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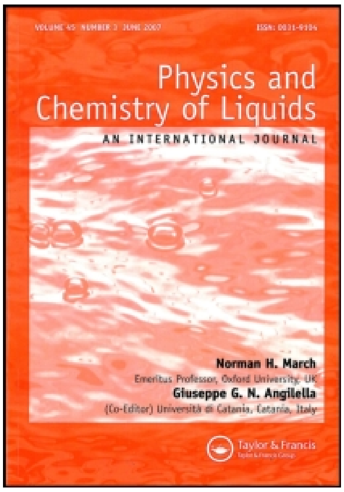

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THERMODYNAMIC PROPERTIES OF LIQUID *n*-PENTADECANE

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This paper reports experimental results concerning the isobaric heat capacity C_P of liquid *n*-pentadecane as a function of pressure (up to 100 MPa) and temperature (313.15 to 373.15 K). The measurements were performed with a modified C80 SETARAM calorimeter based on the Calvet principle. These calorimetric data, combined with density data, were used to evaluate derived thermophysical properties such as the isochoric heat capacity C_V, the isentropic compressibility κ_S and the speed of sound u in the same ranges of pressure and temperature. The pressure-temperature behaviour of these properties was then discussed as a model for simple organic liquids.

Keywords: *n*-pentadecane; Heat capacity; Speed of sound

INTRODUCTION

The thermophysical properties of simple organic liquids are of considerable interest for fundamental and industrial purposes. For example, oil and gas industry has devoted a specific attention to these properties' behaviour under the conditions of storage, *i.e.*, high pressure and high temperature; whereas from a microscopic point of view, the peculiar behaviours of the liquid isobaric heat capacity or thermal expansion can be explained on the basis of the molecular interaction.

As the experimental determination of these properties remains extremely difficult to undertake, indirect methods [1–3] were developed in which several important thermodynamic quantities are

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*Corresponding author.

evaluated from the experimental knowledge of a single property *versus* pressure and temperature. For example, the speed of sound data were confirmed very useful in view of calculating the volumetric properties. However, none of these indirect methods was able to provide reliable values of the thermal properties, isobaric C_P or isochoric C_V heat capacities because of the propagation of errors [4] with the double volume differentiation *versus* temperature that these calculations involve.

The aim of the present study consists primarily to present direct calorimetric measurements of liquid isobaric heat capacity C_P of *n*-pentadecane from the atmospheric pressure to 100 MPa over the temperature range from 313.15 to 373.15 K. Then, these measurements, combined with density data, allow one to determine derived thermophysical properties of *n*-pentadecane such as isochoric heat capacity C_V, isentropic compressibility κ_S and the speed of sound u within the same ranges of pressure and temperature. Calculation of the effects of pressure and temperature on these properties requires that the density data be expressed in an appropriate analytical form.

n-Pentadecane was chosen knowing that experimental information on high molecular weight hydrocarbons is much less documented than studies on lower molecular weight samples (*n*-hexane, *n*-heptane, or toluene..). Concerning the *n*-pentadecane, except the density, we only dispose of speed of sound data [5] in the literature which enabled us to verify the reliability of our calculated data.

EXPERIMENTAL SECTION

The basis of the calorimetric device consists of a SETARAM C80 differential heat flux calorimeter based on the Calvet principle [6]. Two wells for inserting the vessels are located in a massive regulated calorimetric block. These cells are surrounded by thermopiles composed by 162 conductive thermocouples (copper/constantan) which thermally link the vessels to the block. The two thermopiles are disposed in such a way the resulting signal is proportional to the difference in the heat exchange between the cells and the block. Indeed, this signal (μV) is converted to energy (mW) after an electric calibration by Joule effect. Data acquisition is entirely automated: the calorimeter C80 is monitored, *via* a power interface CS32, by a software which simultaneously records the following experimental data (time t, temperature T, Heat flow $S = \dot{q} = (\delta q/dt)$) during all the experiments. The temperature is indicated by a 100 Ω resistance platinum probe placed inside the block.

Specific cells have been developed in order to work at high pressures (100 MPa, [7]). The pressurisation circuit of this calorimetric arrangement (see Fig. 1), in which has been incorporated a high-pressure densimeter (Anton Paar 712P), has been detailed in previous papers [8, 9].

