

PROJECT ADMINISTRATION DATA SHEET

ORIGINAL REVISION NO. _____

Project No. E-19-618 GTRI/GIT DATE 8/15/84

Project Director: Dr. Aryn S. Teja School/lab ChE

Sponsor: National Science Foundation

Type Agreement: Grant No. CPE-8405126

Award Period: From 8/1/84 To 1/31/86 (Performance) 4/30/86 (Reports)

Sponsor Amount: This Change Total to Date

Estimated: \$ _____ \$ 17,000

Funded: \$ _____ \$ 17,000

Cost Sharing Amount: \$ 17,000 Cost Sharing No: E-19-321

Title: "Equipment for High Temperature, High Pressure Physical Properties Measurements"

ADMINISTRATIVE DATA

OCA Contact Lynn Boyd x4820

1) Sponsor Technical Contact:

2) Sponsor Admin/Contractual Matters:

Robert M. Wellek

Richard E. Hastings

Program Director, Thermodynamics

Grants Official

NSF

NSF

Washington, DC 20550

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(202) 357-9606

(202) 357-9626

Defense Priority Rating: n/a Military Security Classification: n/a

(or) Company/Industrial Proprietary: n/a

RESTRICTIONS

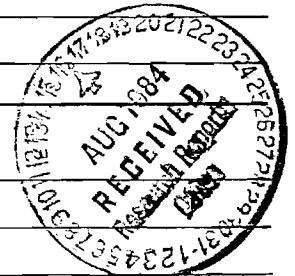
See Attached NSF Supplemental Information Sheet for Additional Requirements.

Travel: Foreign travel must have prior approval - Contact OCA in each case. Domestic travel requires sponsor approval where total will exceed greater of \$500 or 125% of approved proposal budget category.

Equipment: Title vests with GIT.

COMMENTS:

*Includes usual 6 month unfunded flexibility period.



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SPONSORED PROJECT TERMINATION/CLOSEOUT SHEET

Date 5/14/86

Project No. E-19-618

School/~~Lab~~ ChE

Includes Subproject No.(s) N/A

Project Director(s) A. S. Teja

GTR/~~CH~~

Sponsor National Science Foundation

Title - "Equipment for High Temperature, High Pressure Physical Properties Measurements"

Effective Completion Date: 1/31/86 (Performance) 4/30/86 (Reports)

Grant/Contract Closeout Actions Remaining:

- None
- Final Invoice or Final Fiscal Report
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FINAL REPORT

**EQUIPMENT FOR HIGH TEMPERATURE, HIGH
PRESSURE PHYSICAL PROPERTIES MEASUREMENT**

Dr. Aryn S. Teja

Final Report for Period August 1, 1984 to January 31, 1986

Under

Grant No. CPE 8405126

April 1986

GEORGIA INSTITUTE OF TECHNOLOGY

A Unit of the University System of Georgia

Atlanta, Georgia 30332



1986



FINAL PROJECT REPORT
NSF FORM 98A

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PART I—PROJECT IDENTIFICATION INFORMATION

1. Institution and Address Georgia Institute of Technology Atlanta, GA 30332	2. NSF Program Thermodynamics & Trans.	3. NSF Award Number CPE 8405126
	4. Award Period From 8/1/84 To 1/31/86	5. Cumulative Award Amount \$17,000

6. Project Title
Equipment for High Temperature, High Pressure Physical Properties Measurement

PART II—SUMMARY OF COMPLETED PROJECT (FOR PUBLIC USE)

High pressure, high temperature properties of fluids are important in chemical processing and in supercritical extraction. Such properties also provide an understanding of intermolecular forces under extreme conditions. This equipment grant was used to purchase calibration equipment (dead weight gauge, standard platinum resistance thermometer, cathetometer) and other equipment (high pressure, high temperature transducers) to greatly enhance and improve the experimental capabilities of the fluid properties research laboratory of the Principal Investigator.

