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Solubility in supercritical carbon dioxide

Syed Asif Abdullah
New Jersey Institute of Technology

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ABSTRACT

SOLUBILITY IN SUPERCRITICAL CARBON DIOXIDE

by
Syed Asif Abdullah

The different techniques used for the determination of the solubility of compounds in supercritical carbon dioxide are described. A comparative study is made of the methods used in the measurement of the concentration of benzoic acid and caffeine in supercritical carbon dioxide. The difference in measurement techniques did not have much effect on the solubility data at lower pressures. However, at higher pressures, the solubility data obtained by different techniques began to deviate from each other.

Further, the method used by different researchers in generating the solubility data in supercritical carbon dioxide is also discussed. A number of data sheets have been prepared from these articles containing important information like the solubility, pressure range, temperature range, error in measurement of solubility, and also a brief explanation of the measurement technique. These data sheets form part of an IUPAC-NIST solubility data series project.

SOLUBILITY IN SUPERCRITICAL CARBON DIOXIDE

by
Syed Asif Abdullah

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Submitted to the Faculty of
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Master of Science in Chemical Engineering

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APPROVAL PAGE

SOLUBILITY IN SUPERCRITICAL CARBON DIOXIDE

Syed Asif Abdullah

Dr. Dana E. Knox, Thesis Advisor   Date
Professor of Chemical Engineering, NJIT

Dr. Reginald P. T. Tomkins, Committee Member   Date
Professor of Chemical Engineering, NJIT

Dr. Ralph D. Gray, Committee Member   Date
Adjunct Professor of Chemical Engineering, NJIT

BIOGRAPHICAL SKETCH

Author: Syed Asif Abdullah

Degree: Master of Science

Date: January 2007

Undergraduate and Graduate Education:

- Master of Science, Chemical Engineering
  New Jersey Institute of Technology, Newark, NJ, 2007

- Bachelor of Science, Chemical Engineering
  Regional Engineering College, Surat, India, 2003

Major: Chemical Engineering

To my niece, Zainab,

For bringing so much love and happiness

in our lives

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CHAPTER 1

INTRODUCTION

1.1 Preview

Research on solubility in supercritical fluids has grown drastically in recent years. This is due to the large range of applications of supercritical fluids in the fields of pharmaceutical, food, environmental and a host of other process industries. The fact that the properties of supercritical fluid can be varied from gas-like to liquid-like values by changing the pressure makes it very interesting and useful.

Baron Cagniard de la Tour in 1822 was said to be the first to have observed the occurrence of a supercritical phase [1]. He was able to observe that an increase in temperature of certain compounds could remove the gas-liquid boundary. This also led to the discovery of the critical point [2]. Hannay and Hogarth in 1879 were the first to display the solvating power of supercritical fluids for solids [3].

1.2 Present Scenario

The primary focus during the early research was on a single processing fluid and extractions. However, more recent work focuses on binary and multi-component fluids. [4]

Supercritical water oxidation reactions are being studied by NASA (National Aeronautics and Space Administration) for possible extraterrestrial explorations [4].

The determination of the critical temperature of systems containing binary and multi-component fluids is another area of interest, which may help to predict the chemical and physical properties and the behavior of the system with the changing environment. [4]

Solubility data obtained are often modeled with the Chrastil equation [20]. The relationship between solubility and density proposed by Chrastil is

$$s = \rho^k \exp(a/T + b)$$

where, s is the solubility (g/l) of solute in solvent, ρ is the density (g/l) of pure solvent, at empirical temperature and pressure, T is the absolute temperature, k is an average equilibrium association number, which is a characteristic constant for a given gas and solute, a is dependent on sum of enthalpies of vaporization and solvation and the gas constant, and b is dependent on the molecular weights of solvent and solute.

Other models have also been used by different authors to describe the relationship between the solubility and different thermodynamic quantities. One such model was proposed by Bartle [29], where,

$$\ln(xP/P_{ref}) = A + C(\rho - \rho_{ref})$$

$$A = a + b/T, \text{ and}$$

$$\ln(xP/P_{ref}) = a + b/T + C(\rho - \rho_{ref})$$

where, x is the mole fraction solubility, P is the pressure, P_{ref} is a reference pressure of 1 bar, ρ is the density (taken as the density of pure carbon dioxide), and ρ_{ref} is a reference density, for which a value of 700 kg m^{-3} was used.

Various other applications of supercritical fluids are discussed in this thesis. However, the main thrust of the work is on the comparative study of the measurement techniques used in the calculation of the solubility data. The analytical study makes use of the information provided by the different articles and is based on the prominence of certain factors that are more important as far as the accuracy of the data is concerned. The measurement techniques of caffeine and benzoic acid are compared in detail.

This thesis also includes 121 data sheets, which are part of the IUPAC-NIST Solubility Data Series project. These data sheets contain the solubility data of compounds soluble in supercritical carbon dioxide. A detailed description is given in chapter 4. The data sheets are attached in the appendix.

CHAPTER 2

BASICS OF SUPERCRITICAL FLUIDS

2.1 Definition of a Supercritical Fluid

When the temperature and pressure of a one-component fluid exceed its critical temperature and pressure, respectively; it is said to be supercritical.

When the difference between the coexisting liquid and vapor phase disappears, the fluid becomes critical. At this juncture, the one-phase fluid has an isothermal compressibility of infinity. A change of temperature or pressure in the supercritical region changes the phase properties of the compound. This can be achieved without the formation of a distinct interface. This easy transformation of liquid-like and gas-like properties can be done with less complexity even further from the critical point. [5]

The phenomenon shown in the Figure 2.1 on the following page [6]. Pressure is plotted on the Y-axis of the graph and temperature along the X-axis. The three different phases of the compound - solid, liquid and gas - are also shown. With an increase in temperature and pressure, there comes a point when the compound shows intermediate properties; between those of a pure liquid and a pure gas. That is the critical point. The region beyond that critical point is the supercritical fluid, which is also depicted on the graph.

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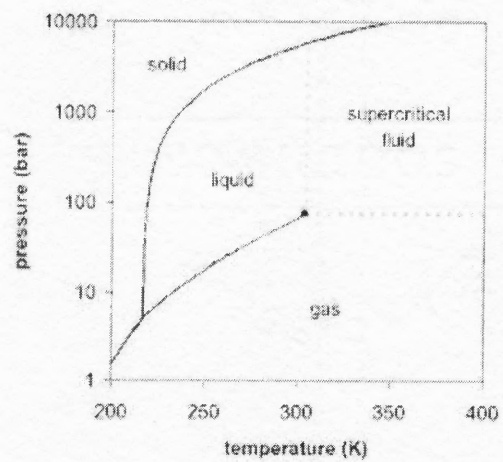
Figure 2.1 Supercritical region. [6]
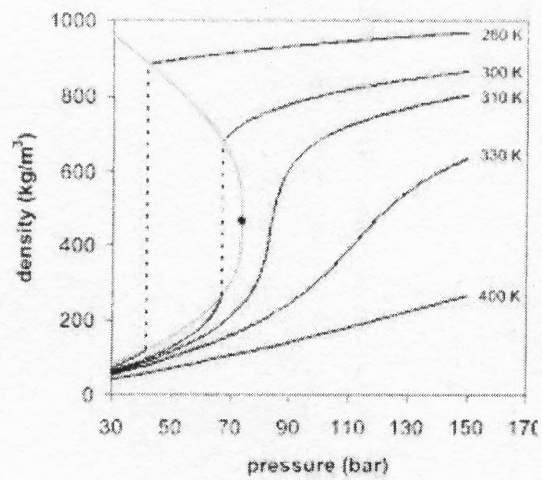
Figure 2.2 Pressure vs. Density. [6]

In Figure 2.2, the variation of density with pressure is depicted. The pressure is plotted along the X-axis and the density is plotted along the Y-axis of the graph.

The critical properties are very crucial for the study of supercritical fluids. A list of some of the most commonly used compounds is given in the table below.

Table 2.1 Critical Properties of Some Solvents [6]

Solvent	Molecular weight (g/mol)	Temperature (K)	Pressure (MPa)	Density (g/cm^3)
Carbon Dioxide	44.01	304.1	7.38	0.469
Water	18.02	647.3	22.12	0.348
Methane	16.04	190.4	4.60	0.162
Ethane	30.07	305.3	4.87	0.203

2.2 Properties of Supercritical Fluids

The properties of supercritical fluids can be explained as follows [5]:

1. Critical properties.

   A one-component fluid has the ensuing conditions of criticality

   $$(\partial P / \partial V)_T = -(\partial^2 A / \partial V^2)_T = 0$$

   $$(\partial^2 P / \partial V^2)_T = -(\partial^3 A / \partial V^3)_T = 0$$

where, A is the Helmholtz free energy

   V is the volume

P is the pressure

T is the temperature.

The condition indicate the attainment of stability.

2. Intermediate properties of vapor and liquid.

The assumption that a supercritical fluid lies between liquid and vapor can be based on the fact that the density of a supercritical fluid is intermediate between those of vapor and liquid.

3. Properties not intermediate between vapor and liquid.

Isothermal compressibility, isobaric expansion coefficient and heat capacity which exhibit extreme values near the critical point, are examples of properties of supercritical fluids that do not lie between those of the liquid and vapor phases.

Supercritical carbon dioxide has some peculiar features. It is found as a gas in air, and in dry ice carbon dioxide is observed as a solid. However, above its critical temperature (31.1°Celsius) and pressure (73 atmospheres), it behaves as a supercritical fluid with the density of a liquid. It shows properties that are midway between a pure liquid and a pure gas. [7]

Supercritical carbon dioxide has been widely used as an industrial solvent. It has a number of applications, which will be discussed in the later sections.

2.3 Phases of Supercritical Carbon Dioxide [8]

The appearance or disappearance of the phase boundary in the critical region can be quite illustrative, as can be seen in Figures 2.3 through 2.6. [8]
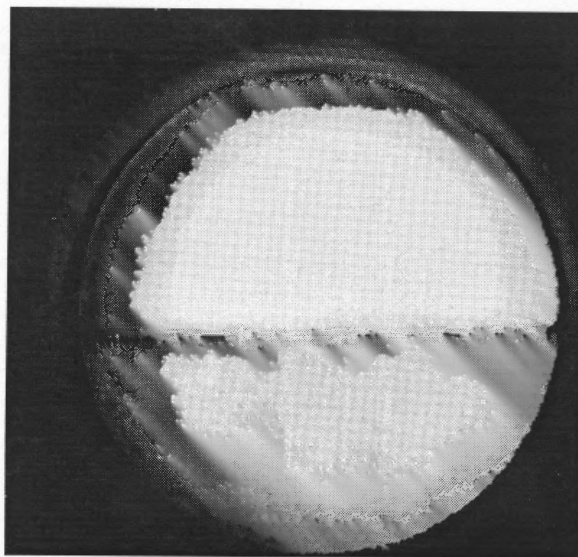
Figure 2.3 Separate phases of carbon dioxide. The separation is distinct and the meniscus is observed with ease. [8]
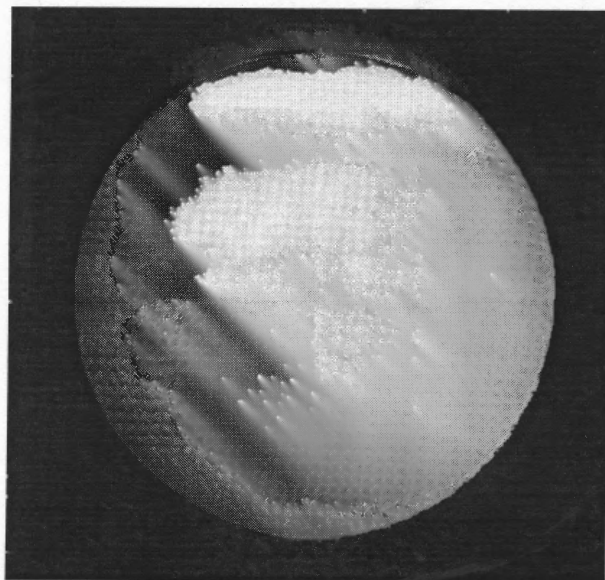
Figure 2.4 Meniscus becomes vague with increase in temperature. [8]
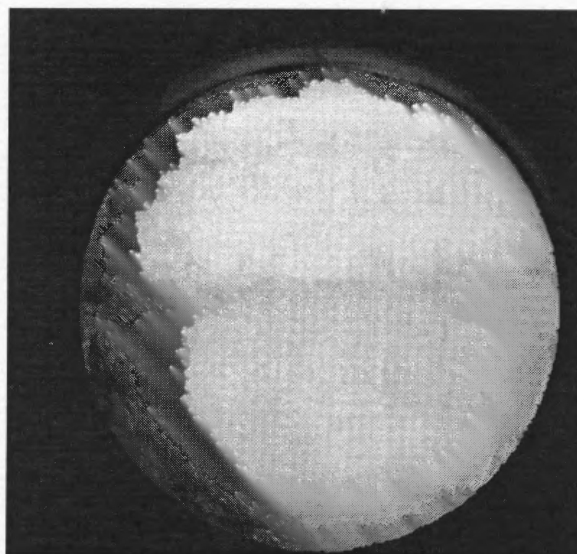
Figure 2.5 The gas and liquid becomes similar with increase in temperature. [8]
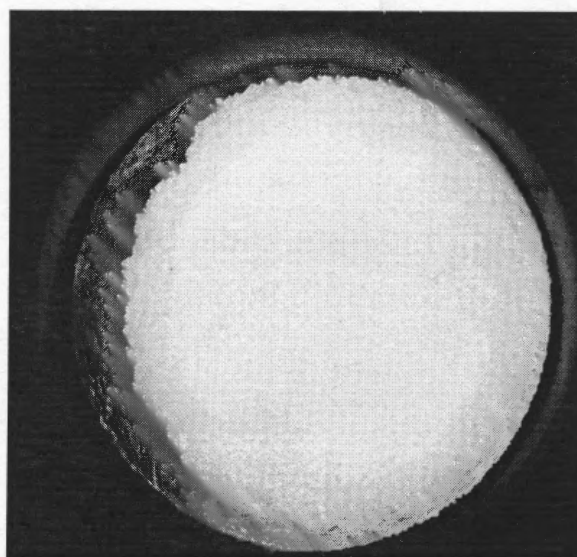
Figure 2.6 The two distinct phases are no longer seen once the critical point is reached. This homogeneous phase is the supercritical fluid having intermediate properties. [8]

2.4 Applications of Supercritical Carbon Dioxide

The application of supercritical carbon dioxide as an industrial solvent has been on the rise. This is because of its low toxicity and its usage in compound extraction. Further, it has a higher stability for the different process reactions and needs a low temperature for any process to occur compared to other solvents. [7]

Perhaps, the most common use of supercritical carbon dioxide is in the coffee industry. Supercritical carbon dioxide has replaced other solvents such as trichloroethylene, ethyl acetate, etc as a decaffeinating agent. It is mixed with the coffee beans and sprayed with water at high pressure. The caffeine can then be recycled and obtained for further use in other industries. As an example, it can be used by the cola industry by using reverse osmosis, crystallization or distillation. [7]

The application of supercritical carbon dioxide has found its way into the pharmaceutical industry. It is used in the production of micro- and nano-scale particles. [7]

Polymerization is another area that makes use of supercritical carbon dioxide. The work revolves around flouropolymer synthesis and making use of its affinity for organofluorine compounds. The latest surge in its use is the urge to replace chlorofluorocarbons which are found to be harmful to the environment. A large scale specialty flouropolymer manufacturing plant using supercritical carbon dioxide worth $40 million is being constructed by Dupont in the United States. This project if it works out well, may proceed to a $235 million facility. [9]

Supercritical carbon dioxide has proved to be a good non-polar solvent for organic compounds. Alkanes, alkenes, aromatics, and alcohols (up to a relative mass of around 400) can easily dissolve in supercritical carbon dioxide. It is seen that polar molecules such as sugars and other inorganic salts are insoluble but by increasing the pressure of the supercritical fluid, the dissolving power can be enhanced.[10]

It is also used for cleaning purposes instead of tetrachloroethylene or water. Further, supercritical carbon dioxide is used in the perfumery industry in extracting oils and fragrance compounds from raw materials.[7]

Sahle-Damessie et al. (2003) reported the increased use of supercritical carbon dioxide in the food, pharmaceutical and biotechnology industry. The reasons attributed were the increasing cost of energy-intensive separation techniques using traditional approaches and the associated environmental problems. [12]

Figure 2.7 gives an idea of an apparatus used in an extraction process by supercritical carbon dioxide. The supercritical fluid is nowadays extensively used for extraction in the industry. Carbon dioxide and ethyl acetate were mixed in an equilibrium cell and the final solution was collected in the collector trap, after passing through the adsorption/desorption column.
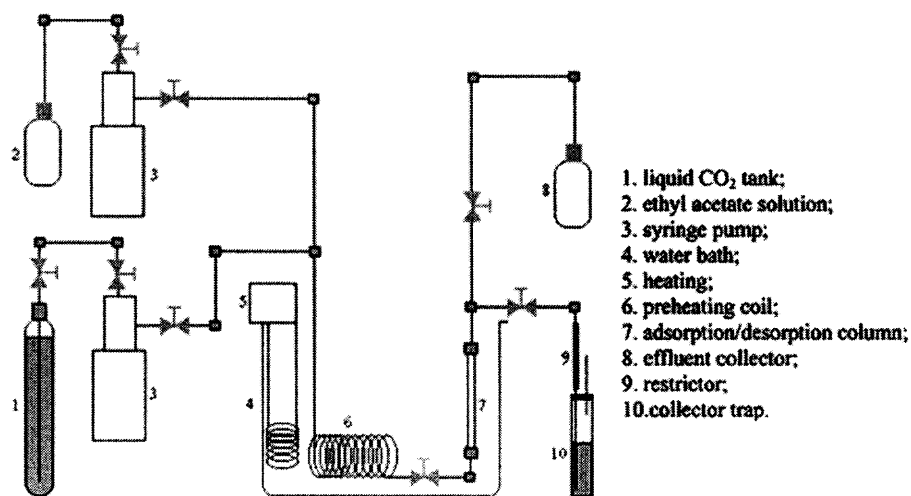
Figure 2.7 Apparatus used for adsorption and supercritical extraction of ethyl acetate from organoclays. [13]

Dragan et al. [14] stated that supercritical fluid technology has been applied in the extraction of pharmaceutical and nutraceutical materials. Supercritical carbon dioxide has replaced conventional solvents in the detoxification of organic wastes and purification of pharmaceuticals.

Shamsipur et al. [15] noted that the advantages of supercritical carbon dioxide are that it is inexpensive, non-toxic, it can be recycled, and has easily accessible critical conditions. The dyeing of polyester fabrics with supercritical carbon dioxide can be utilized as the dye dissolved in the Supercritical fluid can be recovered easily.

Sane et al. [16] discussed the reasons for the application of supercritical processing methods. An example of a drug delivery system was described, where the drug must first be dissolved into the supercritical fluid before the mobile phase can be used to impregnate the polymer matrix.

Duarte et al. (2004) [17] stated that the international regulations require that the traditional organic solvents be properly controlled and this may cause unnecessary problems for process industries. Supercritical carbon dioxide has been used as an environmentally friendly substitute.

CHAPTER 3

SOLUBILITY

3.1 Definition

Solubility can be defined as the concentration (w/w) or mole fraction of a substance in the supercritical phase at equilibrium with the pure fluid. A.W. Francis [2] investigated the solubility of 261 compounds in near-critical liquid carbon dioxide (900 psi, 25°C). He concluded that nearly half of the studied solutes were miscible with carbon dioxide.

The maximum equilibrium amount of solute which can normally dissolve per amount of solvent is the solubility of the solute in that solvent. They form two distinct heterogeneous phases. The term miscible refers to the property of various substances, particularly liquids, that allows them to be mixed together and form a single homogeneous phase.

These solubility data can be used to assess the compatibility of the supercritical fluid for a particular process application in the industry. They can also be used to determine the conditions where these fluids work best and to establish the optimized temperature, pressure and experimental measurement technique to be applied.

3.2 Measurements Techniques of Solubility

There are different techniques for measurement of solubility of compounds in supercritical carbon dioxide. Knowledge of the phase behavior is also extremely important in determining the methods to be employed in supercritical carbon dioxide. It is significant to determine the P-T (Pressure-Temperature) point in LLV (Liquid-Liquid-Vapor) and SLV (Solid-Liquid-Vapor) curves. [18]

The techniques can be dynamic, where the solute is continually carried with the supercritical fluid or static, where both solute and solvent are placed in some type of a cell. [18]

Apart from this very broad classification, there are also other methods. These are supercritical chromatographic methods, and there are bubble and dew point methods. However, static methods can also include bubble and dew point techniques. Dynamic methods may include recirculation methods, flow methods, and saturation methods. The supercritical chromatographic method is not very popular.

The location of the phase-border curves in the P-T graph and the solubility of a high molecular weight solute in a supercritical fluid can be best determined by a static method. The determination of solute solubilities in a supercritical fluid can be best carried out in a dynamic method. [18]

Dynamic method:

Solvent

Vent

THERMOSTAT
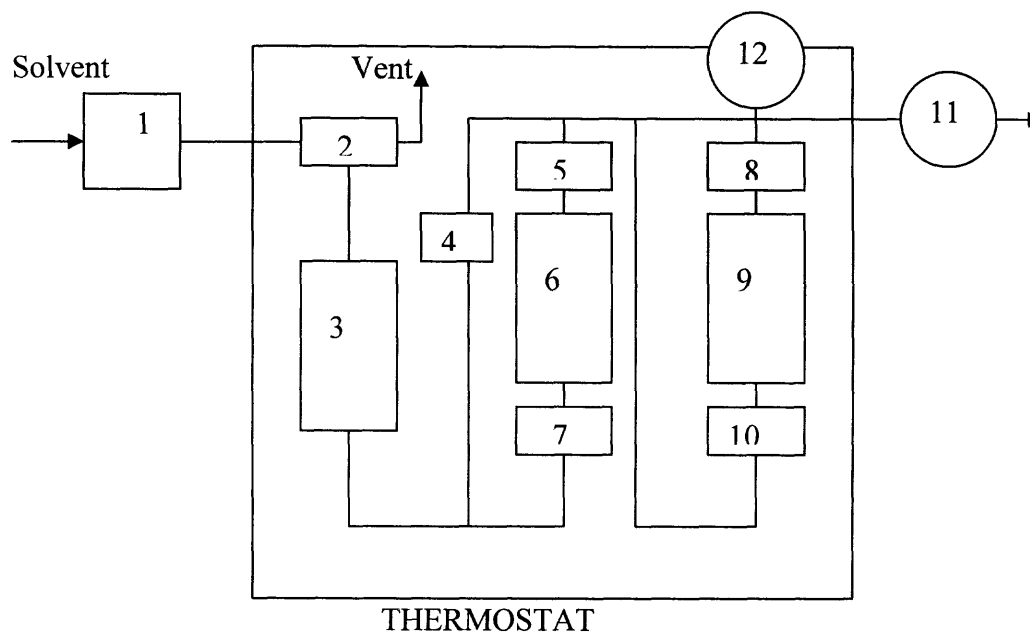
Figure 3.1 Schematic diagram of a dynamic flow apparatus. [18]
1= Feed Pump, 2,4,5,7,8,10= valves, 3= Preheater, 6,9= Equilibrium Cell,
11= metering valve, 12= pressure gauge.

A flow diagram of a dynamic method is shown in Figure 3.1 [18]. There is

no one single apparatus to represent dynamic methods. The dynamic method can

be modified according to the needs of the situation.

A basic outline of this method is explained in the work by Van Leer and

Paulaitis [18]. It embodies the main features found in any dynamic method.

A high pressure pump is used to inject liquid carbon dioxide to the system.

A high pressure liquid chromatographic pump is used in most cases. Carbon

dioxide can also be supplied with a compressor. The flow rate is adjusted to the system depending on the required equilibrium time. The entire system should be optimized to minimize loss of energy and materials.

The solute is placed in a constant temperature bath. The carbon dioxide must pass through the tubings before it can reach the equilibrium cell where the solute is kept. The high pressure columns allow carbon dioxide to pass slowly and for saturation of the solute to take place. The column is packed with glass wool plugs or other similar packing systems. The carbon dioxide after the second cell is expanded to atmospheric pressure, causing the solute to come out of solution. The heavy component is collected in a trap.

The solute is measured gravimetrically or spectrophotometrically. A rotameter or bubble-flow meter is used to measure the flow rate. A dry-test meter or wet-test meter is used to measure the volume of carbon dioxide.

There are many advantages to a dynamic method. It has a simple sampling procedure. It also has a high reproducibility and it takes less time for equilibrium to occur.

The disadvantages include the clogging due to the pressure of solutes. Entrainment can also occur in the presence of a liquid solute. There are chances of an undetected phase change occurring in the saturation columns.

However, these drawbacks can be corrected and modified for different applications.

As mentioned earlier, the dynamic method can be further classified as a recirculation method, flow method or saturation method.
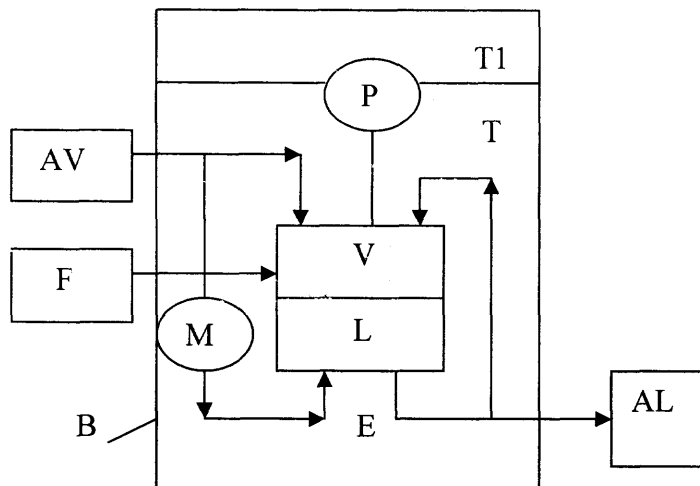
Figure 3.2 Dynamic recirculation method [19]
AL- Batch analysis of liquid phase, AV- Batch analysis of vapor phase, B-
Boundary of thermostated compartments, E- Equilibrium cell, F- Batch feed of
degassed components, L- Liquid phase, M- Pump, P- Pressure meter, T-
Temperature of the cell, T1- Temperature of pressure sensor, V- Vapor phase.

In the recirculation method, the main cell has an external mechanically

operated flow. It is the intermediate between a static cell and a fully developed

dynamic method. This external circulation helps in the proper and uniform mixing

of the solute and the solvent and helps to reduce equilibrium time. It can also

minimize clogging compared to a static cell. However, the drawback is that it can

lead to the appearance of dead volumes and more leaks. [19]
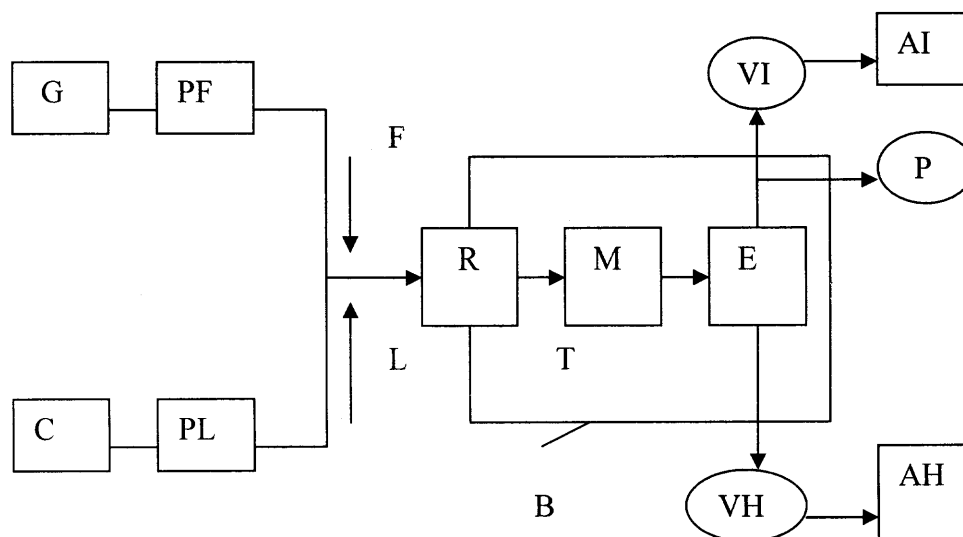
Figure 3.3 Flow method [19]
AH- Analysis of heavier phase, AI- Analysis of lighter phase, B- Boundary of thermostated compartment, C- Solute container, E- Equilibrium cell, F- Steady flow of pure supercritical fluid, G- Liquefied gas tank, L- Steady flow of pure liquid solute, M- Mixer, PF- Pump for solvent fluid, PL- Pump for solute liquid, R- Preheater, T- Temperature, VH, VI- Metering valve.

The flow method is good for unstable compounds. It reduces the residence time of the mixture and also the chances of thermal decomposition. A significant disadvantage of this method is that the pressure fluctuations get suppressed and it cannot be measured properly. [19]

The saturation or transpiration method is the most widely used in the supercritical processing industry. Even a very low volatility can be measured. [19]
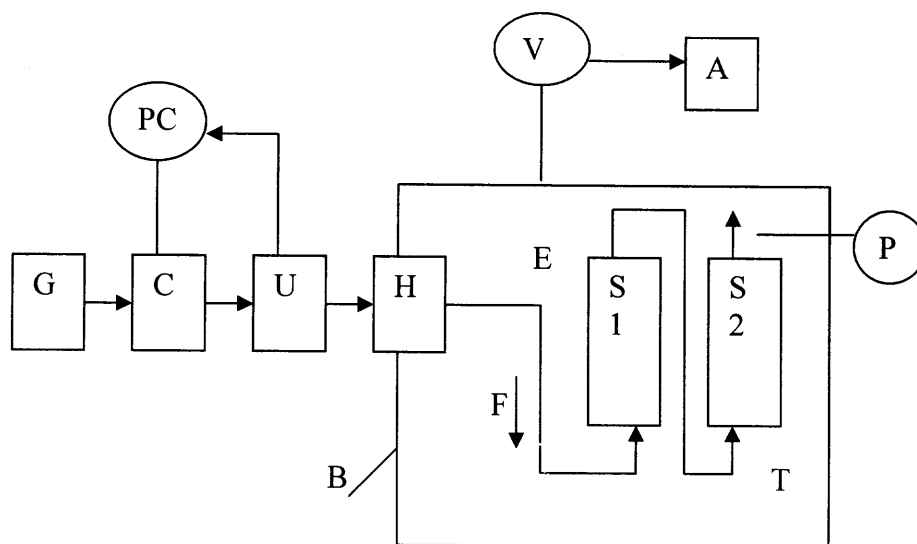
Figure 3.4 Dynamic saturation (transpiration) method [19]
A- Analysis (of de-pressurized fluid phase stream), B- Boundary of thermostated compartment, C- Compressor, E- Steady stream of supercritical fluid saturated with solute, F- Steady flow of pure supercritical fluid solvent, G- Gas cylinder, H- Preheater, P-pressure meter, S1,S2- Saturators (equilibrium columns), T- System temperature, U- Surge tank, V- Metering valve

The non-volatile phase is loaded batch-wise in a saturator, or, in a series of saturators. It stays as a stationary phase throughout the experiment. The saturation time is fixed after repeating the tests a number of times. A steady stream of supercritical fluid passes through a preheater. It is then fed to the saturator, the stationary heavy phase is stripped from the solute and leaves the saturator. [19]

The solubility of the heavy phase is determined by either direct sampling and analysis of the effluent stream or from the total volume of the gas passed through the saturator and from the known mass of solute extracted during a

sample-collecting period [19]. The saturation time can be determined after repeating the experiment 2-3 times .

