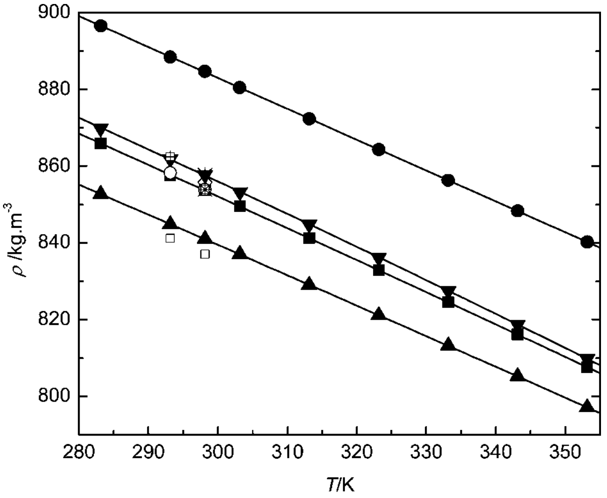

Figure 1. Plot of the density ρ against temperature T for: ■, α-pinene; ▲, *d*-limonene; ▼, (±)-linalool; and ●, citral. ○, ref 32; ×, ref 12; □, ref 8; +, ref 13. Continuous lines are the least-squares representation by means of eq 1.

Apparatus and Procedure. Density and refractive index for the sodium D-line were measured using the equipment and procedures previously reported,[30] while viscosity was measured with an Anton Paar Stabinger viscometer (SVM 3000/G2) calibrated by the manufacturer. The densimeter and viscometer are equipped with their own Peltier effect control systems, which maintained the temperature of both apparatuses at ± 0.01 K. The uncertainties were $\pm\ 0.1\ \text{kg}\cdot\text{m}^{-3}$ for density, ± 0.35 % of the measured value for viscosity, and ± 0.00005 for refractive index. On the other hand, vapor pressure data were obtained with a Fischer Labodest, model 602-S (Germany). The uncertainties of boiling temperatures and total pressure in the still were within ± 0.02 K and ± 0.1 kPa, respectively, as indicated by the manufacturer. Further experimental details can be found elsewhere.[30]

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* To whom correspondence should be addressed. Tel.: +54-381 4364093. E-mail: agomezmarigliano@herrera.unt.edu.ar.

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Table 1. Experimental Data of Density ρ, Viscosity η, and Refractive Index n_D at Several Temperatures and Calculated Normal Boiling Temperature at Atmospheric Pressure for α-Pinene, *d*-Limonene, (±)-Linalool, and Citral

		α-pinene		*d*-limonene		(±)-linalool		citral	
property	T/K	exptl	lit.	exptl	lit.	exptl	lit.	exptl	lit.
$\rho/\text{kg}\cdot\text{m}^{-3}$	283.15	865.9		852.7		869.9		896.5	
	293.15	857.6	858.2[a] 858.06[l]	844.8	841.15[b,n] 846.8[e]	861.8	862.45[b,n] 862.40[g]	888.4	
	298.15	853.7	853.9[a,b,f] 854.4[r] 853.88[g,p] 853.91[o]	841.0	837.0[a,r] 837.31[p] 838.8[q]	857.7	857.60[f] 858.10[g] 858.09[h]	884.7	
	303.15	849.5		837.0	834.7[d]	853.3		880.4	
	313.15	841.2	841.40[j]	829.0		844.8	845.43[h]	872.3	
	323.15	832.8		821.1	817.78[i]	836.2		864.3	
	333.15	824.5		813.2		827.5		856.3	
	343.15	816.1		805.2		818.7		848.3	
	353.15	807.6		797.1		809.8		840.2	
$\eta/\text{mPa}\cdot\text{s}$	283.15	1.676		1.078		9.07		2.822	
	293.15	1.409	1.40[a]	0.932		5.53		2.225	
	298.15	1.303	1.293[f] 1.296[g]	0.897	0.923[a]	4.47	4.381[f] 4.465[g]	2.111	
	303.15	1.203		0.816		3.63		1.807	
	313.15	1.039		0.722		2.541	2.552[h]	1.502	
	323.15	0.908		0.645		1.868		1.274	
	333.15	0.804		0.580		1.432		1.097	
	343.15	0.710		0.524		1.137		0.956	
	353.15	0.635		0.476		0.930		0.842	
n_D	283.15	1.47034		1.47650		1.46658		1.48930	
	293.15	1.46508	1.4658[a] 1.4657[m]	1.47212	1.4735[a] 1.4726[b,n]	1.46152	1.46202[b,n]	1.48669	
	298.15	1.46330	1.4632[a] 1.4636[f] 1.4631[r] 1.46589[k] 1.4630[o]	1.47106	1.4701[a,r]	1.45965	1.4605[f] 1.4601[g] 1.4603[h]	1.48485	
	303.15	1.46034		1.46798		1.45665		1.48294	
	313.15	1.45597		1.46368		1.45204	1.4535[h]	1.47869	
	323.15	1.45114		1.45907		1.44738		1.47420	
	333.15	1.44622		1.45408		1.44229		1.46900	
nbt/K[s]		428.9	429.1[r] 428.75[v]	450.3	448.2[r]	470.9	471 to 473[t] 470 to 472[w]	decomposes before boiling	

