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Effect of Dissolved Gases on the Densities of Hydrocarbons

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Measurements have been made, using a vibrating tube densitometer, of the effects of dissolved air, nitrogen, oxygen, hydrogen, methane, and carbon dioxide on the densities of liquid hydrocarbons at 298.15 K and a total pressure of 101 060 Pa. The hydrocarbons were heptane, octane, nonane, decane, dodecane, tetradecane, hexadecane, cyclohexane, methylcyclohexane, and methylbenzene (toluene). Saturation of the liquids with gases other than carbon dioxide decreases the density by amounts ranging from about 0.004% for oxygen in hexadecane up to about 0.1% for methane in heptane. Saturation with carbon dioxide increases the densities by a maximum of almost 0.1%. For all gases, values of $|\Delta\rho|$ decrease with increasing carbon number in an approximately linear manner in the alkane series. Partial molar volumes of the gaseous solutes have been calculated by combining the density results with literature gas solubility data.

Introduction

During an earlier investigation (Ashcroft et al., 1992) of the shrinkage that occurs when crude oils are spiked with light alkanes, it became apparent that there was incomplete information available in the literature on the effect of dissolved gases on the densities of pure hydrocarbons. The magnitude of the effect is important for the measurement of absolute densities, and to a lesser extent excess volumes. The measurements reported in this paper were undertaken to determine the change in density when a liquid hydrocarbon is saturated with a gas at room temperature and pressure. The effect on density of dissolved gases, particularly air, which may be present in hydrocarbons to the extent of about 0.1 mol %, may partially account for the discrepancies in the literature values for the densities of hydrocarbons. In the course of this work, another research group published two papers (Cibulka and Heintz; 1995, Izak et al., 1995) giving data on the effect of dissolved air-component gases on the density of alkanes and alkanols. There is some overlap between the two sets of data, with generally reasonable agreement.

The measured densities were combined with literature gas solubility data to calculate the partial molar volumes of the gases in solution in a manner similar to that first described by Moore et al. (1982). A literature review of partial molar volumes (Handa and Benson, 1982) revealed a paucity of information on gases dissolved in hydrocarbons. These quantities are useful in a number of ways, for example in the calculation of high-pressure gas solubility with the Krichevsky−Kasarnovsky and Krichevsky−Ilinskaya equations (Prausnitz, 1969).

Experimental Section

Density Measurements. To determine the effect on densities of dissolved gases and to determine partial molar volumes to reasonable accuracy by this method, the precision of the density measurements needs to be rather high. In this work an Anton Paar DMA 602 vibrating tube densitometer was used for the density measurements (Ashcroft et al., 1990). It was operated in conjunction with a frequency counter (Racal Instruments, Type 1991) and a precision thermostat designed in these laboratories. For water the coefficient of thermal expansion, $V^{-1}(\partial V/\partial T)_P$, where V is volume, T temperature, and P pressure, is about 2×10^{-4} K^{-1}, but for hydrocarbons values are about five times larger. To achieve a precision in density of ±0.001 kg m^{-3}, temperature control to ±0.001 K is required. A precision thermostat was designed for this purpose, and temperature was measured by a thermistor calibrated against a short-range (6 K) mercury thermometer certificated on the IPTS-68 scale by the National Physical Laboratory, U.K.

Table 1. Densities ρ and Na d-line Refractive Indices n^{d}_{298} of Air-Saturated Solvents at T = 298.15 K and P = Atmospheric Pressure in Comparison with Some Literature Values

	measured		literature		
solvent	ρ/kg m^{-3}	n^{d}_{298}	ρ/kg m^{-3}	n^{d}_{298}	ref
heptane	679.67	1.385 23	679.50	1.385 13	*a*
			679.49		*b*
octane	698.65	1.395 16	698.54	1.395 12	*a*
			698.64		*b*
nonane	713.82	1.403 26	713.75	1.403 11	*a*
			713.67		*b*
decane	726.28	1.409 68	726.14	1.409 67	*a*
			726.14		*b*
dodecane	745.35	1.419 50	745.18	1.419 49	*a*
			745.39		*b*
tetradecane	759.23	1.426 77	759.20	1.426 85	*a*
			759.53		*b*
hexadecane	769.89	1.432 53	769.94	1.432 50	*a*
			770.01		*b*
cyclohexane	773.91	1.423 50	773.87	1.423 53	*a*
			773.86		*c*
methylcyclohexane	765.04	1.420 70	765.02	1.420 58	*a*
			765.00		*c*
methylbenzene	862.32	1.494 18	862.20	1.494 11	*a*

[a] TRC Thermodynamic Tables (1996). [b] Landolt-Börnstein (1996). [c] Landolt-Börnstein (1997).