Static method:
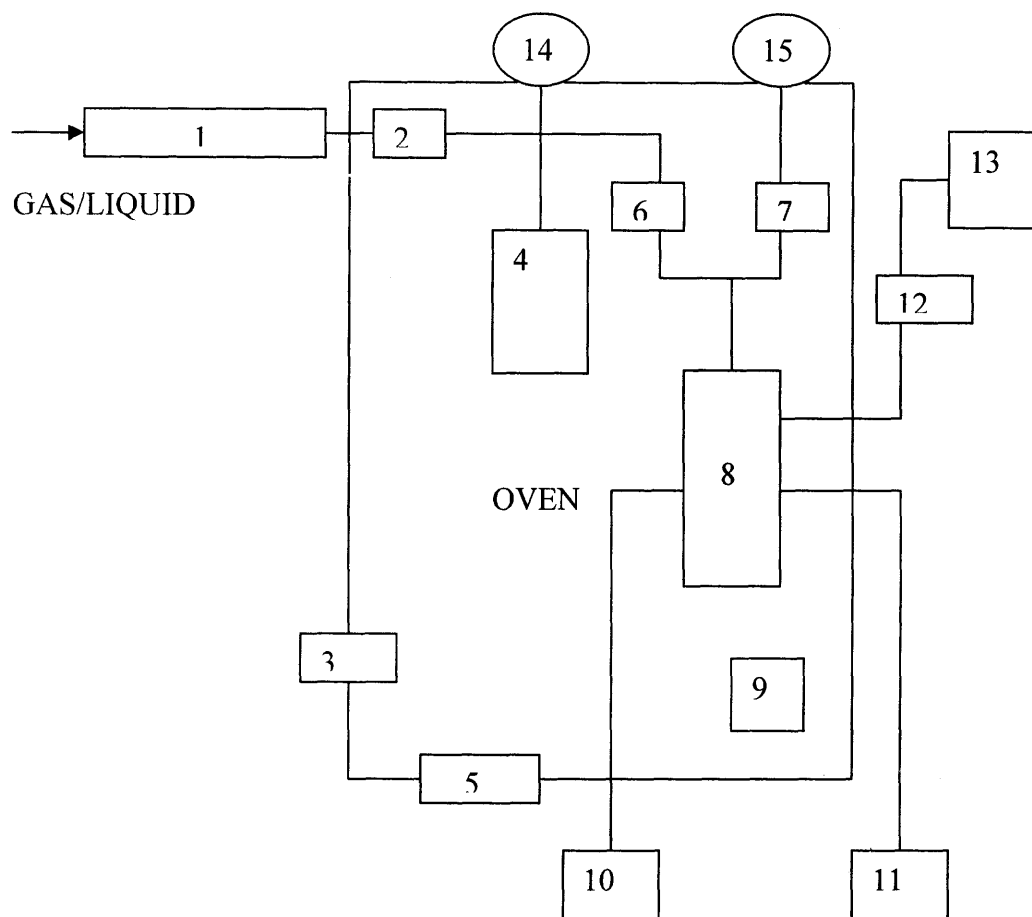
Figure 3.5 Schematic diagram of a constant-composition view cell apparatus [18]
1= compressor, 2,6,7,12= valves, 3= fan, 4= holding tank, 5= heating coil,
8= equilibrium cell, 9= magnetic stirrer, 10= fiber optics, 11= temperature readout, 13= pressure generator.

In a static method, both the solute and the solvent are placed in some type of a cell. A constant temperature environment is created with a thermostat. A modified static apparatus is used for the calculation of the solubility data for different compounds. The solute remains enclosed in the cell and no phase undergoes a mechanically driven flow. [19]

Figure 3.5 depicts the basic static apparatus according to the apparatus described by McHugh, Seckner, and Yogan [18]. The main component of the system consists of a variable volume view cell. Carbon dioxide is passed through a compressor and then placed in a tank.

The pressure of the system is measured by a Bourdon-tube Heise gauge. The gas is kept in the tank until it reaches equilibrium. The gas is then mixed with solute in the equilibrium cell.

A magnetic stirrer is used for proper mixing. The operating pressure is regulated with the help of a piston. Isothermal conditions are maintained using a thermostat. The sample can be collected and measured gravimetrically or spectrophotometrically.

Static methods can be classified as either analytical or synthetic methods. In a synthetic method, the composition is known from the beginning but in an analytical method, the initial compositions are not known. They are analyzed during the process depending upon the needs. Both these methods are used in the supercritical processing technology based on the objective of the study and the properties of the compounds.

Apart from these methods, bubble and dew point methods may also be used for the determination of the solubility in supercritical fluids. They are considered as synthetic methods. They are based on the location of a phase transition state and the pressure is determined at the point where the total composition at isothermal conditions is equal to the value of the phase, which was set from the beginning. This can be used for both low- and high-pressure systems. [19]

Finally, the last technique of measurement, which is different from all the above-mentioned methods, is supercritical fluid chromatography. It makes use of chromatographic techniques to determine the solubility data.
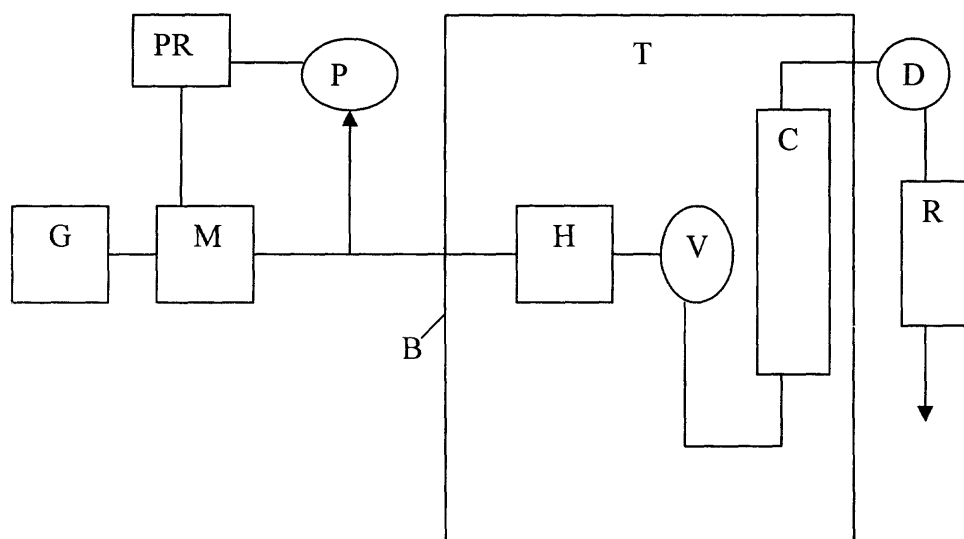
Figure 3.6 Supercritical fluid chromatography assembly. [19]
B- Boundary of thermostated compartment, C- Column, D- Detector, H- Preheater, M- Pump, P- Pressure transducer, PR- Pressure programmer, R- Restrictor, T- System temperature, V- Injection valve.

The operation of supercritical fluid chromatography method is simple. The supercritical fluid flows at high pressure through a column where the stationary phase is loaded. The solute is injected and is distributed between the mobile and the stationary phases. [19]

This technique involves the relationship between the retention time and the equilibrium properties. It requires only a small amount of solute and can be used for complex mixtures and organic solvents. [19]

CHAPTER 4

SOLUBILITY DATA EVALUATION

4.1 Methodology

Due to the widespread application of supercritical carbon dioxide in industry, a great deal of research work has been carried out for a range of solutes in this solvent. The data on solubility are needed for the design of industrial processes. The measurement techniques also differ for different compounds and for different sets of conditions.

There are many articles on the above topic. However, access to most of them is not available due to patent and other issues. In this research, the articles [14-18, 20-58] were collected using Scifinder Scholar. It is a search tool from ACS (American Chemical Society) that displays abstracts from the literature. A step-by-step approach was followed in this research.

The articles were identified from Scifinder Scholar on the topic "Solubility in supercritical carbon dioxide". They were sorted out for the period 2003 - June 2005. Article titles and abstracts were obtained and these abstracts were then studied and, based on the availability of solubility data for different temperature and pressure ranges, pertinent articles were identified for retrieval using the resources of the NJIT library.

These articles were then used to produce data sheets containing all the relevant information pertaining to the compound, such as author names, year of

publication, name of the journal, temperature and pressure range, solubility data in mole fraction, measurement techniques, source and purity of materials, error in the calculation of temperature, pressure and solubility, etc. Calculations were sometimes required to convert reported values to the units used for the data sheets.

There was not a fixed pattern in the type of compounds whose solubility data were obtained. Some of the compounds were:

- ➢ Terfenadine
- ➢ Griseofulnin
- ➢ Caffeine
- ➢ Benzoic acid
- ➢ Naphthalene, etc

Various possible classifications of the compounds were considered and five approaches were short-listed:

1. Based on the number of carbon atoms: 10-20, 20-30, 30-40 etc.

2. Compounds which are hydrocarbons or non-hydrocarbons.

3. Compounds containing elements like O, N, S etc besides carbon and hydrogen.

4. Based on application as in, drug industry, food industry, polymeric industry, etc.

5. Compounds which are aliphatic or aromatic.

Finally, these compounds were classified based on the compounds containing C, H, O, N. This classification was found to be the most acceptable for uniform distribution.

A total of 121 data sheets were compiled for compounds that are soluble in supercritical carbon dioxide covering the period 2003 - June 2005. These data sheets form part of the IUPAC-NIST Solubility Data Series project. The classification of these compounds are as follows:

1. Compounds containing C-H-O/C-H: 59 data sheets

2. Compounds containing C-H-O-N/C-H-N: 34 data sheets

3. Others: 28 data sheets.

4.2   Measurement Techniques

The articles retrieved for the period 2003-June 2005 for solubility in supercritical carbon dioxide contained different techniques for obtaining the solubility data.

Matias et al. [20] measured the solubility of the coenzyme Q10 in supercritical carbon dioxide. A static analytical method was used for a pressure range of 9 to 26 MPa and at temperatures of 305, 313 and 323K. The system consisted of a carbon dioxide compressor, rupture disk, thermostated water bath, six port sampling HPLC-valves, a pressure transducer, temperature controller, equilibrium cell with sapphire windows, glass trap and expansion calibrated cylinder. Ethanol was used as a cosolvent and the solubility effect was analyzed in terms of the entire system. The solubility was increased when ethanol was added and it kept on increasing with the increase in temperature. The average absolute relative deviation in the calculation of the solubility data in mole fractions was reported as 6%. The extraction of Q10 was also observed with supercritical

carbon dioxide at 323K and 15 MPa. The Chrastil model was used for the correlation of the experimental data and a high level of consistency was achieved.

Sahle-Demessie et al. [12] studied the solubility of organic biocides in supercritical carbon dioxide and also with cosolvent mixtures. A dynamic flow apparatus was used over a pressure range of 10 to 30 MPa and a temperature range of 35 to 80°C. The four compounds whose solubility were measured are amical-48 (diiodomethyl p-tolyl sulfone), chlorothalonil (tetrachlorolsophthalonitrile), TCMTB (2-(thiocyanomethylthio) benzothiazole), and tebuconazole (alpha-[2-(4-chlorophenyl)ethyl]-alpha-(1,1-dimethyl-ethyl)-1H-1,2,4-triazole-1-ethanol). The density of the solvent was used to correlate the measured solubilities using the Chrastil model. Methanol and acetone were used as cosolvents and their effect on the solubility was discussed. It was found that the bulk properties of the fluid changed with the increase in concentrations of the cosolvent. The overall accuracies of the temperature and pressure measurements were $\pm$ 0.1°C and $\pm$ 0.1 MPa. The average standard deviation of the solubility data was $\pm$ 1.3%.

Dragan et al. [14] studied the solubility of drugs in supercritical carbon dioxide and examined the effect of entrainers. The drugs investigated were terfenadine and griseofulvin. The solubility data were given for a pressure range of 140-180 bar and at temperatures of 313.2K and 343.2K. The results were modeled with the Chrastil approach. Ethanol was studied as a co-solvent and its effect was found to increase the overall solubility of the solute. The experimental system consisted of a carbon dioxide tank, pressure gauge, gas compressor, safety

valves, heat exchanger, backpressure regulator and flowmeter. The experimental errors in the measurement of temperature and pressure were ± 1°C and ± 5 bar respectively. The average standard deviation of the solubility data for terfenadine was 0.55%-7.35% and for griseofulvin was in the range 1.09%-11.9%.

Ghaziaskar et al. [21] studied the solubility of hexanoic acid and butyl acetate in supercritical carbon dioxide. The pressure range was 75-180 bar and the temperature range was 313-353K. Hexanoic acid had less solubility than butyl acetate. Volatility of butyl acetate was found to be higher than hexanoic acid. A constant flow rate of 120 ± 5 ml/min was maintained throughout the experiment. The system calculations were done at equilibrium conditions. A continuous-flow type apparatus was used consisting of a carbon dioxide cylinder, filter, cooling coil, reciprocating HPLC pump, oven, preheating coil, 6-port two-position switching valve, equilibrium cell, temperature controller, collection flask and a wet gas meter. The overall accuracy of the temperature and pressure was ± 0.5K and ± 1 bar.

Jara-Morante et al. [22] measured the solubilities of imipramine HCl in supercritical carbon dioxide. The major application of this drug is in the treatment of depression. A gravimetric technique was used in the solubility determination. The major components of the apparatus were a syringe pump, a thermostatic chamber, an equilibrium cell, a variable flow-rate restrictor, and an ice trap. The measurements were done for a pressure range of 30-50MPa and at temperatures of 40 and 50°C. In order to ensure consistency, the solute concentration was measured both gravimetrically and spectrophotometrically. The error in the

experimental measurement was around 30%. The main source of error was the quantification of the collected mass of the solute.

Xing et al. [23] studied the solubility of artemisinin in supercritical carbon dioxide. This drug is used for the treatment of cerebral malaria. The pressure range was 10 to 27 MPa and at temperatures of 310.1 to 338.1 K. A flow type apparatus with a high pressure UV detector was used. A wavelength of 210 nm was fixed. The time required to reach this equilibrium was within 5-7 minutes. The accuracy of the temperature and pressure were within $\pm$ 0.1K and $\pm$ 0.1 MPa. The data were correlated with a semi-empirical model and an average absolute relative deviation of 4.24% was obtained.

Ghaziaskar et al. [24] measured the solubility of 2-ethyl-1-hexanol, 2-ethylhexanoic acid, and their mixtures in supercritical carbon dioxide. The data were obtained for a pressure range of 68 to 180 bar for temperatures of 313 and 323 K for the binary systems and at a pressure of 138 bar and a temperature range of 313 to 373 K for the ternary systems. The Chrastil model was used for correlation. A flow type apparatus was used where liquid carbon dioxide was transferred from a cylinder using a HPLC pump. The other components of the measuring apparatus consisted of washing liquid bottle, valve, carbon dioxide purifier, liquid carbon dioxide pump with cooled head, pressure transducer, six-port two-position valve, prewarming coil, heating coil, thermocouple and temperature controller, oven, equilibrium cell, backpressure regulator, collection vial and wet gas meter. The solubility data show an increasing trend with increase in pressure with more solubility for alcohol than acid. The deviation in

temperature and pressure were ± 0.1K and ± 1 bar respectively, whereas the relative standard deviation for the solubilities was less than ± 10%.

Shamsipur et al. [15] measured the solubilities of some aminoanthraquinone derivatives in supercritical carbon dioxide. The four synthesized derivatives disperse dyes were, 1-amino-2-methyl-9,10-anthraquinone (A1), 1-amino-2-ethyl-9,10-anthraquinone (A2), 1-amino-2,3-di-methyl-9,10-anthraquinone (A3) and 1-amino-2,4-di-methyl-9,10-anthraquinone (A4). The temperature range was 308-358K and the pressure range was 122-358 bar. A modified static system with glass beads packing was used for the determination of the solubility and the data were correlated using an empirical model. The solubilities calculated in terms of mole fraction had a deviation of ±5%. An equilibration time of 30 minutes was obtained. The data were obtained using wavelengths of 474 nm and 579.4 nm. The solubilities of the derivatives were in the order A3> A1> A2 >>A4.

Shamsipur et al. [25] studied the solubilities of some thioxanthane derivatives in supercritical carbon dioxide. The data were obtained for a pressure range of 122 to 355 bar and at temperatures of 308, 318, 328, 338 and 348 K. The compounds were thioxanthone (T1), 1-hydroxythioxanthane (T2), 1-hydroxy-3-methylthioxanthone (T3) and 1,4-dihydroxy-3-methyl-thioxanthone (T4). A modified simple static system was used. The accuracies for the equilibrium temperature and pressure were ± 1K and ± 0.5 bar respectively. The standard deviation for the solubility data was ±3%. An absorbance method was used for the

determination of data for T1 to T3 where as fluorescent intensity measurements were used for T4 determination.

Kiyoshi et al. [26] studied the solubilities of 7,8-dihydroxyflavone and 3,3',4',5,7-pentahydroxyflavone in supercritical carbon dioxide. They were measured for a pressure range of 9.1-25.3MPa and at temperatures of 308.2K and 318.2K. A flow type apparatus was used for this purpose. The temperature and pressure control had a variation of ± 0.1K and ± 0.3% respectively. The standard deviation of the solubility data was ± 4.0%.

Khosravi-Darani et al. [27] measured the solubility of poly (beta-hydroxybutyrate) in supercritical carbon dioxide. The pressure range was 122 to 355 bar and the temperature range was 308 to 348 K. The Chrastil model was used for correlation. A modified static system with supercritical fluid extraction mode was used. Equilibration time was minimized as this method increased the contact surface between the sample and the supercritical fluid. The deviation in temperature and pressure was ± 1K and ± 0.5 bar respectively. The accuracy of the solubilities varied around ± 5%. The pressure effect on the solubility was more at higher temperatures.

Leeke et al. [28] worked on the solubilities of p-tolylboronic acid, bromobenzene, and 4-phenyltoluene in carbon dioxide at elevated pressures. The pressure range was 98 - 317 bar and temperature range was 353 – 383 K for the experiment. The Chrastil model was used for correlation and found to have a high level of accuracy. A cloud point apparatus was used for the measurement of the solubility data. It consisted of a pressure generator, view cell, cold collector,

stirrer, flow totalizer, and camera and monitor. The deviation of temperature and pressure was $\pm 0.1°C$ and ± 1.1 bar respectively.

Bahramifar et al. [29] measured the solubilities of some 2H-chromene derivatives in supercritical carbon dioxide. The compounds were 2H-1-benzopyran-2,3-dicarboxylate (A1), di-tert-butyl-3H-naphtho[2,1-b]pyran-2,3-dicarboxylate (A2), dimethyl 6-bromo-2H-1-benzopyran-2,3-dicarboxylate (A3), and dimethyl –3H-naphthol[2,1,b]pyran-2,3-dicarboxylate (A4). The temperature range was 308K to 348K and the pressure range was 12.2 to 35.5 MPa for the experiments. Naphthalene solubility data was used to test the accuracy and the efficiency of the system. A modified static system was used. The accuracy of the temperature and pressure was $\pm 1K$ and $\pm 0.1MPa$ respectively. The deviation of the solubility was $\pm 2\%$.

Sane et al. [16] discussed a semi-continuous flow apparatus for measuring the solubility of solids in supercritical solutions. The experiments were conducted at temperatures 40, 70 and 100 °C and in a pressure range of 103.4-324.1 bar. The compounds investigated were phenanthrene and 5,10,15,20-tetrakis (3,5-bis(trifluoromethyl)phenyl)porphyrin (TBTPP). The overall deviation in the temperature and pressure measurements was ± 0.2 °C and ± 1 bar respectively. The accuracy in the solubility measurements was within $\pm 5\%$. This technique is most suitable for pharmaceutical solids having mg-sized quantities and also for dyes and porphyrins which form opaque solutions when dissolved in supercritical solvents. TBTPP proved to be an appropriate solute for studying the effect of

concentration on rapid expansion processing due to its wide range of solubilities in supercritical carbon dioxide.

Duarte et al. [17] measured the solubility of flurbiprofen in supercritical carbon dioxide. The measurements were done for a pressure range of 8.0 to 25.0 MPa and at temperatures of 303, 313 and 323K. Ethanol was used as a co-solvent and was studied at 18 MPa and 313K. This is significant for predicting solubilities in supercritical fluid technologies. A static analytical high-pressure apparatus was used consisting of a carbon dioxide compressor, rupture disk, thermostatic water bath, six port sampling HPLC valve, manual syringe, pressure transducer, temperature controller, equilibrium cell with sapphire windows, glass trap and an expansion calibrated cylinder. The variation in temperature and pressure were ± 0.1°C and ± 0.04 MPa respectively.

Garmroodi et al. [30] measured the solubilities of the drugs benzocaine, metronidazole benzoate, and naproxen in supercritical carbon dioxide. The temperature and pressure range were 308 to 348 K and 122 to 355 bar respectively for the experiments. An empirical model was used for correlation of the obtained solubility data. A static system was used with a standard deviation of ±4% for at least three replicated measurements. The method of absorbance was used for evaluation of solubilities. High solubility data were obtained which implied that supercritical fluids could be used as alternatives to organic solvents in the impregnation process.

Tamura et al. [31] studied the binary and ternary solubilities of disperse dyes and their blend in supercritical carbon dioxide. The compounds were C.I.

disperse blue 134 (1,4-bis(isopropylamino)anthraquinone, C.I. disperse yellow 16 (3-methyl-1-phenyl-5-pyrazolone) and their dye mixtures. The measurement was done for a pressure range of 10.0 to 25.0 MPa at temperatures of 323.15 to 383.15 K for binary systems and at 383.15 K for ternary systems. The accuracy for temperature and pressure were ± 0.1K and ± 0.2 MPa respectively. The deviation in the solubility was ±4%.

Ferri et al. [32] worked on an experimental technique for measuring high solubilities of dyes in supercritical carbon dioxide. The compounds were (C.I.) disperse orange 3, red 324, blue 79 and quinizarin. The measurements were carried out at the temperatures of 353.2, 373.2, 393.2K and a pressure range of 18 to 30 MPa. A modified flow apparatus consisting of the three sections of compression, equilibrium and expansion was used. The system comprised a carbon dioxide cylinder, cryogenic bath, on-off valve, regulating valve, restrictor, extractor, heating coil, pump and solvent trap. The accuracy of solubility measurements was within ± 4%.

Sauceau et al. [33] measured the solubility of eflucimibe in supercritical carbon dioxide with and without a co-solvent. The values were calculated at 308.15 and 318.15 K and the pressure range was 8-30 MPa. The two co-solvents used were ethanol and dimethylsulphoxide. Molecular interactions and solvent density play a pivotal role in the enhancement of solubility with the addition of the two co-solvents. A continuous flow apparatus was used consisting of 2-way valves, high pressure pumps, mixer, checking valve, heater, thermostated oven, heat exchanger, 6 way-2 position valve, equilibrium cell, temperature transducer,

volume stirred vessel with visual access for determining the cloud point. Pindolol was the only compound found to be soluble in carbon dioxide under the observed conditions. The lower basicity of pindolol played a role in preventing its reaction with carbon dioxide.

Vatanara et al. [37] measured the solubility of some inhaled glucocorticoids in supercritical carbon dioxide. The temperature range was 338 to 358 K and the pressure range was 213 to 385 bar. The three compounds were beclomethasone, dipropionate and budesonide. A semi-emperical model was used for correlation. The low solubility of these compounds was indicative of the fact that supercritical antisolvent methods may be suitable for particle production from the three compounds.

Huang et al. [38] studied the solubility of cholesterol and its esters in supercritical carbon dioxide with and without co-solvents. The cholesterol esters are cholesteryl acetate, cholesteryl butyrate and cholesteryl benzoate. A dynamic flow method was used for the solubility determination. The deviation in temperature and pressure was $\pm$ 0.01°C and $\pm$ 0.2% respectively. The standard deviation in solubility was $\pm$ 1%. The difference in structure of the three esters contributed to the large differences in the solubility data. There is also an indication of more molecular interaction with the addition of a co-solvent.

Khajeh et al. [39] measured the solubilities of some phenyl derivatives of dialkyl 1,4 – dihydro - 2, 6 – dimethyl – 4 - (1-methyl-5-nitro-imidazol-2-yl)-3,5-pyridinedicarboxylates in supercritical carbon dioxide. The temperature ranged from 338 to 358 K and the pressure from 12.2 to 35.5 MPa for the experiments.

A modified static system was used for solubility determination with temperature and pressure variation of ± 1K and ± 0.1 MPa, respectively. The relative standard deviation was ± 7%.

Asghari-Khiavi et al. [40] measured the solubilities of two steroids and their mixtures in supercritical carbon dioxide. The steroid drugs were medroxyprogesterone and cyproterone acetate. The temperature range was 308 to 348 K and the pressure range was 122 to 355 bar. A modified static system was used which had a temperature and pressure accuracy of ± 1K and ± 0.5 bar respectively. The standard deviation for the solubility data was ± 4%. Research pertaining to the separation of binary mixtures was also accomplished.

Pauchon et al. [41] worked on a new apparatus for the dynamic determination of solid compound solubility in supercritical carbon dioxide by studying triphenylmethane. The technique was validated by measuring the solubilities of naphthalene. The measurements for triphenylmethane were carried out at 308.15, 318.15 and 328.15K and for the pressure range of 65 to 280 bar. A dynamic saturation method was used consisting of a carbon dioxide cylinder, ice-water bath, HPLC pump, pressure regulator, autoclave, safety pressure gauge, pressure gauge, check valve, saturation cell, six-port valve, mercury autoclave, reducing valve, mercury tank, graduated cylinder, piston pump, silica column, UV spectrophotometer, volumetric flask, head loss, thermostated bath and valves. The average standard deviation was 6%.

Shamsipur et el. [42] measured the solubilities of some 1-hydroxy-9,10-anthraquinone derivatives in supercritical carbon dioxide. The pressure range was

12.2-35.5 MPa and at temperatures of 308, 318, 338 and 348K. The seven compounds were 1-hydroxy-9,10-anthraquinone (AQ1), 1-hydroxy-2-methyl-9,10-anthraquinone (AQ2), 1-hydroxy-2-(methoxymethyl)-9,10-anthraquinone (AQ3), 1-hydroxy-2-(ethoxymethyl)-9,10-anthraquinone (AQ4), 1-hydroxy-2-(1-propoxymethyl)-9,10-anthraquinone (AQ5), 1-hydroxy-2-(1-butoxymethyl)-9,10-anthraquinone (AQ6), 1-hydroxy-2-(n-amyloxymethyl)-9,10-anthraquinone (AQ7). A modified static system was used for solubility determination. The accuracy in measurement of equilibrium temperature and pressure was ± 1K and ± 0.1 MPa respectively. The average standard deviation was ± 5%.

Chen et al. [43] studied the solubility measurement of alpha-asarone in supercritical carbon dioxide. The pressure range was 9.0 to 18.0 MPa and the temperature range was 35-49°C. The Chrastil model was used for correlation. A dynamic method was used with the system consisting of a carbon dioxide supply tank, needle valve, pressure sensor, compressor, cell, filtration and mini-regulator, temperature and pressure sensor, heat preservation jacket, constant temperature tank, thermocouple, pressure equilibrium cell, sampling valve, U-shape tube, rotor flowmeter and wet flowmeter.

Asghari-Khiavi et al. [44] measured the solubility of the drugs bisacodyl, methimazole, methylparaben, and iodoquinol in supercritical carbon dioxide. The temperature range was 308 to 348 K and the pressure range was 122 to 355 bar. A simple and reliable static method was used with a standard deviation of ± 3%. An extraction chamber was placed with a packing of glass beads. The uncertainties in

the measurement of temperature and pressure was ± 1K and ± 0.5 bar respectively.

Li et al. [45] measured the solubility of the solid solutes 2-naphthol and anthracene solutes in supercritical carbon dioxide with and without co-solvents. The measurements were carried out for a pressure range of 10-30 MPa and at temperatures 308.1, 318.1 and 328.1 K in a flow apparatus. The effect of three co-solvents, namely, acetone, ethanol and cyclohexane, was also discussed. The variation in the accuracy of the temperature and the pressure was ± 0.1 K and ± 0.5 bar, respectively. The average standard deviation was ± 2%.

Gaspar et al. [46] studied the solubilities of echium, borage, and lunaria seed oils in compressed carbon dioxide. The measurement of the solubility data were carried out for a temperature and pressure range of 10 to 55°C and 60 to 300 bar respectively. The overall accuracy in the temperature and pressure was ± 0.5°C and ± a bar. The average and maximum standard deviations were 4% and 9% respectively. A single pass flow apparatus was used and the data was correlated with the help of the Chrastil model. Echium oil was found to have the highest solubility for the same range of temperature and pressure among all other compounds.

Ghaziaskar et al. [47] measured the solubility of 2-ethyl-hexyl-2-ethyl hexanoate in binary and quaternary systems in supercritical carbon dioxide. These were measured for different sets of conditions at a constant flow rate of 250 ± 10 ml/min. The pressure range was 90-253 bar for the measurement at temperature 313K, the temperature range was 313-353K for the measurement at a pressure of

170 bar and finally, at a pressure and temperature of 170 bar and 313K respectively for a flow rate range of 100-300 ml/min. A continuous flow apparatus was used with components consisting of a carbon dioxide cylinder, filter, cooling coil, reciprocating HPLC pump, equilibrium cell, 6-port, two position valve, back pressure regulator, collection flask, temperature controller and oven. The standard average deviation was around ± 10%. The method was validated by measuring the solubility of oleic acid at 50°C and 151 bar. Acid and alcohol was found to display a higher solubility than 2-ethyl-hexyl-2-ethyl-hexanoate.

Huang et al. [48] measured the solubility of aspirin in supercritical carbon dioxide with and without acetone. The pressure range was 10 to 25 MPa and temperature was 308.15 to 328.15 K for the experiments. Addition of acetone as a co-solvent resulted in the increase of the solubility by a factor of five. A dynamic method was used for determination of solubility consisting of carbon dioxide cylinder, circulating freezer, HPLC pump, switching valve, premixing coil, equilibrium vessel, oven, back-pressure regulator, collection U-tube, saturator, wet gas meter, and cosolvent flask. The estimated error in the measurement of temperature and pressure was ± 0.01K and ± 1 bar respectively. The average standard deviation did not exceed 6%.

A few articles on the measurements in supercritical carbon dioxide were obtained beyond the year 2003 in order to assess the work done on this topic in the previous years. These are described briefly in the following paragraphs.

Galia et al. [49] worked on a new simple static method for the determination of solubilities of condensed compounds in supercritical fluids. The compounds studied were benzenecarboxylic acid, 2-hydroxy-benzenecarboxylic acid, 4-hydroxy-benzenecarboxylic acid and 1,8-dihydroxyanthraquinone. The temperature range was 318-357 K and the pressure range was 7-23 MPa. A simple static method based on gravimetric measurements was used consisting of a pressure transducer, thermowell, metering valve, steel cylindrical containers, vessel magnetic stir bar, perforated steel platform and container magnetic stir bar. Steps must be taken to avoid turbulence, which can lead to increase in experimental errors.

