Journal of Thermal Analysis and Calorimetry, Vol. 62 (2000) 621–632

THERMOPHYSICAL PROPERTIES OF *n*-TRIDECANE FROM 313.15 TO 373.15 K AND UP TO 100 MPa FROM HEAT CAPACITY AND DENSITY DATA

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(Received January 4, 2000; in revised form May 2, 2000)

Abstract

Isobaric heat capacities of liquid *n*-tridecane were measured at temperatures from 313.15 to 373.15 K and at pressures up to 100 MPa using a calorimetric device based on a Calvet calorimeter (Setaram C80). These experimental data combined with the additional knowledge of density data were used to calculate the following properties at pressures up to 100 MPa: isochoric heat capacity, isentropic compressibility and ultrasound velocity.

Keywords: density, isentropic compressibility, isobaric heat capacity, isothermal compressibility, *n*-tridecane, pressure, ultrasound velocity

Introduction

Accurate thermodynamic properties of pure substances are of considerable interest for both theoretical and industrial purposes. For example, pressure and temperature dependence of the thermophysical properties of liquid hydrocarbons are of importance because of their wide use in oil and gas industry. As these derivatives at higher pressures are rather difficult to obtain, an indirect method is often favoured in which several important thermodynamic quantities are calculated from some properties precisely measured experimentally. Thermophysical properties have been evaluated by derivations of (P,V,T) data. However, such methods [1, 2] offer a limited accuracy, in particular for the second-order derivatives like heat capacities C_P and C_V. Improvements were obtained by combining (P,V,T) data and measured speed of sound c; a method originally developed by Davis and Gordon [3] enables to obtain reliable values for the density ρ and the first order derivatives (isentropic κ_S and isothermal κ_T compressibilities) [4, 5]. A more consistent method has been based on the direct determination of the isobaric thermal expansion $\alpha(P,T)$ [6, 7]. Using an appropriate

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1418–2874/2000/ $ 5.00

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Kluwer Academic Publishers, Dordrecht

functional form [8] to fit $\alpha(P,T)$, the derivation and the integration of this analytical form provide accurate values for the properties evaluated.

The aim of the present study is to report a set of thermodynamic properties (C_V, κ_S and c) of the liquid phase obtained from direct calorimetric measurements of the isobaric heat capacity C_P as a function of pressure and temperature. This article focuses on *n*-tridecane ($C_{13}H_{28}$) for which C_P data are available in literature only at the atmospheric pressure. Calorimetric measurements were performed over the temperature range from 313.15 to 373.15 K under pressures from 0.1 up to 100 MPa. Calculation of pressure effects requires the volumetric data, also needed for the heat capacity determination, to be selected in the literature and fitted by appropriate analytical equations. This computational method will be detailed after a brief description of the calorimetric device.

Experimental device

The calorimetric device, which has been previously described [9, 10], consists in using a Setaram C80 differential heat-flux calorimeter (sensitivity 10 μW) based on the Calvet principle. The apparatus is entirely automated: the calorimeter C80 is monitored, via a power interface CS32, by a software which simultaneously requires the acquisition of experimental data (time t, temperature T, heat-flow $\dot{q}$) during all the ex-