Uncertainties. Although the apparatus was capable of detecting density changes of less than 0.001 kg m^{-3}, an uncertainty of ±0.01 kg m^{-3} is considered realistic for the precision of the density measurements owing to uncertainties in the temperature and the densities of the calibration standards, water (Wagenbreth and Blanke, 1968) and dry air (Strobridge, 1962). However, the measurement of small differences in density is relatively insensitive to the accuracy of the calibration data. Replicate measurements were made of the density differences between saturated and degassed samples in order to calculate the precision (twice the standard deviation of the mean), which was

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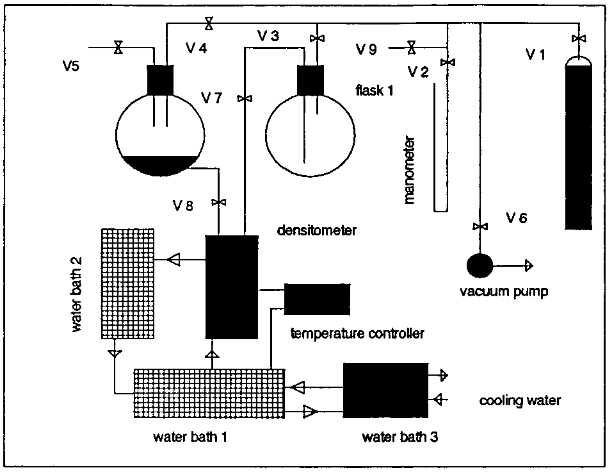

Figure 1. Apparatus for measurement of the densities of degassed and saturated solvents.

found to be in the range ±0.0058 kg m^{-3} to ±0.0070 kg m^{-3}. Sample viscosities greater than about 1 mPa s influence the density readings from vibrating tube densitometers (Ashcroft et al., 1990), but density differences are likely to be largely unaffected. Published gas solubility data are disappointingly scattered, and it proved difficult to assign uncertainties to the values. This point is discussed later in this paper in connection with the uncertainties to be attached to the calculated partial molar volumes.

Materials. Analytical grade heptane, cyclohexane, and methylbenzene (toluene), laboratory grade decane, dodecane, and methylcyclohexane from BDH Ltd, and other hydrocarbons from Sigma were further purified either by distillation in a 10-plate Oldershaw fractionation column for those with a normal boiling temperature of less than 120 °C or by fractional crystallization. The level of volatile impurities was checked by gas chromatography using a Hewlett-Packard 5890 GC equipped with a TCD detector and a Porapak Q-S column or, for the higher boiling hydrocarbons, a column of OV101 on Chromosorb W. For all solvents the GC method showed impurity levels of less than 0.02 mass %. Refractive indices at 298.15 K for the sodium d-line were measured using an Abbé refractometer (Bellingham and Stanley type 60/ED) with a manufacturer's claimed accuracy of ±0.000 04. Our measured densities and refractive indices are compared with literature values for air-saturated materials in Table 1. The gases, supplied by BOC Ltd and Air Products PLC, had the following manufacturers' purity specifications: nitrogen 99.998 mol %; carbon dioxide 99.98 mol %; hydrogen 99.992%; methane 99.995%. Dry air and BDH AnalaR grade water were used for the calibration of the densitometer. The effect of dissolved air on the density of water is reported to be negligibly small at 298 K (Wagenbreth and Blanke, 1968), and this was confirmed experimentally.