Specific attention has been paid to the fact the sample studied can crystallize at the ambient temperature for the high pressures. Therefore, the study of these components with low pressure of solidification

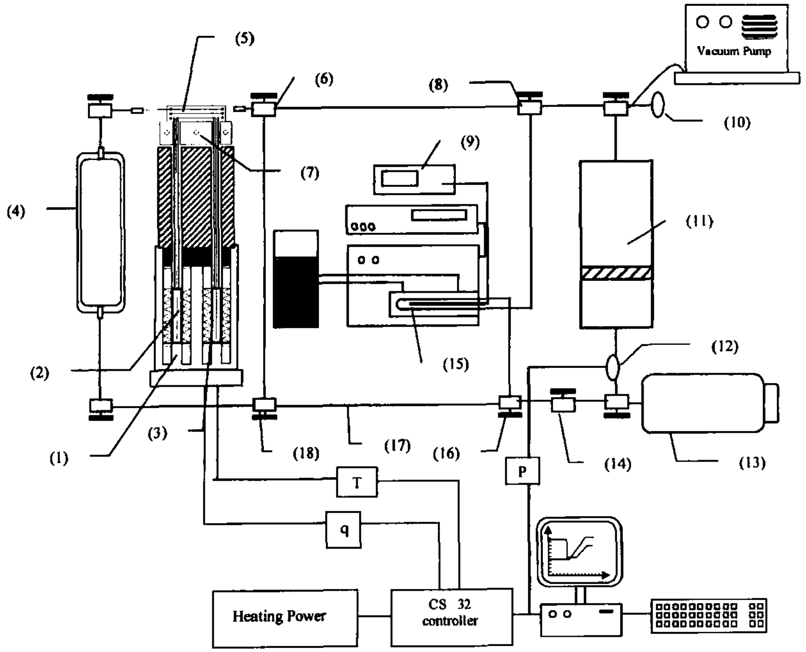

FIGURE 1 **Experimental device.** (1) Calorimeter C80, (2) Measurement vessel, (3) Reference vessel, (4) Buffer volume, (5) Capillary (i.d.: 1 mm) enclosed in the block, (6) Three-way valve, (7) Heating cartridge, (8) Three-way valve, (9) Anton Paar DMA 60, (10), (12) HBM manometer [0.1–200 MPa], (11) Separator (Top Industrie), (13) Pneumatic pump [0.1–100 MPa] (Top Industrie), (15) DMA 512P high pressure cell, (14), (16), (18) Three-way valve, (17) By-pass line capillary (i.d.: 2.4 mm).

has lead to a new modification in the design of the calorimetric device. A metallic block which is used as an auxiliary thermostat was installed on the lid of the calorimeter (this process was already used by Dordain *et al.* [10] in their calorimeter adapted for measuring heat capacities of gases). The *T*-shape connection in the cells as well as the capillary (5) linking the cell and the pressurisation system are enclosed in this block. This high pressure capillary represents a 4 cm^3 buffer volume which is regulated at the temperature of the experimentation (5 m of a capillary 1.5 mm o.d., 1 mm i.d.). The block regulation is achieved through a PID regulator (Eurotherm 2408) knowing that the set temperature is communicated by a thermocouple mounted on the top of the measurement cell. The heating is implemented by a heating cartridge (Mesurex, 100W, (7)) located in a cavity especially drilled in the block.

Moreover, this configuration enables the reduction of the thermal leaks caused by a convection phenomenon between the sample heated up in the cell and the same sample at ambient temperature in the upper tubing. The utilisation of this thermostat permitted to visibly improve the repeatability, *i.e.*, the accuracy of the measurements (0.2%).

A method generally used [11], to determine the heat capacity at constant pressure C_P consists in carrying several experiments, keeping the reference cell in the same state (*e.g.*, empty). In the first run, known as the blank, both vessels (measuring and reference) are empty: it traduces the asymmetry of the apparatus. In the second and in the third runs, the measuring vessel is respectively filled with a calibration compound and with the studied sample.