PART III—TECHNICAL INFORMATION (FOR PROGRAM MANAGEMENT USES)

1. ITEM (Check appropriate blocks)	NONE	ATTACHED	PREVIOUSLY FURNISHED	TO BE FURNISHED SEPARATELY TO PROGRAM	
				Check (✓)	Approx. Date
a. Abstracts of Theses	XX				
b. Publication Citations		XX			
c. Data on Scientific Collaborators	XX				
d. Information on Inventions	XX				
e. Technical Description of Project and Results		XX			
f. Other (specify)					
2. Principal Investigator/Project Director Name (Typed) Dr. Aryn S. Teja	3. Principal Investigator/Project Director Signature C			4. Date 4/21/86	

FINAL REPORT

NSF Grant No.: CPE 8405126

Amount: \$17,000

Title: "Equipment for High Temperature, High Pressure Physical Properties Measurement"

Principal Investigator: Dr. Aryn S. Teja

TECHNICAL DESCRIPTION OF PROJECT AND RESULTS

High pressure and/or high temperature data are important in a number of applications, including supercritical extraction and chemical processing. There is a scarcity of data in the literature at such conditions, particularly for mixtures containing high boiling substances. Mixtures of low and high-boiling substances are also of theoretical interest from the point of view of developing mixing rules for equations of state. Finally, knowledge of the behavior of fluids under extreme conditions of pressure and/or temperature can lead to new materials and new processes.

As a result of these interests, a laboratory has been established at the Georgia Institute of Technology under the direction of the Principal Investigator. The laboratory has the following capabilities.

- (i) Vapor-liquid equilibrium measurement at temperatures up to 525K using a Stage-Muler still.
- (ii) High pressure phase equilibrium measurement at pressures up to 34.5 MPa using a recirculation apparatus (for liquid-supercritical fluid equilibria) and a flow apparatus (for solid-supercritical fluid equilibria).
- (iii) High pressure PVT measurement at pressures up to 34.5 MPa using a vibrating-tube densitometer and a high pressure pycnometer.

- (iv) Critical properties measurement using a novel rapid-heating apparatus suitable for thermally-labile substances.
- (v) High pressure, high temperature viscosity measurements using a capillary and a rolling-ball viscometer.
- (vi) High temperature thermal conductivity measurement using a transient hot wire apparatus.

The present grant (CPE 8405126) plus a matching grant of \$17,000 from the Georgia Institute of Technology were used to purchase calibration equipment for the laboratory and to extend the range of the above equipment to high pressures and temperatures.

The equipment purchased included:

1. A dead weight gauge for accurate calibration of pressures to 68 MPa
2. A standard platinum resistance thermometer calibrated by the National Bureau of Standards to 973K.
3. A cathetometer for accurate calibration of heights, and hence volumes.
4. An accurate flow measurement device (wet test meter).
5. A high pressure, high temperature pressure transducer.
6. Pressure gauges and miscellaneous valves and fittings.

The purchase of this equipment has greatly enhanced the measurement capabilities of the fluid properties laboratory at Georgia Tech. In one case, it has enabled us to upgrade our existing equipment and a paper acknowledging the NSF contribution has been published (Moradina and Teja, 1986). The calibration equipment will, of course, have an indirect and positive effect on all experimental results published from the fluid properties laboratory.

REFERENCE

1. Moradinia I and Teja A. S., "Solubilities of Solid n-Octacosane, n-Triacontane and n-Dotriacontane in supercritical Ethane", Fluid Phase Equilibria (1986).

PUBLICATION CITATION

AUTHOR

SOLUBILITIES OF SOLID n-OCTACOSANE, n-TRIACONTANE, AND n-DOTRIACONTANE IN SUPERCRITICAL ETHANE

IRAJ MORADINIA and AMYN S. TEJA

School of Chemical Engineering, Georgia Institute of Technology, Atlanta, 30332-0100 GA (U.S.A.)

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Presented at the International Symposium on Macromolecules, Prague, 1985. This article not later than 22nd April, '86

ABSTRACT

Moradina, I. and Teja, A.S., 1986. Solubilities of solid n-octacosane, n-triacontane, and n-dotriacontane in supercritical ethane. *Fluid Phase Equilibria*,

The solubilities of solid n-octacosane ($n-C_{28}H_{58}$), n-triacontane ($n-C_{30}H_{62}$), and n-dotriacontane ($n-C_{32}H_{66}$) in supercritical ethane at temperatures between 308.15 K and 319.15 K and pressures up to 20 MPa are reported here. The results have been correlated using an equation of state. A new correlation based on the solute reduced density at the triple point is also reported for this homologous series.