Sovova et al. [50] measured the solubility of beta-carotene in supercritical carbon dioxide and the effect of entrainers. The measurements were done for pressures up to 28 MPa and at temperatures 313.2, 323.2 and 333.2 K. A flow pass flow apparatus was used consisting of a compressor, pressure regulator, vessel with ethanol solution, extractor, switching valve, micrometer valve, trap and gas meter. A flow rate of 40-50 ml/min was maintained. The Chrastil model was used for correlation.

Mishima et al. [51] worked on the measurement of the solubilities of undecanolide and pentadecanolactone in supercritical carbon dioxide by supercritical fluid chromatography. The measurements were done at temperatures of 308.2K and 318.2K for a pressure range of 12.2 to 25.3 MPa. The concept of the degree of retention was applied where a cell constant was obtained from the flow type apparatus. The quantity of absorbed fluid in the stationary phase was

ignored when making thermodynamic calculations. The estimated error in temperature and pressure was ± 0.1K and $\pm 0.3\%$ respectively.

Borg et al. [52] measured the solubility of alpha-tetralol in pure carbon dioxide and in a mixed solvent formed by ethanol and carbon dioxide. The experiment was carried out at high pressures at temperatures of 303.15 to 333.15 K. A static apparatus was used for determining dew point pressures for alpha-tetralol, carbon dioxide and ethanol. The apparatus consisted of an equilibrium cell with a sapphire window, carbon dioxide tank, removable screwed cap for feeding the alcohol, thermostated water outlet, thermostated water inlet, TV connected to the video camera aimed at observing inside the cell, stirring magnet, graduated piston allowing the measurement of the cell volume, and a manual system allowing the pressure to vary by changing the cell volume.

Sovova [53] measured the solubility of ferulic acid in supercritical carbon dioxide with ethanol as a co-solvent. A flow type apparatus was used for the measurement of the solubility data. The experiment was performed for temperatures of 313.2, 323.2, and 333.2 K for a pressure range of 12 to 28 MPa. The mass of solute was found by a spectrophotometric method. An uncertainty in pressure and temperature was ± 0.01 MPa and ± 0.1K. A flow rate of 40 to 50 cubic cm/min was maintained using a micrometer valve. The deviation of the solubility with pure carbon dioxide was $\pm 8\%$ and with e   thanol as a co-solvent was $\pm 20\%$. The maximum solubility for ferulic acid was 0.03 mass percentage.

The amount of research done on the measurement in supercritical carbon dioxide is enormous. Several examples were dealt with in the previous

paragraphs. Different methods and different conditions were used for different types of compounds.

Table 4.1   Details of Some Measurement Techniques

Author	Measurement Technique	Error in Temp. measurement	Error in Pressure measurement	Error in sol. measurement
Matias [20]	Static	± 0.01°C	± 0.1%	± 6%
Sahle [12]	Dynamic	± 0.1°C	± 0.1 MPa	± 13%
Dragan [14]	Static	± 1°C	± 5 bar	± 0.55-11.9%
Ghaziaskar[21]	Flow	± 0.5 K	± 1 bar	varies
Jara [22]	Static	Unspecified	Unspecified	± 30%
Xing [23]	Flow	± 0.1K	± 0.1 MPa	± 4.24%
Shamsipur[15]	Static	Unspecified	Unspecified	± 5%
Khosravi [27]	Static	± 1K	±0.5 bar	± 5%
Bahramifar[29]	Static	± 1K	± 0.1MPa	± 2%
Duarte [17]	Static	± 0.1°C	± 4 bar	± 9%
Sane [16]	Flow	± 0.2°C	± 1 bar	± 5%
Ferri [32]	Flow	Unspecified	Unspecified	± 4%
Sauceau [33]	Flow	Unspecified	Unspecified	± 5%
Huang [38]	Flow	± 0.01°C	± 0.2%	± 1%
Khajeh [39]	Static	± 1K	± 0.1 MPa	± 7%

Table 4.1 gives an idea of the major measurement techniques used in the articles, which are described in this chapter. Static and flow methods are the most commonly used. These techniques are modified depending on the application.

The most commonly used measurement technique used in industry was the saturation (transpiration) method. It is a modification of the dynamic method. The saturation cell was sometimes referred to as an equilibrium cell. It was mainly used for solids or highly viscous heavy liquids. This method was also used often to study co-solvent effects. However, a major concern in this method was the ability to control the area of contact with the solute and solvent, the type of packing, the flow rate and the equilibration time. [19]

CHAPTER 5

COMPARATIVE STUDY OF TECHNIQUES

5.1 The Criterion

There are different measurement techniques used for solubility determinations in supercritical carbon dioxide. The method used to obtain the solubility data in supercritical carbon dioxide differs depending on the properties of the compounds and the experimental conditions.

A comparative and analytical study of these techniques is made. Most of the criteria compared were based on the data (numerical and graphical) given in different literature articles. A major basis of comparison was the data sheet compilation, which is attached in the Appendix.

The major difficulty encountered was the setting of conditions that can form the basis for a comparative study. Some important conditions are based on the data provided by the articles. They are as follows:

1. Information about error in temperature, pressure and solubility measurements

2. Information about purity and source of materials

3. The value of solubility data provided. For example, the accuracy of the data to two or three decimal places

4. The flow-rate and pressure control techniques

5. Information about packing of the column in the equilibrium cell

6. Various instruments used and their accuracy.

Some other criteria have been detailed as [19]:

1. Minimum consumption of the investigated species

2. Minimum equilibrium time

3. Minimum overall time needed for experiments

4. Direct temperature and pressure determination

5. Visual observation of the system

6. Readiness of analysis

7. Convenience

8. Low-Cost Equipment

However, among these different conditions, some are given more priority than others for the analytical study. Accuracy is a very important condition. Factors such as minimum consumption of species, minimum equilibrium time and minimum overall time, etc are more important for industrial scale plants where, along with accuracy, other issues like profitability, low-cost etc, are also important.

5.2 Compounds for analytical study

The systems analyzed for the comparative study were benzoic acid and caffeine. The measurement techniques were studied in detail for these compounds and a final conclusion was reached. A recommended set of values was obtained.

The abstracts of the relevant articles were obtained from Scifinder Scholar, which is a search tool from ACS (American Chemical Society). The abstracts were retrieved for articles containing the solubility data of benzoic acid and caffeine in pure supercritical carbon dioxide. The articles were retrieved from the NJIT print journals and also electronically. Some of the articles were retrieved through the inter-library loan. It was extremely difficult to get the articles containing solubility data in pure supercritical carbon dioxide at a particular temperature.

Around ten articles were short-listed for benzoic acid based on the abstracts obtained. However, five articles contained solubility data in mixtures containing carbon dioxide with different co-solvents with different concentrations at different temperatures. Five articles contained solubility data of benzoic acid in pure supercritical carbon dioxide. One out of the five articles had data at a different temperature.

Finally, four articles were obtained for solubility data of benzoic acid in pure carbon dioxide. Out of these, three articles contained solubility data of benzoic acid in pure carbon dioxide at 318K and three articles had data at 328K.

A similar approach was followed to obtain the articles containing the solubility data of caffeine in pure carbon dioxide at a certain temperature. Just three articles were obtained after an extensive search. However, only two articles contained the solubility data of caffeine in pure carbon dioxide at 313K for a pressure range.

5.3 Analysis of Benzoic Acid Measurement Techniques

The comparative study for benzoic acid was performed using four different

articles. They were Jin et al. [54], Lee et al. [55], Galia et al. [56] and Kurnik et

al. [57].

Jin et al. [54] conducted the experiments at pressures from 8.0 to 23.0

MPa at 308.15K, 318.15K and 328.15K.
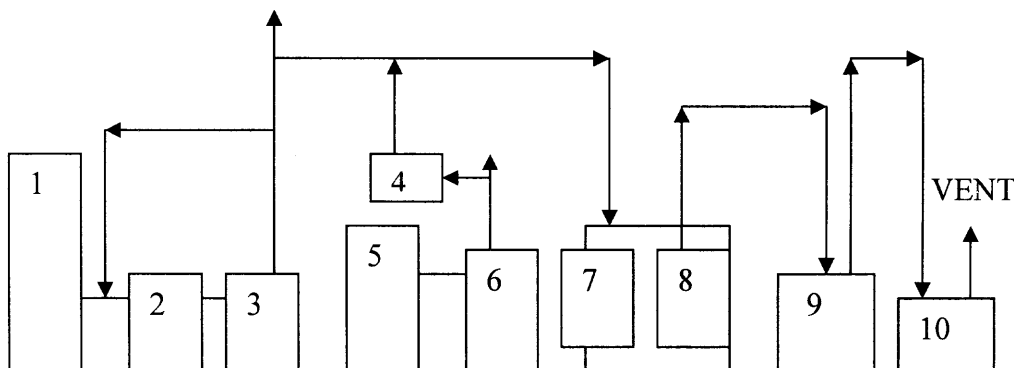
Figure 5.1 Experimental apparatus used by Jin et al. [54].
1- carbon dioxide cylinder, 2- syringe pump, 3- buffer, 4-pressure gauge, 5-cosolvent vessel, 6- high pressure pump, 7- heater, 8- equilibrium cell, 9- U-shape tube, 10- wet gas flow meter.

Figure 5.1 shows a flow type apparatus which was used to determine the

solubility. It consisted of a 376 ml high-pressure equilibrium cell. Isothermal

conditions were maintained using a temperature controller. The deviation in the

measurement of the temperature and pressure were ± 0.1K and ± 0.05 MPa. A

syringe pump was used to pressurize carbon dioxide. A flow rate was maintained

with a variation of ± 0.01ml/min. Solute was placed in the equilibrium cell and

the equilibration time was 30 min. The saturated carbon dioxide was trapped in a

U-tube and measured using a pan balance with an accuracy of ± 0.1 mg. The volume of carbon dioxide was measured by a wet gas flow meter.

The significant point of this set up was that each experiment was carried out three or more times. An equilibration time of 30 minutes was determined after several trials. In order to ensure proper saturation, a time of 40 minutes was set. A flow rate of 0.61 l/min was fixed after a range of flow rates were tested.

The second article studied was by Lee et al. [55]. The solubilities of solid benzoic acid, phenanthrene and 2,3-dimethylhexane in supercritical carbon dioxide were determined. A semiflow phase equilibrium apparatus was used. The solubility data were measured at a temperature of 318.15K and 328.15K and a pressure range of 120 bar to 240 bar.

The main component was an equilibrium cell. The uncertainties in the measurement of the temperature and pressure were ± 0.02K and ± 0.2%. A water bath was used for temperature control.
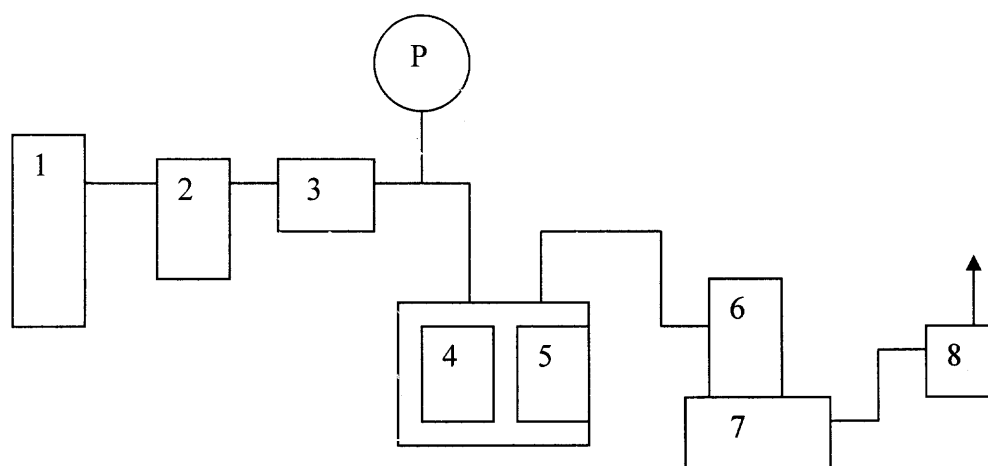
Figure 5.2 Experimental apparatus used by Lee et al. [55].
1- Carbon Dioxide cylinder, 2-Filter, 3-pump, 4-preheater, 5-equilibrium cell, 6- U-tube, 7-ice-bath, 8- gas flow meter.

The main features of the apparatus are described in great detail. A displacement liquid pump was used to introduce carbon dioxide. A gas purifier was used with 5 Angstrom and 2μm molecular sieves. This was done to ensure that the impurities were minimized. A back-pressure regulator, safety valve and check valve were attached. The equilibrium cell had dimensions of 32 cm length, 14 cm inside diameter and 50 mL volume. Precaution was taken to ensure the safety and accuracy in every unit of the apparatus.

The solute was made into a powder and dried. For proper mass transfer, it was spread into uniform layers.

A heating tape was used to prevent deposition of the solute on the line, as it may lead to experimental error in the determination of the solubility data.

Packing was given due importance as it may lead to channeling. A flow rate of 0.1 L/min was maintained. A wet test meter was used to measure the volume of carbon dioxide. Five runs were carried out and out of specification data were removed. The average deviation was ± 3.34%.

The third article studied for benzoic acid was Galia et al. [49]. A simple static method for the determination of solubilities was described. The temperature and pressure range for the experiments was 318-357K and 7-23 MPa. The proposed method allows one to measure mole fraction as low as 10^{-5}. The method can also be extended to multicomponent systems provided a suitable analytical technique is used.

The experimental apparatus consisted of a high-pressure equilibrium cell able to operate with pressures up to 30 MPa and temperatures up to 358K. The

equilibrium cell was equipped with a magnetic stir bar to promote mixing. The solute was placed inside the container. The total amount of carbon dioxide introduced was measured using an electronic scale. The efflux of carbon dioxide from the vessel was measured with a flowmeter.
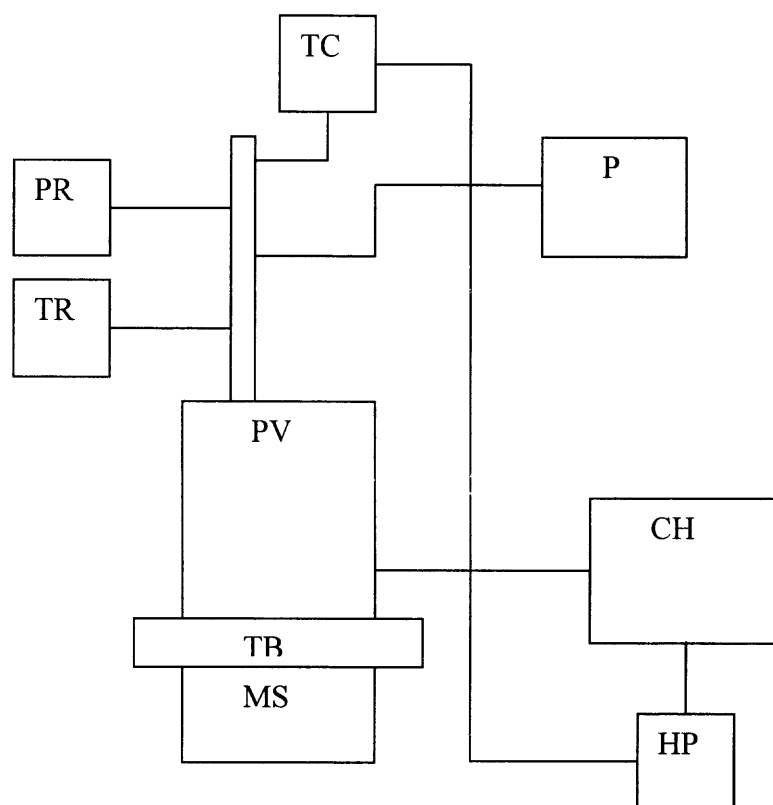
Figure 5.3 Experimental apparatus used by Galia et al. [49].
MS- magnetic stirrer, TB- thermostatized bath, PV- pressure vessel, TR-temperature readout, PR- pressure readout, TC- temperature controller, P- pump, CH- circulating heater, HP- heater power supply.

The containers were carefully cleaned, weighed and inserted in the autoclave chamber. The vessel was repeatedly purged with gaseous carbon

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dioxide. The value of the flow rate was kept low enough to avoid turbulence that could carry the solute out of the container. The amount of solute was 20-30% higher than that necessary to saturate the supercritical fluid phase. The vessel was immersed in the thermostatic bath and the stirring of its contents was commenced. The amount of time needed for equilibrium depends on the nature of the solute and on the mixing efficiency. The solubility had an average standard deviation of lower than 5%.

Finally, Kurnik et al. [56] studied the solubility of the benzoic acid. A one-pass flow system was used to measure equilibrium solubilities.
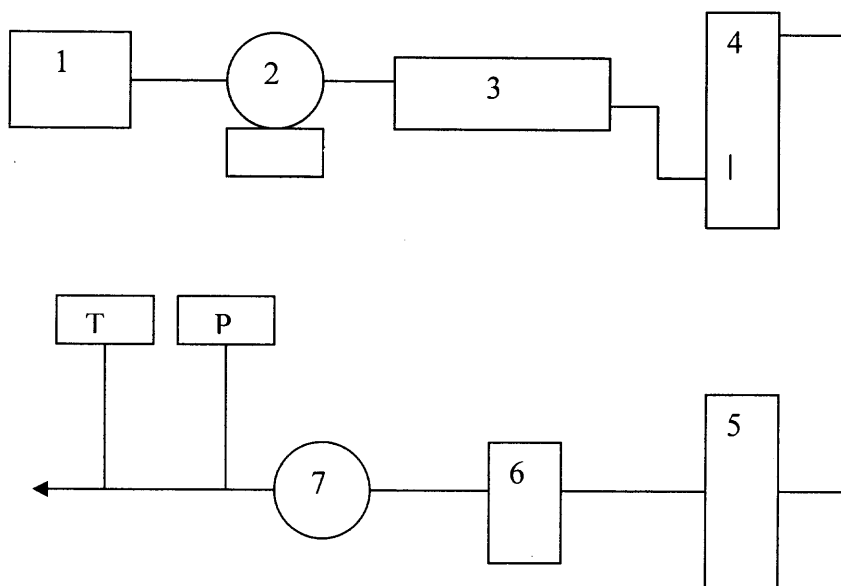
Figure 5.4 Apparatus used by Kurnik et al. [56].
1- gas cylinder, 2- compressor, 3-surge tank, 4- extractor, 5- U-tubes, 6- rotameter, 7- dry test-meter.

The range in temperature covered was 308-338 K and the range in pressure was 80-220 bar for the experiments conducted. Carbon dioxide was compressed to the system pressure with a single stage compressor. Pressure

fluctuations were dampened with an on-line surge tank fitted with a pressure controller at the outlet. The temperature and pressure were controlled to ± 0.5 K and ± 1 bar. The high-pressure fluid, at ambient temperature, was allowed to flow into a vertical extraction tube. The extraction tube was packed with alternate layers of the test solid and quartz wool.

The supercritical extractant with dissolved solid passed from the extractor into a heated regulating valve. The dissolved solids precipitated in a U-tube following the valve. The solute-free, low-pressure extractant gas flow was measured by a rotameter and by a calibrated dry-test meter. The mass of the precipitated solid was found by weighing the tubes after an experiment on a balance. The average deviation was 1.3%.

5.4 Results and Discussion of Benzoic acid solubility data

Figure 5.5 shows the solubility data of benzoic acid at 318K for the different articles studied. Pressure is plotted along the X-axis and mole fraction along the Y-axis. Galia et al. [49] showed an almost linear increase in solubility. Kurnik et al. [56] and Lee et al. [55] showed a flattening tendency at higher pressures. Lee et al. [55] had the lowest solubility for the higher-pressure range.

The solubility data for all three articles for the pressure range 5-17 MPa were almost similar for the same increase in pressure. However, the solubility data began to deviate from the general trend for the higher- pressure range, mostly in the range 17-30 MPa.
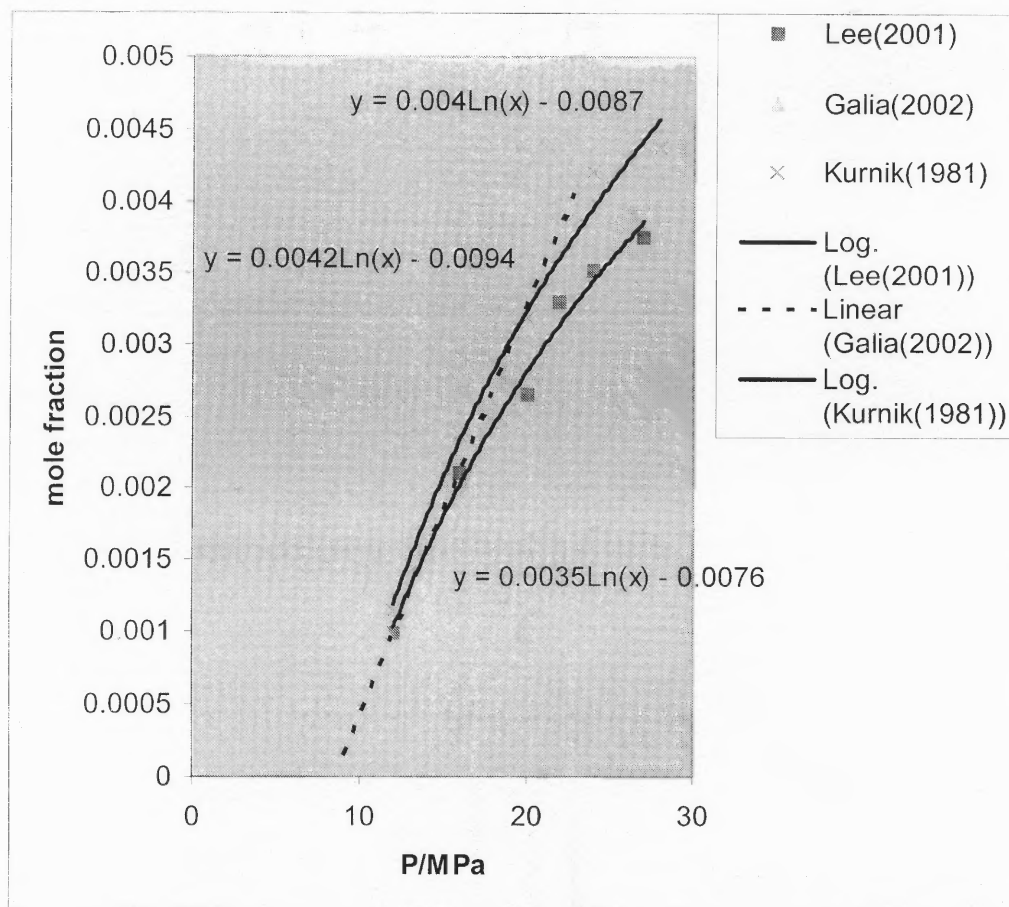
Figure 5.5 Solubility data of benzoic acid in pure supercritical carbon dioxide at 318K.

The best-fit equations for the three data series were obtained as:

$y = 0.004\ln(x) - 0.0087$ (Kurnik et al. [56])

$y = 0.0042\ln(x) - 0.0094$ (Galia et al. [49])

$y = 0.0035\ln(x) - 0.0076$ (Lee et al. [55])

Figure 5.6 shows the common trend-line for the three data series. The common curve-fitting equation for the three data series was as:

$y = 0.0039\ln(x) - 0.0085$

Comparing the individual equations with the common curve equation, it can be inferred that Kurnik et al. [56] may be a better technique for determination

of solubility of benzoic acid at 318 K. This was also observed from the trend

lines.
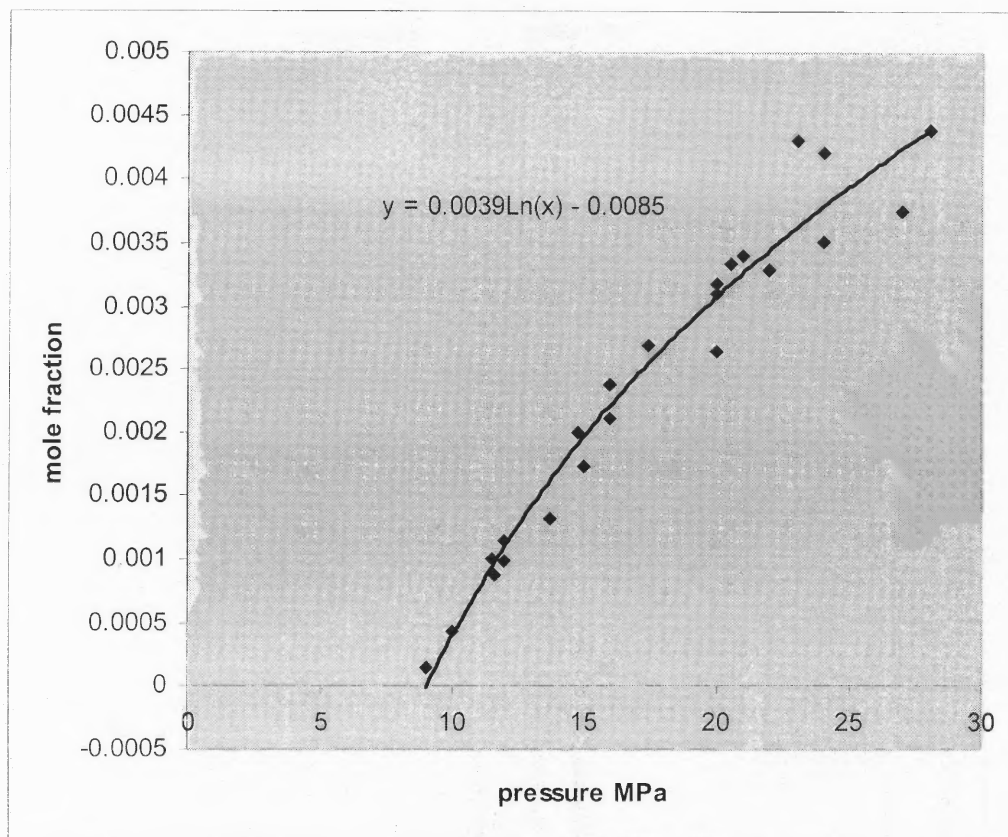
Figure 5.6 Curve-fitting equation and trend line for the solubility data of benzoic acid obtained at 318 K

It can also be inferred that for a low-pressure range [5-17 MPa], the

solubility data does not depend on the measurement technique used. Lee et al.

[55] used a flow apparatus consisting of an equilibrium cell. Galia et al. [49] used

a static method, while; Kurnik et al. [56] used a single-pass flow apparatus.

The recommended set of values found from the curve-fitting equation at 318 K was as follows:

Table 5.1 Recommended Values of Benzoic Acid at 318 K

Pressure (MPa)	Solubility (mole fraction)
10	.00048
12	.00119
16	.00231
20	.00318
24	.00389
30	.00476

A similar trend was also witnessed when the solubility data for benzoic acid was plotted at 328K. The solubility data in terms of mole fraction were plotted along the Y-axis for Jin et al. [54], Lee et al. [55] and Kurnik et al. [56]. Pressure was plotted along the X-axis.

The data at a pressure range of 5-17 MPa were close to each other for each study, but they deviate at higher pressures.
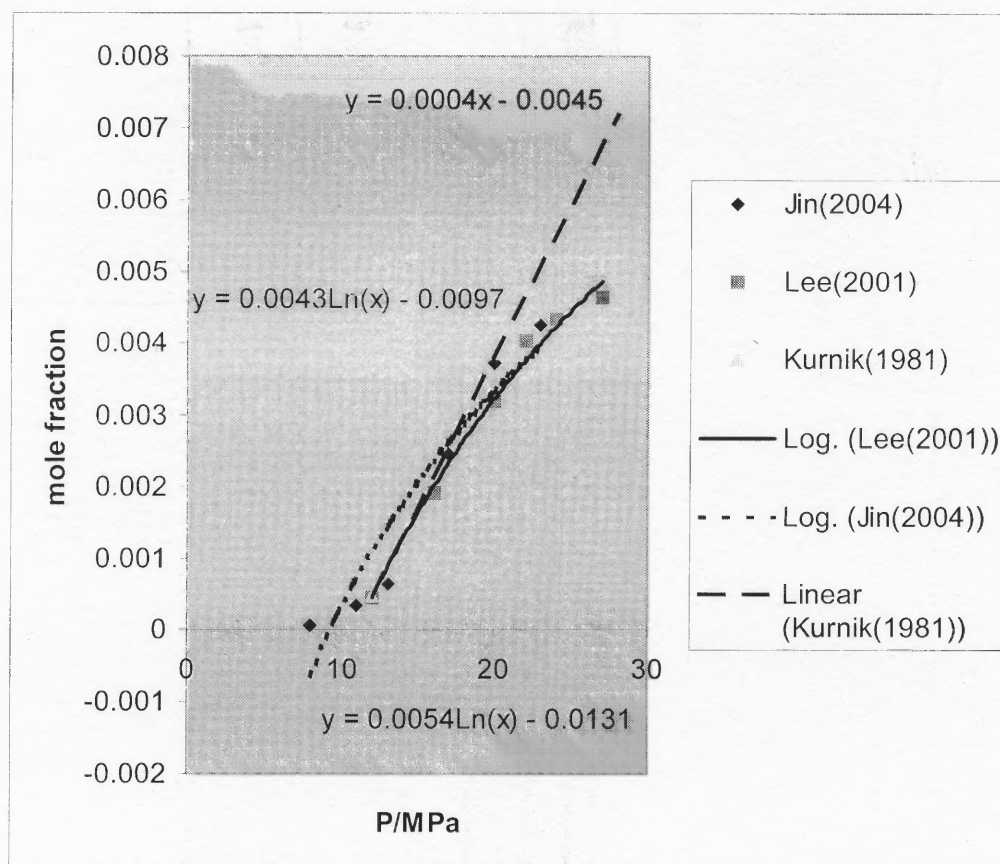
Figure 5.7 Solubility data of benzoic acid in pure supercritical carbon dioxide at 328K.