The combination of the two first experiments enables to determine the inner volume of the measuring vessel under the entire ranges of pressure and temperature which is in fact a sort of calibration-value:

$$v_{c,\mathrm{mes}} = \frac{\dot{q}(\mathrm{water}, \mathrm{empty}) - \dot{q}(\mathrm{empty}, \mathrm{empty})}{(\rho C_{pL})_{\mathrm{water}}(dT/dt)} \tag{1}$$

Then, the third experiment provides the heat capacity at constant pressure C_P corresponding to a volume:

$$C_{px} = \frac{\dot{q}(X, \mathrm{empty}) - \dot{q}(\mathrm{empty}, \mathrm{empty})}{\rho_x v_{c,\mathrm{mes}}(dT/dt)} \tag{2}$$

This value must be divided by the density in order to express it by mass unit. The heat capacity data were obtained using the scanning method [12]. The value of the heating speed (0.15 K/min) has been fixed in order to establish a quasi-stationary rating, *i.e.*, the block and the calorimeter vessels remain in thermal equilibrium. The validity of the experimental device was checked by comparison with best literature data available for *n*-hexane [3] (maximum deviation 0.6% at the higher pressures), with calibration performed with water [13].

EXPERIMENTAL RESULTS

Chemical of the best available purity was used: *n*-Pentadecane (> 99 moles percent) was procured from Aldrich. This study was carried out within the liquid state compatible pressure and temperature conditions. Thus, the ranges of temperature and pressure extended respectively from 313.15 K to 373.15 K and from 0.1 to 100 MPa, the steps adopted during the experiments being 10 K and 10 MPa. Naturally, the determinations were conducted along eleven isobars. The results of the calorimetric measurements of C_P, listed in Table I, show the same characteristic features as those of liquid hydrocarbons measured previously [14]. All the experimental curves show a decrease of C_P with increasing pressure (see Fig. 2); then, a flat minimum is reached at the higher pressures only for the lower temperatures. The significant increase of the heat capacity certainly begins at higher

TABLE I Isobaric heat capacity $C_P(\mathrm{J \cdot K^{-1} \cdot kg^{-1}})$ of *n*-pentadecane *versus* pressure and temperature

T(*K*)	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P(*MPa*)				$C_P(J \cdot K^{-1} \cdot kg^{-1})$			
0,1	2249.7	2280.7	2312.9	2345.9	2378.0	2411.7	2444.1
10	2245.7	2276.1	2307.1	2338.7	2366.0	2400.1	2435.7
20	2242.0	2274.3	2303.5	2335.4	2361.5	2395.1	2428.4
30	2240.1	2272.9	2301.7	2332.4	2358.6	2391.4	2424.1
40	2238.5	2272.4	2300.0	2331.0	2357.1	2390.1	2422.7
50	2238.6	2272.5	2299.4	2329.9	2356.4	2389.7	2421.5
60	2238.9	2272.1	2298.0	2328.8	2355.9	2388.1	2420.9
70	2238.5	2272.1	2297.8	2328.1	2355.0	2387.4	2419.1
80	2239.1	2272.5	2297.5	2328.0	2354.2	2386.1	2418.5
90	2239.2	2272.8	2297.0	2327.7	2354.0	2385.6	2418.0
100	2240.1	2272.9	2297.0	2327.4	2353.2	2384.2	2417.1

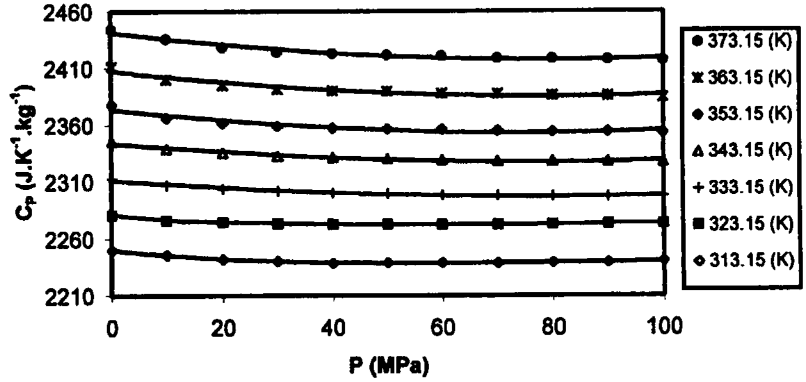

FIGURE 2 Heat capacity at constant pressure C_P ($J \cdot K^{-1} \cdot kg^{-1}$) *versus* pressure (isotherm curves) for the *n*-pentadecane.

pressures than 100 MPa. However, these results seems to display that the higher the pressure of solidification of a sample, the higher the pressure of the minimum on its C_P curve. Indeed, these minima were not observed in previous calorimetric measurements carried out on *n*-dodecane [15] and *n*-tridecane [16], in the same ranges of pressure and temperature.

