

PHASE BEHAVIOR OF LIGHT GASES IN HYDROCARBON  
AND AQUEOUS SOLVENTS

Report for the Period  
from April 1, 1997 to August 31, 1997

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# PHASE BEHAVIOR OF LIGHT GASES IN HYDROCARBON AND AQUEOUS SOLVENTS

## ABSTRACT

Under previous support from the Department of Energy, an experimental facility has been established and operated to measure valuable vapor-liquid equilibrium data for systems of interest in the production and processing of coal fluids. To facilitate the development and testing of models for prediction of the phase behavior for such systems, we have acquired substantial amounts of data on the equilibrium phase compositions for binary mixtures of heavy hydrocarbon solvents with a variety of supercritical solutes, including hydrogen, methane, ethane, carbon monoxide, and carbon dioxide.

The present project focuses on measuring the phase behavior of light gases and water in Fischer-Tropsch (F-T) type solvents at conditions encountered in indirect liquefaction processes and evaluating and developing theoretically-based correlating frameworks to predict the phase behavior of such systems. Specific goals of the proposed work include (a) developing a state-of-the-art experimental facility to permit highly accurate measurements of equilibrium phase compositions (solubilities) of challenging F-T systems, (b) measuring these properties for systematically-selected binary, ternary and molten F-T wax mixtures to provide critically needed input data for correlation development, (c) developing and testing models suitable for describing the phase behavior of such mixtures, and (d) presenting the modeling results in generalized, practical formats suitable for use in process engineering calculations.

During the present reporting period, the solubility of carbon monoxide, hydrogen, and nitrogen in n-dodecane were measured using a static equilibrium cell over the temperature range from 344.3 to 410.9 K and pressures to 13.2 MPa. The uncertainty in these new solubility measurements is estimated to be less than 0.001 in mole fraction. The data were analyzed using the Peng-Robinson (PR) equation of state (EOS). In general, the PR EOS represents the experimental data well when two interaction parameters ( $C_{ij}$  and  $D_{ij}$ ) are used for each isotherm. The data suggest that the EOS interaction parameters are highly temperature dependent for the carbon monoxide and hydrogen systems and less so for the nitrogen system. Also a trend of increasing solubility with increased temperature and pressure is observed.

A manuscript we have prepared for publication is attached which provides detailed technical information.

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PROJECT TITLE: "Phase Behavior of Light Gases in Hydrocarbon and Aqueous Solvents"

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### **EXECUTIVE SUMMARY**

During the present reporting period, the solubility of carbon monoxide, hydrogen, and nitrogen in n-dodecane were measured using a static equilibrium cell over the temperature range from 344.3 to 410.9 K and pressures to 13.2 MPa. The uncertainty in these new solubility measurements is estimated to be less than 0.001 in mole fraction. The data were analyzed using the Peng-Robinson (PR) equation of state (EOS). In general, the PR EOS represents the experimental data well when two interaction parameters ( $C_{ij}$  and  $D_{ij}$ ) are used for each isotherm. The data suggest that the EOS interaction parameters are highly temperature dependent for the carbon monoxide and hydrogen systems and less so for the nitrogen system. Also a trend of increasing solubility with increased temperature and pressure is observed.

**Solubilities of Carbon Monoxide, Hydrogen and Nitrogen in n-Dodecane  
from 344 to 410 K at Pressures to 13.2 MPa**

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## **Abstract**

The solubilities of carbon monoxide, hydrogen, and nitrogen in n-dodecane were measured using a static equilibrium cell over the temperature range from 344.26 to 410.93 K at pressures to 13.24 MPa. The uncertainty in these new solubility measurements is estimated to be less than 0.001 in mole fraction. The data were analyzed using the Peng-Robinson (PR) equation of state (EOS). In general, the PR EOS represents the experimental data well when two interaction parameters ( $C_{ij}$  and  $D_{ij}$ ) are used for each isotherm. The data suggest that the EOS interaction parameters are highly temperature dependent for the carbon monoxide and hydrogen systems while they are less temperature dependent for the nitrogen system. Also a trend of increasing solubility with increased temperature and pressure is observed.

## **Introduction**

An experimental facility has been established and operated to measure vapor-liquid equilibrium data for systems of interest in the production and processing of coal fluids. To facilitate the development and testing of models for prediction of the phase behavior for such systems, we have acquired substantial amounts of data on the equilibrium phase compositions for binary mixtures of heavy hydrocarbon solvents with a variety of supercritical solutes, including hydrogen, methane, ethane, carbon monoxide, and carbon dioxide (Anderson et al., 1986; Darwish, 1991; Darwish et al., 1993; Gasem et al., 1989; Gasem and Robinson, 1985; Park et al., 1995; Srivatsan et al., 1995). These asymmetric mixtures which contain small gas molecules in heavy hydrocarbon solvents provide a severe test of current predictive models and their associated mixing theories (Park et al., 1995).