Degassing and Saturating of the Liquids. The apparatus used in the present work, shown in Figure 1, was designed to ensure complete degassing and saturating while minimizing partial distillation of the solvent and eliminating contact of the solvent with any soluble material other than the gas. Its design took into account the principles established by previous work in this area, particularly the apparatus developed by Battino et al. (1971) and by Gilmour et al. (1967). Glass and PTFE materials were used in all parts of the apparatus in contact with gas or solvent, thereby avoiding the use of mercury. The samples (150 cm^3 to 180 cm^3) were degassed under vacuum with vigorous stirring in flask 1 and then transferred to flask 2 where further cycles of heating and cooling under vacuum were carried out. During transfer, the liquid entered the flask as a thin film on the walls to aid degassing. The liquid was transferred to the density measuring cell by gravity, and, by closing valves 7 and 8, the degassed liquid was trapped in the measuring cell under its own vapor pressure. The measuring cell was then pressurized to that used in the calibrations (10 106 ± 10 Pa) by admitting one of the gases to flask 1 at a point

Table 2. Density Differences[a] $\Delta\rho = \rho_{deg} - \rho_{sat}$ between Degassed and Saturated Liquids at T = 298.15 K and Total P = 101 060 Pa (Gas Partial Pressure = P_2)

		$\Delta\rho$/kg m^{-3}					
solvent	P_2/Pa	N_2	O_2	air	CO_2	H_2	CH_4
heptane	94 947	0.090	0.064	0.089	−0.627	0.142	0.707
octane	99 175	0.083	0.060	0.081	−0.559	0.126	0.671
nonane	100 488	0.079	0.055	0.072	−0.531	0.111	0.642
decane	100 885	0.071	0.050	0.067	−0.507	0.094	0.613
dodecane	101 059	0.063	0.042	0.059	−0.430	0.083	0.564
tetradecane	101 060	0.057	0.041	0.054	−0.373	0.074	0.523
hexadecane	101 060	0.050	0.034	0.047	−0.336	0.066	0.498
cyclohexane	88 046	0.073	0.034	0.068	−0.265	0.070	0.667
methylcyclohexane	94 881	0.081	0.043	0.077	−0.285	0.086	0.626
methylbenzene	97 267	0.059	0.056	0.062	−0.346	0.092	0.579

[a] Uncertainties ± 0.006 kg m^{-3} except for cyclohexane and methylbenzene, which are ± 0.007 kg m^{-3}.

Table 3. Comparison of Measured with Literature Values of Density Differences $\Delta\rho = \rho_{deg} - \rho_{sat}$ at T = 298.15 K, Adjusted to Gas Partial Pressure P_2 = 101 325 Pa

	$\Delta\rho$/kg m^{-3}				
solvent	N_2	O_2	air	CO_2	ref
heptane	0.096 ± 0.006	0.068 ± 0.006	0.095 ± 0.006	−0.669 ± 0.006	*a*
	0.098 ± 0.006	0.027 ± 0.002	0.086 ± 0.003	−0.634 ± 0.003	*b*
octane	0.085 ± 0.006	0.061 ± 0.006	0.083 ± 0.006	−0.571 ± 0.006	*a*
	0.091 ± 0.003	0.024 ± 0.002	0.072 ± 0.003	−0.534 ± 0.006	*b*
dodecane	0.063 ± 0.006	0.042 ± 0.006	0.059 ± 0.006	−0.431 ± 0.006	*a*
	0.072 ± 0.002	0.020 ± 0.002	0.058 ± 0.003	−0.445 ± 0.003	*b*
hexadecane	0.050 ± 0.006	0.034 ± 0.006	0.047 ± 0.006	−0.337 ± 0.006	*a*
	0.049 ± 0.002	0.015 ± 0.002	0.038 ± 0.002	−0.289 ± 0.003	*b*

[a] This paper. [b] Cibulka and Heintz (1995).

Table 4. Mole Fraction Gas Solubilities x_2 at T = 298.15 K and Partial Pressure P_2 = 101 325 Pa

solvent	$10^4x_2(N_2)$	$10^4x_2(O_2)$	$10^4x_2(CO_2)$	$10^4x_2(H_2)$	$10^4x_2(CH_4)$
heptane	13.7[a]	20.4[a]	120[c]	6.8[d]	50.7[a]
octane	13.1[a]	20.5[a]	122[c]	6.9[d]	51.2[a]
nonane	13.3[a]	21.3[a]	123[c]	7.0[d]	52.1[a]
decane	12.8[a]	21.8[a]	125[c]	7.0[d]	53.1[a]
dodecane	13.1[a]	23.0[a]	129[c]	7.2[d]	54.5[a]
tetradecane	13.2[a]	23.7[a]	134[c]	7.4[d]	58.0[a]
hexadecane	13.4[a]	24.7[a]	139[c]	7.5[d]	61.0[a]
cyclohexane	7.6[b]	12.5[b]	76.0[b]	4.1[b]	32.8[b]
methylcyclohexane	9.5[b]	16.0[b]	92.8[b]	4.9[f]	39.6[e]
methylbenzene	5.7[b]	9.1[b]	101.3[b]	3.2[b]	22.4[e]

[a] Hesse et al. (1996). [b] Wilhelm and Battino (1973). [c] Equation 2. [d] Equation 3. [e] Field et al. (1974). [f] Ipatiev and Levin (1935).