Others calorimetric measurements will be undertaken on higher molecular weight hydrocarbons to confirm this striking behaviour.

CALCULATION OF DERIVED THERMOPHYSICAL PROPERTIES

Density

The choice of the form which enables to describe the volume behaviour *versus* pressure and temperature is primordial in view of evaluating the partial volume derivatives.

The modified Tait equation was already applied to traduce the volume variations *versus* pressure of liquids. This form is actually an integrated form *versus* pressure of the original Tait equation [17], (3), for the isothermal compressibility:

$$\left(\frac{\partial v}{\partial P}\right)_T = -\frac{A}{B+P} \qquad (3)$$

There appear in the literature a number of attempts to link the temperature variation of $A(T)$ and $B(T)$ and the structure of liquids, and so to represent the P, V, T behaviour of liquids. The best representation advised by several authors [18, 19] for $A(T)$ and $B(T)$ is a second-order polynomial function. Thus, the following expression (4) was finally chosen in view of obtaining reliable values of the partial volumes derivatives *versus* pressure $(\partial\rho/\partial P)_T$ and temperature $(\partial\rho/\partial T)_P$:

$$\rho(T,P) = \frac{\rho_o(T)}{\left(1 + (A_0 + A_1 * T + A_2 * T^2) * \ln\frac{P+(B_0+B_1 * T+B_2 * T^2)}{P_0+(B_0+B_1 * T+B_2 * T^2)}\right)} \qquad (4)$$

with:

$$\rho_o(T) = \rho_{01} + \rho_{02} * T + \rho_{03} * T^2 + \rho_{04} * T^3 \qquad (5)$$

where $\rho_o(T)$ is the density at the atmospheric pressure for the various temperatures.

Selected literature data of liquid *n*-pentadecane [20] density were then fitted on the basis of a modified TAIT Eq. (4) (coefficients listed in Tab. II) from the atmospheric pressure up to 100 MPa at temperatures from 303.15 to 373.15 K, with an average absolute deviation (AAD) better than 0.003%. As mentioned above, the knowledge of the density data is also required for the determination of the heat capacity C_P.

TABLE II Parameters of the modified Tait equation in the pressure (0.1 – 100 MPa) and temperature (313.15 – 373.15 (K)) ranges

	n-pentadecane
ρ_{01}	1038.677
$\rho_{02}/\mathrm{K}^{-1}$	−1.3320108
$\rho_{03}/\mathrm{K}^{-2}$	0.00203628
$\rho_{04}/\mathrm{K}^{-3}$	−2.17E-06
A_0	−0.05646387
A_1	−0.00017923
A_2	2.68E-07
B_0/MPa	346.62207
$B_1/\mathrm{MPa}\cdot\mathrm{K}^{-1}$	−1.0990849
$B_2/\mathrm{MPa}\cdot\mathrm{K}^{-2}$	0.00088129
AAD (%)	0.003
MAD (%)	0.009

AAD (%): Average Absolute Deviation between the experimental and calculated values.

MAD (%): Maximum Absolute Deviation between the experimental and calculated values.

Isochoric Heat Capacity C_V

The isochoric heat capacity at selected temperatures as a function of pressure up to 100 MPa was calculated with Eq. (6):

$$C_V = C_P - \frac{T(\partial\rho/\partial T)_P}{\rho^2(\partial\rho/\partial P)_T} \tag{6}$$

This equation was deduced from the following thermodynamic relation:

$$C_V = C_P - \frac{Tv\alpha_P}{\kappa_T} \tag{7}$$

The results of the calculations of $C_V(\mathrm{J\cdot K^{-1}\cdot kg^{-1}})$ are listed in Table III. The isochoric heat capacity (see Fig. 3) continuously increases with pressure for all the isotherms without any peculiarities in these ranges of pressure and temperature.