In this work, measurements were made of the solubilities of carbon monoxide, hydrogen and nitrogen in n-dodecane. The data have been correlated using the Peng-Robinson (PR) (Peng and Robinson, 1976) EOS. No experimental data for these systems have been reported before; thus, the present measurements provides a valuable complement to our previous work.

## **Experimental Method**

Details regarding the apparatus and experimental procedure have been described by Darwish (Darwish, 1991; Darwish et al., 1993) and are summarized briefly here.

A schematic diagram of the apparatus is shown in Figure 1. Phase equilibrium is established in a variable-volume, thermostated, static-type blind cell. This equilibrium cell (EC, refer to Fig. 1) is a 25 cc, 316 stainless steel micro-reactor. It is housed in a constant-temperature bath (BATH 1). Two steel balls are placed in the equilibrium cell and the cell is rocked 45 degrees above and below the horizontal position to hasten the establishment of equilibrium. The effective volume of the cell can be varied by the introduction or withdrawal of mercury.

Degassed liquid solvent is stored, at the operating temperature of the experiment, in a solvent storage cell (SV) contained inside the high-temperature air oven (BATH 1). The equilibrium cell and solvent storage cell are from High Pressure Equipment, Inc.

Injection of solvent into the initially-evacuated equilibrium cell is done volumetrically by injecting mercury at the bottom of the solvent cylinder, thus displacing an equal quantity of solvent into the rocking cell. The solvent injection pump (SIP) is from Temco, Inc. Solute gas is then injected into the rocking cell from a second Temco gas-injection pump (GIP). After each solute injection, the bubble point pressure of the mixture is determined. This is done by sequentially injecting known amounts of mercury into the cell to alter the system volume. After



each mercury injection, the equilibrium cell is rocked to bring the system to equilibrium and the pressure is recorded. The bubble point pressure (for the mixture of known composition) is identified graphically from the discontinuity in the pressure vs. total-volume-of-mercury-injected relation as the mixture passes from the more compressible two-phase state to the less compressible single-liquid-phase state.

The air bath housing the equilibrium cell (BATH 1) is thermostated by a proportional-integral (PI) controller supplied by Omega Engineering, Inc. The temperature of the air bath housing the injection pump (BATH 2) is controlled by a PI controller from Halikainen Instruments. The temperatures in the air baths are measured using platinum resistance thermometers equipped with digital displays from Fluke, Inc. Pressures are measured with transducers from Sensotec, Inc. The transducers are calibrated periodically against a Ruska Instrument Corporation dead weight tester (DWG) with certification traceable to the National Bureau of Standards.

Measurement uncertainties are estimated to be 0.1 K in temperature and less than 0.001 in composition (mole fraction). The uncertainty in the bubble point pressure is dependent on the steepness of the pressure vs. composition relation; it is estimated to be approximately 0.06 MPa for the systems studied here.

The experimental apparatus and procedures used in this work were evaluated by conducting measurements of the solubility of CO<sub>2</sub> in benzene at 344.3 K. The results were in agreement with our previous measurements and data reported in the literature for this test system (Darwish, 1991; Park et al., 1995; Srivatsan et al., 1995), as illustrated in Figure 2. We then began our current measurements, which cover temperatures from 344.3 to 410.9 K and pressures to 13.2 MPa.

## Materials

The hydrogen used in this study had a stated purity of 99.995 mol % and was supplied by Union Carbide Corporation. The carbon monoxide had a stated purity of 99.99 mol % and was supplied by Matheson Gas Products. The nitrogen had a stated purity of 99.995+ mol % and was supplied by Sooner Airgas, Inc. The n-dodecane was supplied by the Aldrich Chemical Company and had stated purity of 99+ mol %. No further purification of these chemicals was attempted.

## Results and Data Correlations

The measurements of carbon monoxide, hydrogen and nitrogen solubilities in n-dodecane are presented in Table 1. The effects of temperature and pressure on the solubility are illustrated for carbon monoxide in n-dodecane in Figure 3. A trend of increasing solubility with increased temperature and pressure is observed; results for the other systems are similar.

Equation of state representations of the experimental data are presented for each isotherm in Figures 4-6. In these figures, the data are shown in terms of the deviation,  $\delta x_1$ , in the solubility (liquid mole fraction) predicted by the PR EOS from the measured value, i.e.,  $\delta x_1 = x_1(\text{PR}) - x_1(\text{expt'l.})$ . The solubility values for the PR EOS were calculated using values of the interaction parameters,  $C_{ij}$  and  $D_{ij}$ , regressed from our data at each temperature of interest.