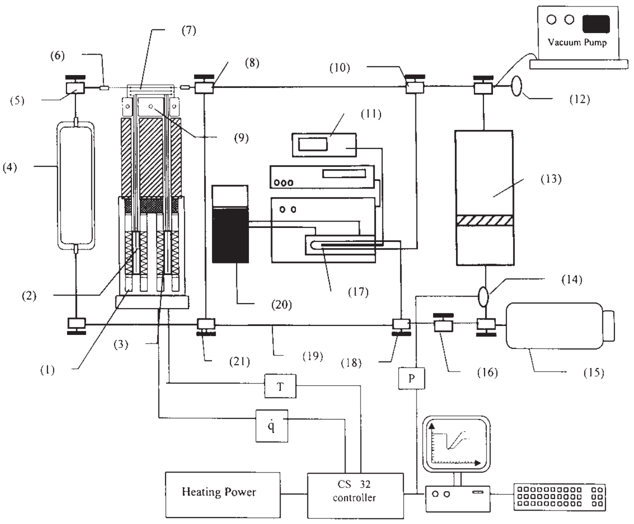

Fig. 1 Experimental device. 1 – calorimeter C80; 2 – measurement vessel; 3 – reference vessel; 4 – buffer volume; 5 – two-way valve; 6 – reductor 1/4–1/16; 7 – capillary (i.d.: 1 mm) enclosed in the block; 8 – two-way valve; 9 – heating cartridge; 10 – two-way valve; 11 – Anton Paar DMA 60; 12, 14 – HBM manometers (0.1–200 MPa); 13 – separator (Top Industrie); 15 – pneumatic pump (0.1–100 MPa) (Top Industrie); 17 – DMA 512P high pressure cell; 16, 18 – three-way valves; 19 – by-pass line capillary (i.d.: 2.4 mm); 20 – temperature regulation; 21 – three-way valve

periments. Specific cells have been developed in order to work at high pressures (100 MPa) [9]. A general view of the calorimetric arrangement, which also contains a high-pressure densimeter (Anton Paar 712P), is presented in Fig. 1.

It is worth noting that a new modification was achieved in order to reduce the thermal leaks caused by convection phenomena between the liquid at the working temperature in the cell and the same liquid at ambient temperature in the upper tubing. For this purpose, a thermostated block was placed in the upper part of the calorimeter. The T-shape connection in the cells as well as the capillary Fig. 1 (7) linking the cell and the pressurisation system are enclosed in this block. The regulation is achieved through a PID regulator knowing that the set temperature is established by a thermocouple mounted on the top of the measuring cell. The heating is implemented by a heating cartridge (Mesurex, 100 W, Fig. 1 (9)) located in a cavity especially drilled in the block. With this arrangement, the temperature of the liquid in the upper tubing is always identical to the temperature in the cell. This thermostat has permitted to noticeably improve the repeatability, i.e. the accuracy of the measurements.

The heat capacity of the sample is determined in three experiments carried out under the same conditions: first, both vessels (measuring and reference) are empty and the quantity $\dot{q}$ (empty, empty) is recorded; second, the measuring vessel is filled with water and the quantity $\dot{q}$ (water, empty) is recorded. The combination of these two experiments enables to determine the inner volume $v_{\mathrm{c,meas}}$ (P,T) – which is actually a parameter of calibration – of the measuring vessel under the entire ranges of pressure and temperature. Water was chosen as a reference liquid since heat capacity is well established under the entire range of pressure [11]:

$$v_{\mathrm{c,meas}}(P,T)=\frac{\dot{q}(\text{water, empty})-\dot{q}(\text{empty, empty})}{(\rho C_{\mathrm{PL}})_{\mathrm{water}}\dfrac{\mathrm{d}T}{\mathrm{d}t}} \tag{1}$$

with $\mathrm{d}T/\mathrm{d}t$: heating rate fixed 0.15 K min^{-1}.

Then, in a third experiment, with the measuring vessel filled with the sample X the quantity $\dot{q}$ (X, empty) is obtained to yield the heat capacity at constant pressure C_{P}:

$$C_{\mathrm{PX}}=\frac{\dot{q}(\text{X, empty})-\dot{q}(\text{empty, empty})}{\rho_{\mathrm{X}} v_{\mathrm{c,meas}}\dfrac{\mathrm{d}T}{\mathrm{d}t}} \tag{2}$$

This procedure leads to the volumic heat capacity and one needs the density of the liquid *vs*. pressure and temperature in order to obtain the molar C_{P}. Density data were selected in the literature and correlated on the basis of a modified Tait [12] equation valid in the entire range of pressure and temperature.