The best-fit equations of the data series at 328 K were as:

y = 0.0004x – 0.0045   (Kurnik et al. [56])

y = 0.0043ln(x) – 0.0097   (Jin et al. [54])

y = 0.0054ln(x) – 0.0131   (Lee et al. [55])

The common trend line showed best fit in the logarithmic curve. Other curves were tried but they could not fit well into the data. The curve-fitting equation for the three data series at 328 K was as:

y = 0.0055ln(x) – 0.0127
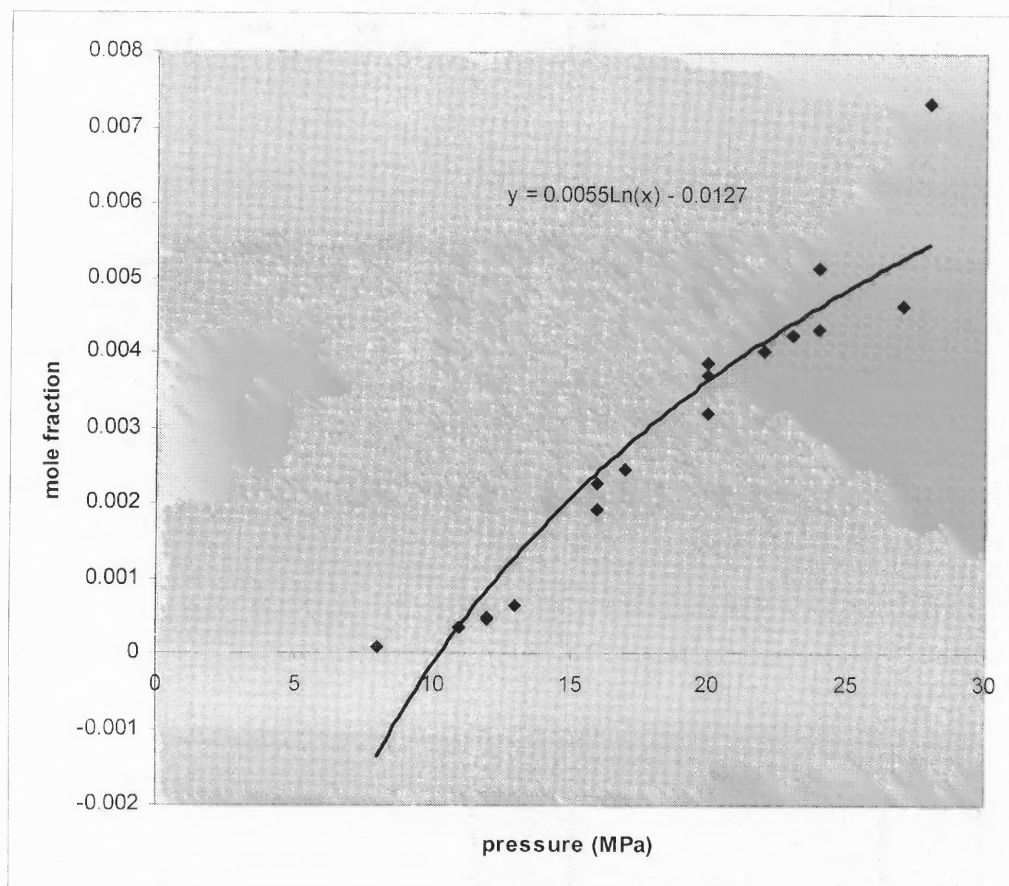
Figure 5.8 Curve-fitting equation and trend line for solubility data of benzoic acid obtained at 328 K.

Comparison of the individual equations with the common equation show that Jin et al. [54] and Lee et al. [55] give a better data that Kurnik et al. [56] for benzoic acid at 328 K.

The data points for Jin et al. [54] and Lee et al. [55] were similar. At higher pressures, Kurnik et al. [56] again showed a greater increase in solubility with pressure.

The recommended set of values for the system found from the curve fitting equation at 328 K was as follows:

Table 5.2 Recommended Values of Benzoic Acid at 328 K

Pressure (MPa)	Solubility (mole fraction)
10	.000035
12	.00096
16	.00254
20	.00377
24	.00477
30	.00600

The differences in the solubility data at higher pressures can be attributed to a number of factors. It is therefore instructive to examine factors such as calibration procedures, purity of the substances, reproducibility, etc.

Jin et al. [54] mentions the purity of carbon dioxide to be greater than 99.9% and the purity of the rest of the material used to be greater than 99.5%. Lee et al. [55] used carbon dioxide with a purity of greater than 99.5% and other materials with purity of 99%. Kurnik [56] mentions the purity of benzoic acid to be 99%.

Jin et al. [54] had experimental uncertainty of ± 0.1 K and ± 0.05 MPa. The error in measurement in Lee et al. [55] was ± 0.02 K and ± 0.2 MPa. Kurnik

et al. [56] had an uncertainty of ± 0.5 K and ± 1 bar. Further, the measurement uncertainty in the solubility for Jin et al. [54] was ± 5%.

Galia et al. [49] mentions a standard deviation of 5% and the average deviation for Kurnik et al. [56] was 1.3%.

Jin et al. [54] had an equilibrium time in the range of 20-60 minutes. After testing, it was optimized to 30 minutes, but to enhance proper saturation, the data was collected after 40 minutes. The suitable flow rate was in the range of 0.4-1.01 l/min. This flow rate was optimized at 0.6 l/min after careful testing. Galia et al. [49] had an equilibration time of 4 hours, which depended on the nature of solute used and also the mixing efficiency. The flow rate was 60 ml/hr. Further, all the methods had a low fluid flow rate to avoid turbulence, which may wash away the solute.

Moreover, the uncertainty in the measurement of mass for Galia et al. [49] was ± 0.1g and for Kurnik et al. [56] was ± 5 mg.

These major differences may account for the large deviation in the solubility data for benzoic acid at higher pressures.

5.5 Analysis of caffeine measurement techniques

The method for caffeine was based on the work by Kopcak et al. [57] and Solorzano et al. [58].

Kopcak et al. [57] investigated the caffeine solubility in supercritical carbon dioxide. The experiment was performed in the pressure range 15.2 to 30.0

MPa and at temperatures of 323.2 K and 343.2 K. A high-pressure semi-continuous flow apparatus was used to determine the solubility.

Figure 5.9 shows the apparatus used by Kopcak et al. [57]. The semi-continuous apparatus is from Autoclave Engineers (USA) and has an operating range up to 37 MPa at 473 K. The major components include a liquid displacement pump (46-460 mL/h), 300 mL high-pressure extraction vessel and separator flasks.
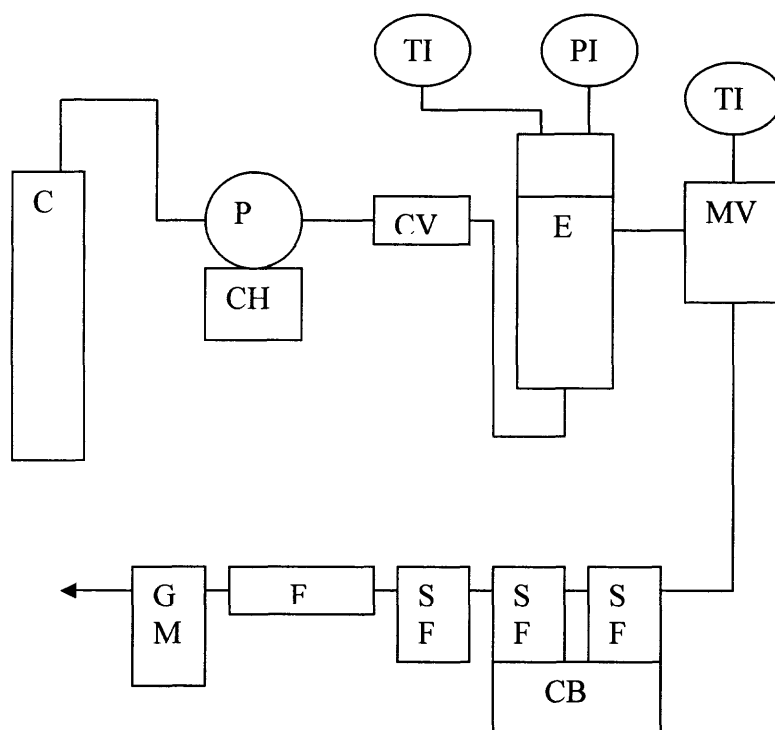
Figure 5.9 Experimental Apparatus used by Kopcak et al. [57]. C- Carbon Dioxide/ co-solvent cylinder, CB- Cooling bath (ice), CH- chiller, E-Extracted (Heated), F- Carbon adsorption filter, GM- gas flow meter, P-pump, PI-pressure indicator, TI-temperature indicator, SF-Separator flask, CV-check valve, MV-micrometering valve.

Isothermal conditions are maintained by heating jackets and a temperature controller. A constant flow rate is maintained using a pump head connected to the chiller. A flow computer-measuring unit is used. Valves are used to control the flow rate throughout the system. A digital transducer system is used to measure the pressure.

The uncertainty in the measurement of the temperature and pressure was ±0.5°C and ±0.03MPa.

The solubility was obtained using 50 grams of sample. A flow rate of 1.4 g/min was used. Each experiment was conducted thrice to ensure reproducibility. A valve depressurizes the effluent (supercritical carbon dioxide saturated with caffeine) from the extractor. A UV spectrophotometer was used to analyze the concentration at a wavelength of 270 nm.

A comparative study of this method was made with the one as described by Solorzano et al. [58] who worked on the solubility measurements and modeling of molecules of biological and pharmaceutical interest with supercritical carbon dioxide.

The solubility data for caffeine, uracil and eythromycin were obtained for a temperature range of 40-60°C and up to 300 bar. However, the data of interest for this thesis is the solubility of caffeine in supercritical carbon dioxide.

Figure 5.10 shows the custom-built recirculating high-pressure apparatus used for the determination of the solubility data. The five major components of the system are pumping, equilibration, heating, sampling, and cleaning /venting system.

The pumping system has an ISCO syringe pump for initial pressurization. The second is a recirculation pump used for the flow of solute-carbon dioxide mixture. Finally, there is a vacuum pump used in the cleaning. The deviation in the measurement of the pressure was ± 0.34 bar.
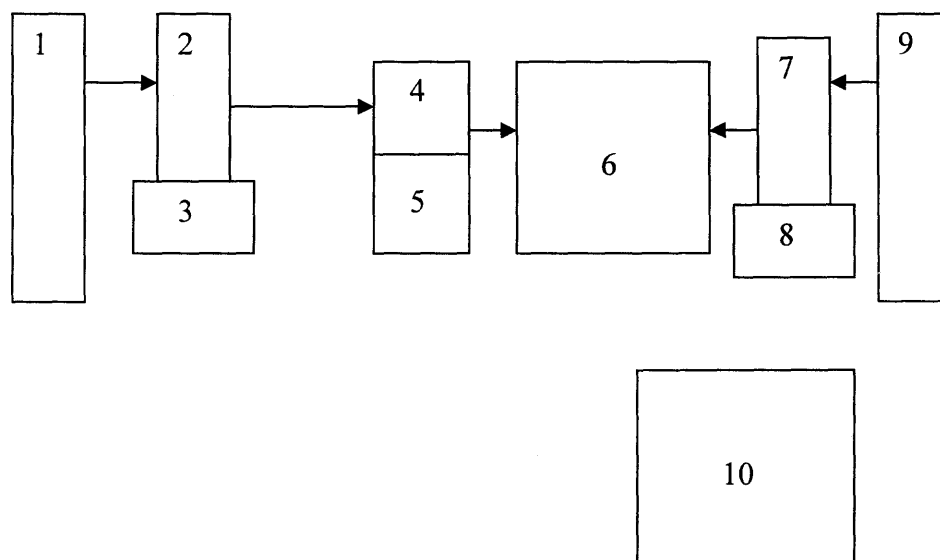
Figure 5.10 Experimental apparatus for Solorzano et al. [58].
1,9- carbon dioxide tanks, 2,5,7-pressure indicator, 3-ISCO syringe pump, 4-Temperature indicator, 6-equilibrium cell, 8- vacuum pump, 10- UV-Vis-system spectrometer.

The equilibration system consists of a steel vessel where the solute is placed and glass wool is kept at the entrance to minimize entrainment. The uncertainty in the measurement of the temperature is ± 0.1K.

The heating system consists of cartridge heaters and an oven. Thermocouples are used for temperature control.

The sampling system consists of valves and loops. The valves prevent entrainment and contamination.

Finally, the cleaning/ventilation system consists of a vacuum valve and a vent valve. All the five major components are well integrated to the system and work in cohesion for smooth functioning.

The equilibrium cell consists of the solute and the glass beads to increase surface contact and facilitate mass transfer. The carbon dioxide is pumped in at 10% of the maximum rpm. Thermodynamic equilibrium occurs at 30-90 minutes. The final sample collected in the vial is analyzed by UV-Vis system spectrometer. At least four measurements were taken for each pressure.

5.6 Results and discussion of caffeine solubility data

The analysis of the articles by Kopcak et al. [57] and Solorzano et al. [58] showed some interesting results. The solubility data obtained in pure supercritical carbon dioxide are compared.

Figure 5.11 shows the comparison of the solubility data of caffeine in pure supercritical carbon dioxide by Kopcak et al. [57] and Solorzano et al. [58]. The solubility data are plotted for the same range of pressure and at a temperature of

313 K It shows that for the same temperature and pressure range, the data obtained by Kopcak et al. [57] is higher than the data of Solorzano et al. [58]. Higher solubility data may result from various factors like a higher amount of solute, higher flow rate, etc. Higher amount of solute may result in experimental error, in case; some of the excess amount gets deposited along the line. Higher flow rate, may lead to turbulence and may cause error is data.
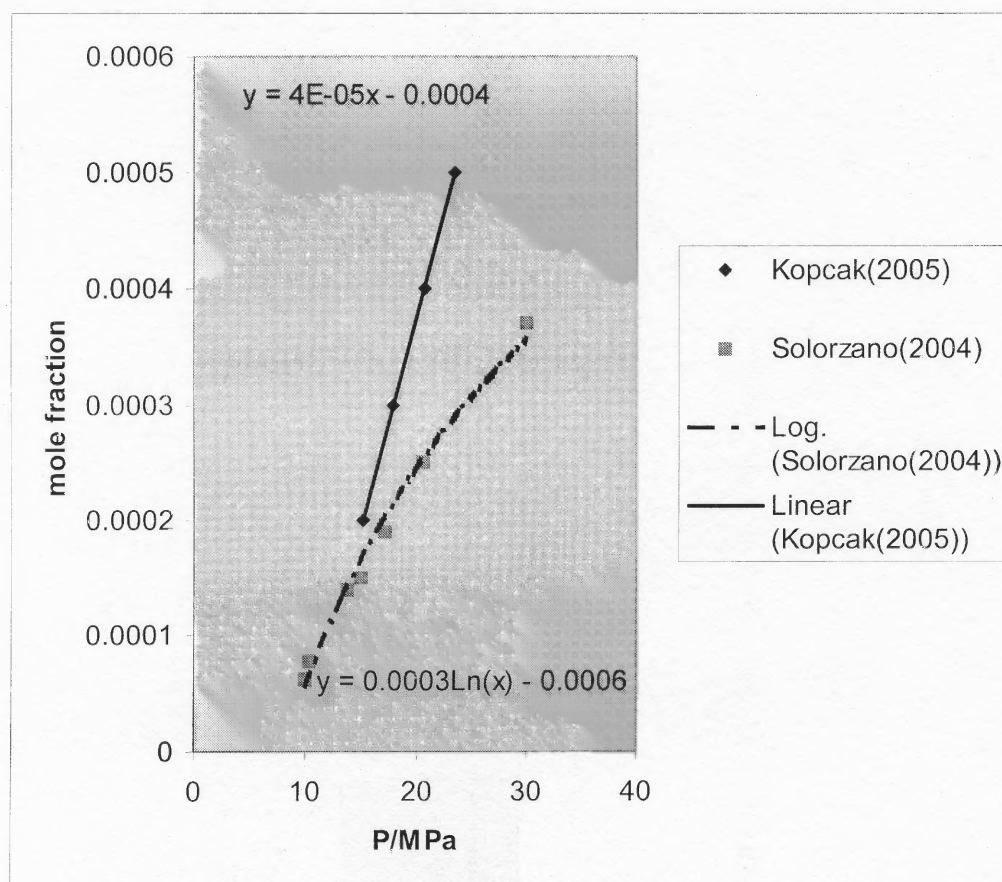
Figure 5.11 Solubility data of caffeine in pure supercritical carbon dioxide at 313 K.

The best fit equation for the data series were as:

y = 4exp(-05)x − 0.0004   (Kopcak et al. [57])

y = 0.0003ln(x) − 0.0006   (Solorzano et al. [58])

The common curve-fitting equation for the solubility data was as:

y = 0.0004ln(x) − 0.0008

Comparison of the equation and the trend lines show that Solorzano et al. [58] may give better solubility data.

The recommended set of values for the system found from the curve fitting equation was as follows:

Table 5.3 Recommended Values of Caffeine at 313 K

Pressure (MPa)	Solubility (mole fraction)
10	.00012
12	.00019
16	.00030
20	.00039
24	.00047
30	.00056

The trend observed for benzoic acid solubility data can be seen for the solubility data of caffeine too. At lower pressures, the data obtained from the two different techniques were closer. In the pressure range of 10-17 MPa, the different techniques do not appear to have much effect on the solubility data. However, as

the pressure was increased from 17 MPa, the data obtained from the different
methods show a deviation.
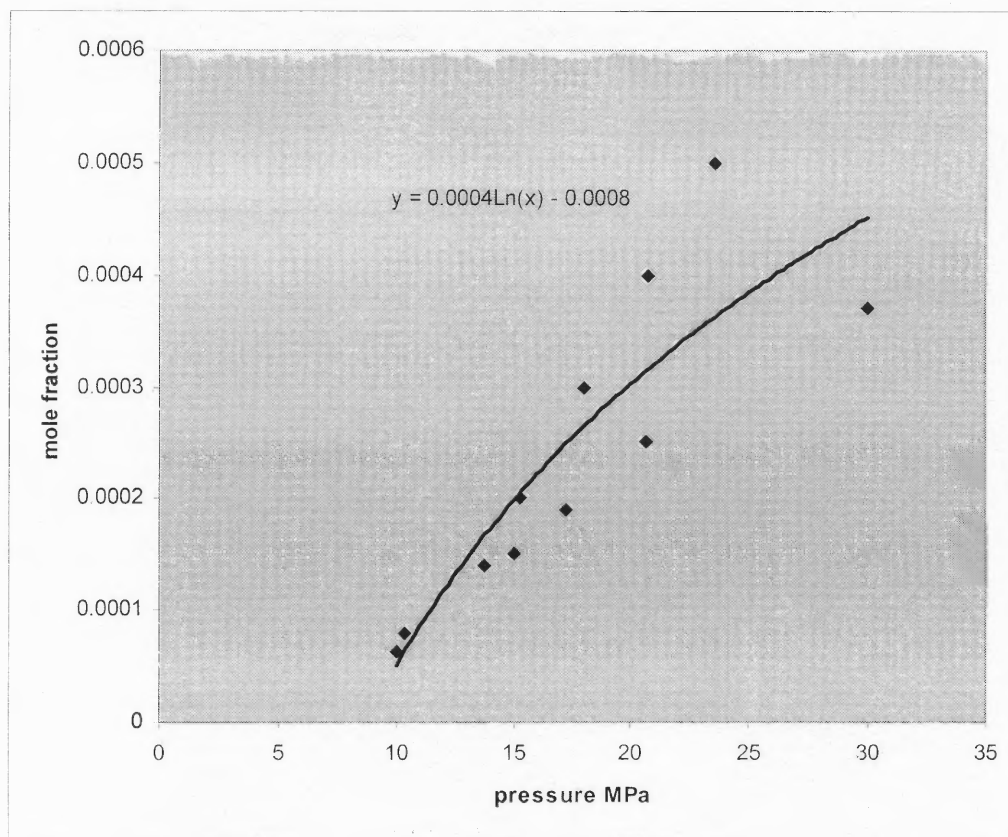
Figure 5.12 Curve-fitting equation and trend line for solubility data of caffeine at
313 K.

Factors such as the difference in measurement techniques, uncertainty in
measurement of temperature and pressure, equilibrium time, etc seem to play a
greater role at higher pressures.

Kopcak et al. [57] showed a uniform increase of the solubility with increasing pressures while, Solorzano et al. [58] showed the data to be flattening at higher pressures.

The uncertainty in the measurement of pressure for both techniques was ± 0.03 MPa. The error in the measurement of temperature for Kopcak et al. [57] was ± 0.5 °C and for Solorzano was ± 0.1 K. Further, the uncertainty in the measurement of the solubility data for Solorzano et al. [58] was ± 10%. However, the error in measurement of the solubility data for Kopcak et al. could not be ascertained.

A comprehensive comparison was not possible because of the non-availability of articles containing the solubility of the compound in pure supercritical carbon dioxide at a certain temperature. However, it can be concluded from the study of the solubility data of benzoic acid and caffeine that at pressures lower than 17 MPa, the measurement techniques does not have much effect on the solubility data obtained. The differences in techniques start to play a role as the pressure is increased from 17 MPa.

5.7 Optimized conditions for higher accuracy

The different measurement techniques for the solubility of caffeine and benzoic acid in supercritical carbon dioxide have been discussed. A general review of the supercritical phenomenon and description of the different measurement techniques was also done by Knox [59].

There was scope for improvement in each of the techniques. The accuracy of the techniques can be increased if proper calibration and validation of the equipment and the process is done. The important factors are the data related to the error in the measurement of the solubility, temperature and pressure. Calibration of measuring balance, flow meter, water- bath temperature should be known.

CHAPTER 6

CONCLUSION

The discussion on supercritical carbon dioxide gives a brief idea about the different measurement techniques used in the determination of the solubility data for different sets of compounds and conditions. The definition, the basic properties and the behavior of the supercritical fluids in the critical region was observed.

Further, the concept of solubility and its relation to supercritical fluids was studied. The various techniques and methods used in the study of solubility in supercritical fluids were explored. The primary techniques were the static method, the dynamic method, the dew and bubble point method, and finally, the supercritical fluid chromatography method. These methods were extensions of each other and have different modifications.

The method used in the solubility determination in supercritical carbon dioxide by different researchers was described. The methodology used in the selection and retrieval of the articles was also discussed.

A number of data sheets were made from these articles. They form part of the IUPAC-NIST Solubility Data Series project. Several volumes have been compiled by different authors on the solubility data in supercritical carbon dioxide. These data sheets contain the information on the solubility concentration,

temperature range, pressure range, the error in measurement of temperature, pressure and solubility, and also a brief description of the measurement techniques. The possible classifications of the compounds whose data sheets were made were also discussed. These data sheets are attached in the appendix.

Finally, a comparative study of the measurement techniques was done. The different methods for the calculation of the solubility of benzoic acid and caffeine in supercritical carbon dioxide were analyzed.

APPENDIX

DATA SHEETS

The appendix consists of 121 data sheets, which are obtained from the articles on the solubility in supercritical carbon dioxide for the year 2003- June 2005. The data sheets have information about the compound name, CAS number, chemical formula, temperature range, pressure range, measurement techniques, source and purity of materials and any references, if provided. They also provide information about the error in measurement of the temperature, pressure and solubility.

The table layout consists of data on the solubility of the compound in mole fractions with changing pressure of carbon dioxide at different temperatures.

Components:	Original Measurements:
(1) Griseofulvin; $C_{17}H_{17}NclO_6$; [126-07-8] (2) Carbon Dioxide; CO_2; [124-38-9]	Simion Dragan, Mihaela Dragan, Studia Universitatis Babes- Bolyai, Chemia, 48(1), 191 – 200, 2003
Variables: T/K = 313.2, 343.2 P/MPa = 14.0 - 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Griseofulvin(1) in Carbon Dioxide(2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
313.2	140	14.0	3.6006
313.2	160	16.0	6.2735
313.2	180	18.0	6.9466
343.2	140	14.0	0.4458
343.2	160	16.0	1.7656
343.2	180	18.0	6.5678

Auxiliary Information

The main of the experimental equipment is the autoclave. A specific amount of the substance 99.9% was filled in a metal cage, weighed and placed on the bottom of the autoclave. Carbon dioxide gas comes from the tank and preheated into the autoclave. After equilibrium was reached, carbon dioxide was vented out. The metal cage with the rest of the substance was weighed and the amount of the dissolved substance calculated.

Source and Purity of Materials:
1. Griseofulvin source Merck Co., purity
2. Carbon Dioxide source AGA Gas GmbH Hamburg, purity 99.99%;

Estimated Error:
Temperature: ±1C
Pressure: ±5 bar
Solubility: 1.09%-11.9%

Figure A.1 Solubility of Griseofulvin in Carbon Dioxide.

Components:	Original Measurements:
(1) Griseofulvin; $C_{17}H_{17}NclO_6$;[126-07-8].	Simion Dragan, Mihaela Dragan, Studia Universitatis Babes-Bolyai, Chemia, 48(1), 191-200 (2003).
(2) Carbon Dioxide; CO_2; [124-38-9], with Ethanol; C_2H_5OH; [64-17-5]	
Variables: T/K = 313.2 P/MPa = 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Griseofulvin(1) in Carbon Dioxide with Ethanol(2)

T/K	P/MPa	% ethanol	$x_1 \times 10^6$
313.2	18.0	0.0	6.9466
313.2	18.0	2.5	31.350
313.2	18.0	5.0	57.048

Auxiliary Information

The main of the experimental equipment is the autoclave. A specific amount of the substance was filled in a metal cage, weighed and placed on the bottom of the autoclave. Carbon dioxide gas comes from the tank and preheated into the autoclave. After equilibrium was reached, carbon dioxide was vented out. The metal cage with the rest of the substance was weighed and the amount of the dissolved substance calculated.	Source and Purity of Materials: 1. Griseofulvin source Merck Co., purity 99.9% 2. Carbon Dioxide source AGA Gas GmbH Hamburg, purity 99.99%; Ethanol unstated. Estimated Error: Temperature: $\pm$1C Pressure: $\pm$5 bar Solubility: 0.0%-0.17%

Figure A.2 Solubility of Griseofulvin in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) Terfenadine; $C_{32}H_{41}NO_2$; [50679-08-8] (2) Carbon Dioxide; CO_2; [124-38-9]	Simion Dragan, Mihaela Dragan, Studia Universitatis Babes – Bolyai, Chemia, 48(1), 191 - 200 (2003).
Variables: T/K = 313.2, 343.2 P/MPa = 14.0 - 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of Terfenadine(1) in Carbon Dioxide(2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
313.2	140	14.0	0.9985
313.2	160	16.0	1.2939
313.2	180	18.0	1.5344
343.2	140	14.0	0.3337
343.2	160	16.0	1.0435
343.2	180	18.0	1.9914

Auxiliary Information

The main apparatus of the experimental equipment is autoclave. A specific amount of the substance was filled in a metal cage, weighed and placed on the bottom of the autoclave. The carbon dioxide gas comes from the tank and preheated into the autoclave. After equilibrium was reached, carbon dioxide was vented out. The metal cage with the rest of the substance was weighed and the amount of the dissolved substance calculated.

Source and Purity of Materials:
1. Terfenadine source Merck, purity 99.9%
2. Carbon Dioxide source AGA Gas GmbH
   Hamburg, purity 99.99%;

Estimated Errors:
Temperature: ± 1C
Pressure : ±5 bar
Solubility: 0.55% - 7.35%

Figure A.3 Solubility of Terfenadine in Carbon Dioxide.

Components:	Original Measurements:
(1) Caffeine; $C_8H_{10}N_4H0_20_2$; [58-08-2]	Uriam Kopack, Rahoma Sadeg Mohamed, J. of
(2) 95%Carbon Dioxide; CO_2; [124-38-9], with 5%Ethanol; C_2H_5OH; [64-17-5]	Supercritical Fluids, *34*, 209-214 (2005).

Variables:	Prepared By:
T/K = 313.2, 323.2, 343.2	Dana E. Knox, Syed A. Abdullah
P/MPa = 15.2 - 23.4	

Experimental Data
Solubility of Caffeine(1) in 95%Carbon Dioxide/5% Ethanol(2)

T/K	P/MPa (compiler -read from graph in original article)	$x_1 \times 10^3$ (compiler -read from graph in Original article)
313.2	15.2	0.60
313.2	18.0	1.30
313.2	20.4	1.75
313.2	23.4	1.80
323.2	15.2	0.40
323.2	18.0	0.85
323.2	20.4	1.25
323.2	23.4	1.70
343.2	15.2	0.30
343.2	18.0	0.70
343.2	20.4	1.00
343.2	23.4	1.60

Auxiliary Information

The experimental apparatus used was a semi-continuous flow high pressure system and designed for working pressures up to 37MPa at 473K.Samples of Caffeine were placed in the extractor. A super-critical mixed flow rate of 1.4g/min was used. The supercritical mixed solvent was delivered by the pump as a liquid and slowly allowed into the extractor until extraction pressure was reached. The extractor is heated to the extraction temperature and the micro metering valve located downstream of the extractor was slowly opened while maintaining the pressure constant in the extractor. The effluents of the extractor (super-critical fluid saturated with caffeine) are de-pressurized through the micro metering valve. precipitated caffeine was recovered in three separators flasks placed in cooling bath. The solution was analyzed with UV spectrophotometer.

Source and Purity of Materials:
(1) Caffeine source Synth Ltd.(Sao Paulo, Brazil) purity not stated.
(2) Carbon Dioxide/ Ethanol source White Martins Gases Industrials S.A (Campinas, Brazil), purity unstated.

Estimated Error:
Temperature: $\pm$ 0.5C
Pressure: $\pm$0.03 MPa
Solubility: not stated

Figure A.4 Solubility of Caffeine in 95% Carbon Dioxide/5% Ethanol.

Components:	Original Measurements:
(1) Caffeine; $C_8H_{10}N_4H0_2O_2$; [58-08-2] (2) 90%Carbon Dioxide; CO_2; [124-38-9], with 10%Ethanol; C_2H_5OH; [64-17-5]	Uriam Kopack, Rahoma Sadeg Mohamed, J. of Supercritical fluids, *34*, 209- 214 (2005).
Variables: T/K = 323.2, 343.2 P/MPa = 15.2 - 30.0	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data

Solubility of Caffeine(1) in 90%Carbon Dioxide/10%Ethanol(2)

T/K	P/MPa (compiler-read from graph in original article)	x_1 X 10^4 (compiler-read from graph in original article)
323.2	15.2	4.00
323.2	18.0	7.50
323.2	21.0	10.0
323.2	23.5	13.0
323.2	26.5	13.5
323.2	30.0	16.0
343.2	15.2	15.0
343.2	18.0	22.5
343.2	21.0	27.5
343.2	23.5	28.0
343.2	26.5	35.0
343.2	30.0	57.0

Auxiliary Information

The experimental apparatus used was a semi-continuous flow high pressure system and designed for working pressures up to 37MPa at 473K.Samples of Caffeine were placed in the extractor. A super-critical mixed flow rate of 1.4g/min was used. The supercritical mixed solvent was delivered by the pump as a liquid and slowly allowed into the extractor until extraction pressure was reached. The extractor is heated to the extraction temperature and the micro metering valve located downstream of the extractor was slowly opened while maintaining the pressure constant in the extractor. The effluents of the extractor (super-critical fluid saturated with caffeine) are de-pressurized through the micro metering valve. precipitated caffeine was recovered in three separator flasks placed in cooling bath. The solution was analyzed with UV spectrophotometer.

Source and Purity of Materials:
(1) Caffeine source Synth Ltd.(Sao Paulo, Brazil) purity not stated.
(2) Carbon Dioxide/ ethanol source White Martins Gases Industrials S.A (Campinas, Brazil), purity unstated.

Estimated Error:
Temperature: $\pm$ 0.5C
Pressure: $\pm$0.03 MPa
Solubility: not stated

Figure A.5 Solubility of Caffeine in 90% Carbon Dioxide/ 10% Ethanol.