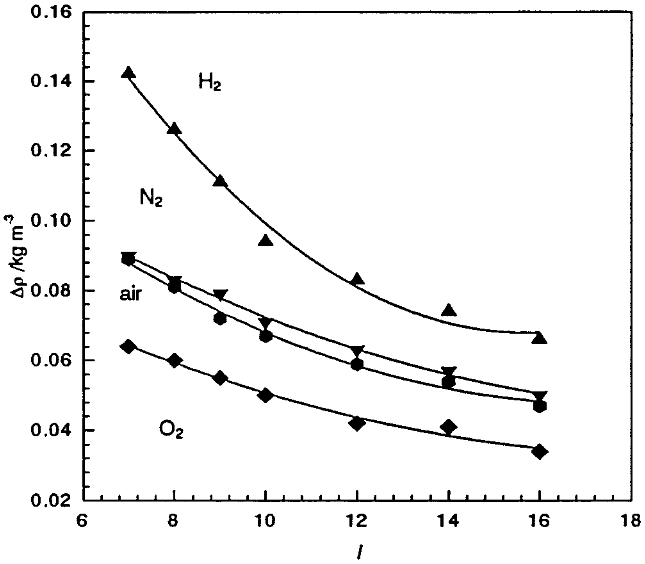

Figure 2. Variation of $\Delta\rho = (\rho_{deg} - \rho_{sat})$ for N_2, O_2, air, and H_2 dissolved in *n*-alkanes of chain length *l*.

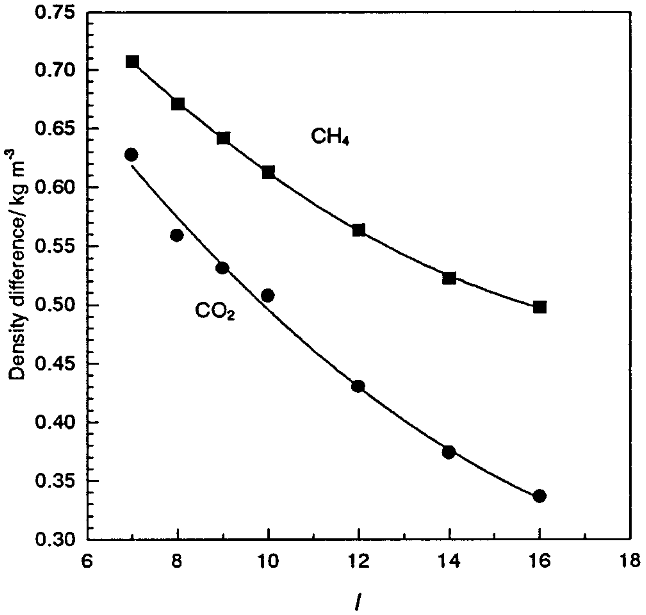

Figure 3. Variation of $\Delta\rho = (\rho_{deg} - \rho_{sat})$ for CH_4 and $-\Delta\rho$ for CO_2 dissolved in *n*-alkanes of chain length *l*.

sufficiently remote from the measuring cell that there was no possibility of diffusion of the solute to the liquid in the cell. This procedure was repeated until at least three successive density readings agreed.

The saturation of the solvents was carried out by slowly admitting the gas through valves 3 and 4 to flask 2 containing the thermostated solvent, initially under partial vacuum, until a total pressure of (101 060 ± 10) Pa was attained. The pressure of the solute was adjusted through valve 9 and monitored by a water manometer. A small purge of gas was allowed through valve 9 to prevent the diffusion of water vapor from the manometer fluid to the gas. The temperature of flask 2 was held constant at (298.15 ± 0.05) K and the solvent was agitated by a PTFE-coated stirrer operated magnetically. Gas was periodically fed to the saturating vessel to maintain the pressure. Liquid was transferred to the measuring cell by gravity. At least four repetitions of the entire experiment were made for each solute/solvent pair.