The heat capacity data were obtained by using the scanning (or continuous) method. It means that the value measured is the signal amplitude directly proportional to the power requested for the temperature increase at a given rate. The value of the heating speed (0.15 K min^{-1}) was adjusted in order to establish a quasi-stationary rating, i.e. the block and the calorimeter vessels remain in thermal equilibrium. The repeatability of the measurements with the scanning is identical to that of the stepwise method, which is more time consuming [13]. The reliability of the measurements was tested by measuring the heat capacity of liquid *n*-hexane, with calibration performed with water. Comparison with the best literature data [6] shows maximum deviation of 0.6% at higher pressures.

Results

n-Tridecane was supplied by Sigma with a purity higher than 99%. Experimentally determined heat capacities of *n*-tridecane at temperatures from 313.15 to 373.15 K and at pressures from 0.1 to 100 MPa are summarised in Table 1.

Table 1 *n*-Tridecane heat capacity C_P as a function of pressure and temperature

T/K	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P/MPa	C_P/J kg^{-1} K^{-1}						
0.1	2249.7	2284.3	2312.9	2345.9	2378.0	2411.7	2444.1
10	2245.7	2280.7	2307.1	2338.7	2366.0	2400.1	2435.7
20	2242.0	2276.1	2305.5	2335.4	2361.5	2395.1	2428.4
30	2240.1	2274.3	2301.7	2332.4	2358.6	2391.4	2424.1
40	2238.5	2272.9	2300.0	2331.1	2357.1	2390.1	2422.7
50	2238.6	2272.4	2299.4	2329.9	2356.4	2389.7	2421.5
60	2238.9	2272.5	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

At the atmospheric pressure, we observe excellent agreement between the literature data [14, 15] (when smoothed and extrapolated) and our data. The results of calorimetric measurements of C_P in these (P,T) ranges show a classical behaviour previously observed [16] for the liquid state: isobaric heat capacity C_P increases quasi-linearly with temperature and decreases with pressure. At high pressures and low temperatures a plateau is reached, which tends to show that the minima of heat capacity *vs*. pressure, for *n*-tridecane, are close to 100 MPa (Fig. 2). Other calorimetric measurements of C_P on higher molecular mass components will be undertaken to confirm this peculiarity. Because in the series of hydrocarbons the pressure for which the minimum is observed tends to become less as the molecular mass increases [17]. And, 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 (called cross-over point) constitute primordial thermodynamic information which, in turn, serve as a consistency test for the predictive models.

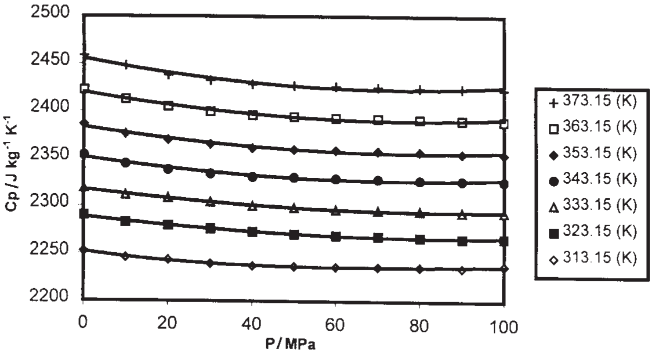

Fig. 2 *n*-Tridecane isobaric heat capacity C_P (J kg^{-1} K^{-1}) *vs*. pressure (isothermal curves)

Calculation of derived thermophysical properties

Outline of the computational method

The starting point of this procedure is the simultaneous knowledge of isobaric heat capacity C_P and density ρ under extended ranges of pressure and temperature. Through the thermodynamic relationships, we determine several thermophysical properties (C_V, κ_S, c).