The PR EOS (Peng and Robinson, 1976) was used to correlate the experimental data. The PR EOS is given below.

$$p = \frac{RT}{v-b} - \frac{a(T)}{v(v+b) + b(v-b)} \quad (1)$$

where

$$a = a_c \alpha(T) \quad (2)$$

$$b = 0.0778RT_c / P_c \quad (3)$$

and

$$a_c = 0.45724R^2T_c^2 / P_c \quad (4)$$

$$\alpha(T)^{1/2} = 1 + k(1 - T_r^{1/2}) \quad (5)$$

$$k = 0.37464 + 1.54226\omega + 0.26992\omega^2 \quad (6)$$

where  $p$  is the pressure,  $R$  is the gas constant,  $T$  is the temperature,  $a$  and  $b$  are EOS constants,  $v$  is the molar volume,  $T_c$  is the critical temperature,  $p_c$  is the critical pressure,  $T_r$  is the reduced temperature,  $\alpha(T)$  expresses the temperature dependence in the parameter  $a$ , and  $\omega$  is the acentric factor.

To apply the PR equations of state to mixtures, the values of  $a$  and  $b$  were determined using the mixing rules (Gasem et al., 1989):

$$a = \sum_i^N \sum_j^N z_i z_j (1 - C_{ij})(a_i a_j)^{1/2} \quad (7)$$

$$b = 0.5 \sum_i^N \sum_j^N z_i z_j (1 + D_{ij})(b_i + b_j) \quad (8)$$

where  $z_i$  is the mole fraction of component "i" in a mixture,  $z_j$  is the mole fraction of component "j" in a mixture, and  $N$  is the number of components in mixture.

In Equations 7 and 8, the summations are over all chemical species, and  $C_{ij}$  and  $D_{ij}$  are empirical interaction parameters characterizing the binary interactions between components "i" and "j". Values of these parameters were determined by fitting the experimental data to minimize the objective function,  $SS$ , which represents the sum of squared errors in predicted bubble point pressures.

$$SS = \sum_i^n (P_{\text{exp}} - P_{\text{calc}})_i^2 \quad (9)$$

where  $n$  is the number of data points,  $P_{\text{exp}}$  is the experimental pressure, and  $P_{\text{cal}}$  is the calculated pressure. Further details of the data reduction technique used in this study are given by Gasem (Gasem et al., 1985; Gasem, 1986). The input parameters for the pure components (acentric factors, critical temperatures and critical pressures) required by the PR EOS, together with their literature source, are presented in Table 2.

Details of the EOS representations of the solubilities for the systems considered are shown in Table 3. In general, the PR EOS is capable of describing the data with RMS errors within 0.002 in mole fraction when one interaction parameters,  $C_{ij}$ , is used for each isotherm. However, when two interaction parameters,  $C_{ij}$  and  $D_{ij}$ , are used, the quality of the predictions improves resulting in RMS errors within 0.001 in mole fraction. When interaction parameters are regressed for a given hydrocarbon solvent without considering the temperature effect, the RMS errors are within 0.002 for carbon monoxide and hydrogen, and within 0.001 for nitrogen. For carbon monoxide and hydrogen, there is a strong temperature dependence of the interaction parameters. In comparison, less temperature dependence is exhibited by the nitrogen interaction parameters, which is consistent with our previous studies of hydrocarbon mixtures involving methane, ethane, and  $\text{CO}_2$  (Anderson et al., 1986; Darwish, 1991; Darwish et al., 1993; Gasem et al., 1989; Gasem and Robinson, 1985; Park et al., 1995; Srivatsan et al., 1995).

## Conclusions

Measurements have been obtained for the solubilities of carbon monoxide, hydrogen and nitrogen in n-dodecane at temperatures from 344.3 K to 410.9 K at pressures up to 13.2 MPa. The data are well described by the PR EOS. Interaction parameters for the PR EOS have been obtained for the binary systems studied.

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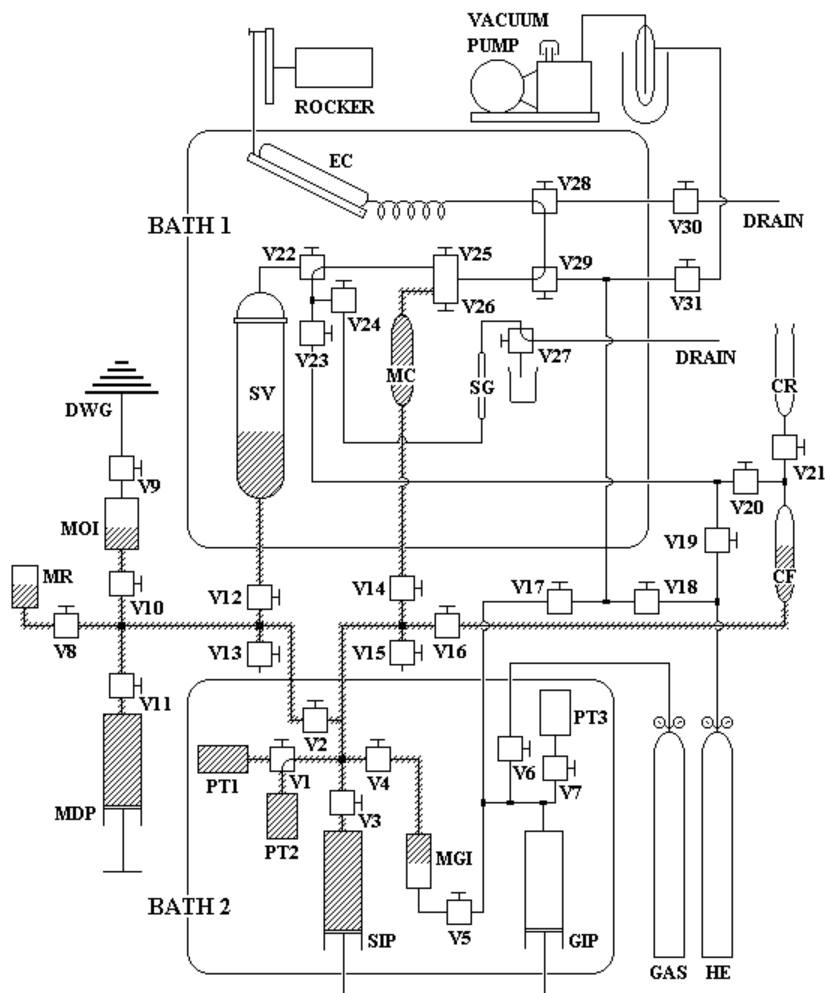
### **Financial Support**