INTRODUCTION

The n-alkanes are an interesting homologous series because the members of the series display great regularity in their behavior. Many of their fluid phase properties, for example, can be correlated with the number of carbon atoms in the molecules (see, for example, Ambrose, 1976; Chase, 1984). To develop general relations for supercritical extraction, therefore, we have studied the solubilities of solid n-alkanes ($n-C_{28}H_{58}$, $n-C_{30}H_{62}$, and $n-C_{32}H_{66}$) in supercritical ethane. In addition to possible supercritical extraction applications, these longer chain n-alkanes are also of importance in coal liquefaction and in secondary and tertiary oil recovery.

An additional reason for studying the n-alkane series is that even-numbered n-alkanes exhibit different trends in their solid phase properties (e.g., sublimation pressure, heat of fusion etc.) than the odd-numbered members of the series (Broadhurst, 1962; Bondi, 1963; Morawetz, 1972). This is shown for the heat of fusion and the heat of sublimation in Figs. 1 and 2 and is a consequence of the different packing arrangements in the solid phase. It may be possible using supercritical extraction to exploit these differences to separate close-boiling members of the series. Any generaliza-

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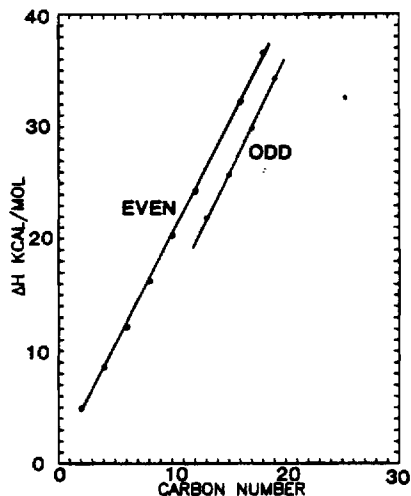


Fig. 1. Heat of sublimation versus carbon number for the n-alkanes.

tions for supercritical extraction behavior must, however, take account of this difference in behavior.

Figure 3 shows how the even and odd-numbered alkanes pack at their end groups. If we assume there is no appreciable interaction between end groups and that the chain axes are at right angles to the end layers, the packing arrangement for more than one molecule is shown in Fig. 4. It is seen that

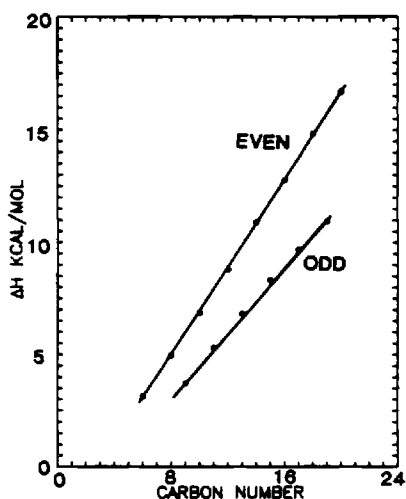


Fig. 2. Heat of fusion versus carbon number of the n-alkanes.

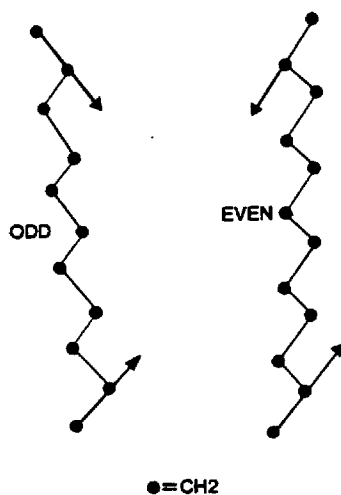


Fig. 3. Packing arrangements of single n-alkane molecules.

the packing arrangement for odd-numbered molecules repeats itself every other molecule, whereas all successive even-numbered molecules are identically situated. In Fig. 4, A_2 and A_3 are identical points on the solid lattice and the angle β_0 measures the inclination of the chain axis relative to the plane A_1A_2 . The angle θ_0 , which is the angle between the end C-C bond and the chain axis is identical to β_0 . This is not true for even-numbered members of the series, i.e., the angle θ_e is no longer equal to the angle β_e . A more comprehensive treatment of these differences in packing can be found in Müller (1929).