The isochoric heat capacity C_V was computed according to the following relation:

$$C_V=C_P-\frac{Tv\alpha_P^2}{\kappa_T} \tag{3}$$

where v, the specific volume (v=1/ρ, ρ being the density) and C_P are known experimentally whereas $\alpha_P=1/v(\partial v/\partial T)_P$ and $\kappa_T=-1/v(\partial v/\partial p)_T$ directly linked to the first derivatives and are evaluated by derivations of the volume. These derivations will induce the accuracy of the calculated properties. It is worth noting that the term involving the first derivatives represents only 20% of the C_V value. The method applied to calculate these derivatives will be detailed hereafter.

The ratio:

$$\gamma=\frac{C_P}{C_V}=\frac{\kappa_T}{\kappa_S} \tag{4}$$

enables to determine the isentropic compressibility κ_S; then, the speed of sound c can be evaluated by the relation

$$c=\left(\frac{1}{\rho\kappa_S}\right)^{\frac{1}{2}} \tag{5}$$

Calculation of the first derivatives $(\partial v/\partial T)_P$ *and* $(\partial v/\partial p)_T$*: choice of an equation*

From the data volume as function of P and T, we can deduce by derivations *vs*. pressure and temperature the values of the isobaric thermal expansion α_p and the isothermal compressibility κ_T. Instead of performing numeric calculations, we prefer to represent the experimental (P,ρ,T) data with an equation of state which will be analytically derived. Thus, the accuracy on the calculations of these first (volume) derivatives *vs*. pressure and temperature will depend on the functional form used to describe the volume behaviour over the entire ranges of pressure and temperature.

The first step of this work consisted of testing two well-known liquid state equations (Tait [12]: Eq. (6); Secant Bulk Modulus [18]: Eq. (7)). Indeed, these equations satisfactorily represent, from a phenomenological point of view, the volume behaviour *vs*. pressure. Consequently, the derived form of this equation *vs*. pressure provides reliable values of $(\partial v/\partial p)_T$.

$$\frac{v-v_{\mathrm{ref}}}{v_{\mathrm{ref}}}=J\ln\left[\frac{L+p}{L+p_0}\right] \tag{6}$$

$$K=\frac{V_0P}{V_0-V}=A+BP+CP^2 \tag{7}$$

Several authors [19, 20] have modified these equations by assuming the coefficients J and L in Eq. (6) and A, B and C in Eq. (7) are functions of temperature in order to represent the volume behaviour not only with pressure but also with temperature. In order to represent the variation of J, L and A, B and C with temperature in an empirical way, we choose a linear form, a second order polynomial form and a three order form for each parameter of the two equations.

The tests for evaluating the reliability of these analytical forms is first the capability to interpolate the volume over the entire ranges of pressure and temperature; an other criterion is the faculty to restitute the peculiar pressure-temperature behaviour of the isobaric thermal expansion (the single crossing point of isotherms) from the calculation of the analytical derived $(\partial V/\partial T)_P$. The accuracy of this analytical procedure was checked by comparing the calculated thermal expansion for *n*-hexane – previously suggested as reference sample [21–23] with reliable experimental data published by Randzio *et al*. [6]. This comparative study has shown that the best results are provided by the modified Tait equation with a second-order polynomial form which shows an absolute average deviation (*AAD*) of 3.5% between experimental and calculated isobaric thermal expansion data α_P; the maximal deviation observed between the fitted and the experimental volumes is always inferior to 0.05%.

Concerning the coefficient of isothermal compressibility κ_T, the agreement is good (*AAD* always inferior to 0.6%) with the Randzio's data in the entire ranges (P,T) investigated.

In view of the incapability of the Tait and the Secant Bulk Modulus equations to well determine the isobaric thermal expansion, we performed test with simple second order polynomial functions linking the three variables (P,V,T) and permuting the explicit variable T, P or V. Tests were also performed on *n*-hexane.

$$V(T,P)=A(T)+B(T)P+C(T)P^2 \tag{8}$$

$$T(V,P)=A(P)+B(P)V+C(P)V^2 \tag{9}$$

$$P(T,V)=A(T)+B(T)V+C(T)V^2 \tag{10}$$

Surprisingly enough, the original expression Eq. (9) provides good results not only for the volume fitted (AAD=0.095%) but also for the determination of thermal expansivity α_P (AAD=3.2%) and isothermal compressibility κ_T (AAD=0.3%). Furthermore, even if the accuracy of the α_P calculated is not excellent, we observed a good qualitative (P,T) behaviour with the appearance of a cross-over-point.