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## Legend to Figures

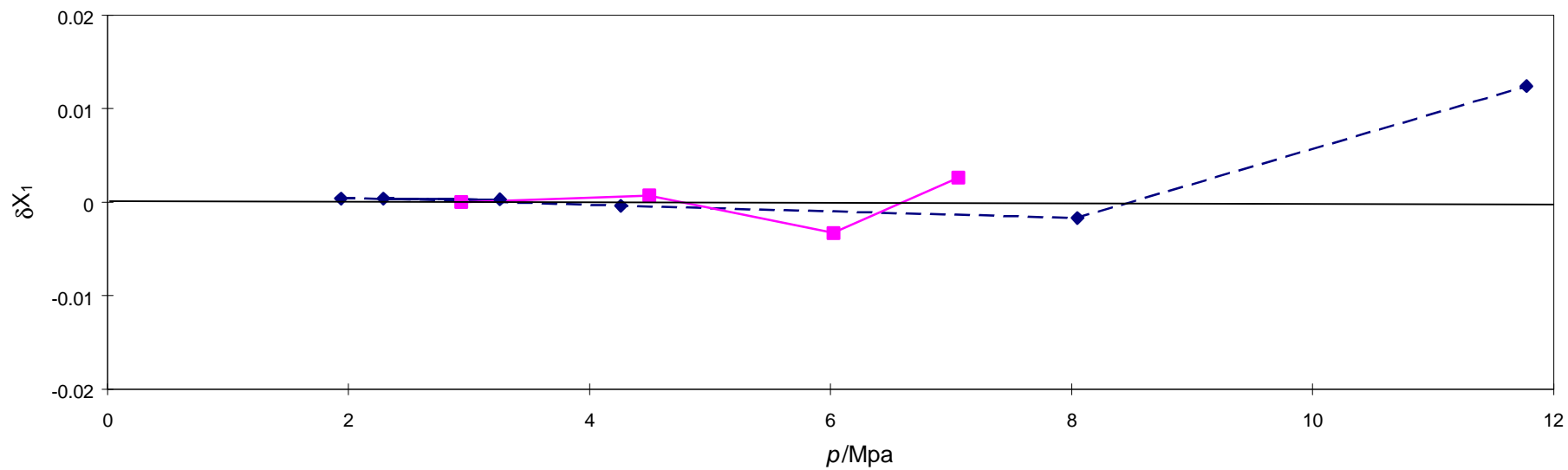
- Figure 1.** Schematic diagram of experimental apparatus.
- Figure 2.** Comparison of the solubility of carbon dioxide (1) in benzene (2): (◆) Darwish, 1991; (■) this work.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.
- Figure 3.** Solubility of carbon monoxide (1) in n-dodecane (2) at (◆) 344.3 K, (■) 377.6 K, (#) 410.9 K.
- Figure 4.** EOS representation of the solubility of carbon monoxide (1) in n-dodecane (2): (◆) 344.26 K, (■) 377.59 K, (#) 410.93 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.
- Figure 5.** EOS representations of the solubility of hydrogen (1) in n-dodecane (2): (◆) 344.3 K, (■) 377.6 K, (#) 410.9 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.
- Figure 6.** EOS representation of the solubility of nitrogen (1) in n-dodecane (2): (◆) 344.3 K, (■) 377.6 K, (#) 410.9 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.