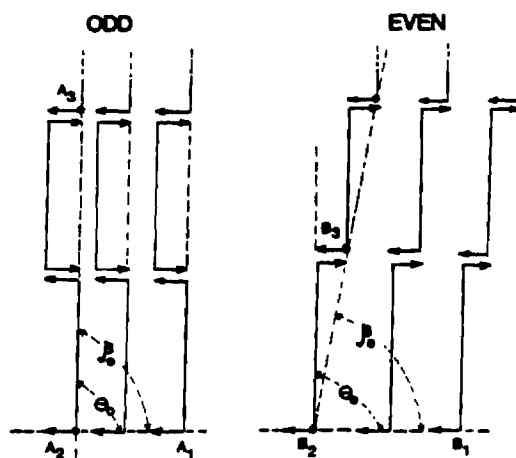


Fig. 4. Packing arrangements in the even and odd numbered n-alkane solids.

Because of these interesting differences, we have studied the solubilities of solid *n*-octacosane ($n\text{-C}_{28}\text{H}_{58}$), *n*-triacontane ($n\text{-C}_{30}\text{H}_{62}$) and *n*-dotriacontane ($n\text{-C}_{32}\text{H}_{66}$) in supercritical ethane. These solubilities are reported below. The solubilities of odd-numbered *n*-alkanes in supercritical ethane will be reported in a later publication.

EXPERIMENTAL SECTION

The apparatus used in this study was a single-pass flow system shown schematically in Fig. 5. The equipment is similar in principle to that used by a number of workers (McHugh and Paulaitis, 1980; Johnston and Eckert, 1981; Kurnik et al., 1981; Adachi et al., 1983). Solvent gas (ethane, in our case) is first liquefied and then compressed past its critical pressure by means of a Milton Roy minipump A. Its temperature is increased to supercritical conditions in the heating coil B immersed in a constant-temperature water bath C. The bath temperature was maintained constant within ± 0.1 K and the temperature was monitored by means of a copper-constantan thermocouple D calibrated against an NBS-certified platinum resistance thermometer. The pressure of the system was measured by a Heise gauge E calibrated against a dead-weight gauge. The pressure was maintained constant by a back pressure regulator F. The supercritical solvent was brought into contact with the solute (*n*-alkane) in an equilibrium cell G (modified Jerguson gage). The equilibrium temperature was measured by a second copper-constantan thermocouple K calibrated against an NBS-certified platinum resistance thermometer. This thermocouple was inserted into the equilibrium chamber where it could be in intimate contact with the contents. For better contact between the supercritical fluid and the solid solute, layers of glass beads were packed with the solute in the equilibrium cell. The loaded supercritical fluid was then depressurized at the micrometering valve H and the deposited solute was collected in a separation vessel J. The micrometering valve was heated to compensate for the Joule-Thompson effect upon depressurization and all the lines outside the constant temperature bath were heat traced. In addition, the micrometering valve was placed just outside the bath to minimize the length of tubing used. In spite of these precautions, however, some of the long-chain *n*-alkanes still precipitated inside the micrometering valve. This deposited solid was flushed with liquid hexane at the end of each run. For this purpose, the metering valve was isolated from the equilibrium cell by means of a two-way block valve installed just upstream from the valve. This ensured that only the precipitated solid inside the metering seat assembly would be removed by flushing. The relative amounts of solid collected by flushing to that in the separator ranged from 0 to as much as 80%. The solute collected was