We first calculate $\partial T/\partial V=B(P)+2(P)V=1/(\partial V/\partial T)$ which allows to deduce $\partial V/\partial T$. In order to calculate κ_T we determine $(\partial T/\partial P)_V$, and knowing $(\partial T/\partial V)_P$ we can obtain $(\partial V/\partial P)_T$.

Finally, we selected this polynomial form for its simplicity, the small number of empirical coefficients and the reliable values obtained therefrom. The coefficients of these equations were fitted by a least squares method.

Derived thermophysical properties of n-tridecane

The computational method outlined in the preceding section was used for deriving, as a function of pressure, various thermophysical (C_V, κ_S and c) of *n*-tridecane from the values of its isobaric heat capacity C_P measured experimentally.

Table 2 Coefficients for the fitting equation $T(\rho,P)=A(P)+B(P)\rho+C(P)\rho^2$ with $A(P)=a_1+a_2P+a_3P^2$; $B(P)=b_1+b_2P+b_3P^2$; $C(P)=c_1+c_2P+c_3P^2$

Coefficient	*n*-Hexane	*n*-Tridecane
a_1/K	681.285703	1199.97958
b_1/K g^{-1} cm^3	–0.11589721	0.099917938
c_1/K g^{-2} cm^6	–0.00071664	0.00026435
a_2/K MPa^{-1}	21.85597	21.6082924
b_2/K MPa^{-1} g^{-1} cm^3	–0.05406234	–0.0470056
c_2/K MPa^{-1} g^{-3} cm^6	3.439E–5	2.6091E–5
a_3/K MPa^{-2}	–0.02161604	–0.01826924
b_3/K MPa^{-2} g^{-1} cm^3	6.8586E–5	5.012E–5
c_3/K MPa^{-2} cm^6	–5.2842E–2	3.4345E–8
T_{min}/K	303.15	303.15
T_{max}/K	383.15	383.15
P_{min}/MPa	0.1	0.1
P_{max}/MPa	150	150

First, experimental density (directly linked to the specific volume $\rho=1/V$) of *n*-tridecane [24] were fitted with the polynomial form $T(V,P)$ (coefficients reported in Table 2) with an *AAD* better than 0.09% in the ranges of pressure (0.1–110 MPa) and temperature (303.15–383.15 K). This function was derived *vs*. pressure and temperature, therefore, the respective ranges (P,T) for the quantities deduced were truncated of their extrema values, thus leading to the working following domain: (10–100 MPa) and (313.15–373.15 K).

Isochoric heat capacity C_V

The isochoric heat capacity C_V, at selected pressures and temperatures, was calculated with Eq. (3). The results of these calculations are recorded in Table 3. As illustrated by Fig. 3, this property continuously increases with pressure for all the temperatures. There is no peculiarities (minimum) in the range covered by our study.

Table 3 *n*-Tridecane heat capacity at constant volume C_V

T/K	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P/MPa	C_V/J kg^{-1} K^{-1}						
10	1885.09	1922.23	1951.23	1985.17	2017.16	2054.41	2090.55
20	1892.73	1929.45	1958.50	1989.66	2021.83	2057.84	2091.57
30	1897.74	1935.21	1963.84	1994.85	2026.71	2061.73	2096.00
40	1903.05	1939.60	1967.44	1999.59	2030.61	2066.14	2100.06
50	1909.38	1944.15	1972.87	2006.08	2036.59	2071.76	2105.81
60	1915.53	1949.18	1977.21	2011.26	2042.12	2077.01	2111.38
70	1920.91	1954.07	1981.90	2015.99	2046.81	2081.75	2116.48
80	1925.90	1957.99	1986.54	2020.25	2051.45	2086.16	2120.99
90	1929.70	1961.61	1990.10	2024.60	2054.23	2090.33	2124.97
100	1936.81	1966.70	1994.56	2028.59	2058.19	2094.01	2128.71