Isentropic Compressibilities κ_S and Speed of Sound u

The simultaneous knowledge of the isobaric and isochoric heat capacities provides the value of the specific heat ratio γ.

$$\gamma = \frac{C_P}{C_V} = \frac{\kappa_T}{\kappa_S} = \frac{(\partial\rho/\partial P)_T}{(\partial\rho/\partial P)_S} \quad \left(\frac{\partial\rho}{\partial P}\right)_S = \frac{(\partial\rho/\partial P)_T}{\gamma} \tag{8}$$

TABLE III Isochoric heat capacity $C_V(\mathrm{J\cdot K^{-1}\cdot kg^{-1}})$ of *n*-pentadecane *versus* pressure and temperature

$T(K)$	313.15	323.15	333.15	343.15	353.15	363.15	373.15
$P(MPa)$				$C_P(J\cdot K^{-1}\cdot kg^{-1})$			
0,1	1885.6	1917.4	1949.6	1982.0	2012.9	2044.9	2074.9
10	1894.3	1926.6	1958.8	1991.2	2018.6	2052.4	2087.2
20	1900.9	1935.9	1967.2	2000.6	2027.7	2061.8	2095.1
30	1907.6	1943.6	1975.0	2007.8	2035.5	2069.2	2102.3
40	1913.2	1950.7	1981.3	2014.6	2042.5	2076.6	2109.7
50	1919.4	1957.1	1987.3	2020.4	2048.6	2083.1	2115.4
60	1924.9	1962.2	1991.4	2024.8	2053.8	2087.1	2120.3
70	1929.1	1966.9	1996.0	2028.9	2057.6	2091.0	2122.9
80	1933.7	1971.4	1999.8	2032.9	2060.8	2093.5	2125.9
90	1937.4	1975.2	2002.8	2036.0	2063.9	2096.2	2128.3
100	1941.4	1978.5	2005.9	2038.8	2066.0	2097.5	2129.7

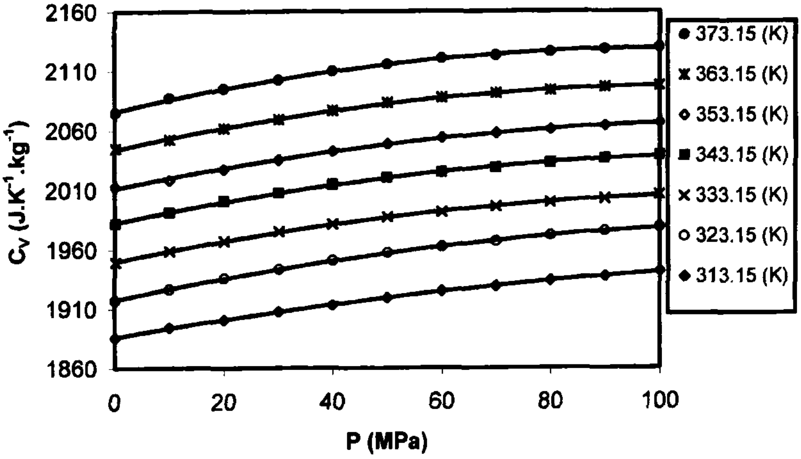

FIGURE 3 Isochoric heat capacity $C_V(\mathrm{J\cdot K^{-1}\cdot kg^{-1}})$ *versus* pressure (isotherm curves) for the *n*-pentadecane.

This specific heat ratio enables to determine $(\partial\rho/\partial p)_S$ and so the isentropic compressibility:

$$\kappa_S = \frac{1}{\rho}\left(\frac{\partial\rho}{\partial P}\right)_S. \tag{9}$$

The isentropic compressibility κ_S which is directly linked to the speed of sound u by the following relation:

$$u = \frac{1}{\rho\kappa_S} \tag{10}$$

The results of the isentropic compressibility κ_S and the speed of sound u data are respectively presented in Tables IV and V. It's interesting to note that the temperature dependence of the thermomechanical coefficient κ_S is less marked at high pressures.