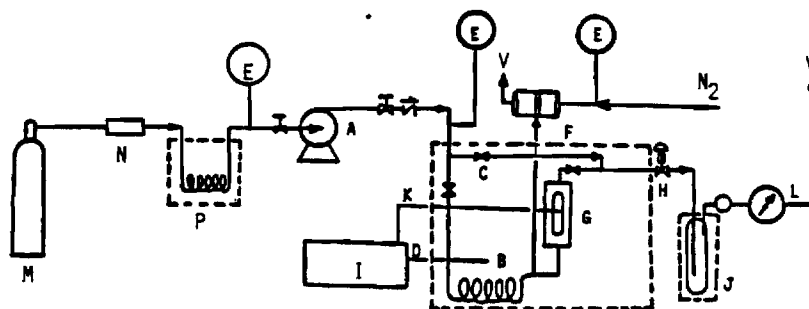


Fig. 5. Supercritical flow apparatus: A-minipump, B-heating coil, C-constant temperature bath, D-bath thermocouple, E-Heise pressure gauges, F-back pressure regulator, G-equilibrium cell, H-micro-metering valve, I-temperature measuring unit, J-separator vessel, K-equilibrium cell thermocouple, L-wet test meter, M-solvent gas cylinder, N-filter, P-condenser coil, V-vent to atmosphere, N_2 -high pressure nitrogen supply.

weighed to ± 0.10 mg using a Mettler electronic balance. The solvent volume was recorded using a wet test meter L. We estimate the precision of our temperature, pressure and volume measurements to be ± 0.10 K, ± 0.05 MPa and $\pm 1/2\%$, respectively. The accuracies were typically twice the magnitude of the precision.

Source and purity of the materials

The solid n-alkanes were obtained from Wiley Organics and had a stated purity of 99% or better. They were used without further purification. Ethane was furnished by the Matheson Gas Co. with 99 + % purity and was also used without further purification. The crystallinity of the solid samples used in this study was confirmed in two ways. First, a melting-freezing point check was carried out on a Thomas Hoover Capillary apparatus. Both the melting and freezing of each hydrocarbon occurred at a single temperature which was within $\pm 0.5^\circ\text{C}$ of the value reported by the supplier. In addition one sample of each hydrocarbon was analyzed on a Philips θ - 2θ X-ray Diffractometer. Sharp peaks in the X-ray diffraction scans were noted in each case, confirming that the hydrocarbons were crystalline.

RESULTS

For comparison purposes the solubilities of 2,3-dimethylnaphthalene (2,3-DMN) in carbon dioxide at three temperatures were first measured and the results compared with those of Kurnik et al. (1981). These results are presented in Table 1 for the 308.15 K isotherm. In no case did out

TABLE 1

Comparison of CO₂(1)+2,3-dimethylnaphthalene(2) data at 308.15 K

System pressure (MPa)	$y_2 \times 10^3$	
	this work	Kurnik et al.
9.87	2.28	2.2
14.25	4.36	4.41
19.60	5.45	5.42
24.12	5.76	5.82
27.92	6.45	6.43

solubilities differ from those of Kurnik et al. by more than 3.6%. Our results for C₂H₆ + n-C₂₈H₅₈, C₂H₆ + n-C₃₀H₆₂, and C₂H₆ + C₃₂H₆₆ systems are given in Table 2.

The region of the C₂H₆ + n-alkane phase diagram covered in the experiments is shown shaded in Fig. 6. It is seen that this region is bounded by the solid-liquid-gas (S^oLG) curve at higher pressures. In other words, the supercritical gas phase is in equilibrium with a liquid phase at higher pressures and this type of equilibrium cannot be studied in our apparatus. Hence, solid solubilities have been measured only in the range 308.15–319.15 K and 6–20 MPa. Solubilities of n-octacosane (n-C₂₈H₅₈) in carbon dioxide have been reported by McHugh, et al. (1984). The reported solubilities at 35°C are approximately one order of magnitude smaller than the solubilities measured here, confirming that ethane is a more efficient solvent for the n-alkanes than carbon dioxide. However, as the data reported by McHugh et al. (1984) show, one can measure solubilities of n-octacosane in carbon dioxide over a wider range of temperature (307.9–325.2 K) compared with the much smaller range of temperatures possible when ethane is the solvent due to the formation of a liquid phase.