Components:	Original Measurements:
(1) Caffeine;$C_8H_{10}N_4H0_2O_2$;[58-08-2]	Uriam Kopack, Rahoma Sadeg Mohamed, J. of
(2) 95%Carbon Dioxide; CO_2; [124-38-9], with	Supercritical fluids,*34*,209-214(2005).
5%Isopropanol; $(CH_3)_2CHOH$; [67-63-0]	
Variables:	Prepared By:
T/K = 323.2, 343.2	Dana E. Knox, Syed A. Abdullah
P/MPa = 15.2 - 30.0	

Experimental Data
Solubility of Caffeine(1) in 95%Carbon Dioxide/5%Isopropanol(2)

T/K	P/MPa (compiler-read from graph in original article)	x_1 X 10^4 (compiler-read from graph in Original article)
323.2	15.2	4.0
323.2	18.0	7.0
323.2	21.0	10.0
323.2	23.5	13.0
323.2	26.5	15.0
323.2	30.0	16.0
343.2	15.2	2.5
343.2	18.0	−
343.2	21.0	−
343.2	23.5	−
343.2	26.5	16.0
343.2	30.0	−

Auxiliary Information

The experimental apparatus used was a semi-continuous flow high pressure system and designed for working pressures up to 37MPa at 473K.Samples of Caffeine were placed in the extractor. A super-critical mixed flow rate of 1.4g/min was used. The supercritical mixed solvent was delivered by the pump as a liquid and slowly allowed into the extractor until extraction pressure was reached. The extractor is heated to the extraction temperature and the micro metering valve located downstream of the extractor was slowly opened while maintaining the pressure constant in the extractor. The effluents of the extractor (super-critical fluid saturated with caffeine) are de-pressurized through the micro metering valve. precipitated caffeine was recovered in three separator flasks placed in cooling bath. The solution was analyzed with UV spectrophotometer.

Source and Purity of Materials:
(1) Caffeine source Synth Ltd.(Sao Paulo, Brazil) purity not stated.
(2) Carbon Dioxide/Isopropanol source White Martins Gases Industrials S.A (campinas, Brazil), purity unstated.

Estimated Error:
Temperature: ± 0.5C
Pressure: ±0.03 MPa
Solubility: not stated

Figure A.6 Solubility of Caffeine in 95% Carbon Dioxide/ 5% Isopropanol.

Components:	Original Measurements:
(1) Naphthalene; $C_{10}H_8$; [91-20-3]	Veronique Pauchon, Zeini Cisse, Monique Chavret,
(2) Carbon Dioxide; CO_2; [124-38-9]	Jacques Jose, J. supercritical fluids, *32*, 115-121 (2004)
Variables:	Prepared By:
T/K = 308.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 7.19 - 32.15	

Experimental Data
Solubility of Naphthalene(1) in Carbon Dioxide(2)

P/bar	P/MPa	$x_1 \times 10^4$	P/bar	P/MPa	$x_1 \times 10^4$	P/bar	P/MPa	$x_1 \times 10^4$
71.9	7.19	7.40	94.4	9.44	103.00	181.5	18.15	171.00
77.5	7.75	5.15	96.2	9.62	105.00	187.8	18.78	173.00
84.8	8.48	36.60	96.2	9.62	109.00	188.8	18.88	169.00
86.6	8.66	33.40	97.4	9.74	110.00	189.0	18.90	169.00
86.9	8.69	76.70	97.4	9.74	105.00	194.6	19.46	161.00
87.6	8.76	78.70	97.6	9.76	114.00	197.4	19.74	177.00
89.4	8.94	64.30	102.3	10.23	121.00	209.0	20.90	182.00
89.7	8.97	88.10	102.3	10.23	121.00	209.2	20.92	183.00
90.0	9.00	88.00	106.6	10.66	124.00	214.4	21.44	181.00
90.3	9.03	81.10	106.6	10.66	126.00	228.0	22.80	179.00
90.8	9.08	85.20	115.9	11.59	134.00	260.1	26.01	181.00
90.8	9.08	79.60	116.5	11.65	135.00	260.8	26.08	176.00
91.4	9.14	93.40	124.8	12.48	152.00	279.5	27.95	174.00
92.0	9.20	95.30	125.0	12.50	144.00	279.7	27.97	183.00
92.9	9.29	104.00	136.6	13.66	155.00	300.5	30.05	183.00
92.9	9.29	106.00	136.6	13.66	147.00	300.8	30.08	177.00
93.8	9.38	97.10	146.0	14.60	154.00	319.3	31.93	176.00
93.8	9.38	96.60	146.3	14.63	156.00	321.5	32.15	185.00
94.1	9.41	91.40	165.6	16.56	153.00			
94.1	9.41	99.10	166.1	16.61	163.00			

Auxiliary Information

The apparatus allows a dynamic solubilisation and an on-line or off-line analysis. The liquid carbon-dioxide is filtered, then charged to the system with HPLC pump, working at constant pressure and compressed to desired regulated pressures. A correct regulation and a rapid stabilization of pressure are obtained by means of a carbon dioxide leak rate and a buffer vessel. The carbon dioxide flows continuously through the saturation cell kept at a constant temperature. It becomes saturated with the solute prior to flowing through a Rheodyne six-port valve. Samplings of the saturated fluid are carried out for HPLC placed in the thermostat bath. The solute is dissolved in heptanes and after passing through an UV spectrophotometer, the solution is collected in a volumetric flask.

Source and Purity of Materials:
(1) Naphthalene source Prolabo, purity99%
(2) Carbon Dioxide source Air Liquide, purity 99.5%

Estimated Error:
Temperature: ±0.02 K
Pressure: ± 0.3 bar
Solubility: not stated.

Figure A.7 Solubility of Naphthelene in Carbon Dioxide.

Components:	Original Measurements:
(1) Triphenylmethane; $C_{19}H_{16}$; [519-73-3]	Veronique Pauchon, Zeini Cisse, Monique Chavret,
(2) Carbon Dioxide; CO_2; [124-38-9]	Jacques Jose, J. Supercritical fluids, *32*, 115-121 (2004)
Variables·	Prepared By:
T/K = 308.15, 318.15, 328.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 6.5 - 28	

Experimental Data
Solubility of Triphenylmethane(1) in Carbon Dioxide(2)

T/K	P/bar	P/MPa	$x_1 X$ 10^4	T/K	P/bar	P/MPa	$x_1 X$ 10^4	T/K	P/bar	P/MPa	$x_1 X$ 10^4
308.15	70.5	7.05	0.387	318.15	65.0	6.50	0.748	328.15	66.3	6.63	0.479
308.15	70.8	7.08	0.546	318.15	65.3	6.53	0.614	328.15	67.0	6.70	0.764
308.15	73.7	7.37	0.359	318.15	68.9	6.89	0.618	328.15	70.2	7.02	0.741
308.15	75.0	7.50	0.609	318.15	70.5	7.05	0.315	328.15	70.5	7.05	2.930
308.15	75.3	7.53	0.369	318.15	74.0	7.40	1.100	328.15	75.3	7.53	0.850
308.15	78.1	7.81	2.300	318.15	74.7	7.47	1.430	328.15	75.6	7.56	0.979
308.15	78.5	7.85	3.150	318.15	79.1	7.91	.0733	328.15	79.7	7.97	1.280
308.15	82.5	8.25	4.830	318.15	79.4	7.94	0.464	328.15	79.7	7.97	0.746
308.15	83.1	8.31	4.850	318.15	85.3	8.53	0.422	328.15	85.0	8.50	0.902
308.15	87.5	8.75	5.680	318.15	93.0	9.30	0.909	328.15	85.0	8.50	0.554
308.15	87.5	8.75	6.710	318.15	93.6	9.36	0.912	328.15	89.9	8.99	0.747
308.15	96.6	9.66	7.760	318.15	102.2	10.22	4.570	328.15	90.2	9.02	0.703
308.15	97.5	9.75	7.890	318.15	103.1	10.31	3.540	328.15	96.9	9.69	1.420
308.15	108.1	10.81	9.220	318.15	111.0	11.10	6.700	328.15	97.5	9.75	1.740
308.15	108.4	10.84	9.180	318.15	112.5	11.25	6.470	328.15	106.4	10.64	2.810
308.15	117.9	11.79	10.500	318.15	121.9	12.19	9.450	328.15	107.2	10.72	2.500
308.15	118.8	11.88	10.700	318.15	122.2	12.22	9.060	328.15	116.8	11.68	4.600
308.15	127.5	12.75	11.600	318.15	132.0	13.20	11.100	328.15	117.1	11.71	4.930
308.15	136.4	13.64	12.500	318.15	132.3	13.23	11.300	328.15	128.1	12.81	7.580
308.15	137.8	13.78	12.300	318.15	140.5	14.05	13.700	328.15	128.4	12.81	7.380
308.15	147.0	14.70	13.300	318.15	140.8	14.08	13.100	328.15	137.2	13.72	10.70
308.15	147.3	14.73	13.300	318.15	149.4	14.94	15.600	328.15	145.7	14.57	13.40
308.15	162.0	16.20	14.300	318.15	150.2	15.02	15.800	328.15	147.0	14.70	13.60
308.15	162.0	16.20	14.200	318.15	160.1	16.01	18.100	328.15	155.3	15.53	16.30
308.15	170.9	17.09	15.100	318.15	160.1	16.01	16.500	328.15	155.9	15.59	16.60
308.15	171.4	17.14	15.600	318.15	178.2	17.82	19.400	328.15	189.9	18.99	23.10
308.15	206.6	20.66	16.900	318.15	240.6	24.06	28.200	328.15	243.0	24.30	35.00
308.15	283.7	28.37	20.500	318.15	280.8	28.08	30.400	328.15	280.2	28.02	39.80

Auxiliary Information

The apparatus allows a dynamic solubilisation and an on-line or off-line analysis. The liquid carbon-dioxide is filtered, then charged to the system with HPLC pump, working at constant pressure and compressed to desired regulated pressures. A correct regulation and a rapid stabilization of pressure are obtained by means of a carbon dioxide leak rate and a buffer vessel. The carbon dioxide flows continuously through the saturation cell kept at a constant temperature. The solubility is measured using spectrometer

Source and Purity of Materials:
(1) Triphenylmethane source Janssen, purity 97%
(2) Carbon Dioxide source Air Liquide, purity 99.5%

Estimated Error:
Temperature: $\pm$ 0.02K
Pressure: $\pm$ 0.3 bar
Solubility: not stated.

Figure A.8 Solubility of Triphenylmethane in Carbon Dioxide.

Components:	Original Measurements:
(1) Benzoic acid; $C_7H_6O_2$; [65-85-0]	Jun-Su Jin, Ze-ting Zhang, Qun-Sheng Li, Ying Li
(2) CarbonDioxide; CO_2; [124-38-9]	En-ping Yu, J. Chem. Eng. Data, 50, 801 – 803 (2005)
Variables:	Prepared By:
T/K = 328.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 8 - 24	

Experimental Data
Solubility of Benzoic acid(1) in Carbon Dioxide(2)

T/K	P/MPa (compiler-read from graph in original article)	$x_1 \times 10^3$ (compiler-read from graph in original article)
328.15	8.0	0.0
328.15	11.0	0.2
328.15	13.0	0.3
328.15	17.0	2.3
328.15	24.0	4.1

Auxiliary Information

A flow type apparatus was used to determine the solubility. The experimental apparatus is described in the previous paper[1].Low pressure carbon dioxide was passed through the cell at a low flow rate to remove the air trapped inside. Afterwards carbon-dioxide was compressed to a mixer which was heated by an electric coil and they were transferred into the equilibrium cell from the bottom to a desired pressure and temperature. In the equilibrium cell, the solvent and solute reached equilibrium through mass transfer. Then the saturated carbon dioxide flowed from the top of the cell through a decompression valve wrapped with a coiled heater into 2 weighed U-type tubes. These were partially packed to catch the small particles of solute in the expanded gas system. After vacuum drying, the solid solute collected in the U-tubes was weighed.

Source and Purity of Materials:
(1) Benzoic acid source not stated, purity not stated.
(2)carbon dioxide source Beijing Tianbao chemical factory, purity 99.9%

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubility: not stated

References:
1. Li,Q.S.; Zhang,Z.T.;Zhong,C.L.;Lui,Y,C.; Zhou,Q.R., Fluid Phase Equilibria, 207, 183 – 192 (2003).

Figure A.9 Solubility of Benzoic acid in Carbon Dioxide.

Components:	Original Measurements:
(1) Propyl p- hydroxybenzoate; $C_{10}H_{12}O_3$; [94-13-3]	Jun-Su Jin, Ze-ting Zhang, Qun -Sheng Li, Ying Li En-ping Yu ,J. Chem. Eng. Data, *50*, 801-803 (2005)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables:	Prepared By:
T/K = 308.15, 318.15, 328.15 P/MPa = 8.0 - 23.0	Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Propyl p-hydroxybenzoate(1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^4$
308.15	8.0	0.3461
308.15	11.0	0.6595
308.15	13.0	0.7592
308.15	17.0	1.0059
308.15	20.0	1.9504
308.15	23.0	2.6990
318.15	8.0	0.2335
318.15	11.0	0.6242
318.15	13.0	1.3320
318.15	17.0	2.6708
318.15	20.0	3.3589
318.15	23.0	4.0853
328.15	8.0	0.0378
328.15	11.0	0.7004
328.15	13.0	1.6064
328.15	17.0	4.4370
328.15	20.0	5.3255
328.15	23.0	6.1751

Auxiliary Information

A flow type apparatus was used to determine the solubility. The experimental apparatus is described in the previous paper[1].Low pressure carbon dioxide was passed through the cell at a low flow rate to remove the air trapped inside. Afterwards carbon-dioxide was compressed to a mixer which was heated by an electric coil and they were transferred into the equilibrium cell from the bottom to a desired pressure and temperature. In the equilibrium cell, the solvent and solute reached equilibrium through mass transfer. Then the saturated carbon dioxide flowed from the top of the cell through a decompression valve wrapped with a coiled heater into 2 weighed U-type tubes. These were partially packed to catch the small particles of solute in the expanded gas system. After vacuum drying, the solid solute collected in the U-tubes was weighed.

Source and Purity of Materials:
(1) Propyl p-benzoate source Beijing chemical regent factory, purity 99.0% .
(2) Carbon Dioxide source Beijing Tianbao chemical factory, purity 99.9%

Estimated Error:
Temperature:$\pm$ 0.1K
Pressure:$\pm$ 0.05MPa
Solubility: $\pm$5%

References:
1. Li,Q.S.; Zhang,Z.T.;Zhong,C.L.;Lui,Y,C.; Zhou,Q.R., Fluid Phase Equilibria, *207*, 183-192 (2003)

Figure A.10 Solubility of Propyl-p-hydroxybenzoate in Carbon Dioxide.

Components:	Original Measurements:
(1) Propyl p-hydroxybenzoate; $C_{10}H_{12}O_3$; [94-13-3]	Jun-Su Jin, Ze-ting Zhang, Qun-Sheng Li, Ying Li En-ping Yu, J. Chem. Eng. Data, *50*, 801-803 (2005)
(2) Carbon Dioxide; CO_2; [124-38-9], with Cyclohexane; C_6H_{12}; [110-82-7]	

Variables:	Prepared By:
T/K = 308.15 P/MPa = 8.0 - 23.0	Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Propyl p-hydroxybenzoate(1) in Carbon Dioxide and Cyclohexane (2)

T/K	P/MPa	$x_1 \times 10^4$
308.15	8.0	0.4405
308.15	11.0	1.4983
308.15	13.0	1.6149
308.15	17.0	1.9176
308.15	20.0	2.4504
308.15	23.0	3.4228

Auxiliary Information

A flow type apparatus was used to determine the solubility. The experimental apparatus is described in the previous paper[1].Low pressure carbon dioxide was passed through the cell at a low flow rate to remove the air trapped inside. Afterwards carbon-dioxide was compressed to a mixer which was heated by an electric coil and they were transferred into the equilibrium cell from the bottom to a desired pressure and temperature. In the equilibrium cell, the solvent and solute reached equilibrium through mass transfer. Then the saturated carbon dioxide flowed from the top of the cell through a decompression valve wrapped with a coiled heater into 2 weighed U-type tubes. These were partially packed to catch the small particles of solute in the expanded gas system. After vacuum drying, the solid solute collected in the U-tubes was weighed.

Source and Purity of Materials:
(1) Propyl p-benzoate source Beijing chemical regent factory, purity 99.0% .
(2) Carbon Dioxide source Beijing Tianbao chemical factory, purity 99.9%;cyclo-hexane source Beijing chemical factory, purity 99.5%

Estimated Error:
Temperature: ± 0.1K
Pressure: ± 0.05 MPa
Solubility: ± 5%

References:
1.Li,Q.S. ;Zhang ,Z.T.;Zhong, C.L.;Lui,Y.C.;Zhou,Q.R, Fluid Phase Equilibria ,*207*, 183-192 (2003)

Figure A.11 Solubility of Propyl p-hydroxybenzoate in Carbon Dioxide with Cyclohexane.

Components:	Original Measurements:
(1) Propyl p-hydroxybenzoate; $C_{10}H_{12}O_3$; [94-13-3]	Jun-Su Jin, Ze-ting Zhang, Qun-Sheng Li, Ying Li En-ping Yu, J. Chem. Eng. Data, *50* ,801-803 (2005)
(2) Carbon Dioxide; CO_2; [124-38-9],with Cyclohexane; C_6H_{12}; [110-82-7], and Acetone; $(CH_3)_2CO$; [67-64-1]	

Variables: T/K = 308.15 P/MPa = 8.0 - 23.0	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data

Solubility of Propyl p-hydroxybenzoate(1) in Carbon Dioxide with cyclohexane and acetone (2)

T/K	P/MPa	$x_1 \times 10^4$
308.15	8.0	0.4367
308.15	11.0	3.2538
308.15	13.0	6.0511
308.15	17.0	9.1679
308.15	20.0	10.1896
308.15	23.0	11.6134

Auxiliary Information

A flow type apparatus was used to determine the solubility. The experimental apparatus is described in the previous paper[1].Low pressure carbon dioxide was passed through the cell at a low flow rate to remove the air trapped inside. Afterwards carbon-dioxide was compressed to a mixer which was heated by an electric coil and they were transferred into the equilibrium cell from the bottom to a desired pressure and temperature .In the equilibrium cell, the solvent and solute reached equilibrium through mass transfer. Then the saturated carbon dioxide flowed from the top of the cell through a decompression valve wrapped with a coiled heater into 2 weighed U-type tubes. These were partially packed to catch the small particles of solute in the expanded gas system. After vacuum drying, the solid solute collected in the U-tubes was weighed.

Source and Purity of Materials:
(1) Propyl p-benzoate source Beijing chemical regent factory, purity 99.0%
(2) Carbon Dioxide source Beijing Tianbao chemical factory, purity 99.9%;cyclo-hexane and acetone source Beijing chemical factory, purity 99.5%

Estimated Error:
Temperature: ±0.1 K
Pressure: ± 0.05 MPa
Solubility: ± 5%

References:
1.Li,Q.S. ;Zhang ,Z.T.; Zhong, C.L.; Lui, Y.C.; Zhou, Q.R, Fluid Phase Equilibria, *207*, 183-192 (2003)

Figure A.12 Solubility of Propyl p-hydroxybenzoate in Carbon Dioxide with Cyclohexane and Acetone.

Components:	Original Measurements:
(1) Propyl p-hydroxybenzoate; $C_{10}H_{12}O_3$; [94-13-3]	Jun-Su Jin, Ze-ting Zhang ,Qun-Sheng Li, Ying Li En-ping Yu, J. Chem. Eng. Data, *50* ,801-803 (2005)
(2) Carbon Dioxide; CO_2; [124-38-9],with Acetone; $(CH_3)_2CO$; [67-64-1]	

Variables:	Prepared By:
T/K = 308.15 P/MPa = 8.0 - 23.0	Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Propyl p-hydroxybenzoate(1) in Carbon Dioxide and Acetone(2)

T/K	P/MPa	$x_1 \times 10^4$
308.15	8.0	0.4472
308.15	11.0	4.0538
308.15	13.0	7.2101
308.15	17.0	10.6505
308.15	20.0	13.1172
308.15	23.0	14.1332

Auxiliary Information

A flow type apparatus was used to determine the solubility. The experimental apparatus is described in the previous paper[1].Low pressure carbon dioxide was passed through the cell at a low flow rate to remove the air trapped inside. Afterwards carbon-dioxide was compressed to a mixer which was heated by an electric coil and they were transferred into the equilibrium cell from the bottom to a desired pressure and temperature .In the equilibrium cell, the solvent and solute reached equilibrium through mass transfer. Then the saturated carbon dioxide flowed from the top of the cell through a decompression valve wrapped with a coiled heater into 2 weighed U-type tubes. These were partially packed to catch the small particles of solute in the expanded gas system. After vacuum drying, the solid solute collected in the U-tubes was weighed.

Source and Purity of Materials:
(1) Propyl p-benzoate source Beijing chemical regent factory, purity 99.0%
(2) Carbon Dioxide source Beijing Tianbao chemical factory, purity 99.9% Acetone source Beijing chemical factory, purity 99.5%

Estimated Error:
Temperature: $\pm$ 0.1K
Pressure: $\pm$ 0.05 MPa
Solubility: $\pm$ 5%

References:
1.Li,Q.S. ;Zhang ,Z.T; Zhong, C.L.; Lui,Y.C.; Zhou,Q.R, Fluid Phase Equilibria, *207*, 183-192 (2003)

Figure A.13 Solubility of Propyl p-hydroxybenzoate in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) Trans-$Co_2(CO)_6[3,5$-bis$(CF_3)C_6H_3P(i$-$C_3H_7)_2]$; [850700-56-0]	Nicolaus Dahmen, Pia Griesheimer, Piotr Makarczyk, Stephan Pitter, Olaf Walter, Journal of
(2) Carbon Dioxide; CO_2; [124-38-9]	Organometallic chemistry, *690*, 1467-1473 (2005)

Variables:	Prepared By:
T/K = 313 - 343	Dana E. Knox, Syed A. Abdullah
P/MPa = 13.35 - 26.0	

Experimental Data

Solubility of Trans-$Co_2(CO)_6[3,5$-bis$(CF)_3C_6H_3P(i$-$C_3H_7)_2$ (1) in Carbon Dioxide(density $0.75gcm^{-3}$)(2)

T/K	P/bar	P/MPa-compiler	Solubility(gl^{-1})	x_1 X 10^5
313	133.5	13.35	1.1±0.1	6.81
323	175.0	17.50	2.5±0.3	15
333	217.5	21.75	2.6±0.3	16.1
343	260.0	26.00	2.5±0.5	15

Auxiliary Information

An extraction apparatus to allow for solubility measurements by a dynamic procedure was used. if a sufficiently low flow rate is adjusted, the carbon-dioxide passing the extraction cell is loaded with an equilibrium substance amount in the steady state. The solid is placed on abed of glass beads inside the extraction cell having a volume of 10ml which is heated to the temperature of the measurement. The carbon dioxide is compressed to the desired pressure. The experiment proceeds by pumping carbon dioxide at a constant flow rate. The pressure is released through a back-pressure regulator. A separator is used to vent-off the gas. Knowing the flow rate, the time of sampling the solid, and the mass of the precipitated solid, its solubility is measured.

Source and Purity of Materials:
(1) compound 1 was synthesized, purity not stated.
(2) carbon dioxide source not stated, purity not stated.

Estimated Error:
Temperatue: not stated
Pressure: not stated
Solubility: varies

Figure A.14 Solubility of Trans-compound in Carbon Dioxide with 0.75 gcm^{-3} density.

Components:	Original Measurements:
(1) Trans-$Co_2(CO)_6[3,5$-bis$(CF_3)C_6H_3P($i-$C_3H_7)_2$; [850700-56-0]	Nicolaus Dahmen, Pia Griesheimer,Piotr Makarczyk, Stephan Pitter, Olaf Walter, Journal of
(2) Carbon Dioxide; CO_2; [124-38-9]	Organomettalic chemistry, *690*, 1467-1473 (2005)
Variables:	Prepared By:
T/K = 323	Dana E. Knox, Syed A. Abdullah
P/MPa = 10.0 - 23.7	

Experimental Data

Solubility of Trans-$Co_2(CO)_6[3,5$-bis$(CF)_3C_6H_3P($i-$C_3H_7)_2$ (1) in Carbon Dioxide(2)

P/bar	P/MPa-compiler	CO_2density(gl^{-1})	solubility(gl^{-1})	x_1 X 10^5
100	10.0	380	<0.1	<1.21
125	12.5	610	0.7±0.1	5.33
150	15.0	700	2.1±0.2	13.9
175	17.5	750	2.5±0.3	15.5
200	20.0	780	2.7±0.1	16.1
213	21.3	800	7.0±0.7	40.6
225	22.5	810	11.4±0.4	65.4
237	23.7	820	15.8±0.8	89.7

Auxiliary Information

An extraction apparatus to allow for solubility measurements by a dynamic procedure was used. If a sufficiently low flow rate is adjusted, the carbon-dioxide passing the extraction cell is loaded with an equilibrium substance amount in the steady state. The solid is placed on abed of glass beads inside the extraction cell having a volume of 10ml which is heated to the temperature of the measurement. The carbon dioxide is compressed to the desired pressure. The experiment proceeds by pumping carbon dioxide at a constant flow rate. The pressure is released through a back-pressure regulator. A separator is used to vent-off the gas. Knowing the flow rate, the time of sampling the solid ,and the mass of the precipitated solid, its solubility is measured.

Source and Purity of Materials:
(1) compound 1 was synthesized, purity not stated.
(2) carbon dioxide source not stated, purity not stated.

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubility: varies

Figure A.15 Solubility of Trans-compound in Carbon Dioxide.

Components:	Original Measurements:
(1) Bis(cyclohexyl-1,4-dihydro-2,6-dimethyl-4-(1-methyl -5-nitro-imidazol-2-yl)-3,5-pyridinedicarboxylates; NP1; $C_{25}H_{34}N_4O_6$; [196707-73-0] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad,, J. chem. Eng. Data, *50*, 344-347 (2005)
Variables: T/K = 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of NP1 (1) in Carbon Dioxide(2)

T/K	P/MPa	x_1 X 10^6
328	12.2	---
328	15.2	2.6
328	18.2	4.8
328	21.3	8.9
328	33.4	18.8
328	35.5	20.4
338	12.2	---
338	15.2	2.0
338	18.2	4.9
338	21.3	9.6
338	33.4	20.8
338	35.5	22.3
348	12.2	---
348	15.2	---
348	18.2	9.1
348	21.3	14.0
348	33.4	25.9
348	35.5	27.5

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Source and Purity of Materials:
(1) NP1 was synthesized, purity 99.5%
(2) Carbon Dioxide source Sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 6%

Figure A.16 Solubility of NP1 in Carbon Dioxide.

References:
(1) Yamini,Y.; Bahrmifar,N, J .Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng .Data, *43* ,400-402 (1998)

Components:	Original Measurements:
(1) Bis(cyclohexylmethyl-1,4-dihydro2,6-dimethyl-4-(1-Methyl-5-nitro-imidazol-2-yl)-3,5-pyridinecarboxylates; NP2; $C_{27}H_{38}N_4O_6$; [196707-74-1] (2) Carbon Dioxide; CO_2;[124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. chem. Eng. Data, *50*, 344-347 (2005)
Variables: T/K = 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of NP2 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
328	12.2	---
328	15.2	7.2
328	18.2	11.5
328	21.3	11.5
328	33.4	27.5
328	35.5	31.2
338	12.2	---
338	15.2	7.2
338	18.2	11.5
338	21.3	11.5
338	33.4	27.4
338	35.5	31.2
348	12.2	---
348	15.2	2.9
348	18.2	11.8
348	21.3	15.9
348	33.4	58.0
348	35.5	62.8

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2]. The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.17 Solubility of NP2 in Carbon Dioxide.

Source and Purity of Materials:
(1) NP2 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$ 0.1 MPa
Solubility: $\pm$ 6%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Components:	Original Measurements:
(1) Bis(cyclohexylethyl-1,4-dihydro2,6-dimethyl-4-(1-Methyl-5-nitro-imidazol-2-yl)-3,5-pyridinecarboxylates; NP3; $C_{29}H_{42}N_4O_6$; [196707-75-2] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. chem. Eng. Data, *50*, 344 – 347 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of NP3 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	3.1
338	15.2	6.2
338	18.2	11.6
338	21.3	14.7
338	33.4	21.2
338	35.5	22.0
348	12.2	3.7
348	15.2	6.5
348	18.2	11.9
348	21.3	17.1
348	33.4	27.7
348	35.5	27.1
358	12.2	2.6
358	15.2	5.6
358	18.2	12.4
358	21.3	19.4
358	33.4	34.0
358	35.5	36.1

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.18 Solubility of NP3 in Carbon Dioxide.

Source and Purity of Materials:
(1) NP3 was synthesized, purity 99.5%
(2) Carbon Dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 6%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53 – 56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng. Data, *43*, 400 – 402 (1998)

Components:	Original Measurements:
(1) Bis(cyclohexylpropyl-1,4-dihydro2,6-dimethyl-4-(1-Methyl-5-nitro-imidazol-2-yl)-3,5-pyridinecarboxylates; NP4; $C_{31}H_{46}N_4O_6$; [196707-76-3] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. chem. Eng. Data, *50*, 344 – 347 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of NP4 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	---
338	18.2	3.9
338	21.3	4.2
338	33.4	5.9
338	35.5	5.7
348	12.2	---
348	15.2	---
348	18.2	4.2
348	21.3	5.1
348	33.4	7.3
348	35.5	7.5
358	12.2	---
358	15.2	---
358	18.2	3.8
358	21.3	5.4
358	33.4	9.1
358	35.5	9.6

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2]. The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.19 Solubility of NP4 in Carbon Dioxide.

Source and Purity of Materials:
(1) NP4 was synthesized, purity 99.5%
(2) carbon dioxide source sabaían, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 6%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)

(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Components:	Original Measurements:
(1) Bis(cyclohexylbutyll-1,4-dihydro2,6-dimethyl-4-(1-Methyl-5-nitro-imidazol-2-yl)-3,5-pyridinecarboxylates; NP5; $C_{33}H_{50}N_4O_6$; [196707-76-4]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. chem. Eng. Data, *50*, 344 – 347 (2005)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables:	Prepared By:
T/K = 338, 348, 358	Dana E. Knox, Syed A. Abdullah
P/MPa = 12.2 - 35.5	

Experimental Data
Solubility of NP5 (1) in Carbon Dioxide(2)

T/K	P/MPa	x_1 X 10^6
338	12.2	---
338	15.2	---
338	18.2	3.1
338	21.3	4.3
338	33.4	13.4
338	35.5	15.1
348	12.2	---
348	15.2	---
348	18.2	3.3
348	21.3	4.6
348	33.4	20.3
348	35.5	23.1
358	12.2	---
358	15.2	---
358	18.2	2.3
358	21.3	2.9
358	33.4	28.8
358	35.5	34.0

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2]. The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.20 Solubility of NP5 in Carbon Dioxide.

Source and Purity of Materials:
(1) NP5 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 6%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A1); $C_{27}H_{26}N_4O_6$; [196707-62-7] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. Chem. Eng Data, 50, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A1 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	---
338	18.2	---
338	24.3	0.1
338	33.4	0.4
338	35.5	0.5
348	12.2	---
348	15.2	---
348	18.2	---
348	24.3	0.2
348	33.4	0.8
348	35.5	1.3
358	12.2	---
358	15.2	---
358	18.2	---
358	24.3	0.7
358	33.4	2.8
358	35.5	3.6

Auxiliary Information

A suprex MPS/225 system equipped with a modified
static system for solubility determination in the SFE
mode was used. A detailed description of the equipment
and operating procedures has been given [1,2].The solid
solutes were mixed well with the proper number of
glass beads and packed into a 1.0mL extraction vessel.
supercritical carbon dioxide was pressurized and passed
into the extraction. Then it was loaded into an injection
loop. The loop was depressurized into the collection vial.
Finally the solubility was calculated by absorbance
measurements using an UV-vis spectrophotometer.

Figure A.21 Solubility of A1 in Carbon Dioxide.