[a] From ref 32. [b] From ref 8. [c] From ref 9. [d] From ref 33. [e] From ref 37. [f] From ref 12. [g] From ref 13. [h] From ref 14. [i] From ref 15. [j] From ref 16. [k] From ref 19. [l] From ref 20. [m] From ref 23. [n] From ref 24. [o] From ref 25. [p] From ref 26. [q] From ref 28. [r] From ref 31. [s] nbt/K denotes calculated normal boiling temperatures in Kelvin. Note: boiling temperatures obtained at (95.2, 96.0, and 96.7) kPa for α-pinene, *d*-limonene, and (±)-linalool, respectively, were corrected to normal pressure using eq 2.1 in ref 31, while for citral no experimental value was obtained because it decomposes. [t] From ref 38. [v] From ref 11. [w] From ref 39.

Table 2. Experimental Boiling Temperatures Over the P = (1.0 to 96.7, or 96.0, or 95.2, or 50) kPa Pressure Range for α-Pinene, *d*-Limonene, (±)-Linalool, and Citral, Respectively

	boiling temperatures/K					
	α-pinene		*d*-limonene		(±)-linalool	citral
P/kPa	this work	lit.[a]	this work	lit.[b]	this work	this work
1.0	308.9	308	326.8	327	352.3	371.6
1.5	315.8	315	333.7	333	358.8	379.7
2.5	324.9	324	343.7	343	368.2	389.8
5.0	339.0	339	358.7	358	382.4	405.2
10.0	355.8	356	375.5	374	398.7	424.4
20.0	374.4	374	394.5	395	417.1	444.3
30.0	386.4	386	406.9	406	428.9	456.7
40.0	395.5	396	416.2	416	438.1	465.9
50.0	403.0	403	423.9	424	445.5	473.6
60.0	409.4	410	430.5	431	451.8	
70.0	414.9	415	436.2	436	457.3	
80.0	419.8	420	441.2	441	462.2	
90.0	424.2	425	445.6	446	466.6	
95.2					468.7	
96.0			448.2	448		
96.7	427.1	427				

[a] Interpolated from ref 8. [b] Interpolated from ref 10. Note: In this reference, some experimental values of the vapor pressure correspond to *d*-linalool. Therefore, these values are not included.

Results

Experimental results for density ρ, viscosity η, and refractive index n_D at several temperatures at atmospheric pressure for α-pinene, *d*-limonene, (±)-linalool, and citral are summarized in Table 1, while vapor pressure data over the P = (1.0 to 96.7, or 96.0, or 95.2, or 50) kPa pressure range are given in Table 2 for the same chemicals, respectively. For comparison, existing values found in the literature are also included in both tables.

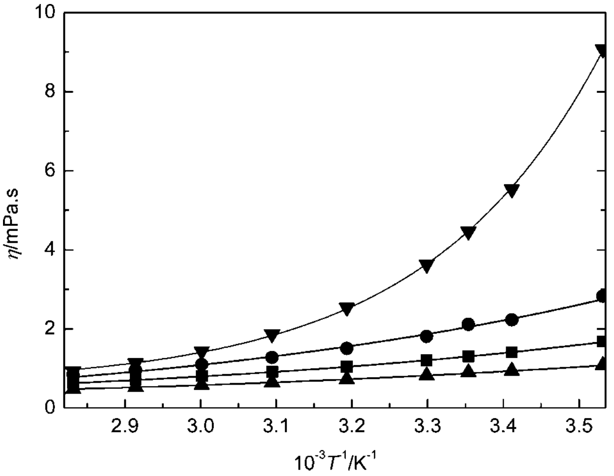

Figure 2. Plot of the viscosity η against temperature T for: ■, α-pinene; ▲, *d*-limonene, ▼, (±)-linalool, and ●, citral. ○, ref 32; ×, ref 12; □, ref 8; +, ref 13. Continuous lines are the least-squares representation by means of eq 3.