TABLE 2

Experimental solubilities ($y_2 \times 10^3$) of solid n-alkanes in supercritical ethane

P (MPa)	T = 308.15 K			T = 313.15 K		T = 319.15 K
	q = 28	q = 30	q = 32	q = 30	q = 32	q = 32
6.57	1.89	0.549	0.216	0.486	0.177	0.149
10.10	3.38	1.24	0.713	1.95	0.953	1.28
12.02	6.43	1.45	0.801	2.31	1.15	1.44
13.64	7.53	1.71	0.959	3.50	1.44	2.14
16.67	10.80	2.24	1.26	–	1.73	–
20.02	15.18	3.20	1.81	–	2.18	–

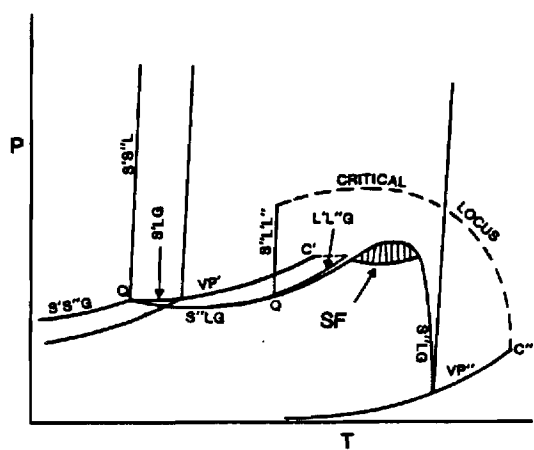


Fig. 6. $P-T$ diagram of a system in which liquid immiscibility is bounded by a solid phase, e.g., ethane + n -docosane, and methane + n -heptane.

Data correlation

Experimental data were correlated as follows:

2/

- (1) using the Patel and Teja (1983) equation of state; and
- (2) using a new empirical relationship.

Equation of state correlation

For a pure solid phase in equilibrium with a supercritical gas phase, we may write (Prausnitz et al., 1986)

$$P_2^{vap} \phi_2^{vap} \exp \left\{ \int_{P_2^{vap}}^P \frac{v_2^s}{RT} dP \right\} = y_2 \phi_2 P \tag{1}$$

where P_2^{vap} is the sublimation (vapor) pressure of the solute 2, ϕ_2^{vap} is the fugacity coefficient of the solute at its sublimation pressure, v_2^s is the molar volume of the pure solute, y_2 is the composition (solubility) of the solute in the supercritical solvent, ϕ_2 is the fugacity coefficient of the solute in the supercritical solvent, P is the pressure and all properties are evaluated at the system temperature T . Since the sublimation pressure is usually very small, we may assume $\phi_2^{vap} \sim 1$ and the integral to be evaluated from zero pressure to the pressure P . Also, since the solid volume is approximately constant with pressure, we can integrate and rewrite eqn. (1) as follows

$$y_2 = \frac{P_2^{vap} \exp(Pv_2^s/RT)}{\Phi_2} \tag{2}$$

Thus, if the solid phase properties (density and sublimation pressure) are

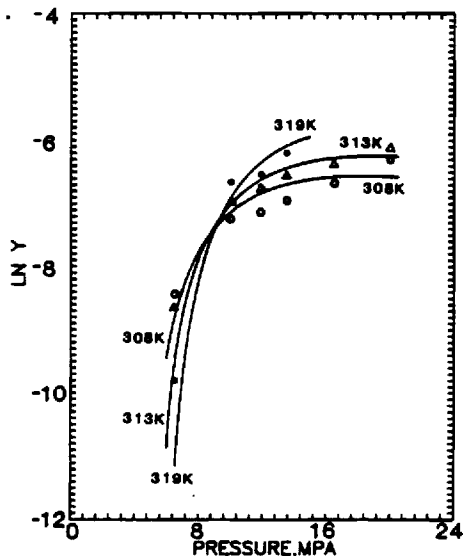


Fig. 7. Solubility of n-dotriacontane in supercritical ethane. \circ -experimental data at 308.15 K; Δ -experimental data at 313.15 K; $*$ -experimental data at 319.15 K. The solid lines were calculated using the Patel-Teja equation of state with $k_{ij} = -0.0383$ (308.15 K), -0.0315 (313.15 K), and -0.0227 (319.15 K).

known, then the solubility of the solute in the supercritical solvent at any pressure and temperature can be calculated provided an equation of state is available for the calculation of ϕ_2 . Typical results using the Patel-Teja equation of state are shown in Fig. 7. Note that a temperature dependent binary interaction coefficient (k_{ij}) is required in the equation of state calculations. At a given temperature, the binary interaction coefficient decreases as the number of carbon atoms in the solute increases, denoting increasing deviations from the geometric mean rule with increasing size difference