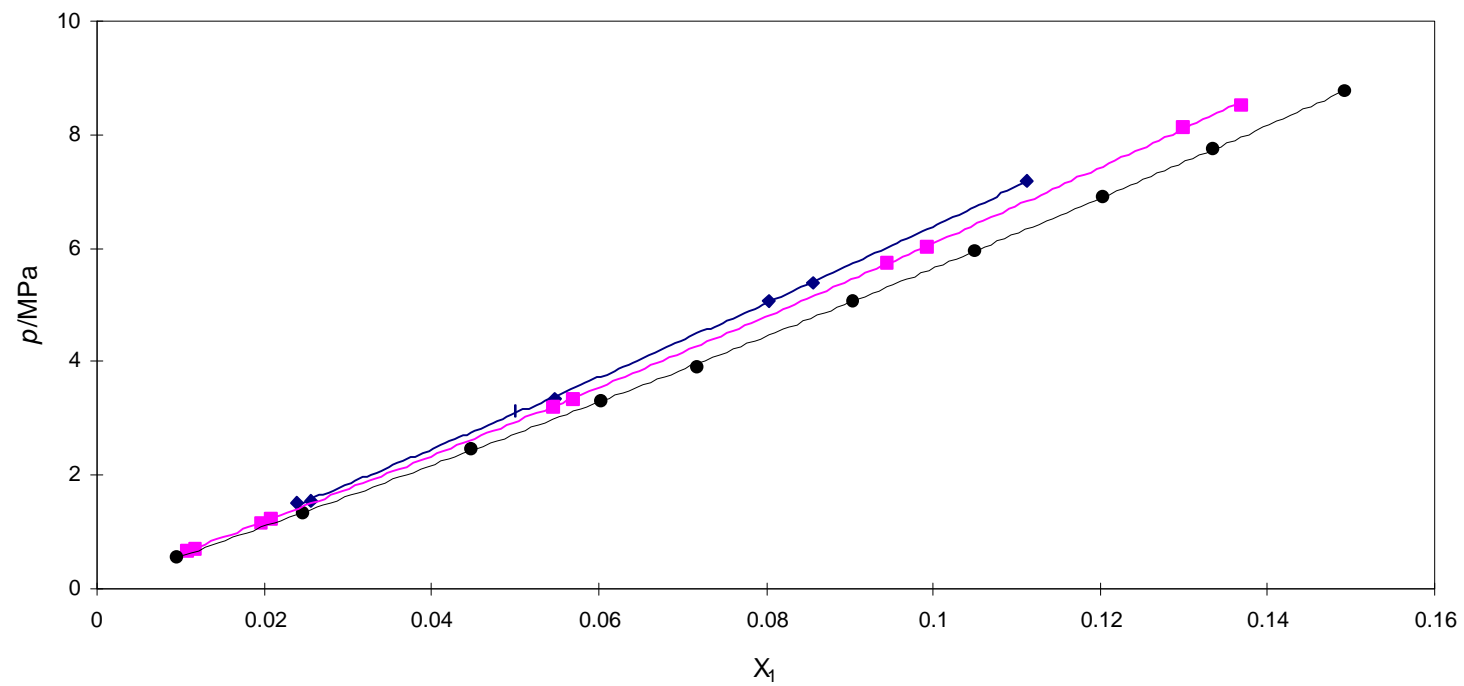


CF	Cleaning Fluid Cylinder	MGI	Mercury-Gas Interface
CR	Cleaning Fluid Reservoir	MC	Mercury Storage Cell
DWG	Dead Weight Gauge	MOI	Mercury-Oil Interface
EC	Equilibrium Cell	MR	Mercury Reservoir
GAS	Solute Gas	PT's	Pressure Transducers
EC	Equilibrium Cell	SG	Sight Glass
GIP	Solute Gas Injection Pump	SIP	Solvent Injection Pump
HE	Helium Gas	SV	Solvent Storage Cell
MDP	Mercury Displacement Pump	V1-V31	Valves