Source and Purity of Materials:
(1) A1 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran,
 purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem.Eng
 Data, 45, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.;
 Shamshipur,M, J. Chem. Eng. Data
 43, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A2); $C_{33}H_{38}N_4O_6$; [196707-70-7] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. Chem. Eng Data, 50, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A2 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	---
338	18.2	0.8
338	24.3	1.3
338	33.4	3.7
338	35.5	4.2
348	12.2	---
348	15.2	---
348	18.2	0.4
348	24.3	1.5
348	33.4	5.7
348	35.5	6.2
358	12.2	---
358	15.2	---
358	18.2	0.6
358	24.3	2.5
358	33.4	8.2
358	35.5	10.3

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.22 Solubility of A2 in Carbon Dioxide.

Source and Purity of Materials:
(1) A2 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem.Eng Data, 45, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamshipur,M, J. Chem. Eng. Data 43, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A3); $C_{35}H_{42}N_4O_6$; [196707-72-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. Chem. Eng Data, *50*, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A3 (1) in Carbon Dioxide(2)

T/K	P/MPa	x_1 X 10^6
338	12.2	---
338	15.2	---
338	18.2	2.8
338	24.3	4.1
338	33.4	7.8
338	35.5	8.9
348	12.2	---
348	15.2	---
348	18.2	2.9
348	24.3	4.6
348	33.4	10.7
348	35.5	12.1
358	12.2	---
358	15.2	---
358	18.2	3.0
358	24.3	6.0
358	33.4	13.6
358	35.5	16.6

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.23 Solubility of A3 in Carbon Dioxide.

Source and Purity of Materials:
(1) A3 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$ 0.1 MPa
Solubility: $\pm$ 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H; Shamshipur,M, J. Chem. Eng. Data *43*, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A4); $C_{31}H_{34}N_4O_6$; [196707-68-3] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. Chem. Eng Data, *50*, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A4 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	---
338	18.2	3.2
338	24.3	4.3
338	33.4	12.2
338	35.5	13.8
348	12.2	---
348	15.2	---
348	18.2	2.8
348	24.3	4.9
348	33.4	14.9
348	35.5	16.9
358	12.2	---
358	15.2	---
358	18.2	3.2
358	24.3	6.3
358	33.4	19.2
358	35.5	23.1

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.24 Solubility of A4 in Carbon Dioxide.

Source and Purity of Materials:
(1) A4 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H; Shamshipur,M, J. Chem. Eng. Data *43*, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A5); $C_{33}H_{38}N_4O_6$; [848354-96-1] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh, Yadollah Yamini, Ramin Miri, Bahram Hemmateenejad, J. Chem. Eng Data, *50*, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A5 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	0.7
338	18.2	0.9
338	24.3	3.2
338	33.4	4.6
338	35.5	4.7
348	12.2	---
348	15.2	0.7
348	18.2	2.01
348	24.3	3.3
348	33.4	6.9
348	35.5	7.3
358	12.2	---
358	15.2	1.4
358	18.2	0.9
358	24.3	5.0
358	33.4	9.5
358	35.5	11.0

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.25 Solubility of A5 in Carbon Dioxide.

Source and Purity of Materials:
(1) A5 was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$ 0.1 MPa
Solubility: $\pm$ 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.;S harghi,H; Shamshipur,M, J. Chem. Eng. Data *43*, 400-402 (1998)

Components:	Original Measurements:
(1) 1,4-Dihydro-2,6-dimethyl-4-(1-methyl-5-nitroimidazol -2-yl)-3,5-pyridinedicarboxylates derivative; (A6); $C_{29}H_{30}N_4O_6$;[196707-64-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Mostafa Khajeh,Yadollah Yamini, Ramin Miri,Bahram Hemmateenejad, J. Chem. Eng Data, *50*, 348-351 (2005)
Variables: T/K = 338, 348, 358 P/MPa = 12.2- 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of A6 (1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^6$
338	12.2	---
338	15.2	---
338	18.2	1.5
338	24.3	3.1
338	33.4	4.1
338	35.5	4.4
348	12.2	---
348	15.2	---
348	18.2	1.8
348	24.3	3.6
348	33.4	5.7
348	35.5	5.9
358	12.2	---
358	15.2	---
358	18.2	2.3
358	24.3	4.4
358	33.4	7.2
358	35.5	7.8

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given [1,2].The solid solutes were mixed well with the proper number of glass beads and packed into a 1.0mL extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction. Then it was loaded into an injection loop. The loop was depressurized into the collection vial. Finally the solubility was calculated by absorbance measurements using an UV-vis spectrophotometer.

Figure A.26 Solubility of A6 in Carbon Dioxide.

Source and Purity of Materials:
(1) A6was synthesized, purity 99.5%
(2) carbon dioxide source sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: ± 1K
Pressure: ± 0.1 MPa
Solubility: ± 7%

References:
(1) Yamini,Y.; Bahrmifar,N, J. Chem. Eng Data, *45*, 53-56 (2000)
(2) Fathi,M.R.; Yamini,Y.; Sharghi,H; Shamshipur,M, J. Chem. Eng. Data *43*, 400-402 (1998)

Components:	Original Measurements:
(1) Acetazolamide; $C_4H_6N_4O_3S_2$; [59-66-5]	Ana Rita C.Duarte, Susana Santiago, Herminio C. de
(2) Carbon Dioxide; CO_2; [124-38-9],with	Sousa, Catarina M.M.Duarte;J.Chem.Eng.Data,*50*,
Ethanol; C_2H_5OH; [64-17-5]	216-220(2005)

Variables:	Prepared By:
T/K = 313, 323	Dana E. Knox, Syed A. Abdullah
P/MPa = 13 - 21	

Experimental Data
Solubility of Acetazolamide(1) in Carbon Dioxide/Ethanol(2)

T/K	%mole fraction ethanol	P/MPa	$x_1 \times 10^5$
313	5.0	15.0	0.570
313	5.0	17.2	0.669
313	5.0	18.1	0.726
313	5.0	20.2	0.888
313	5.0	15.0	0.830
313	5.0	17.1	0.842
313	7.5	18.2	1.011
313	7.5	13.1	1.050
313	7.5	15.1	1.190
313	10.0	17.1	1.277
313	10.0	18.1	1.266
313	10.0	20.1	1.392
323	10.0	15.1	0.735
323	10.0	17.1	0.799
323	5.0	18.1	0.839
323	5.0	20.1	0.898

Auxiliary Information

The solubility of acetazolamide in the presence of a co solvent was measured using a static analytical high pressure apparatus. The determination of solubility was performed in a similar manner to that described by Matias et al[1].A stainless steel equilibrium cell is immersed in a thermostatic water bath. The cell was pressurized with the mixture of ethanol and carbon dioxide inside the syringe pump. The mixture of carbon dioxide, ethanol and acetazolamide is stirred for 1 hour, then taken through HPLC valve. The samples were collected by depressurization and expansion and calculated from measurement of the resulting sub-atmospheric pressure at the working temp.

Source and purity of materials:
(1) acetazolamide source sigma-aldrich, purity 99%.
(2) ethanol source Riedel-de Haen, purity 99.8%
(3) carbon dioxide source Air Liquide, purity 99.9%

Estimated Errors:
Temperature: ± 0.1C
Pressure: ± 0.04 MPa
Solubility: ± 0.5%

References:
1.Matias A.A.; Nunes A.V.M.; Casimiro T.; Duarte C.M.M, J. of supercritical Fluids, *28*, 201-206 (2004)

Figure A.27 Solubility of Acetazolamide in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) Benzoic acid; $C_7H_6O_2$; [65-85-0]	Jun-Su Jin, Chongli Zhong, Zeting Zhang, Ying Li,
(2) 98%Carbon Dioxide; CO_2; [124-38-9]; with 2% mixture of ethyl acetate; $CH_3COOC_2H_5$; [141-78-6] and ethanol; C_2H_5OH;[64-17-5].	Fluid Phase Equilibria, 226, 9-13 (2004)

Variables:	Prepared By:
T/K = 308.15, 318.15, 328.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 8 - 23	

Experimental Data

Solubility of Benzoic acid(1) in 98%carbon dioxide with 2%ethyl acetate and ethanol(2)

T/K	P/MPa	x_1 X 10^3
308.15	8.0	0.7141
308.15	11.0	1.1907
308.15	13.0	2.0222
308.15	17.0	3.1731
308.15	20.0	3.8215
308.15	23.0	4.5322
318.15	8.0	0.2025
318.15	11.0	0.8234
318.15	13.0	1.6772
318.15	17.0	3.8745
318.15	20.0	4.9912
318.15	23.0	5.5582
328.15	8.0	0.1021
328.15	11.0	0.6919
328.15	13.0	1.4172
328.15	17.0	4.6957
328.15	20.0	7.4183
328.15	23.0	8.6561

Auxiliary Information

A Flow type apparatus was used to measure the solubility. The experimental apparatus was described in detail previously[1].The cell was immersed in a constant-temperature stirred water bath. The super critical fluid was pressurized by pump. The solute was loaded by solid solute. They reached equilibrium though mass transfer. The saturated carbon dioxide flowed from the top of the cell into two weighed U-type tubes in turn. The solid solute collected in the U-type tubes was weighed by a single pan balance after drying. The volume of carbon dioxide was measured by calibrated wet gas flow meter.

Source and Purity of Materials:

(1) Benzoic acid source Beijing chemical Regent Factory, purity 99.5%

(2) carbon dioxide source Beijing Chemical regent Factory, purity 99.9%

(3) Ethanol and ethyl acetate source Beijing Chemical regent Factory, purity 99.5%

Estimated Error:

Temperature: $\pm$0.1 K

Pressure: $\pm$0.05 MPa

Solubility: $\pm$5%

References:

1. Q.Li, Z.Zhang, C.Zhong ,Y.Liu, Q.Zhou, Fluid Phase equillibria,207,183-192(2003)

Figure A.28 Solubility of Benzoic acid in Carbon Dioxide with Ethanol and Ethyl Acetate.

Components:	Original Measurements:
(1) Benzoic acid; $C_7H_6O_2$; [65-85-0]	Jun-Su Jin, Chongli Zhong, Zeting Zhang, Ying Li,
(2) 98%Carbon Dioxide; CO_2; [124-38-9];with	Fluid Phase Equilibria, *226*, 9-13 (2004)
2% ethyl acetate; $CH_3COOC_2H_5$; [141-78-6]	

Variables:	Prepared By:
T/K = 328.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 8 - 23	

Experimental Data

Solubility of Benzoic acid(1) in 98%carbondioxide with 2%ethyl acetate(2)

T/K	P/MPa	$x_1 \times 10^3$
328.15	8.0	0.1205
328.15	11.0	0.6421
328.15	13.0	1.0725
328.15	17.0	3.3353
328.15	20.0	4.2538
328.15	23.0	4.8961

Auxiliary Information

A Flow type apparatus was used to measure the solubility. The experimental apparatus was described in detail previously[1].The cell was immersed in a constant-temperature stirred water bath. The super critical fluid was pressurized by pump. The solute was loaded by solid solute. They reached equilibrium though mass transfer. The saturated carbon dioxide flowed from the top of the cell into two weighed U-type tubes in turn. The solid solute collected in the U-type tubes was weighed by a single pan balance after drying. The volume of carbon dioxide was measured by calibrated wet gas flow meter.

Source and Purity of Materials:
(1) Benzoic acid source Beijing chemical Regent Factory, purity 99.5%
(2) carbon dioxide source Beijing Chemical regent Factory, purity 99.9%
(3) ethyl acetate source Beijing Chemical regent Factory, purity 99.5%

Estimated error:
Temperature: ± 0.1K
Pressure: ± 0.05 MPa
Solubility: ± 5%

References:
1. Q.Li,Z.Zhang,C.Zhong,Y.Liu,Q.Zhou, Fluid Phase equillibria,*207*,183-192(2003)

Figure A.29 Solubility of Benzoic acid in Carbon Dioxide with Ethyl Acetate.

Components:	Original Measurements:
(1) Benzoic acid; $C_7H_6O_2$; [65-85-0]	Jun-Su Jin, Chongli Zhong, Zeting Zhang, Ying Li,
(2) 98%Carbon Dioxide; CO_2; [124-38-9]; with 2% ethanol; C_2H_5OH; [64-17-5]	Fluid Phase Equilibria, *226*, 9-13 (2004)
Variables: T/K = 328.15 P/MPa = 8 - 23	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Benzoic acid(1) in 98%carbon dioxide with 2%ethanol(2)

T/K	P/MPa	$x_1 \times 10^3$
328.15	8.0	0.3659
328.15	11.0	3.3940
328.15	13.0	8.0922
328.15	17.0	13.594
328.15	20.0	15.305
328.15	23.0	16.821

Auxiliary Information

A Flow type apparatus was used to measure the solubility. The experimental apparatus was described in detail previously[1].The cell was immersed in a constant-temperature stirred water bath. The super critical fluid was pressurized by pump. The solute was loaded by solid solute. They reached equilibrium though mass transfer. The saturated carbon dioxide flowed from the top of the cell into two weighed U-type tubes in turn. The solid solute collected in the U-type tubes was weighed by a single pan balance after drying. The volume of carbon dioxide was measured by calibrated wet gas flow meter.

Source and Purity of Materials:
(1) Benzoic acid source Beijing chemical Regent Factory, purity 99.5%
(2) carbon dioxide source Beijing Chemical regent Factory, purity 99.9%
(3) ethanol source Beijing Chemical regent Factory, purity 99.5%

Estimated error:
Temperature: ±0.1K
Pressure: ±0.05 MPa
Solubility: ±5%

References:
1. Q.Li,Z.Zhang,C.Zhong,Y.Liu,Q.Zhou, Fluid Phase equillibria,*207*,183-192(2003)

Figure A.30 Solubility of Benzoic Acid in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) Benzoic acid; $C_7H_6O_2$; [65-85-0]	Jun-Su Jin, Chongli Zhong, Zeting Zhang, Ying Li,
(2) Carbon Dioxide; CO_2; [124-38-9]	Fluid Phase Equilibria, *226*, 9-13 (2004)
Variables:	Prepared By:
T/K = 328.15	Dana E. Knox, Syed A. Abdullah
P/MPa = 8 - 23	

Experimental Data
Solubility of Benzoic acid(1) in carbon dioxide(2)

T/K	P/MPa	$x_1 \times 10^3$
328.15	8.0	0.0695
328.15	11.0	0.3388
328.15	13.0	0.6373
328.15	17.0	2.4475
328.15	20.0	3.6975
328.15	23.0	4.2325

Auxiliary Information

A Flow type apparatus was used to measure the solubility. The experimental apparatus was described in detail previously[1].The cell was immersed in a constant-temperature stirred water bath. The super critical fluid was pressurized by pump. The solute was loaded by solid solute. They reached equilibrium though mass transfer. The saturated carbon dioxide flowed from the top of the cell into two weighed U-type tubes in turn. The solid solute collected in the U-type tubes was weighed by a single pan balance after drying. The volume of carbon dioxide was measured by calibrated wet gas flow meter.

Source and Purity of Materials:
(1) Benzoic acid source Beijing chemical Regent Factory, purity 99.5%
(2) carbon dioxide source Beijing Chemical regent Factory, purity 99.9%

Estimated error:
Temperature: ±0.1K
Pressure: ±0.05 MPa
Solubility: ±5%

References:
1. Q.Li,Z.Zhang,C.Zhong,Y.Liu,Q.Zhou, Fluid Phase equillibria,*207*,183-192(2003)

Figure A.31 Solubility of Benzoic Acid in Carbon Dioxide.

Components:	Original Measurements:
(1) Pinodol; $C_{14}H_{20}N_2O_2$; [13523-86-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Randy D.Weinstein, Joesph J.Gribbin, Kenneth R.Muske, J. Chem. Eng. Data, *50*, 226-229 (2005)
Variables: T/K = 298, 308, 318 P/MPa = 8 - 27.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Pinodol(1) in carbon dioxide(2)

T/K	P/bar	P/MPa-Compiler	$x_1 \times 10^4$
298	80	8.0	0.660
298	100	10.0	0.842
298	150	15.0	1.330
298	200	20.0	1.580
298	250	25.0	2.100
298	275	27.5	2.240
308	80	8.0	0.308
308	100	10.0	0.380
308	150	15.0	0.938
308	200	20.0	1.410
308	250	25.0	2.000
308	275	27.5	2.110
318	80	8.0	0.337
318	100	10.0	0.282
318	150	15.0	0.875
318	200	20.0	1.210
318	250	25.0	1.780
318	275	27.5	1.920

Auxiliary Information

A dynamic system manufactured by Thar Design Technologies was used. Details of the experimental apparatus are presented in the previous paper along with the expected errors in the measurements[1].To measure the solubility, the vessel was loaded and pressured. Once all the solid particles were visually observed to dissolve, the vessel was isolated from the pump, and it's volume slowly increased until material was seen to drop out of the solution. With the known amount of drug placed in the vessel and density of pure carbon dioxide; the solubility of drug can be calculated.

Source and Purity of Materials:
(1) Pinodol source Sigma-Aldrich, purity 98%
(2) carbon dioxide source BOC gases, purity 99%.

Estimated Errors:
Temperature: not stated
Pressure: not stated
Solubility: not stated

References:
1. Weinstein,R.D; Muske,K.R.; Moriarty,J.; Schmidt,E.K, J. Chem. Eng. Data, *49*, 547-552 (2004)

Figure A.32 Solubility of Pinodol in Carbon Dioxide.

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Components:	Original Measurements:
(1) Beclomethasone dipropionate; $C_{28}H_{37}ClO_7$; [5534-09-8]	Alireza Vatanara, Abdolhossein Rouholamini Najafabadi,Mostafa Khajeh, Yadollah Yamini,
(2) Carbon Dioxide; CO_2; [124-38-9].	J. Of Supercritical Fluids, *33* , 21-25 (2005).
Variables: T/K=338,348,358 P/MPa=21.3-38.5	Prepared By: Dana E. Knox, Syed A. Abdullah.

Experimental Data
Solubility of Beclomethasone dipropionate(1) in carbon dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
338	213	21.3	7.07
338	243	24.3	7.82
338	274	27.4	1.09
338	304	30.4	1.17
338	334	33.4	1.38
338	355	35.5	1.66
338	385	38.5	2.12
348	213	21.3	8.06
348	243	24.3	7.95
348	274	27.4	9.88
348	304	30.4	1.37
348	334	33.4	1.75
348	355	35.5	1.86
348	385	38.5	2.15
358	213	21.3	8.31
358	243	24.3	8.42
358	274	27.4	9.92
358	304	30.4	1.66
358	334	33.4	2.29
358	355	35.5	2.51
358	385	38.5	3.36

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. The solid solutes were mixed well with some glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. A portion of saturated supercritical carbon dioxide was loaded into the injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a model Shimadzu UV-vis spectrophotometer.

Source and Purity of Materials:
1. Beclomethasone dipropionate source Sina Daru, Iran, purity unstated.
2. carbon dioxide source Sabalan, Iran, purity 99.99%.

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubility: ±6%

Figure A.33 Solubility of Beclomethasone Dipropionate in Carbon Dioxide.

Components:	Original Measurements:
(1) Budesonide; $C_{25}H_{34}O_6$; [51333-22-3]	Alireza Vatanara, Abdolhossein Rouholamini Najafabadi, Mostafa Khajeh, Yadollah Yamini,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, *33* , 21-25 (2005).
Variables: T/K = 338, 348, 358 P/MPa = 21.3 - 38.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Budesonide(1) in carbon dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
338	213	21.3	5.93
338	243	24.3	6.67
338	274	27.4	9.62
338	304	30.4	1.04
338	334	33.4	1.23
338	355	35.5	1.50
338	385	38.5	1.98
348	213	21.3	6.79
348	243	24.3	6.74
348	274	27.4	8.58
348	304	30.4	1.22
348	334	33.4	1.57
348	355	35.5	1.68
348	385	38.5	2.51
358	213	21.3	6.95
358	243	24.3	7.12
358	274	27.4	8.58
358	304	30.4	1.48
358	334	33.4	2.08
358	355	35.5	2.29
358	385	38.5	2.91

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. The solid solutes were mixed well with some glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. A portion of saturated supercritical carbon dioxide was loaded into the injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a model Shimadzu UV-vis spectrophotometer.

Source and Purity of Materials:
1. Budesonide source
   Sina Daru, Iran, purity unstated.
2. carbon dioxide source Sabalan, Iran, purity 99.99%

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubility: ±6%

Figure A.34 Solubility of Budesonide in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-9,10-anthraquinone; AQ1; $C_{14}H_8O_3$; [129-43-1]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini , Hashem Sharghi ,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, 32, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ1(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	1.00
308	18.2	1 03
308	24.3	1.23
308	33.4	1.34
308	35.5	1.35
318	12.2	0.30
318	18.2	0.95
318	24.3	1.41
318	33.4	1.63
318	35.5	1.71
328	12.2	0.57
328	18.2	1.01
328	24.3	1.54
328	33.4	2.08
328	35.5	2.32
338	12.2	0.45
338	18.2	1.27
338	24.3	2.08
338	33.4	3.30
338	35.5	3.59
348	12.2	0.40
348	18.2	1.67
348	24.3	2.66
348	33.4	4.18
348	35.5	4.45

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Super critical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ1 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co, Iran, purity 99.99%

Estimated Error;
Temperature: ± 1K
Pressure: ±0.1 MPa
Solubility: ±5%

References:
1..M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J. Chem. Eng. Data, 43, 400-402 (1998)

Figure A.35 Solubility of AQ1 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-2-methyl-9,10-anthraquinone; AQ2; $C_{15}H_{10}O_3$; [6268-09-3] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini , Hashem Sharghi , J. of Supercritical Fluids, 32, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ2(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	0.63
308	18.2	1.51
308	24.3	1.76
308	33.4	2.32
308	35.5	2.44
318	12.2	0.59
318	18.2	1.38
318	24.3	2.06
318	33.4	2.95
318	35.5	3.12
328	12.2	0.67
328	18.2	1.36
328	24.3	2.62
328	33.4	4.06
328	35.5	4.35
338	12.2	0.17
338	18.2	1.56
338	24.3	3.20
338	33.4	5.16
338	35.5	5.51
348	12.2	0.09
348	18.2	1.85
348	24.3	3.65
348	33.4	7.21
348	35.5	7.37

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ2 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co, Iran, purity 99.99%

Estimated Error;
Temperature: $\pm$ 1K
Pressure: $\pm$0.1 MPa
Solubility: $\pm$5%

References:
1. M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J. Chem. Eng. Data, 43, 400-402 (1998)

Figure A.36 Solubility of AQ2 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-2-(methoxymethyl)-9,10-anthra Quinine; AQ3; $C_{16}H_{12}O_4$; [174568-52-6]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini , Hashem Sharghi ,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, *32*, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ3(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	0.77
308	18.2	1.66
308	24.3	1.81
308	33.4	2.05
308	35.5	2.29
318	12.2	0.60
318	18.2	1.52
318	24.3	2.23
318	33.4	2.57
318	35.5	2.55
328	12.2	0.51
328	18.2	1.51
328	24.3	2.48
328	33.4	3.32
328	35.5	3.33
338	12.2	0.57
338	18.2	1.42
338	24.3	2.45
338	33.4	4.25
338	35.5	4.34
348	12.2	0.59
348	18.2	1.37
348	24.3	2.47
348	33.4	5.34
348	35.5	5.37

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the Solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ3 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co,Iran, purity 99.99%

Estimated Error;
Temperature: ± 1K
Pressure: ±0.1 MPa
Solubility: ±5%

References:
1.M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.37 Solubility of AQ3 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-2-(ethoxymethyl)-9,10-anthra Quinine; AQ4; $C_{17}H_{14}O_4$; [101451-69-8]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi ,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, 32, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ4(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	0.80
308	18.2	1.94
308	24.3	2.62
308	33.4	3.07
308	35.5	3.10
318	12.2	1.00
318	18.2	2.63
318	24.3	3.48
318	33.4	4.50
318	35.5	4.86
328	12.2	1.09
328	18.2	2.38
328	24.3	3.79
328	33.4	5.85
328	35.5	6.58
338	12.2	1.15
338	18.2	1.71
338	24.3	3.96
338	33.4	6.97
338	35.5	8.12
348	12.2	0.23
348	18.2	1.75
348	24.3	3.83
348	33.4	9.19
348	35.5	11.00

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ4 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co, Iran, purity 99.99%

Estimated Error;
Temperature: $\pm$ 1K
Pressure: $\pm$0.1 MPa
Solubility: $\pm$5%

References:
1.M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J.Chem.Eng.Data, 43,400-402(1998).

Figure A.38 Solubility of AQ4 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-2-(propoxymethyl)-9,10-anthra Quinine; AQ5; $C_{18}H_{16}O_4$; [174568-52-6] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi , J. of Supercritical Fluids, 32, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ5(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	1.03
308	18.2	2.76
308	24.3	3.22
308	33.4	4.31
308	35.5	5.11
318	12.2	1.22
318	18.2	3.47
318	24.3	5.21
318	33.4	7.10
318	35.5	7.58
328	12.2	1.89
328	18.2	3.43
328	24.3	5.49
328	33.4	9.27
328	35.5	10.17
338	12.2	2.67
338	18.2	3.56
338	24.3	6.76
338	33.4	12.59
338	35.5	13.92
348	12.2	1.04
348	18.2	2.36
348	24.3	7.08
348	33.4	15.06
348	35.5	16.76

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ5 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co, Iran, purity 99.99%

Estimated Error;
Temperature: $\pm$ 1K
Pressure: $\pm$0.1 MPa
Solubility: $\pm$5%

Figure A.39 Solubility of AQ5 in carbon Dioxide.

References:
1.M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J.Chem.Eng.Data, 43,400-402(1998).

Components:	Original Measurements:
(1) 1-hydroxy-2-(butoxymethyl)-9,10-anthra Quinine; AQ6; $C_{19}H_{18}O_4$; [174568-52-6] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi , J. of Supercritical Fluids, 32, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ6(1) in carbon dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	1.83
308	18.2	4.06
308	24.3	5.21
308	33.4	6.41
308	35.5	7.03
318	12.2	1.57
318	18.2	3.78
318	24.3	6.06
318	33.4	8.90
318	35.5	9.71
328	12.2	0.90
328	18.2	3.82
328	24.3	6.51
328	33.4	12.34
328	35.5	13.46
338	12.2	0.87
338	18.2	3.88
338	24.3	7.85
338	33.4	16.06
338	35.5	18.14
348	12.2	0.82
348	18.2	2.81
348	24.3	13.34
348	33.4	24.14
348	35.5	26.99

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ6 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co, Iran, purity 99.99%

Estimated Error;
Temperature: ± 1K
Pressure: ±0.1 MPa
Solubility: ±5%

Figure A.40 Solubility of AQ6 in Carbon Dioxide.

References:
1.M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J.Chem.Eng.Data, 43,400-402(1998).

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Components:	Original Measurements:
(1) 1-hydroxy-2-(n-amyloxymethyl)-9,10-anthra Quinone; AQ7; $C_{20}H_{20}O_4$; [174568-55-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi ,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, *32*, 47-53 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa=12.2-13.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of AQ7(1) in carbon dioxide(2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	2.61
308	18.2	4.80
308	24.3	6.08
308	33.4	8.54
308	35.5	7.82
318	12.2	1.74
318	18.2	4.74
318	24.3	7.99
318	33.4	12.52
318	35.5	12.17
328	12.2	1.64
328	18.2	4.91
328	24.3	9.02
328	33.4	14.42
328	35.5	14.80
338	12.2	0.51
338	18.2	3.53
338	24.3	9.23
338	33.4	16.52
338	35.5	18.44
348	12.2	0.25
348	18.2	2.33
348	24.3	7.59
348	33.4	22.13
348	35.5	26.40

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination was used. A detailed description of the operating procedure is given elsewhere[1].The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized into a collection vial containing methanol. The solubility were calculated by absorbance measurements using a UV-spectrophotometer.

Source and Purity of Materials:
(1) AQ7 was synthesized, purity 99.5%
(2) carbon dioxide source RohamGas Co,Iran, purity 99.99%

Estimated Error;
Temperature: ± 1K
Pressure: ±0.1 MPa
Solubility: ±5%

References:
1.M.R.Fathi,Y.Yamini,H.Sharghi,M.Shamsipur J.Chem.Eng.Data, *43*,400-402(1998).

Figure A.41 Solubility of AQ7 in Carbon Dioxide.

Components:	Original Measurements:
(1) Dibenzoylmethane; $C_{15}H_{12}O_2$; [120-46-7] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Ghiasvand, Yadollah Yamini, J. Chem. Eng. Data, 49, 1483-1486 (2004).
Variables: T/K = 308, 318, 328, 338 P/MPa = 10.1 - 35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of Dibenzoylmethane (1) in carbon dioxide(2)

T/K	P/bar	P/MPa-compiler	x_1 X 10^4
308	101	10.1	7.3
308	152	15.2	13.0
308	203	20.3	12.8
308	253	25.3	16.9
308	304	30.4	17.6
308	355	35.5	21.5
318	101	10.1	6.7
318	152	15.2	10.5
318	203	20.3	15.9
318	253	25.3	21.9
318	355	35.5	37.6
328	101	10.1	3.0
328	152	15.2	8.2
328	203	20.3	24.7
328	253	25.3	37.7
328	304	30.4	45.3
328	355	35.5	60.3
338	101	10.1	1.0
338	152	15.2	7.5
338	203	20.3	31.8
338	253	25.3	56.7
338	304	30.4	76.5
338	355	35.5	99.5

Auxiliary Information

A Suprex MPS/225 integrated SFE-SFC system modified for solubility determination in SFE mode was used.A complete description of the system was reported in the previous paper[1].Solubility measurements were carried out with a 1-ml extraction vessel.30 minutes was found to be adequate to ensure the attainment of equilibrium. The solid solutes were mixed well with 1g of glass beads and packed into the extraction vessel. A 2100 Shimadzu UV-vis spectrophotometer was used for solubility determination by absorbance measurements.

Source and Purity of Materials:
(1) Dibenzoylmethane source Merck, purity unstated.
(2) carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ±1K
Pressure: ±0.5 bar
Solubility: ±3%

References:
1. Yamini,Y.; Fathi,M.R.; Alizadeh,N.; Shamsipur,M, Fluid Phase Equilibria, 152, 229-305 (1998).

Figure A.42 Solubility of Dibenzoylmethane in Carbon Dioxide.

Components:	Original Measurements:
(1) 8-hydroxyquinoline; C₉H₇NO;[148-24-3]	Mojtaba Shamsipur, Ali Reza Ghiasvand,
(2) Carbon Dioxide; CO₂; [124-38-9]	Yadollah Yamini, J. Chem. Eng. Data ,*49*,1483-1486 (2004).
Variables: T/K = 308, 318, 328, 338 P/MPa=10.1-35.5	Prepared By: Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of 8-hydroxyquinoline (1) in carbon dioxide(2)

T/K	P/bar	P/MPa-compiler	$x_1 \times 10^4$
308	101	10.1	23.2
308	152	15.2	35.4
308	203	20.3	42.1
308	253	25.3	47.7
308	304	30.4	52.7
308	355	35.5	54.0
318	101	10.1	10.9
318	152	15.2	43.3
318	203	20.3	56.1
318	253	25.3	63.9
318	355	35.5	79.6
328	101	10.1	8.7
328	152	15.2	49.5
328	203	20.3	72.6
328	253	25.3	90.0
328	304	30.4	103.9
328	355	35.5	116.0
338	101	10.1	14.0
338	152	15.2	51.4
338	203	20.3	83.1
338	253	25.3	106.3
338	304	30.4	144.2
338	355	35.5	167.1

Auxiliary Information

A Suprex MPS/225 integrated SFE-SFC system modified for solubility determination in SFE mode was used. A complete description of the system was reported in the previous paper[1].Solubility measurements were carried out with a 1-ml extraction vessel.30 minutes was found to be adequate to ensure the attainment of equilibrium. The solid solutes were mixed well with 1g of glass beads and packed into the extraction vessel. A 2100 Shimadzu UV-vis spectrophotometer was used for solubility determination by absorbance measurements.