Solid densities and sublimation pressures used in the solubility calculations (eqn. (2)) are given in Table 3. The densities were supplied by the manufacturer whereas the sublimation pressures were estimated as follows. The reported sublimation pressures of n-tetradecane ($n\text{-C}_{14}\text{H}_{30}$), n-hexadecane ($n\text{-C}_{16}\text{H}_{34}$) and n-octadecane ($n\text{-C}_{18}\text{H}_{38}$) reported by Bondi (1963), Bradley and Shellard (1949), and Ubbelohde (1938) were fitted to the following equations

$$\ln P^{\text{vap}} = A + B/T \quad (3)$$

where

$$A = 3.04549 + 2.23175q \quad (4)$$

$$B = 2080.07 - 1129.73q \quad (5)$$

TABLE 3

Solid properties required in eqn. (2)

q (n-alkane)	V^s (l mol ⁻¹)	P_c (KPa)	T_c (K)	ω	$P_2^{vap} \times 10^{14}$ MPa		
					308.15 K	313.15 K	319.15 K
28	0.4894	892.4	838.3	1.255	6.3762	-	-
30	0.5222	838.2	853.6	1.342	0.3620	1.8802	-
32	0.5550	790.3	867.6	1.428	0.02005	0.1198	0.9274

In eqns. (3)–(5), q is the number of carbon atoms in the (even-numbered) n-alkane and the units of P and T are MPa and K, respectively. The overall absolute average deviation between experimental and calculated sublimation pressure was found to be 16.6% for the three alkanes studied. These equations allow the estimation of the sublimation pressures of other even-numbered n-alkanes.

Critical properties (T_c , P_c) and acentric factors (ω) required in the equation of state calculations were estimated from existing correlations developed by Peng and Robinson (1978) and are given in Table 3.

New empirical correlation

Because of the need to estimate T_c , P_c , ω and P_2^{vap} in the equation of

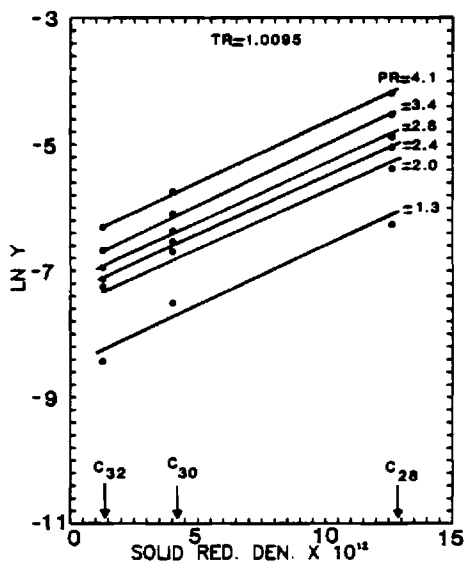


Fig. 8. Solubilities of n-alkanes in supercritical ethane. The solid lines were calculated using eqn. (6) whereas the experimental points are given by circles.

state calculations of the solubility, it was decided to correlate the solubility directly with solid-state properties.

The solubilities of even-numbered n-alkanes at a given P and T were correlated using the following relationship

$$\ln y = A + B(P_1 V^s / RT_1) \quad (6)$$

where P_1 , T_1 are the triple-point pressure and temperature, respectively, and V^s is the molar volume of the solid. The triple point temperature can be approximated by the melting temperature (Prausnitz et al., 1986) and, once this is known, the triple point pressure can be estimated from eqns. (3)–(5). Thus the solubility of any even-numbered n-alkane (say n-C₃₄) in supercritical ethane near the critical point of ethane can be predicted solely from a knowledge (or estimates) of P_1 , T_1 and V^s . Typical results are shown in Fig. 8. It should be emphasized that this is a correlation of data over a limited range of conditions and should only be subjected to judicious extrapolation.

ACKNOWLEDGMENT

Partial support for this work was provided by equipment grant no. CPE8405126 from the National Science Foundation.

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FP: 172 [Equation (1)]
 DP: 421 [Equation (2)]