Figure 4 shows the speed of sound u plotted as a function of pressure at several temperatures. In agreement with the usual observation for the liquid state, these sets of curves show regular and smooth variations corresponding to positive pressure coefficients $(\partial u/\partial P)_T$ and negative temperature coefficients $(\partial u/\partial T)_P$.

TABLE IV Isentropic compressibility κ_S (MPa^{-1}) of *n*-pentadecane *versus* pressure and temperature

T(*K*) *P*(*MPa*)	313.15	323.15	333.15	343.15 κ_S (GPa^{-1})	353.15	363.15	373.15
0,1	0.81281	0.86861	0.92913	0.99491	1.06648	1.14483	1.23061
10	0.74283	0.78955	0.83953	0.89308	0.95023	1.01211	1.07881
20	0.68382	0.72369	0.76568	0.81026	0.85712	0.90723	0.96034
30	0.63392	0.66838	0.70429	0.74201	0.78128	0.82277	0.86620
40	0.59108	0.62128	0.65237	0.68483	0.71828	0.75330	0.78956
50	0.55396	0.58066	0.60791	0.63616	0.66507	0.69509	0.72583
60	0.52143	0.54522	0.56933	0.59421	0.61950	0.64550	0.67199
70	0.49264	0.51405	0.53559	0.55768	0.57999	0.60280	0.62582
80	0.46704	0.48642	0.50579	0.52558	0.54542	0.56558	0.58584
90	0.44407	0.46173	0.47926	0.49712	0.51491	0.53289	0.55085
100	0.42338	0.43953	0.45551	0.47171	0.48776	0.50389	0.51995

TABLE V Speed of sound u ($m \cdot s^{-1}$) of *n*-pentadecane *versus* pressure and temperature

T(*K*) *P*(*MPa*)	313.15	323.15	333.15	343.15 u($m \cdot s^{-1}$)	353.15	363.15	373.15
0,1	1270.8	1234.4	1198.2	1163.0	1128.4	1094.6	1061.8
10	1328.2	1294.0	1260.2	1226.9	1194.7	1163.8	1132.9
20	1381.0	1348.1	1316.7	1285.5	1255.1	1226.1	1196.9
30	1429.8	1399.0	1368.1	1338.4	1310.1	1282.2	1254.8
40	1475.6	1445.8	1416.6	1387.7	1360.4	1334.4	1308.0
50	1518.6	1489.7	1461.6	1433.8	1407.2	1381.8	1357.3
60	1559.2	1531.1	1504.0	1477.2	1450.8	1426.8	1402.5
70	1597.7	1570.1	1543.7	1518.1	1492.9	1469.1	1445.6
80	1634.5	1607.7	1581.5	1556.8	1531.9	1509.0	1486.1
90	1669.4	1643.2	1618.1	1593.7	1570.1	1547.0	1524.7
100	1703.0	1677.5	1652.8	1628.6	1605.6	1583.3	1561.6

Experimental data of the speed of sound in *n*-pentadecane were available in the literature [5]. The comparison of our calculated values with the experimental values reveals an excellent agreement within the entire ranges of pressure and temperature; the maximum deviation (MD) observed is 0.67% whereas the average absolute deviation (AAD) is 0.22%. These deviations are random and do not increase systematically with pressure or temperature.

The excellent results of this comparative study, lead on the speed of sound, tend to show that the calculated isochoric heat capacities are reliable; since, the determination of u involves the previous knowledge of the isochoric heat capacity C_V.

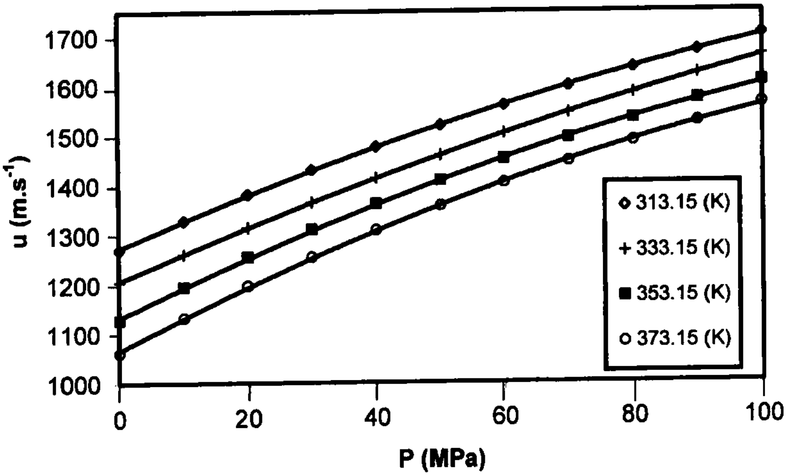

FIGURE 4 Speed of sound u (m · s^{-1}) *versus* pressure (isotherm curves) for the *n*-pentadecane.