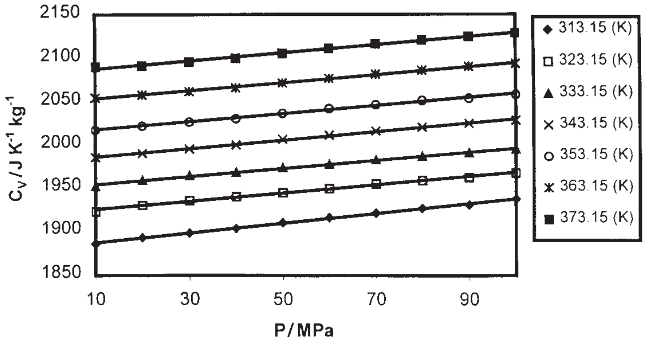

Fig. 3 *n*-Tridecane heat capacity at constant volume C_V (J kg^{-1} K^{-1}) *vs*. pressure (isothermal curves)

Isentropic compressibility κ_S *and ultrasound velocity c*

Isentropic compressibility κ_S and the speed of sound c values are respectively obtained from Eq. (2) and (3) knowing the heat capacity ratio and the isothermal compressibility κ_T (Table 4). The results are recorded in Tables 5 and 6. A representation of selected isotherms of the speed of sound is given in Fig. 4. In agreement with the usual observation for the liquid state, these sets of curves show regular and smooth variations corresponding to positive pressure coefficients $(\partial c/\partial P)_T$ and negative temperature coefficients $(\partial c/\partial T)_P$.

Table 4 *n*-Tridecane isothermal compressibility κ_T

T/K	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P/MPa	κ_T/GPa^{-1}						
10	0.9473	1.0062	1.0683	1.1337	1.2026	1.2748	1.3505
20	0.8583	0.9069	0.9578	1.0110	1.0666	1.1245	1.1847
30	0.7865	0.8274	0.8701	0.9144	0.9605	1.0082	1.0575
40	0.7274	0.7624	0.7988	0.8364	0.8754	0.9155	0.9569
50	0.6778	0.7082	0.7396	0.7721	0.8056	0.8400	0.8753
60	0.6354	0.6622	0.6898	0.7182	0.7474	0.7773	0.8078
70	0.5988	0.6226	0.6471	0.6722	0.6980	0.7243	0.7511
80	0.5668	0.5882	0.6101	0.6325	0.6555	0.6789	0.7027
90	0.5385	0.5578	0.5776	0.5979	0.6185	0.6395	0.6609
100	0.5131	0.5308	0.5489	0.5673	0.5860	0.6050	0.6243

Table 5 *n*-Tridecane isentropic compressibility κ_S

T/K	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P/MPa	κ_S/GPa^{-1}						
10	0.7949	0.8472	0.9018	0.9599	1.0210	1.0856	1.1534
20	0.7240	0.7675	0.8127	0.8602	0.9100	0.9623	1.0165
30	0.6664	0.7033	0.7415	0.7814	0.8230	0.8663	0.9111
40	0.6186	0.6505	0.6833	0.7175	0.7528	0.7896	0.8275
50	0.5785	0.6063	0.6349	0.6646	0.6952	0.7269	0.7594
60	0.5441	0.5687	0.5939	0.6201	0.6469	0.6746	0.7030
70	0.5143	0.5363	0.5587	0.5820	0.6058	0.6303	0.6554
80	0.4881	0.5079	0.5281	0.5490	0.5704	0.5922	0.6146
90	0.4648	0.4828	0.5012	0.5202	0.5394	0.5592	0.5793
100	0.4441	0.4605	0.4773	0.4946	0.5121	0.5302	0.5484