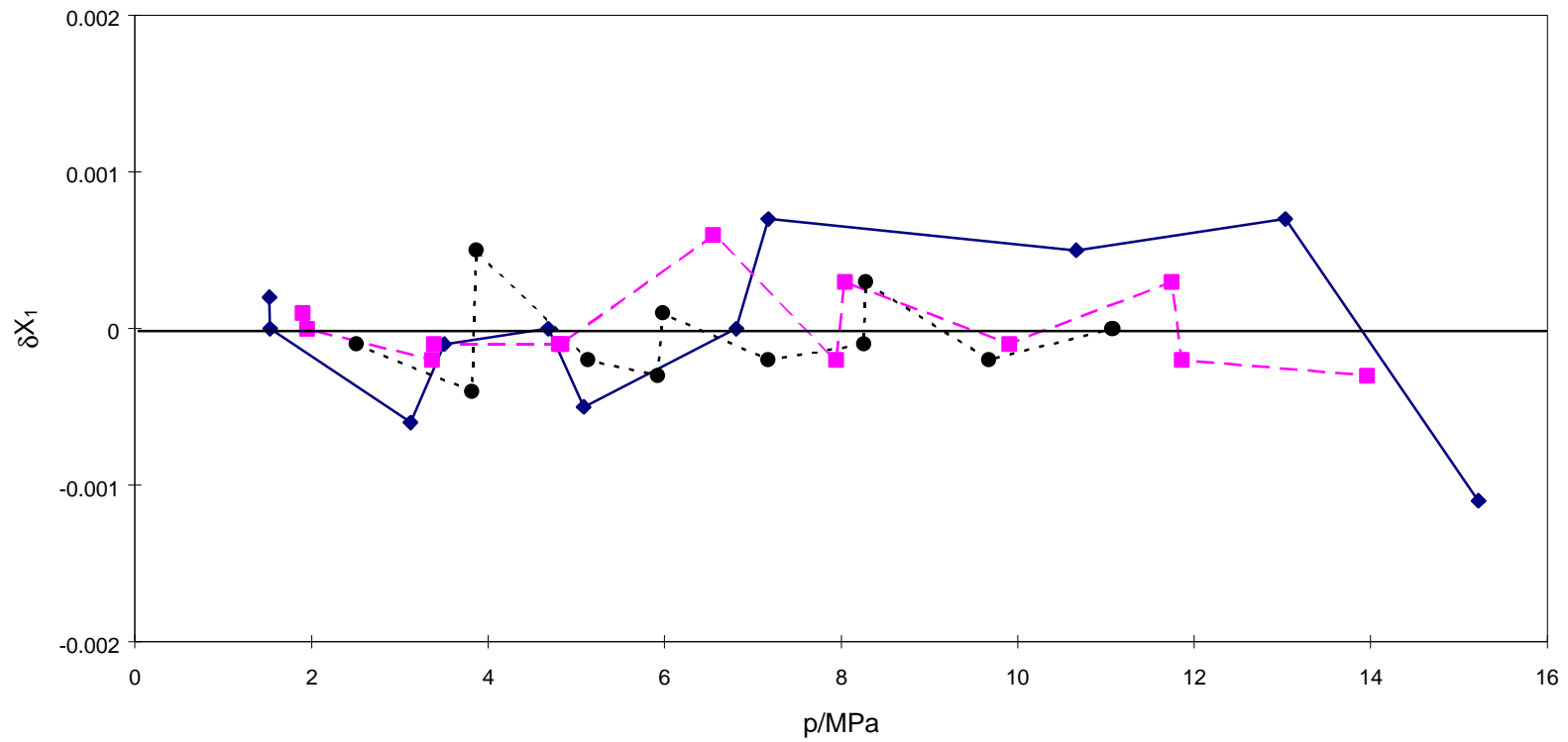
Figure 1. Schematic Diagram of Experimental Apparatus