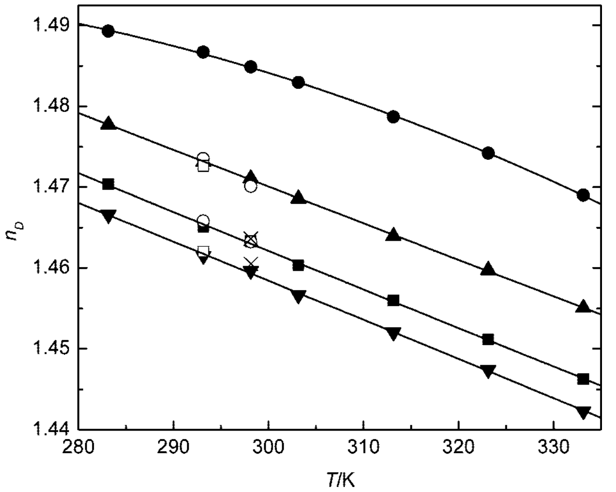

Figure 3. Plot of the refractive index n_D against temperature T for: ■, α-pinene; ▼, (±)-linalool; ▲, *d*-limonene, and ●, citral. ○, ref 32; ×, ref 12; □, ref 8; +, ref 13. Continuous lines are the least-squares representation by means of eq 2.

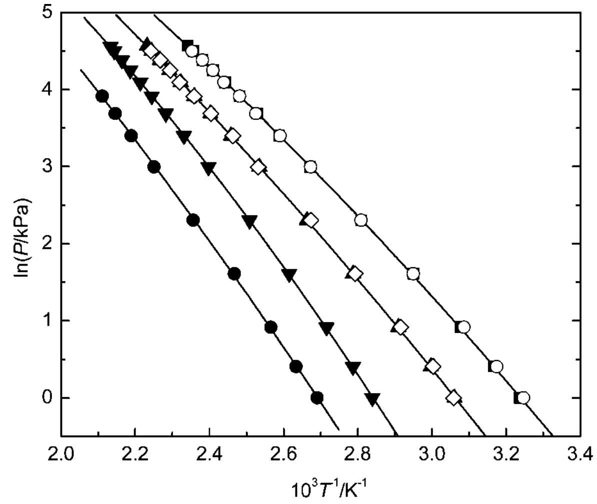

Figure 4. Plot of the logarithm of the vapor pressure P against the reciprocal temperature T over the P = (1.0 to 96.7, or 96.0, or 95.2, or 50) kPa pressure range for: ■, α-pinene; ▲, *d*-limonene; ▼, (±)-linalool, and ●, citral, respectively. ○, ref 32; ◇, ref 10.

Figures 1 to 3 show the experimental density ρ, viscosity η, and refractive index n_D against temperature, respectively. In these figures, the error bars are smaller than the plotted points. Figure 4 shows the logarithm of the experimental vapor pressure data P against the reciprocal temperature over the P = (1.0 to 96.7, or 96.0, or 95.2, or 50) kPa pressure range for α-pinene, *d*-limonene, (±)-linalool, and citral, respectively. For comparison, some existing values found in the literature are also included in Figures 1 to 4.

From these experimental results, empiric equations for the density, viscosity, and refractive index for the sodium D-line and the logarithm of the vapor pressure for α-pinene, *d*-limonene, (±)-linalool, and citral as a function of the temperature were developed, as follows

$$\rho/\text{kg}\cdot\text{m}^{-3} = A + B\cdot(T/\text{K}) \tag{1}$$

$$n_D = C + D\cdot(T/\text{K}) + E\cdot(T/\text{K})^2 \tag{2}$$

$$\eta/\text{mPa}\cdot\text{s} = F + G\cdot\exp\left(\frac{H}{T/\text{K}}\right) \tag{3}$$

$$\ln(P/\text{kPa}) = I + J\cdot(T/\text{K})^{-1} + M\cdot[(T/\text{K})^{-1}]^2 \tag{4}$$

where A to M are parameters listed in Tables 3 and 4 together with the standard deviation for each compound.