Results and Discussion

The measured density differences between the degassed and saturated samples, $\Delta\rho = \rho_{deg} - \rho_{sat}$, are given in Table 2 for a total pressure of 101 060 Pa. The partial pressure of the gas was calculated by subtracting the vapor pressure of the solvent obtained from the Antoine constants (TRC, 1996). For gases other than carbon dioxide and methane, the plots in Figure 2 show that the density differences, $\Delta\rho$, are rather small and they decrease linearly with increasing carbon number for the normal alkanes. Methane and carbon dioxide have a much larger effect on density as shown in Figure 3. Carbon dioxide, in contrast to the other gases, causes an increase in density (negative $\Delta\rho$). However the magnitudes of the density differences, $|\Delta\rho|$, again decrease with increasing carbon number in the *n*-alkane series. The effect of saturation with most gases is to decrease the density by amounts up to 0.1%, the largest effect being found for methane in heptane. Carbon dioxide increases density by a similar percentage. The effect of dissolved gases, most commonly air, should be taken into account when measuring and reporting densities at moderate to high accuracies and when carrying out excess volume measurements on mixtures. The results for N_2 and O_2 can be used to predict $\Delta\rho$ for air with reasonable accuracy in comparison with the measured values, thus confirming the independent behavior of the gases in solution as observed for water by Bignell (1984). Comparison of $\Delta\rho$ with published values (Cibulka and Heintz, 1995) is possible for a few solute/solvent pairs if the values are adjusted to a common pressure of solute. The values in Table 3 were adjusted to 101 325 Pa by assuming that $\Delta\rho$ was proportional to the partial pressure of the gaseous solute, P_2. The agreement between the two sets of data is moderate for N_2, CO_2, and air but not always as good as might be expected from the precision of the measurements. The authors can see no obvious reason for the serious disagreement of a factor of about 2 in the case of the O_2 values. Density differences from Table 2 may be used with gas solubility data in eq 1 to calculate the partial molar volumes of the solutes, V_2, which, since the solutions are highly dilute, may be regarded essentially as infinite

Table 5. Partial Molar Volumes of Gases in Solution V_2^∞ at Infinite Dilution at T = 298.15 K and P = 101 600 Pa Total Pressure

	V_2^∞/cm^3 mol^{-1}				
solvent	N_2	O_2	CO_2	H_2	CH_4
heptane	56.5 ± 0.7	54.3 ± 0.4	52.7 ± 0.6	51.5 ± 2.8	55.8 ± 0.2
octane	54.3 ± 0.8	52.9 ± 0.5	52.1 ± 0.5	47.0 ± 2.6	54.3 ± 0.2
nonane	53.8 ± 0.8	51.3 ± 0.5	50.8 ± 0.5	43.6 ± 2.6	53.8 ± 0.2
decane	53.4 ± 0.9	50.2 ± 0.5	49.7 ± 0.5	39.1 ± 2.5	53.4 ± 0.2
dodecane	54.1 ± 1.1	48.6 ± 0.6	48.9 ± 0.5	38.8 ± 2.6	54.1 ± 0.2
tetradecane	53.1 ± 1.3	48.1 ± 0.6	48.4 ± 0.5	37.5 ± 2.7	53.1 ± 0.3
hexadecane	51.9 ± 1.4	46.8 ± 0.7	48.0 ± 0.5	36.2 ± 2.8	52.4 ± 0.3
cyclohexane	53.6 ± 1.0	45.8 ± 0.6	51.3 ± 0.1	30.0 ± 1.8	53.6 ± 0.7
methylcyclohexane	52.0 ± 1.0	46.7 ± 0.5	52.0 ± 0.1	34.4 ± 2.6	49.3 ± 0.6
methylbenzene	45.6 ± 1.1	45.0 ± 0.7	45.9 ± 0.1	39.7 ± 2.1	49.5 ± 0.7

dilution values, V_2^∞.

$$V_2^\infty = \left(\frac{((1 - x_2)M_1 + x_2M_2)/\rho - M_1/\rho_1}{x_2}\right) + \frac{M_1}{\rho 1} \qquad (1)$$