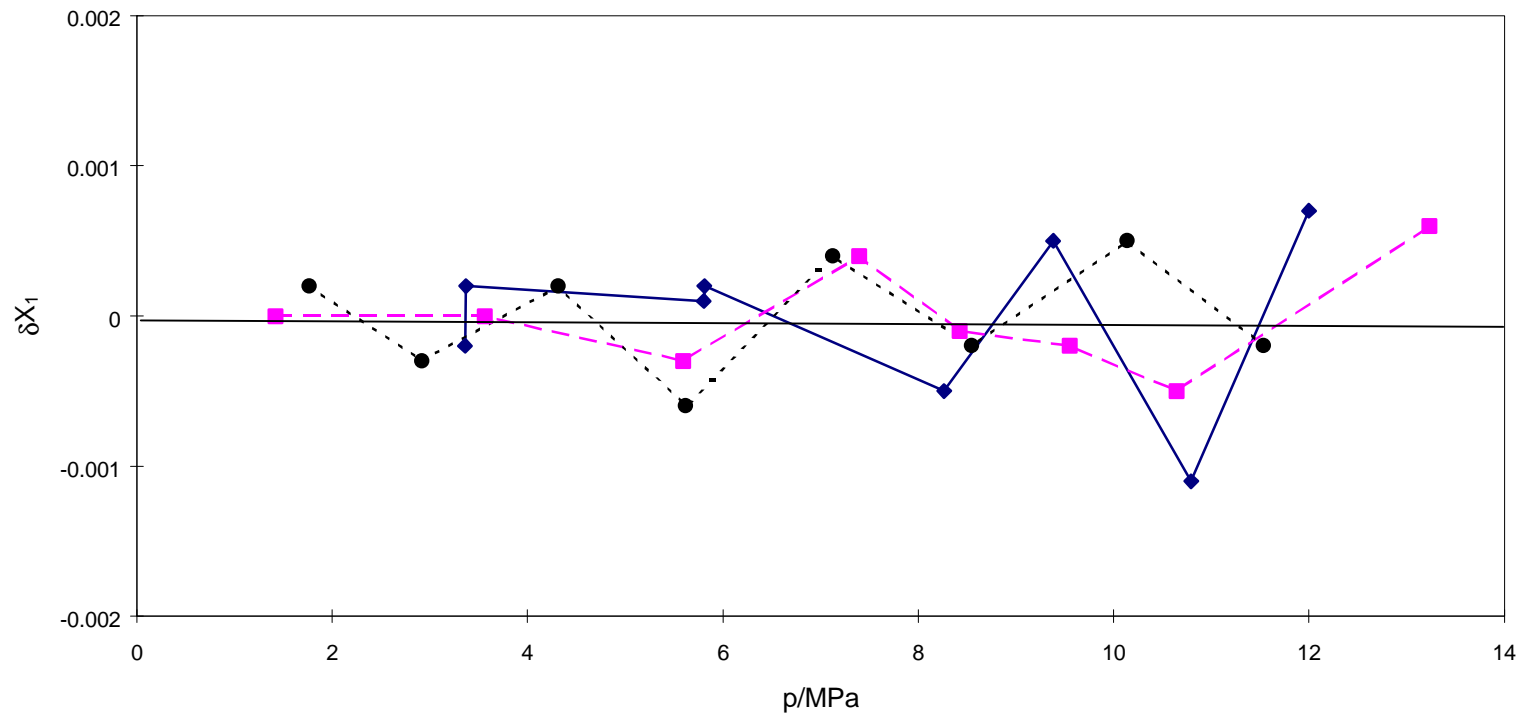
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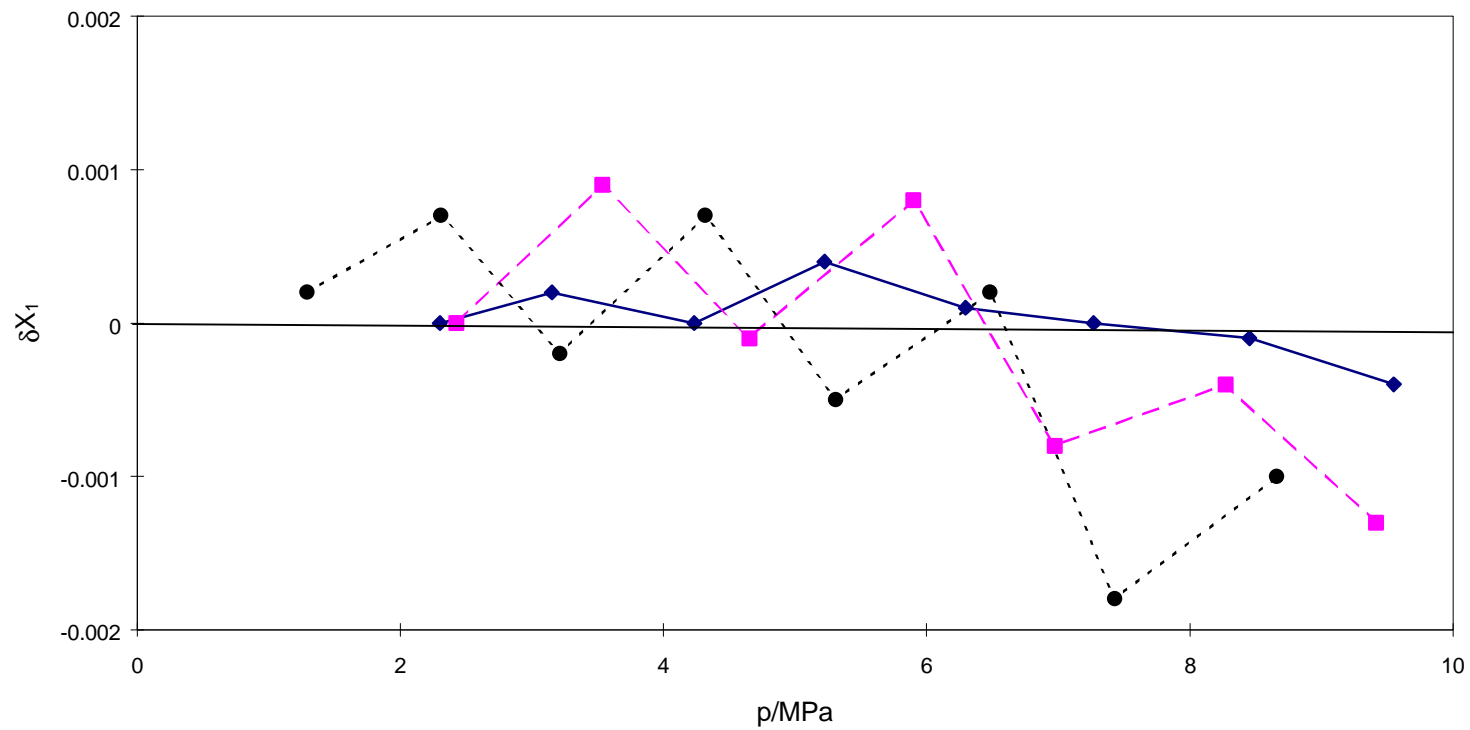
**Figure 3.** Solubility of carbon monoxide (1) in n-dodecane (2) at (◆) 344.3 K, (■) 377.6 K, (#) 410.9 K.



**Figure 4.** EOS representation of the solubility of carbon monoxide (1) in n-dodecane (2): (◆) 344.26 K, (■) 377.59 K, (#) 410.93 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation



**Figure 5.** EOS representations of the solubility of hydrogen (1) in n-dodecane (2): ( $\blacklozenge$ ) 344.3 K, ( $\blacksquare$ ) 377.6 K, ( $\bullet$ ) 410.9 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.