Source and Purity of Materials:
(1)8-hydroxyquinoline source Merck, purity unstated.
(2) carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ±1K
Pressure: ±0.5 bar
Solubility: ±3%

References:
1. Yamini,Y.; Fathi,M.R.; Alizadeh,N.; Shamsipur,M, Fluid Phase Equilibria, *152*, 229-305 (1998).

Figure A.43 Solubility of 8-hydroxyquinoline in Carbon Dioxide.

Components:	Original Measurements:
(1) 1,10-Phenanthroline; $C_{12}H_8N_2$; [66-71-7]	Mojtaba Shamsipur, Ali Reza Ghiasvand, Yadollah
(2) Carbon Dioxide; CO_2; [124-38-9]	Yamini, J. Chem. Eng. Data, *49*, 1483-1486 (2004)
Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348	Dana E. Knox, Syed A. Abdullah
P/MPa = 10.1 - 35.5	

Experimental Data
Solubility of 1,10-Phenanthroline (1) in carbon dioxide(2)

T/K	P/bar	P/MPa-compiler	$x_1 \times 10^4$
308	101	10.1	23.2
308	152	15.2	35.4
308	203	20.3	42.1
308	253	25.3	47.7
308	304	30.4	52.7
308	355	35.5	54.0
318	101	10.1	10.9
318	152	15.2	43.3
318	203	20.3	56.1
318	253	25.3	63.9
318	355	35.5	79.6
328	101	10.1	8.7
328	152	15.2	49.5
328	203	20.3	72.6
328	253	25.3	90.0
328	304	30.4	103.9
328	355	35.5	116.0
338	101	10.1	14.0
338	152	15.2	51.4
338	203	20.3	83.1
338	253	25.3	106.3
338	304	30.4	144.2
338	355	35.5	167.1
348	101	10.1	0.2
348	152	15.2	1.5
348	203	20.3	5.2
348	253	25.3	7.3
348	304	30.4	9.2
348	355	35.5	10.4

Auxiliary Information

A Suprex MPS/225 integrated SFE-SFC system modified for solubility determination in SFE mode was used. A complete description of the system was reported in the previous paper[1].Solubility measurements were carried out with a 1-ml extraction vessel.30 minutes was found to be adequate to ensure the attainment of equilibrium. The solid solutes were mixed well with 1g of glass beads and packed into the extraction vessel. A 2100 Shimadzu UV-vis spectrophotometer was used for solubility determination by absorbance measurements.

Source and Purity of Materials:
(1) 1,10-phenanthroline source Merck, purity unstated.
(2) carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ± 1 K
Pressure: ± 0.5 bar
Solubility: $\pm 3\%$

References:
1. Yamini,Y.; Fathi,M.R.; Alizadeh,N.; Shamsipur,M, Fluid Phase Equilibria, *152*, 229-305 (1998).

Figure A.44 Solubility of 1,10-phenanthroline in Carbon Dioxide.

Components:	Original Measurements:
(1) Aspirin; $C_9H_8O_4$; [50-78-2]	Zhen Huang, Wei D.Lu, Sibudjing Kawi, Yee C.Chiew
(2) 97%CarbonDioxide; CO_2; [124-38-9],with 3%Acetone; CH_3COCH_3; [67-64-1]	J. Chem. Eng. Data , *49*, 1323-1327 (2004)

Variables:	Prepared By:
T/K = 318.15, 328.15	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.0 - 20.0	

Experimental Data
Solubility of aspirin(1) in 97%Carbon Dioxide with 3%acetone(2)

T/K	P/MPa	x_1 x 10^3
318.15	10.0	0.218
318.15	12.0	0.337
318.15	15.0	0.428
318.15	17.2	0.525
318.15	20.0	0.615
328.15	10.0	0.113
328.15	12.0	0.321
328.15	15.0	0.498
328.15	17.2	0.612
328.15	20.0	0.757

Auxiliary Information

The solubility was measured using a continuous flow technique. High purity carbon dioxide was fed into an HPLC pump. The extraction vessel was loaded with alternate layers of aspirin powder. downstream to the extraction vessel, the saturated solvent and the aspirin solution was depressurized to ambient conditions and the aspirin was precipitated in a glass U-tube collector immersed in an ice bath. The collected aspirin was weighed using a balance. By means of the measured solute mass and solvent volume, the solubility value was readily obtained.

Source and Purity of Materials:
1. aspirin source Sigma Chemicals, USA; purity 99.5%
2. carbon dioxide source Soxal, Singapore; purity 99.8%
3. acetone source Tedia Company, USA; purity 99.97%

Estimated Error:
Temperature : $\pm$ 0.01K
Pressure : $\pm$ 1 bar
Solubility : not stated.

Figure A.45 Solubility of Aspirin in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) Aspirin; $C_9H_8O_4$; [50-78-2]	Zhen Huang, Wei D.Lu, Sibudjing Kawi, Yee C.Chiew
(2) Carbon Dioxide; CO_2;[124-38-9]	J. Chem. Eng. Data, 49, 1323-1327 (2004)

Variables:	Prepared By:
T/K = 308.15, 318.15, 328.15	Dana E. Knox; Syed A. Abdullah
P/MPa = 12.0 - 25.0	

Experimental Data
Solubility of aspirin(1) in Carbon Dioxide(2)

T/K	P/MPa	$x_1 \times 10^3$
308.15	12.0	0.89
308.15	15.0	1.12
308.15	17.2	1.22
308.15	18.5	1.29
308.15	20.0	1.33
308.15	21.5	1.42
308.15	23.0	1.45
308.15	25.0	1.51
318.15	12.0	0.72
318.15	15.0	1.39
318.15	17.2	1.75
318.15	18.5	1.95
318.15	20.0	2.12
318.15	21.5	2.28
318.15	23.0	2.34
318.15	25.0	2.58
328.15	12.0	0.63
328.15	15.0	1.37
328.15	17.2	1.82
328.15	18.5	2.34
328.15	20.0	2.77
328.15	21.5	2.86
328.15	23.0	3.03
328.15	25.0	3.47

Auxiliary Information

The solubility was measured using a continuous flow technique. High purity carbon dioxide was fed into an HPLC pump. The extraction vessel was loaded with alternate layers of aspirin powder. downstream to the extraction vessel, the saturated solvent and the aspirin solution was depressurized to ambient conditions and the aspirin was precipitated in a glass U-tube collector immersed in an ice bath. The collected aspirin was weighed using a balance. By means of the measured solute mass and solvent volume, the solubility value was readily obtained.

Source and Purity of Materials:
1. aspirin source Sigma Chemicals, USA; purity 99.5%
2. carbon dioxide source Soxal, Singapore; purity 99.8%

Estimated Error:
Temperature : $\pm$ 0.01K
Pressure : $\pm$ 1 bar
Solubility : not stated.

Figure A.46 Solubility of Aspirin in Carbon Dioxide.

Components:	Original Measurements:
(1) Medroxyprogesterone acetate; $C_{24}H_{34}O_4$; [71-58-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Mehdi Asghari-Khiavi, Yadollah Yamini, Mir Ali J. of Supercritical Fluids, *30*, 111-117 (2004)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of medroxyprogesterone(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	122	12.2	0.40
308	182	18.2	0.74
308	243	24.3	1.07
308	304	30.4	1.42
308	355	35.5	1.86
318	122	12.2	0.30
318	182	18.2	0.65
318	243	24.3	1.09
318	304	30.4	1.75
318	355	35.5	2.26
328	122	12.2	0.23
328	182	18.2	0.56
328	243	24.3	1.14
328	304	30.4	2.08
328	355	35.5	2.66
338	122	12.2	0.19
338	182	18.2	0.55
338	243	24.3	1.24
338	304	30.4	2.53
338	355	35.5	3.39
348	122	12.2	0.16
348	182	18.2	0.54
348	243	24.3	1.34
348	304	30.4	2.98
348	355	35.5	4.13

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with modified static system for the solubility determination was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid samples were mixed with proper amounts of glass bead and packed into the extraction vessel. Carbon Dioxide was pressurized and passed into the extraction vessel. After equilibrium, portion of saturated supercritical carbon dioxide was transferred into the collection vial. Then the sample loop was washed with methanol and transferred into the collection vial. Finally, the quantitative analysis was performed by UV absorption.

Figure A.47 Solubility of Medroxyprogesterone in Carbon Dioxide

Source and Purity of Materials:
1. medroxyprogesterone source Food and Drug Quality Lab, Tehran; purity 99%.
2. carbon dioxide source Sabalan, Iran; purity 99.99%.

Estimated Error:
Temperature : ± 1K
Pressure : ± 0.5 bar
Solubility : ± 4%

References:
1. M.R.Fathi, Y.Yamini, H.Sharghi, M. Shamsipur, J. Chem. Eng. Data, *43*,400 (1998).

Components:	Original Measurements:
(1) Cyproterone Acetate;$C_{24}H_{29}ClO_4$; [427-51-0]	Mehdi Asghari-Khiavi, Yadollah Yamini, Mir Ali
(2) Carbon Dioxide; CO_2; [124-38-9]	J. of Supercritical Fluids, *30*, 111-117 (2004)

Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348 P/MPa=12.2-35.5	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of cyproterone acetate(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	122	12.2	0.45
308	182	18.2	0.81
308	243	24.3	1.35
308	304	30.4	1.76
308	355	35.5	2.53
318	122	12.2	0.35
318	182	18.2	0.72
318	243	24.3	1.16
318	304	30.4	1.65
318	355	35.5	2.61
328	122	12.2	0.23
328	182	18.2	0.52
328	243	24.3	0.97
328	304	30.4	1.58
328	355	35.5	2.50
338	122	12.2	0.17
338	182	18.2	0.36
338	243	24.3	0.83
338	304	30.4	1.45
338	355	35.5	2.55
348	122	12.2	0.13
348	182	18.2	0.23
348	243	24.3	0.63
348	304	30.4	1.34
348	355	35.5	2.45

Auxiliary Information

A Suprex MPS/225 integrated SFE/SFC system equipped with modified static system for the solubility determination was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid samples were mixed with proper amounts of glass bead and packed into the extraction vessel. Carbon Dioxide was pressurized and passed into the extraction vessel. After equilibrium, portion of saturated supercritical carbon dioxide was transferred into the collection vial. Then the sample loop was washed with methanol and transferred into the collection vial. Finally, the quantitative analysis was performed by UV absorption.

Source and Purity of Materials:
1. cyproterone acetate source Food and Drug Quality Lab, Tehran; purity 99%.
2. carbon dioxide source Sabalan, Iran; purity 99.99%.

Estimated Error:
Temperature : ± 1K
Pressure : ± 0.5 bar
Solubility : ± 4%

References:
1. M.R.Fathi, Y.Yamini, H.Sharghi, M. Shamsipur,J.Chem.Eng.Data,*43*,400 (1998).

Figure A.48 Solubility of Cyprotene in Carbon Dioxide.

Components:	Original Measurements:
(1) Caffeine; $C_8H_{10}N_4O_2$; [58-08-2]	G.I.Burgos-Solorzano, J.F.Brennecke, M.A. Stadtherr
	Fluid Phase Equilibria, *220*, 57-69 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 313	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.0 - 30.0	

Experimental Data
Solubility of caffeine(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \ X \ 10^5$
313	100.0	10.00	6.3
313	103.4	10.34	7.8
313	137.9	13.79	14.0
313	150.0	15.00	15.0
313	172.4	17.24	19.0
313	206.8	20.68	25.0
313	300.0	30.00	37.0

Auxiliary Information

Solubility measurements were performed with a custom-built re- circulating high-pressure apparatus[i]. The equilibrium vessel is filled with a large excess of the compound of interest. The system is filled with carbon dioxide to the desired pressure from the ISCO pump. During equilibration the valve allows by-passing of the sample loop. After the pressure stabilizes, the temperature controllers and heaters are activated, taking care to avoid temperature shoot. Once thermodynamic equilibrium is reached, the valve is shifted to the load position to fill the sample loop with the saturated supercritical carbon dioxide /solute solution. Then the sample is led into a liquid collection solvent. Once no more bubbles are observed in the collection solvent, the sample loop is collected in the collection vial. The sample is analyzed using a UV-vis system spectrometer.

Source and Purity of Materials:
1. caffeine source Sigma, purity unstated.
2. carbon dioxide source Mittler supply, purity unstated.

Estimated Errors:
Temperature: $\pm$ 0.1K
Pressure: $\pm$ 0.34bar
Solubility: $\pm$ 10%

References:
1 .A.M. Scurto, High-pressure phase and chemical equilibria of beta-Diketone ligands and chelates with Carbon dioxide, Ph.D. Thesis, University of Norte Dame, Norte dame, 2002.

Figure A.49 Solubility of Caffeine in Carbon Dioxide.

Components:	Original Measurements:
(1) Uracil; $C_4H_4N_2O_2$; [66-22-8]	G.I.Burgos-Solorzano, J.F.Brennecke, M.A. Stadtherr
	Fluid Phase Equilibria, *220*, 57-69 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables:	Prepared By:
T/K = 313, 333	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.0 - 30.0	

Experimental Data
Solubility of uracil(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
313.1	100.0	10.00	2.9
313.0	125.0	12.50	2.9
313.2	150.0	15.00	8.4
313.1	200.0	20.00	12.0
313.1	249.9	24.99	20.0
313.0	299.8	29.98	36.0
333.1	100.0	10.00	2.3
333.0	125.0	12.50	2.5
333.0	150.0	15.00	5.5
333.2	200.1	20.01	27.0
333.1	250.0	25.00	58.0
333.0	299.9	29.99	130.0

Auxiliary Information

Solubility measurements were performed with a custom-built re-circulating high-pressure apparatus[1]. The equilibrium vessel is filled with a large excess of the compound of interest. The system is filled with carbon dioxide to the desired pressure from the ISCO pump. During equilibration the valve allows by-passing of the sample loop. After the pressure stabilizes, the temperature controllers and heaters are activated, taking care to avoid temperature shoot. Once thermodynamic equilibrium is reached, the valve is shifted to the load position to fill the sample loop with the saturated supercritical carbon dioxide /solute solution. Then the sample is led into a liquid collection solvent. Once no more bubbles are observed in the collection solvent, the sample loop is collected in the collection vial. The sample is analyzed using a UV-vis system spectrometer.

Source and Purity of Materials:
1. uracil source Sigma, purity 99%.
2. carbon dioxide source Scott Specialty Gases; purity unstated.

Estimated Errors:
Temperature: ± 0.1K
Pressure: ± 0.34bar
Solubility: ± 15%

References:
1. A.M. Scurto, High-pressure phase and chemical equilibria of beta-Diketone ligands and chelates with Carbon dioxide, Ph.D. Thesis, University of Norte Dame, Norte dame, 2002.

Figure A.50 Solubility of Uracil in Carbon Dioxide.

Components:	Original Measurements:
(1) Erythromycin; $C_{37}H_{67}NO_{13}$; [114-07-8].	G.I.Burgos-Solorzano, J.F.Brennecke, M.A. Stadtherr
(2) Carbon Dioxide; CO_2; [124-38-9]	Fluid Phase Equilibria, *220*, 57-69 (2004)
Variables: T/K = 313, 333 P/MPa = 10.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of erythromycin(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	x_i X 10^4
313.0	100.0	10.00	-
313.0	150.0	15.00	3.8
313.1	200.1	20.01	8.8
313.2	249.9	24.99	8.8
313.0	299.9	29.99	8.9
333.0	100.0	10.00	-
333.0	150.0	15.00	14.0
333.1	200.0	20.00	18.0
333.1	249.9	24.99	18.0
333.2	300.0	30.00	21.0

Auxiliary Information

Solubility measurements were performed with a custom-built re-circulating high-pressure apparatus[1]. The equilibrium vessel is filled with a large excess of the compound of interest. The system is filled with carbon dioxide to the desired pressure from the ISCO pump. During equilibration the valve allows by-passing of the sample loop. After the pressure stabilizes, the temperature controllers and heaters are activated, taking care to avoid temperature shoot. Once thermodynamic equilibrium is reached, the valve is shifted to the load position to fill the sample loop with the saturated supercritical carbon dioxide /solute solution. Then the sample is led into a liquid collection solvent. Once no more bubbles are observed in the collection solvent, the sample loop is collected in the collection vial. The sample is analyzed using a UV-vis system spectrometer.

Source and Purity of Materials:
1. erythromycin source Fluka, purity 97%.
2. carbon dioxide source Scott Specialty Gases; purity unstated.

Estimated Errors:
Temperature: ± 0.1K
Pressure: ± 0.34bar
Solubility: ± 16%

References:
1. A.M. Scurto, High-pressure phase and chemical equilibria of beta-Diketone ligands and chelates with Carbon dioxide, Ph.D. Thesis, University of Norte Dame, Norte dame, 2002.

Figure A.51 Solubility of Erythromycin in Carbon Dioxide.

Components:	Original Measurements:
(1) disperse orange 3; $C_{12}H_{10}N_4O_2$; [730-40-5]	A.Ferri, M.Banchero, L.Manna, S.Sicardi, J. of Supercritical Fluids, *30*, 41-49 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables: T/K = 353.2, 373.2, 393.2 P/MPa = 16.0 - 28.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of disperse orange 3(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^6$
353.2	16.0	1.59
353.2	18.0	3.24
353.2	20.0	5.11
353.2	22.0	8.31
353.2	24.0	9.86
353.2	26.0	13.4
353.2	28.0	15.5
373.2	16.0	1.45
373.2	18.0	3.09
373.2	20.0	6.00
373.2	22.0	9.56
373.2	24.0	14.0
393.2	16.0	1.63
393.2	18.0	3.27
393.2	20.0	4.95
393.2	22.0	10.9
393.2	24.0	17.4

Auxiliary Information

The apparatus used consists of the usual three sections of compression, equilibrium and expansion but some modifications were introduced to improve flow stability of the supercritical medium. A cylinder containing liquid carbon dioxide feeds the pump the pressurizes the fluid at the working pressure. Inside the extractor, the dye charged is blended with glass spheres. The fluid is expanded through the restrictor and the precipitated solute is collected into a solvent trap. The dye concentration is analysed via a UV/VIS spectrophotometer. The knowledge of the total amount of the solvent in the sample and of total mass of carbon dioxide flowed through the extractor permits to calculate the dye solubility at a set value of temperature and pressure.

Source and Purity of Materials:
1. disperse orange 3 source Sigma-Aldrich; purity unstated.
2. carbon dioxide source SIAD S.p.A, purity 99.998%

Estimated Errors:
Temperature: unstated
Pressure : unstated
Solubility : ± 4%

Figure A.52 Solubility of Disperse orange 3 in carbon Dioxide.

Components:	Original Measurements:
(1) disperse red 324; formula unspecified; [87396-78-9]	A.Ferri, M.Banchero, L.Manna, S.Sicardi, J. of Supercritical Fluids, *30*, 41-49 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables: T/K = 353.2, 373.2, 393.2 P/MPa = 18.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of disperse red 324 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^6$
353.2	18.0	0.440
353.2	20.0	0.940
353.2	22.0	1.240
353.2	24.0	1.860
353.2	26.0	3.200
353.2	28.0	3.720
353.2	30.0	4.650
373.2	18.0	0.480
373.2	20.0	1.000
373.2	22.0	1.860
373.2	24.0	3.200
373.2	26.0	4.230
373.2	28.0	4.750
373.2	30.0	5.780
393.2	18.0	0.290
393.2	20.0	0.680
393.2	22.0	1.340
393.2	24.0	2.580
393.2	26.0	4.440
393.2	28.0	5.160
393.2	30.0	7.850

Auxiliary Information

The apparatus used consists of the usual three sections of compression, equilibrium and expansion but some modifications were introduced to improve flow stability of the supercritical medium. A cylinder containing liquid carbon dioxide feeds the pump the pressurizes the fluid at the working pressure. Inside the extractor, the dye charged is blended with glass spheres. The fluid is expanded through the restrictor and the precipitated solute is collected into a solvent trap. The dye concentration is analysed via a UV/VIS spectrophotometer. The knowledge of the total amount of the solvent in the sample and of total mass of carbon dioxide flowed through the extractor permits to calculate the dye solubility at a set value of temperature and pressure.

Source and Purity of Materials:
1. disperse red324 source Ciba-Geigy; purity unstated.
2. carbon dioxide source SIAD S.p A, purity 99.998%

Estimated Errors:
Temperature: unstated
Pressure : unstated
Solubility : $\pm$ 4%

Figure A.53 Solubility of Disperse Red 324 in carbon Dioxide.

Components:	Original Measurements:
(1) disperse blue 79; $C_{24}H_{27}BrN_6O_{10}$; [12239-34-8]	A.Ferri, M.Banchero, L.Manna, S.Sicardi, J. of Supercritical Fluids,*30*,41-49 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 353.2, 373.2, 393.2 P/MPa = 18.0 - 30.0	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of disperse blue 79 (1) in Carbon Dioxide (2)

T/K	P/MPa	x_1 X 10^6
353.2	18.0	0.520
353.2	20.0	1.730
353.2	22.0	3.650
353.2	24.0	7.110
353.2	26.0	13.60
353.2	28.0	18.30
353.2	30.0	23.60
373.2	18.0	0.540
373.2	20.0	1.360
373.2	22.0	3.300
373.2	24.0	8.880
373.2	26.0	16.90
373.2	28.0	29.50
373.2	30.0	36.30
393.2	18.0	0.540
393.2	20.0	1.440
393.2	22.0	3.500
393.2	26.0	17.90
393.2	28.0	28.40
393.2	30.0	49.90

Auxiliary Information

The apparatus used consists of the usual three sections of compression, equilibrium and expansion but some modifications were introduced to improve flow stability of the supercritical medium. A cylinder containing liquid carbon dioxide feeds the pump the pressurizes the fluid at the working pressure. Inside the extractor, the dye charged is blended with glass spheres. The fluid is expanded through the restrictor and the precipitated solute is collected into a solvent trap. The dye concentration is analysed via a UV/VIS Spectrophotometer. The knowledge of the total amount of the solvent in the sample and of total mass of carbon dioxide flowed through the extractor permits to calculate the dye solubility at a set value of temperature and pressure.

Source and Purity of Materials:
1. disperse blue 79 source Ciba-Geigy; purity unstated.
2. carbon dioxide source SIAD S.p.A, purity 99.998%

Estimated Errors:
Temperature: unstated
Pressure : unstated
Solubility : ± 4%

Figure A.54 Solubility of Disperse blue 79 in Carbon Dioxide.

Components:	Original Measurements:
(1) Quinizarin; $C_{14}H_8O_4$ [81-64-1] (2) Carbon Dioxide; CO_2;[124-38-9]	A.Ferri, M.Banchero, L.Manna, S.Sicardi, J. of Supercritical Fluids, *30*, 41-49 (2004)
Variables: T/K = 353.2, 373.2, 393.2 P/MPa=16 0-30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of disperse quinizarin(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^6$
353.2	16.0	12.600
353.2	19.0	27.500
353.2	21.0	52.600
353.2	24.0	67.200
353.2	27.0	79.200
353.2	30.0	110.000
373.2	12.0	2.210
373.2	14.0	7.160
373.2	16.0	13.200
373.2	22.0	60.800
373.2	25.0	107.000
393.2	12.0	0.690
393.2	15.0	9.590
393.2	18.0	21.000
393.2	21.0	69.400

Auxiliary Information

The apparatus used consists of the usual three sections of compression, equilibrium and expansion but some modifications were introduced to improve flow stability of the supercritical medium. A cylinder containing liquid carbon dioxide feeds the pump the pressurizes the fluid at the working pressure. Inside the extractor, the dye charged is blended with glass spheres. The fluid is expanded through the restrictor and the precipitated solute is collected into a solvent trap. The dye concentration is analysed via a UV/VIS spectrophotometer. The knowledge of the total amount of the solvent in the sample and of total mass of carbon dioxide flowed through the extractor permits to calculate the dye solubility at a set value of temperature and pressure.

Source and Purity of Materials:
1. quinizarin source Sigma-Aldrich; purity unstated.
2. carbon dioxide source SIAD S.p.A, purity 99.998%

Estimated Errors:
Temperature: unstated
Pressure : unstated
Solubility : $\pm$ 4%

Figure A.55 Solubility of Quinizarin in Carbon Dioxide.

Components:	Original Measurements:
(1) Cholesterol; $C_{27}H_{46}O$; [57-88-5]	Z.Huang, S.Kawi, Y.C.Chiew, J.of Supercritical Fluids *30*, 25-39 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 318.15 P/MPa = 10.0 - 24.0	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Cholesterol (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^6$
318.15	100	10.0	6.49
318.15	120	12.0	24.00
318.15	140	14.0	37.20
318.15	160	16.0	57.60
318.15	180	18.0	70.90
318.15	210	21.0	79.20
318.15	240	24.0	94.00

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into a premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.

Source and Purity of Materials:
1. Cholesterol source Sigma, purity 99%
2. carbon dioxide source Soxal, purity 99.8%

Estimated Error:
Temperature: ±0.01C
Pressure: ±0.2%
Solubility: ±1%

Figure A.56 Solubility of Cholesterol in Carbon Dioxide.

Components:	Original Measurements:
(1) Cholesteryl acetate; $C_{29}H_{48}O_2$; [604-35-3] (2) Carbon Dioxide; CO_2; [124-38-9]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
Variables: T/K = 308.15, 318.15, 328.15 P/MPa = 9.0 - 24.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Cholesteryl acetate (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308.15	90	9.0	7.9
308.15	110	11.0	---
308.15	140	14.0	25.70
308.15	180	18.0	42.70
308.15	210	21.0	45.50
308.15	240	24.0	52.70
318.15	90	9.0	0.435
318.15	110	11.0	7.55
318.15	140	14.0	20.70
318.15	180	18.0	47.70
318.15	210	21.0	63.30
318.15	240	24.0	81.90
328.15	90	9.0	---
328.15	110	11.0	1.62
328.15	140	14.0	17.60
328.15	180	18.0	54.60
328.15	210	21.0	104.00
328.15	240	24.0	---

Auxiliary Information

The solubility was measured using a continuous flow
type apparatus. Carbon dioxide solvent was supplied
through a gas cylinder. The liquefied carbon dioxide
was fed to an HPLC and pumped into a premixing
coil. It then enters extraction vessels where solute is
kept. A glass U tube was used to collect the deposited
solute. The determination of the solubility was based
on the amount of solute trapped in the collecting tube
and precipitated in the tubing upstream of the BPR.
The mass of the collected solute was gravimetrically
determined using a balance.

Source and Purity of Materials:
1. Cholesteryl acetate source Tokyo Chemicals
   purity 97.8%
2. carbon dioxide source Soxal, purity 99.8%

Estimated Error:
Temperature:±0.01C
Pressure: ±0.2%
Solubility:±1%

Figure A.57 Solubility of Cholesteryl Acetate in Carbon Dioxide.

Components:	Original Measurements:
(1) Cholesteryl butyrate; C$_{31}$H$_{52}$O$_2$;[521-13-1] (2) Carbon Dioxide; CO$_2$; [124-38-9]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
Variables: T/K = 308.15, 318.15, 328.15 P/MPa = 10.0 - 24.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Cholesteryl butyrate (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308.15	100	10.0	1.07
308.15	120	12.0	2.16
308.15	140	14.0	3.59
308.15	160	16.0	4.32
308.15	180	18.0	4.60
308.15	210	21.0	5.70
308.15	240	24.0	5.81
318.15	100	10.0	0.22
318.15	120	12.0	1.46
318.15	140	14.0	2.50
318.15	160	16.0	3.78
318.15	180	18.0	4.98
318.15	210	21.0	6.72
318.15	240	24.0	8.36
328.15	100	10.0	---
328.15	120	12.0	0.38
328.15	140	14.0	1.45
328.15	160	16.0	2.85
328.15	180	18.0	4.15
328.15	210	21.0	6.05
328.15	240	24.0	8.93

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into a premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.

Source and Purity of Materials:
1.Cholesteryl butyrate source Fluka Chemicals purity 99.0%
2. carbon dioxide source Soxal, purity 99.8%

Estimated Error:
Temperature: ±0.01C
Pressure: ±0.2%
Solubility: ±1%

Figure A.58 Solubility of Cholesteryl Butyrate in Carbon Dioxide.

Components:	Original Measurements:
(1) Cholesteryl benzoate; $C_{34}H_{50}O_2$; [604-32-0] (2) Carbon Dioxide; CO_2; [124-38-9]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
Variables: T/K = 308.15, 318.15, 328.15 P/MPa = 12.0 - 27.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Cholesteryl benzoate (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308.15	120	12.0	0.72
308.15	140	14.0	0.75
308.15	180	18.0	1.80
308.15	210	21.0	1.84
308.15	240	24.0	1.98
308.15	255	25.5	---
308.15	270	27.0	2.51
318.15	120	12.0	---
318.15	140	14.0	---
318.15	180	18.0	2.00
318.15	210	21.0	2.47
318.15	240	24.0	3.15
318.15	255	25.5	---
318.15	270	27.0	3.62
328.15	120	12.0	---
328.15	140	14.0	0.52
328.15	180	18.0	2.05
328.15	210	21.0	2.83
328.15	240	24.0	4.23
328.15	255	25.5	4.39
328.15	270	27.0	5.28

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into an premixing 99.8% coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.

Source and Purity of Materials:
1.Cholesteryl benzoate source Aldrich, purity 98%
2. carbon dioxide source Soxal, purity

Estimated Error:
Temperature: ±0.01C
Pressure: ±0.2%
Solubility: ±1%

Figure A.59 Solubility of Cholesteryl Benzoate in Carbon Dioxide.

Components:	Original Measurements:
(1) Cholesteryl benzoate; $C_{34}H_{50}O_2$;[604-32-0] (2) Carbon Dioxide; CO_2; [124-38-9], with methanol; CH_3OH; [67-56-1]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
Variables: T/K = 318.15, 328.15 P/MPa = 13.0 - 27.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Cholesteryl benzoate (1) in Carbon Dioxide with 3%methanol (2)

T/K	P/bar	P/MPa	x_1 X 10^5
318.15	130	13.0	1.53
318.15	140	14.0	---
318.15	160	16.0	2.50
318.15	180	18.0	3.13
318.15	210	21.0	3.98
318.15	240	24.0	5.01
318.15	270	27.0	5.53
328.15	130	13.0	---
328.15	140	14.0	1.38
328.15	160	16.0	2.24
328.15	180	18.0	2.98
328.15	210	21.0	4.25
328.15	240	24.0	5.72
328.15	270	27.0	6.11

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into an premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.	Source and Purity of Materials: 1.Cholesteryl benzoate source Aldrich, purity 98% 2. carbon dioxide source Soxal, purity 99.8% methanol source Tedia Company, purity 99.98% Estimated Error: Temperature: $\pm0.01C$ Pressure: $\pm0.2\%$ Solubility: $\pm1\%$

Figure A.60 Solubility of Cholesteryl Benzoate in Carbon Dioxide with Methanol.