DISCUSSION

Direct measurements of isobaric heat capacity and the simultaneous knowledge of density permit accurate calculations of related thermodynamic properties. A qualitative analysis of the pressure-temperature behaviour of these quantities reveals at least two striking behaviour which could be used in view establishing specific models for compressed simple liquids. First, the influence of pressure on the isobaric heat capacity C_P. In the lower isotherms, the heat capacity first decreases, demonstrates a flat minimum and then certainly increases; the pressure of these minima decreases as the pressure of solidification increases. It seems to be general rule, at least for simple liquids.

The location of these minima in the isotherms of the isobaric heat capacity, as well as the intersection point of isotherms of the isobaric thermal expansion α_p (called cross-over point) constitutes a primordial thermodynamic information which in turn could serves as a consistency test for the predictive models.

In the case of our study, C_V presents a monotonic behaviour *versus* pressure $(\partial C_V/\partial P) > 0$ and temperature $(\partial C_V/\partial T) > 0$.

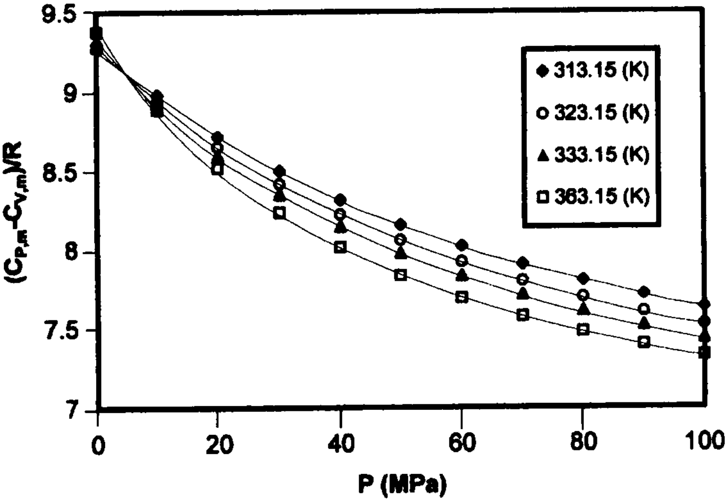

FIGURE 5 Isotherms of the difference between isobaric and isochoric molar heat capacities of *n*-pentadecane.

In several studies [3,4], it was reported that C_V does not always increase with pressure (isotherms curves); for *n*-hexane [3] a minimum appears above 403 K, at low pressures (15 – 30 MPa). The temperature range of the present investigation is not certainly enough extended to confirm the observation of this singularity.

However, an other interesting feature of these experimental results is observed for the quantity $C_P - C_V$ (Fig. 5). Indeed, all the isotherms cross at approximately 7.5 MPa. At this pressure, the value of $C_{P,m} - C_{V,m}$ is $9R$. A similar behaviour was already mentioned for *n*-hexane [3]. Molecular interpretation of this phenomenon awaits further studies.

CONCLUSIONS

The simultaneous knowledge of heat capacity and density in wide ranges of pressure and temperature has allowed a self-consistent set of thermodynamic quantities for *n*-pentadecane to be determined. First of all, this study underlines the decisive contribution the knowledge of the experimental heat capacities can make. Indeed, these thermal properties cannot be evaluated from the volume (or speed of sound) data because of uncertainties in the double volume derivatives [4]. Therefore, scanning calorimetry is a powerful experimental technique which supplies important information in characterizing the liquid state.

The behaviour of these several thermodynamic quantities of liquid *n*-pentadecane as functions of pressure and temperature could be used as a reference to confirm or develop models for liquids without strong molecular interactions.

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