Differentiating eq 4 with respect to T and using the Clausius−Clapeyron equation, the temperature dependence for the molar enthalpy of vaporization ΔH_V of the studied compounds can be obtained

$$\Delta H_V/\text{kJ}\cdot\text{mol}^{-1} = -10^{-3}R[J + 2M/(T/\text{K})] \tag{5}$$

where R is the universal gas constant (= 8.31451 J·mol^{-1}·K^{-1}) and T is the absolute temperature in Kelvin.

Numerical values of the coefficients were obtained from a least-squares analysis of the data. The number of coefficients used for each property was determined as the minimum number needed to adequately represent the data. The number of significant digits was established taking into account the error of each parameter. The standard deviation, σ, between the experimental and calculated values was defined as

$$\sigma = \left[\sum (Y_{\text{exptl}} - Y_{\text{calcd}})^2/(N - p)\right]^{1/2} \tag{6}$$

where Y represents either ln P, ρ, η, or n_D, while N and p are the numbers of experimental points and parameters, respectively.

Discussion

From our experimental results, we conclude that a linear dependence of the density with temperature is obtained for all the chemicals. On the other hand, for the refractive index this relationship is linear for α-pinene, *d*-limonene, and (±)-linalool, while for citral it is quadratic. Furthermore, the viscosity shows an exponential dependence with temperature. Using these relations, the density, viscosity, and refractive index of α-pinene, *d*-limonene, (±)-linalool, and citral can be calculated within the studied temperature range.

A quadratic relationship was also obtained for the dependence of the logarithm of the vapor pressure against the reciprocal temperature. From this dependence, the molar enthalpy of vaporization of each compound was calculated as a function of temperature. Calculated values at 298.15 K are listed in Table 4 nd compared with available reported values.

Figure 2 shows that the dependence of the viscosity with temperature for (±)-linalool is more pronounced than for the other compounds, which is characteristic of chemical species with chemical effects in their pure state.

No modification in the enantiomer ratios of the chiral compounds due to heating to their boiling temperatures at atmospheric pressure was observed (see Table 5 in Appendix

Table 3. Parameters for Equations 1 to 3 in the (283.15 to 353.15) K Temperature Range and Corresponding Standard Deviations σ

	eq 1			eq 2				eq 3			
chemicals	A	B	σ/kg·m^{-3}	C	10^3D	10^6E	σ	F	10^3G	H	σ/mPa·s
α-pinene	1101.6	−0.832	0.1	1.6050	−0.477		0.0003	0	12.4	1389	0.02
d-limonene	1077.4	−0.793	0.1	1.6025	−0.444		0.0008	0	17	1173	0.01
(±)-linalool	1113.8	−0.860	0.3	1.6027	−0.481		0.0002	0.58	0.0017	4373	0.1
citral	1123.9	−0.803	0.1	1.3373	1.34	−2.84	0.001	0	5.3	1775	0.04

Table 4. Parameters for Equation 4 with their Standard Deviations σ and Molar Enthalpy of Vaporization ΔH_V at 298.15 K from Equation 5

	eq 4				ΔH_V/kJ·mol^{-1}	
chemicals	I	$10^{-3}J$	$10^{-5}M$	$10^2\sigma$	calcd	lit.
α-pinene	13.27	−2.78	−3.99	3.4	45.4	41.09[a]
d-limonene	13.84	−3.26	−4.02	3.6	49.5	48.1[b]
(±)-linalool	12.68	−1.94	−8.77	4.4	65.0	51.37[a]
citral	17.51	−6.32	−0.6	4.4	55.9	55.50[a]

[a] From ref 32, p. C-678. Note: The temperature for these literature values is not reported. [b] From ref 31.

A, Supporting Information[34−36]). Therefore, these chemicals are stable at the experimental conditions used in this work.

On the other hand, our VLE, density, viscosity, and refractive index results are in agreement with those available in the literature, as can be seen in Tables 1 and 2.

Supporting Information Available:

Appendix A, containing the experimental procedure used to obtain the enantiomer ratios of the chiral compounds, together with their values (Table 5) and chiral GC chromatograms (Figure S1). This material is available free of charge via the Internet at http://pubs.acs.org.

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