In this equation ρ_1 and ρ represent the densities of the pure solvent and the mixture, respectively, M_1 and M_2 represent the molar masses of the solvent and solute, respectively, and x_2 represents the mole fraction of the dissolved solute. Fogg and Gerrard (1991), Battino et al. (1983, 1984), and the IUPAC Solubility Data Series (1981, 1982, 1987) have critically reviewed the extensive literature solubility data. It appears that disagreements between different determinations of gas solubility in hydrocarbons are commonly up to 10% but can exceptionally be as high as 35%. Given this unsatisfactory situation, the following procedures have been adopted. For nitrogen, oxygen, and methane, the recent experimental measurements of Hesse et al. (1996), of claimed uncertainty ±0.5%, were used directly. These data diverge from previously published values for nitrogen in the higher alkanes. Solubilities in cyclohexane, methylcyclohexane, and benzene, with an uncertainty of ±1%, were taken from Wilhelm and Battino (1973) and Field et al. (1974). For carbon dioxide dissolved in the alkanes, eq 2, taken from Fogg and Gerrard (1991), was used

$$x_2 = 0.01156 + (9.28 \times 10^{-6})l^2 \qquad (2)$$

where l is the carbon chain length. For hydrogen dissolved in n-alkanes, eq 3 was derived from the data of Cook et al. (1957) and Thomsen and Gjaldbaek (1963).

$$x_2 = (6.179 \times 10^{-4}) + (8.53 \times 10^{-6})l \qquad (3)$$

Equations 2 and 3 fit the data with an average absolute deviation of about 3%, but since the data themselves are inaccurate to at least 1%, it is likely that values of x_2 calculated from eqs 2 and 3 could be inaccurate to about 5%. The solubility of hydrogen in methylcyclohexane was derived, with an estimated uncertainty of ±5%, from the high-pressure data of Ipatiev and Levin (1935) using Henry's law. The values of gas solubility used in the calculations of partial molar volumes are listed in Table 4. Table 5 gives the calculated partial molar volumes. The uncertainties include those of $\Delta\rho$ and gas solubility.

From Table 5 it can be seen that the partial molar volumes of all the gases decrease with increasing carbon number of the alkane series and that nitrogen and methane have almost the same partial molar volume when dissolved in n-alkanes at atmospheric pressure and 298.15 K. The review by Handa and Benson (1982) revealed that data for hydrocarbons were scarce and there was very little overlap with results in the present paper. Even with the recent results of Cibulka and Heintz (1995) and Izak et al. (1995), the data in Table 6 show that the extent of duplication of measurements is not large. The values of Handa et al. (1982) were obtained by direct dilatometric measurements, whereas the present results and those of Cibulka and Heintz (1995) and Izak et al. (1995) depend on the interpretation of published solubility data. For some mixtures these solubility data are widely scattered and cannot be regarded as satisfactorily established.

Table 6. Comparison of Published Experimental Values for Infinite Dilution Partial Molar Volumes of Gases in Solution V_2^∞ at T = 298.15 K and P = Atmospheric

	V_2^∞/cm^3 mol^{-1}						
solvent	N_2	O_2	CO_2	H_2	CH_4	ref	method
heptane	56.5	54.3	52.7	51.5	55.8	*a*	*e*
	57.0	50.3	53.2			*b*	*e*
				43.2	54.2	*c*	*f*
octane	54.3	52.9	52.1		54.3	*a*	*e*
	56.3	48.4	52.7			*b*	*e*
	55.4	47.7	52.0		53.8	*d*	*f*
decane	53.4	50.2	49.7		53.4	*a*	*e*
	54.6	46.6	51.0			*b*	*e*
					51.8	*d*	*f*
hexadecane	51.9		48.0		52.4	*a*	*e*
	51.4		49.3			*b*	*e*
					50	*d*	*f*
cyclohexane				30.0		*a*	*e*
				41.0		*c*	*f*
methylbenzene				39.7		*a*	*e*
				35.8		*c*	*f*

[a] This work. [b] Cibulka and Heintz (1995). [c] Handa and Benson (1982). [d] Handa et al. (1982). [e] Density difference method. [f] Dilatometric method.

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Nomenclature

l = carbon chain length in n-alkanes C_lH_{2l+2}
M_1 = molar mass of solvent
M_2 = molar mass of solute
P = total pressure
P_1 = partial pressure of solvent
P_2 = partial pressure of solute
T = temperature
ρ = density of solution
ρ_1 = density of solvent
V = volume
V_2^∞ = partial molar volume of solute at infinite dilution
x_2 = mole fraction of solute

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