**Figure 6.** EOS representation of the solubility of nitrogen (1) in n-dodecane (2): (◆) 344.3 K, (■) 377.6 K, (#) 410.9 K.  $\delta X_1$  is the difference between the measured solubility from that predicted by the PR equation.

Table 1. Solubility of CO, H<sub>2</sub>, N<sub>2</sub> in n-Dodecane

$x_1$	$p/\text{MPa}$		$x_1$	$p/\text{MPa}$
Carbon Monoxide				
344.3 K				
0.0240	1.52		0.0804	5.08
0.0256	1.53		0.0857	5.39
0.5000	3.12		0.1113	7.18
0.0546	3.35		0.1150	7.87
377.6 K				
0.0113	0.69		0.0945	5.73
0.0118	0.71		0.0992	6.03
0.0197	1.17		0.1300	8.14
0.0208	1.23		0.1368	8.54
0.0568	3.34			
410.9 K				
0.0245	1.34		0.1049	5.95
0.0447	2.47		0.1202	6.91
0.0603	3.32		0.1335	7.75
0.0718	3.91		0.1493	8.75
0.0904	5.07			
Hydrogen				
344.3 K				
0.0297	3.36		0.0703	8.26
0.0302	3.37		0.0802	9.38
0.0505	5.80		0.0900	10.79
0.0506	5.81		0.1010	12.00
377.6 K				
0.0144	1.42		0.0804	8.43
0.0355	3.56		0.0904	9.55
0.0550	5.59		0.1000	10.64
0.0707	7.39		0.1204	13.24
410.9 K				
0.0209	1.77		0.0803	7.13
0.0348	2.92		0.0956	8.55
0.0500	4.31		0.1108	10.14
0.0652	5.62		0.1252	11.54

Table 1. Solubility of CO, H<sub>2</sub>, N<sub>2</sub> in n-Dodecane (continued)

$x_1$	$p/\text{MPa}$		$x_1$	$p/\text{MPa}$
		Nitrogen		
		344.3 K		
0.0314	2.30		0.0804	6.30
0.0422	3.15		0.0915	7.27
0.0559	4.23		0.1046	8.45
0.0675	5.22		0.1165	9.55
		377.6 K		
0.0195	1.41		0.0801	5.90
0.0350	2.43		0.0950	6.97
0.0493	3.53		0.1101	8.27
0.0651	4.65		0.1242	9.42
		410.9 K		
0.0202	1.29		0.0801	5.31
0.0354	2.31		0.0955	6.48
0.0498	3.21		0.1100	7.43
0.0649	4.32		0.1251	8.66



Table 2. Critical Pressures,  $p_c$ , Critical Temperatures,  $T_c$ , and Acentric Factors,  $\omega$ , used in the PR Equation of State

Component	$p_c/\text{MPa}$	$T_c/\text{K}$	$\omega$	Ref.
Carbon Monoxide	3.50	132.9	0.066	Reid et al. 1987
Hydrogen	1.30	33.2	-0.218	Reid et al. 1987
Nitrogen	3.39	126.2	0.039	Reid et al. 1987
n-Dodecane	1.82	658.2	0.575	Reid et al. 1987

Table 3. PR EOS Representations of the Solubility of Carbon Monoxide, Hydrogen and Nitrogen in n-Dodecane

T/K	C <sub>12</sub>	D <sub>12</sub>	Error in Solute Mole Fraction	
			RMS	MAX
Carbon Monoxide				
344.3	-0.1353	0.0437	0.0008	0.0015
	0.1120		0.0016	0.0032
377.6	-0.1269	-0.0399	0.0009	0.0016
	0.1004		0.0017	0.0037
410.9	-0.0828	0.0276	0.0009	0.0020
	0.0671		0.0006	0.0012
344.3, 377.6 and 410.9	-0.1073	0.0357	0.0018	0.0038
	0.0973		0.0027	0.0063
Hydrogen				
344.3	-0.0051	0.0343	0.0005	0.0011
	0.3567		0.0011	0.0027
377.6	0.1114	0.0291	0.0003	0.0006
	0.4002		0.0008	0.0013
410.9	0.1704	0.0231	0.0004	0.0006
	0.3863		0.0007	0.0016
344.3, 377.6 and 410.9	0.4292	-0.0051	0.0014	0.0046
	0.3775		0.0014	0.0047
Nitrogen				
344.3	0.2344	-0.0043	0.0002	0.0004
	0.2095		0.0002	0.0003
377.6	0.2171	-0.0015	0.0008	0.0013
	0.2086		0.0007	0.0012
410.9	0.2050	-0.0004	0.0008	0.0017
	0.2049		0.0008	0.0018
344.3, 377.6 and 410.9	0.2097	-0.0004	0.0008	0.0023
	0.2094		0.0008	0.0023