Table 6 *n*-Tridecane ultrasound velocity *c*

T/K	313.15	323.15	333.15	343.15	353.15	363.15	373.15
P/MPa				c/m s^{-1}			
10	1295.59	1260.75	1227.57	1195.37	1164.50	1134.64	1106.05
20	1351.49	1318.29	1286.63	1256.02	1226.50	1197.98	1170.72
30	1402.98	1371.16	1340.88	1311.53	1283.23	1255.87	1229.64
40	1450.59	1420.09	1391.05	1362.77	1335.56	1309.17	1283.88
50	1494.84	1465.58	1437.56	1410.24	1384.01	1358.52	1334.05
60	1536.26	1508.07	1481.04	1454.58	1429.18	1404.50	1380.76
70	1575.29	1548.02	1521.85	1496.19	1471.55	1447.57	1424.49
80	1612.28	1585.83	1560.37	1535.43	1511.43	1488.10	1465.60
90	1647.59	1621.81	1596.97	1572.60	1549.26	1526.41	1504.44
100	1681.25	1656.13	1631.86	1608.02	1585.18	1562.82	1541.29

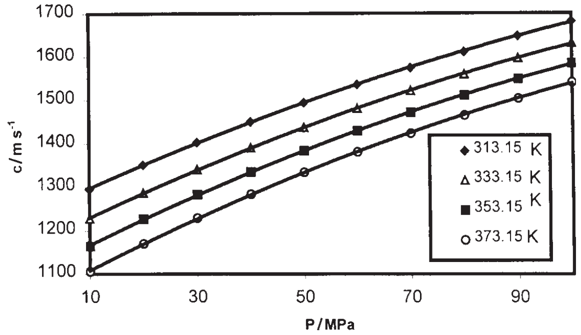

Fig. 4 *n*-Tridecane ultrasound velocity c(m s^{-1}) *vs*. pressure (isothermal curves)

Thermodynamic consistency

To validate the analytical method developed to generate some derived properties, the results obtained in the present work on *n*-tridecane (κ_S and c) were compared with the experimental values provided by Daridon *et al.* [25] in the common range of the two investigations.

Up to now, direct or indirect determinations of isochoric heat capacity C_V of *n*-tridecane were not available in the literature. The deviations obtained in the comparative study performed on the isentropic compressibility are –0.07 and 0.2% for the average and the absolute average deviations, respectively. These deviations are divided by two in sound speed (respectively –0.03 and 0.07% for *AD* and *AAD*), which is explained by the fact that c is related to κ_S through Eq. (3). Thus *AAD*(%) on c, $(\sigma c/c)$, differs little from 1/2 $(\sigma\kappa_S/\kappa_S)$ since $(\sigma\rho/\rho)$ is lower. This excellent agreement observed between the two sets of data, those deduced from direct measurements, on the one hand, and those resulting from our present measurements of heat capacity on the other hand, underlines the thermodynamic consistency of the approach developed to generate derived thermophysical properties. Moreover, this confirms that the values obtained for the isochoric heat capacity are reliable since the calculation of the speed of sound involves the previous knowledge of C_V.

Conclusions

Heat capacity measurements carried out on *n*-tridecane between 0.1 and 100 MPa and 303.15 and 373.15 K were used to calculate the isochoric heat capacity C_V as well as the isentropic compressibility κ_S and the ultrasound velocity c as a function of pressure. The very satisfactory agreement of the speed of sound c deduced from the heat capacity measurements with the speed of sound directly measured confirm the good quality and consistency of both the experimental measurements and of the correlating relationship. Above all, this study emphasises the decisive contribution brought by the heat capacity data experimentally measured.

Moreover, the data given in this paper make *n*-tridecane a useful substance for calibration or test of high pressure instruments and associated correlation methods.

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