Components:	Original Measurements:
(1) Cholesteryl benzoate; $C_{34}H_{50}O_2$;[604-32-0]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9], with acetone; CH_3COCH_3; [67-64-1]	

Variables:	Prepared By:
T/K = 318.15, 328.15	Dana E. Knox; Syed A. Abdullah
P/MPa = 13.0 - 27.0	

Experimental Data
Solubility of Cholesteryl benzoate (1) in Carbon Dioxide with 3%acetone(2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
318.15	130	13.0	1.64
318.15	140	14.0	---
318.15	160	16.0	2.68
318.15	180	18.0	3.06
318.15	210	21.0	3.95
318.15	240	24.0	4.83
318.15	270	27.0	5.40
328.15	130	13.0	1.19
328.15	140	14.0	---
328.15	160	16.0	2.58
328.15	180	18.0	3.31
328.15	210	21.0	4.69
328.15	240	24.0	5.96
328.15	270	27.0	7.20

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into an premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.

Source and Purity of Materials:
1. Cholesteryl benzoate source Aldrich, purity 98%
2. carbon dioxide source Soxal, purity 99.8% acetone source Tedia Company, purity 99.97%

Estimated Error:
Temperature: ±0.01C
Pressure: ±0.2%
Solubility: ±1%

Figure A.61 Solubility of Cholesteryl Benzoate in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) Cholesterol; $C_{27}H_{46}O$; [57-88-5]	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids *30*, 25-39 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9], with methanol; CH_3OH; [67-56-1]	

Variables:	Prepared By:
T/K = 318.15, 328.15	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.0 - 24.0	

Experimental Data
Solubility of Cholesterol (1) in Carbon Dioxide with 3%methanol (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
318.15	100	10.0	2.53
318.15	120	12.0	5.92
318.15	140	14.0	8.20
318.15	160	16.0	9.61
318.15	180	18.0	11.70
318.15	210	21.0	13.50
318.15	240	24.0	16.00
328.15	100	10.0	---
328.15	120	12.0	2.49
328.15	140	14.0	5.95
328.15	160	16.0	9.57
328.15	180	18.0	11.30
328.15	210	21.0	14.70
328.15	240	24.0	17.50

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into an premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.

Source and Purity of Materials:
1. Cholesterol source Sigma, purity 99%
2. carbon dioxide source Soxal, 99.8% methanol source Tedia Company, purity 99.98%

Estimated Error:
Temperature: ±0.01C
Pressure: ±0.2%
Solubility: ±1.0%

Figure A.62 Solubility of Cholesterol in Carbon Dioxide with Methanol.

Components:	Original Measurements:

(1) Cholesterol;
    $C_{27}H_{46}O$;[57-88-5]
(2) Carbon Dioxide; CO_2; [124-38-9], with
    acetone; CH_3COCH_3; [67-56-1]
	Z.Huang, S.Kawi, Y.C.Chiew, J. of Supercritical Fluids
	30, 25-39 (2004)

Variables: T/K = 318.15, 328.15 P/MPa = 10.0 - 24.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of Cholesterol (1) in Carbon Dioxide with 3%acetone (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
318.15	100	10.0	3.73
318.15	120	12.0	6.08
318.15	140	14.0	8.26
318.15	160	16.0	10.10
318.15	180	18.0	11.60
318.15	210	21.0	14.10
318.15	240	24.0	15.40
328.15	100	10.0	---
328.15	120	12.0	4.05
328.15	140	14.0	7.71
328.15	160	16.0	10.60
328.15	180	18.0	12.40
328.15	210	21.0	17.50
328.15	240	24.0	21.80

Auxiliary Information

The solubility was measured using a continuous flow type apparatus. Carbon dioxide solvent was supplied through a gas cylinder. The liquefied carbon dioxide was fed to an HPLC and pumped into an premixing coil. It then enters extraction vessels where solute is kept. A glass U tube was used to collect the deposited solute. The determination of the solubility was based on the amount of solute trapped in the collecting tube and precipitated in the tubing upstream of the BPR. The mass of the collected solute was gravimetrically determined using a balance.	Source and Purity of Materials:
	1. Cholesterol source Sigma,
	   purity 99%
	2. carbon dioxide source Soxal, purity 99.8%
	   acetone source Tedia Company,
	   purity 99.97%
	
	Estimated Error:
	Temperature: $\pm$0.01C
	Pressure: $\pm$0.2%
	Solubility: $\pm$1.0%

Figure A.63 Solubility of Cholesterol in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) C.I. Disperse Blue 134(1,4-bis(isopropylamino) anthraquinone); $C_{20}H_{22}N_2O_{2;}$ [14233-37-5] (2) Carbon Dioxide; CO_2; [124-38-9]	Kazuhiro Tamuro, Takashi Shinoda, Fluid Phase Equilibria , 219, 25-32 (2004)
Variables: T/K = 323.15, 353.15, 383.15 P/MPa = 10.0 - 25.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of C.I. Disperse Blue 134 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
323.15	10.0	0.117
323.15	12.0	1.111
323.15	15.0	2.956
323.15	20.0	5.652
323.15	25.0	8.744
353.15	10.0	0.030
353.15	12.0	0.144
353.15	15.0	0.980
353.15	20.0	6.771
353.15	25.0	13.080
383.15	10.0	0.055
383.15	12.0	0.174
383.15	15.0	0.737
383.15	20.0	4.567
383.15	25.0	15.360

Auxiliary Information

A detailed description of the equipment and operating procedure and the reliability of the solubility measurement is given elsewhere[1]. A flow type apparatus was used. The dye packed with glass beads was loaded into the extraction cell. Carbon dioxide was delivered from the gas cylinder and flowed through the extraction cell. The supercritical mixture including the dye was released at the back pressure regulator. The dye was trapped in a two-step cold trap filled with ethanol. A UV-Vis spectrophotometer was used to analyze the concentration of the dye dissolved. The solubility of the dye can be calculated from the dye concentration and volume of carbon dioxide.

Source and Purity of Materials:
1. C.I. Disperse Blue 134 source Arimoto Chemicals Co. Ltd , purity 97%
2. Carbon dioxide source Uno Sanso Co purity 99.9%.

Estimated Error:
Temperature: ± 0.1K
Pressure: ± 0.2MPa
Solubility: ±4%

References:
1. T. Shinoda, K.Tamura, J. Chem. Eng. Data, 48, 869-873 (2003).

Figure A.64 Solubility of C.I. Disperse Blue 134 in Carbon Dioxide.

Components:	Original Measurements:
(1) C.I.Disperse Yellow 16(3-methyl-1-phenyl-5-pyrazolone); $C_{16}H_{14}N_4O$;[4314-14-1] (2) Carbon Dioxide; CO_2; [124-38-9]	Kazuhiro Tamuro, Takashi Shinoda, Fluid Phase Equilibria , *219*, 25-32 (2004)
Variables: T/K = 323.15, 353.15, 383.15 P/MPa = 10.0 - 25.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of C.I. Disperse Yellow 16 (1) in Carbon Dioxide (2)

T/K	P/MPa	x_1 X 10^5
323.15	10.0	0.224
323.15	12.0	2.111
323.15	15.0	5.453
323.15	20.0	9.613
323.15	25.0	13.650
353.15	10.0	0.092
353.15	12.0	0.331
353.15	15.0	2.090
353.15	20.0	9.988
353.15	25.0	22.030
383.15	10.0	0.208
383.15	12.0	0.542
383.15	15.0	1.775
383.15	20.0	9.616
383.15	25.0	26.940

Auxiliary Information

A detailed description of the equipment and operating procedure and the reliability of the solubility measurement is given elsewhere[1]. A flow type apparatus was used. The dye packed with glass beads was loaded into the extraction cell. Carbon dioxide was delivered from the gas cylinder and flowed through the extraction cell. The supercritical mixture including the dye was released at the back pressure regulator. The dye was trapped in a two-step cold trap filled with ethanol. A UV-Vis spectrophotometer was used to analyze the concentration of the dye dissolved. The solubility of the dye can be calculated from the dye concentration and volume of carbon dioxide.

Source and Purity of Materials:
1. C.I. Disperse Yellow 16 source Arimoto Chemicals Co. Ltd , purity 97%
2. Carbon dioxide source Uno Sanso Co purity 99.9%.

Estimated Error:
Temperature: $\pm$ 0.1K
Pressure: $\pm$ 0.2MPa
Solubility: $\pm$4%

References:
1. T. Shinoda, K.Tamura, J. Chem. Eng. Data, *48*, 869-873 (2003).

Figure A.65 Solubility of C.I. Disperse Yellow 16 in Carbon Dioxide.

Components:	Original Measurements:
(1) Benzocaine; $C_9H_{12}NO_2$; [94-09-7]	Aziz Garmroodi, Jalal Hassan, Yadollah Yamini, J. Chem. Eng. Data, *49*, 709-712 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348	Dana E. Knox; Syed A. Abdullah
P/MPa = 12.2 - 35.5	

Experimental Data
Solubility of benzocaine(1) in Carbon Dioxide (2)

T/K	P/bar	P/ MPa	$x_1 X 10^4$
308	122	12.2	10.30
308	182	18.2	12.80
308	243	24.3	14.70
308	304	30.4	17.60
308	355	35.5	20.00
318	122	12.2	10.60
318	182	18.2	15.30
318	243	24.3	18.20
318	304	30.4	22.80
318	355	35.5	22.50
328	122	12.2	8.30
328	182	18.2	18.90
328	243	24.3	28.30
328	304	30.4	36.00
328	355	35.5	39.00
338	122	12.2	3.90
338	182	18.2	23.00
338	243	24.3	42.60
338	304	30.4	64.40
338	355	35.5	78.20
348	122	12.2	2.70
348	182	18.2	20.30
348	243	24.3	49.30
348	304	30.4	86.50
348	355	35.5	121.20

Auxiliary Information

A suprex MPS/225 integrated SFE/supercritical fluid chromatography system, equipped with a static system in the SFE mode was used. A detailed description is given elsewhere[1]. The solute solids were mixed well with glass beads and packed into the extraction vessel. supercritical carbon dioxide was then pressurized and passed into the extraction vessel. After equilibrium was reached, the loop was pressurized into a collection vial. The solubility was calculated by absorbance at suitable wavelength using a Model 2100 Shimadzo UV-Vis spectrophotometer.

Source and Purity of Materials:
1. Benzocaine source Food and Drug Quality Control Lab, Iran; purity 99%
2. carbon dioxide source Sabalan, Iran; Purity 99.99%

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubilty: ± 4%

References:
1. Yamini,Y.; Fathi,M,R.; Alizeth,N.; Fluid Phase Equilibria , *152*, 299-305 (1998).

Figure A.66 Solubility of Benzocaine in Carbon Dioxide.

Components:	Original Measurements:
(1) Metronidazole benzoate; $C_{13}H_{13}N_3O_4$; [13182-89-3]	Aziz Garmroodi, Jalal Hassan, Yadollah Yamini, J. Chem. Eng. Data, 49, 709-712 (2004)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Metronidazole benzoate(1) in Carbon Dioxide (2)

T/K	P/bar	P/ MPa	$x_1 \times 10^4$
308	122	12.2	4.90
308	182	18.2	6.10
308	243	24.3	8.40
308	304	30.4	10.80
308	355	35.5	13.90
318	122	12.2	2.70
318	182	18.2	7.20
318	243	24.3	8.80
318	304	30.4	12.70
318	355	35.5	14.70
328	122	12.2	1.50
328	182	18.2	5.60
328	243	24.3	10.50
328	304	30.4	14.40
328	355	35.5	17.70
338	122	12.2	0.70
338	182	18.2	5.10
338	243	24.3	12.60
338	304	30.4	22.70
338	355	35.5	27.90
348	122	12.2	1.60
348	182	18.2	5.50
348	243	24.3	17.50
348	304	30.4	30.20
348	355	35.5	45.50

Auxiliary Information

A suprex MPS/225 integrated SFE/supercritical fluid chromatography system, equipped with a static system in the SFE mode was used. A detailed description is given elsewhere[1]. The solute solids were mixed well with glass beads and packed into the extraction vessel. supercritical carbon dioxide was then pressurized and passed into the extraction vessel. After equilibrium was reached, the loop was pressurized into a collection vial. The solubility was calculated by absorbance at suitable wavelength using a Model 2100 Shimadzo UV-Vis spectrophotometer.

Source and Purity of Materials:
1.metronidazole benzoate source Food and Drug Quality Control Lab, Iran; purity 99%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubilty: ± 4%

References:
1. Yamini,Y.; Fathi,M,R.; Alizeth,N.; Fluid Phase Equilibria , 152, 299-305 (1998).

Figure A.67 Solubility of Metronidazole Benzoate in Carbon Dioxide.

Components:	Original Measurements:
(1) Naproxen; $C_{14}H_{14}O_3$; [22204-53-1]	Aziz Garmroodi, Jalal Hassan, Yadollah Yamini,
(2) Carbon Dioxide; CO_2; [124-38-9]	J. Chem. Eng. Data ,*49*, 709-712 (2004)

Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of naproxen(1) in Carbon Dioxide (2)

T/K	P/bar	P/ MPa	$x_1 \times 10^4$
308	122	12.2	0.10
308	182	18.2	0.20
308	243	24.3	0.20
308	304	30.4	0.30
308	355	35.5	0.40
318	122	12.2	0.20
318	182	18.2	0.20
318	243	24.3	0.30
318	304	30.4	0.40
318	355	35.5	0.50
328	122	12.2	0.10
328	182	18.2	0.20
328	243	24.3	0.40
328	304	30.4	0.50
328	355	35.5	0.80
338	122	12.2	0.20
338	182	18.2	0.30
338	243	24.3	0.60
338	304	30.4	0.80
338	355	35.5	1.10
348	122	12.2	0.10
348	182	18.2	0.30
348	243	24.3	0.70
348	304	30.4	1.30
348	355	35.5	2.00

Auxiliary Information

A suprex MPS/225 integrated SFE/supercritical fluid chromatography system, equipped with a static system in the SFE mode was used. A detailed description is given elsewhere[1]. The solute solids were mixed well with glass beads and packed into the extraction vessel. supercritical carbon dioxide was then pressurized and passed into the extraction vessel. After equilibrium was reached, the loop was pressurized into a collection vial. The solubility was calculated by absorbance at suitable wavelength using a Model 2100 Shimadzo UV-Vis spectrophotometer.

Source and Purity of Materials:
1. Naproxen source Food and Drug Quality Control Lab, Iran; purity 99%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: not stated
Pressure: not stated
Solubilty: ± 4%

References:
1. Yamini,Y.; Fathi,M,R.; Alizeth,N.; Fluid Phase Equilibria , *152*, 299-305 (1998).

Figure A.68 Solubility of Naproxen in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-fluoro-alpha-methyl-4-biphenylacetic acid; Flurbiprofen; $C_{13}H_{13}F_1 O_2$; [5104-49-4]. (2) Carbon Dioxide; CO_2; [124-38-9]	Ana Rita C.Duarte, Patricia Coimbra, Herminio C. de Sousa, Catarina M.M.Duarte, J. Chem. Eng. Data, *49*, 449-452 (2004).
Variables: T/K = 303, 313, 323 P/MPa = 8.9 - 24.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Flurbiprofen(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
303	8.9	2.170
303	13.1	3.530
303	15.3	5.312
303	18.5	6.236
303	21.9	7.896
303	24.5	8.337
313	9.8	1.672
313	13.1	5.389
313	15.3	6.646
313	20.2	12.563
313	23.4	13.904
313	24.4	14.950
323	11.2	2.603
323	16.6	10.179
323	17.7	14.012
323	19.7	14.838
323	20.6	16.213
323	23.4	19.683

Auxiliary Information

| The solubility was measured using a static analytic high-pressure apparatus. The determination of solubility was performed in a similar manner to that described by Matias et al [1]. An equilibrium visual cell was immersed in a thermostatic water bath. The cell is initially loaded with the solid and a magnetic internal stirrer. Carbon dioxide is pumped using a pneumatic compressor. After equilibrium is reached, samples are taken out from HPLC valve. To determine the amount of Flurbiprofen, the resulting solution were analyzed by UV – spectrophotometry. | Source and Purity of Materials: 1. Flurbiprofen source Sigma-Aldrich, purity 97%. 2. carbon dioxide source Air Liquide, purity 99.99%

Estimated Error: Temperature: ± 0.1C Pressure: unspecified Solubility: unspecified

References: 1.Matias A.A.; Nunes A.V.M.; Casimiro T.; Duarte C.M.M, J. of Supercritical Fluids, *28*,201-206 (2004). |

Figure A.69 Solubility of Flurbiprofen in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-fluoro-alpha-methyl-4-biphenylacetic acid; Flurbiprofen; $C_{13}H_{13}F_1 O_2$; [5104-49-4] (2) Carbon Dioxide; CO_2; [124-38-9], with Ethanol; C_2H_5OH; [64-17-5]	Ana Rita C.Duarte, Patricia Coimbra, Herminio C. de Sousa, Catarina M.M.Duarte, J. Chem. Eng. Data, *49*, 449-452 (2004).

Variables: T/K = 313 P/MPa = 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Flurbiprofen(1) in Carbon Dioxide with ethanol (2)

T/K	P/MPa	% cosolvent	$x_1 \times 10^4$
313	18.0	0.0	0.4848
313	18.0	2.5	3.7648
313	18.0	5.0	7.8519
313	18.0	7.5	15.5522
313	18.0	10.0	36.7575

Auxiliary Information

The solubility was measured using a static analytic high-pressure apparatus. The determination of solubility was performed in a similar manner to that described by Matias et al [1]. An equilibrium visual cell was immersed in a thermostatic water bath. The cell is initially loaded with the solid and a magnetic internal stirrer. Carbon dioxide is pumped using a pneumatic compressor. After equilibrium is reached, samples are taken out from HPLC valve. To determine the amount of Flurbiprofen, the resulting solution were analyzed by UV – spectrophotometry.

Source and Purity of Materials:
1. Flurbiprofen source Sigma-Aldrich, purity 97%.
2. carbon dioxide source Air Liquide, purity 99.99%;ethanol source Riedel-de Haen purity 99.8%

Estimated Error:
Temperature: ±0.1C
Pressure: unspecified
Solubility: unspecified

References:
1. Matias A.A.; Nunes A.V.M.; Casimiro T.; Duarte C.M.M, J. of Supercritical Fluids, *28*,201-206 (2004).

Figure A.70 Solubility of Flurbiprofen in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) Phenanthrene; $C_{14}H_{10}$; [85-01-8] (2) Carbon Dioxide; CO_2; [124-38-9]	Amporn Sane, Shelby Taylor, Ya-Ping Sun, Mark Tiles J. of Supercritical Fluids, 28, 277-285 (2004)
Variables: T/K = 308, 328 P/MPa = 12.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Phenanthrene(1) in Carbon Dioxide (2)

T/K	P/bar	P/ MPa	$x_1 \ X \ 10^3$
308	120	12.0	0.887
308	150	15.0	1.120
308	200	20.0	1.390
308	250	25.0	1.58
308	300	30.0	1.770
328	120	12.0	0..479
328	160	16.0	1.460
328	200	20.0	2.060
328	240	24.0	2.700
328	280	28.0	3.000

Auxiliary Information

The basic strategy is to first obtain equilibrium in the variable volume view cell, then use a syringe pump and heated nozzle to deliver the contents of the cell through the sampling loop at constant temperature and pressure so as not to disturb equilibrium, and finally to analyze the contents of the equilibrium phase obtained in the sample loop. The contents of the sample loop subsequently depressurized gets collected in a flask located in an ice bath. The solution of solvent and solute is observed by a UV/Vis spectrophotometer.	Source and Purity of Materials 1.Phenanthrene source Aldrich Chemical Co purity 99.5% 2. carbon dioxide source National welders Supply Co, purity 99.99% Estimated Error: Temperature: ± 0.2C Pressure: ± 1 bar Solubility: ± 3%

Figure A.71 Solubility of Phenanthrene in Carbon Dioxide.

Components:	Original Measurements:
(1) 5,10,15,20-tetrakis(3,5-bis(trifluoromethyl)phenyl) porphyrin(TBTPP); $C_{52}H_{22}F_{24}N_4$; [127286-86-6] (2) Carbon Dioxide; CO_2; [124-38-9]	Amporn Sane, Shelby Taylor, Ya-Ping Sun, Mark Tiles J. of Supercritical Fluids,*28*,277-285(2004)
Variables: T/K = 313, 343, 373 P/MPa =10.34 – 32.41	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of TBTPP(1) in Carbon Dioxide (2)

T/K	P/bar	P/ MPa	$x_1 \times 10^4$
313	103.4	10.34	0.103
313	158.6	15.86	0.978
313	213.7	21.37	2.250
313	268.9	26.89	3.570
313	324.1	32.41	5.590
343	103.4	10.34	0.0008
343	158.6	15.86	0.083
343	213.7	21.37	1.060
343	268.9	26.89	3.250
343	324.1	32.41	7.120
373	103.4	10.34	0.001
373	158.6	15.86	0.007
373	213.7	21.37	0.225
373	268.9	26.89	1.740
373	324.1	32.41	6.860

Auxiliary Information

The basic strategy is to first obtain equilibrium in the variable volume view cell, then use a syringe pump and heated nozzle to deliver the contents of the cell through the sampling loop at constant temperature and pressure so as not to disturb equilibrium, and finally to analyze the contents of the equilibrium phase obtained in the sample loop. The contents of the sample loop subsequently depressurized gets collected in a flask located in an ice bath. The solution of solvent and solute is observed by a UV/Vis spectrophotometer.

Source and Purity of Materials
1.TBTPP was synthesized in laboratory purity 99.0%
2. carbon dioxide source National welders Supply Co, purity 99.99%

Estimated Error:
Temperature: ± 0.2C
Pressure: ± 1 bar
Solubility: ± 4%

Figure A.72 Solubility of TBTPP in Carbon Dioxide.

Components:	Original Measurements:
(1) Coenzyme Q10 (ubiquinone) $C_{59}H_{90}O_4$; [303-98-0] (2) Carbon Dioxide; CO_2; [124-38-9]	Ana A.Matias, Ana V.M.Nunes, Teresa Casimiro, Catarina M.M.Duarte, J. of Supercritical Fluids, *28*, 201-206 (2004)
Variables: T/K = 305, 313, 323 P/MPa = 9.2 - 26.2	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Coenzyme Q10 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
305	11.6	1.57
305	14.1	2.99
305	17.8	5.21
305	22.2	6.51
305	22.9	6.97
305	25.9	9.91
313	12.1	0.99
313	15.8	2.78
313	18.4	4.24
313	21.1	6.62
313	26.2	11.29
323	9.2	0.00
323	12.1	0.00
323	14.2	0.74
323	15.8	1.30
323	18.1	2.51
323	21.7	4.68
323	25.3	9.56

Auxiliary Information

The solubility was measured using a static analytical high pressure apparatus. The equilibrium cell, immersed in the thermostated water bath, is initially loaded with the drug and the system is pressurized with fresh carbon dioxide until the desired pressure is brought into the cell, then the mixture is stirred to reach equilibrium. Samples from the gas phase are taken through a six-port sampling HPLC valve. These are expanded into a glass trap. The amount of collected solute is determined by spectrophotometric analysis.

Source and Purity of Materials:
1. Coenzyme Q10 source Sigma, 98%.
2. carbon dioxide source Air Liquide, purity 99.99%.

Estimated Error:
Temperature: ±1.0C
Pressure: ±0.1%
Solubility: ± 6%

Figure A.73 Solubility of Coenzyme Q10 in Carbon Dioxide.

Components:	Original Measurements:
(1) p-tolylboronic acid; C₇H₉BO₂ ; [5720-05-8] (2) Carbon Dioxide; CO₂; [124-38-9]	Gary A.Leeke, Regina C.Santos, Jonathan Seville, Bushra Al-Duri, Connie K.Y.Lee, Andrew B. Holmes, J. Chem. Eng. Data, 49, 48-52 (2004)
Variables: T/K = 353, 383 P/MPa = 9.56 - 31.72	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of p-tolylboronic acid (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
353	106.1	10.61	3.936
353	127.2	12.72	3.693
353	139.8	13.98	3.745
353	150.3	15.03	3.962
353	167.5	16.75	3.862
353	202.2	20.22	4.095
353	238.0	23.80	4.276
353	245.1	24.51	4.345
353	252.9	25.29	4.367
353	296.7	29.67	4.639
353	312.0	31.20	4.762
383	95.6	9.56	10.705
383	111.4	11.14	10.893
383	125.5	12.55	10.702
383	159.0	15.90	10.940
383	166.8	16.68	10.990
383	172.4	17.24	10.818
383	194.1	19.41	11.016
383	227.3	22.73	10.938
383	241.4	24.14	11.123
383	255.8	25.58	11.256
383	278.4	27.84	11.356
383	305.2	30.52	11.421
383	317.2	31.72	11.875

Auxiliary Information

The apparatus consists of an equilibrium cell into which the solid was loaded. Liquid carbon dioxide was compressed by the use of a pressure generator and directed to the equilibrium cell. The temperature in the cell was increased to the desired isothermal condition and the pressure raised until dissolution of the compound occurred and a homogeneous phase existed. Once the compound had been fully dissolved, the pressure was lowered to the onset of cloud point. The volume of carbon dioxide present in the vessel at cloud point was recorded by depressurizing across micro metering valve and passing the expanded stream through a wet-test meter. For safety reasons any heavy component present in the expanded gas stream was trapped in the glass collector.

Source and Purity of Materials:
1. p-Tolylboronic acid source Sigma-Aldrich, purity 97%.
2. carbon dioxide source BOC, purity 99.9%

Estimated Error:
Temperature: ± 0.1C
Pressure: ± 1.1 bar
Solubility: unstated

Figure A.74 Solubility of p-Tolylboronic acid in Carbon Dioxide.

Components:	Original Measurements:
(1) bromobenzene; C$_6$H$_5$Br ; [108-86-1] (2) Carbon Dioxide; CO$_2$; [124-38-9]	Gary A.Leeke, Regina C.Santos, Jonathan Seville, Bushra Al-Duri, Connie K.Y.Lee, Andrew B. Holmes, J. Chem. Eng. Data, *49*, 48-52 (2004)
Variables: T/K = 353, 383 P/MPa = 9.83 - 20.12	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Bromobenzene(1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	x_i X 10^3
353	98.3	9.83	8.977
353	109.8	10.98	11.606
353	115.3	11.53	10.851
353	117.5	11.75	9.965
353	120.2	12.02	10.782
353	128.5	12.85	11.604
353	134.7	13.47	17.523
353	138.4	13.84	20.774
353	139.8	13.98	23.618
353	140.0	14.00	23.578
353	142.6	14.26	26.572
353	148.9	14.89	31.235
353	153.1	15.31	41.724
353	161.2	16.12	74.328
353	162.7	16.27	73.484
353	164.9	16.49	78.679
383	104.7	10.47	10.453
383	114.5	11.45	13.249
383	129.4	12.94	17.636
383	141.9	14.19	20.217
383	144.1	14.41	18.638
383	162.3	16.23	29.448
383	179.0	17.90	48.102
383	180.2	18.02	50.902
383	194.1	19.41	74.371
383	198.5	19.85	86.800
383	201.2	20.12	88.256

Auxiliary Information

The apparatus consists of an equilibrium cell into which the solid was loaded. Liquid carbon dioxide was compressed by the use of a pressure generator and directed to the equilibrium cell. The temperature in the cell was increased to the desired isothermal condition and the pressure raised until dissolution of the compound occurred and a homogeneous phase existed. Once the compound had been fully dissolved, the pressure was lowered to the onset of cloud point. The volume of carbon dioxide present in the vessel at cloud point was recorded by depressurizing across micro metering valve and passing the expanded stream through a wet-test meter.

Source and Purity of Materials:
1.Bromobenzene source Sigma-Aldrich, purity 99 .
2. carbon dioxide source BOC, purity 99.9%

Estimated Error:
Temperature: ± 0.1C
Pressure: ± 1.1 bar
Solubility: unstated

Figure A.75 Solubility of Bromobenzene in Carbon Dioxide.

Components:	Original Measurements:
(1) 4 –phenyltoiuene $C_{13}H_{12}$; [644-08-6] (2) Carbon Dioxide; CO_2; [124-38-9]	Gary A.Leeke, Regina C.Santos, Jonathan Seville, Bushra Al-Duri, Connie K.Y.Lee, Andrew B. Holmes, J. Chem. Eng. Data, *49*, 48-52 (2004).
Variables: T/K = 353, 383 P/MPa = 10.08 - 30.76	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 4- phenyltoluene (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
353	100.8	10.08	6.629
353	104.6	10.46	6.622
353	120.7	12.07	12.370
353	137.7	13.77	9.990
353	148.9	14.89	21.670
353	162.8	16.28	35.580
353	178.6	17.86	46.790
353	201.6	20.16	79.140
353	215.4	21.54	118.110
353	227.5	22.75	144.37
353	255.3	25.53	223.14
383	94.4	9.44	10.940
383	97.3	9.73	10.900
383	124.7	12.47	11.600
383	140.2	14.02	21.320
383	169.5	16.95	36.650
383	178.3	17.83	35.150
383	185.4	18.54	63.230
383	191.9	19.19	67.330
383	215.8	21.58	104.750
383	244.1	24.41	134.060
383	251.9	25.19	154.010
383	252.5	25.25	154.960
383	307.6	30.76	246.50

Auxiliary Information

The apparatus consists of an equilibrium cell into which the solid was loaded. Liquid carbon dioxide was compressed by the use of a pressure generator and directed to the equilibrium cell. The temperature in the cell was increased to the desired isothermal condition and the pressure raised until dissolution of the compound occurred and a homogeneous phase existed. Once the compound had been fully dissolved, the pressure was lowered to the onset of cloud point. The volume of carbon dioxide present in the vessel at cloud point was recorded by depressurizing across micro metering valve and passing the expanded stream through a wet-test meter. For safety reasons any heavy component present in the expanded gas stream was trapped in the glass collector.

Source and Purity of Materials:
1. 4-phenyltoluene source Sigma-Aldrich, purity 97%.
2. carbon dioxide source BOC, purity 99.9%

Estimated Error:
Temperature: $\pm$ 0.1C
Pressure: $\pm$ 1.1 bar
Solubility: unstated

Figure A.76 Solubility of 4-phenyltoluene in Carbon Dioxide.

Components:	Original Measurements:
(1) n - Hexadene $C_{16}H_{34}$; [544-76-3] (2) Carbon Dioxide; CO_2; [124-38-9]	Gary A.Leeke, Regina C.Santos, Jonathan Seville, Bushra Al-Duri, Connie K.Y.Lee, Andrew B. Holmes, J. Chem. Eng. Data, *49*, 48-52 (2004).
Variables: T/K = 333 P/MPa = 9.8 - 15.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of n-hexadene (1) in Carbon Dioxide (2)

T/K	P/bar (compiler – From graph)	P/MPa	x_1 X 10^3 (compiler – from graph)
333	98	9.8	2.815
333	100	10.0	3.016
333	115	11.5	5.631
333	120	12.0	7.240
333	130	13.0	8.447
333	150	15.0	12.067

Auxiliary Information

The apparatus consists of an equilibrium cell into which the solid was loaded. Liquid carbon dioxide was compressed by the use of a pressure generator and directed to the equilibrium cell. The temperature in the cell was increased to the desired isothermal condition and the pressure raised until dissolution of the compound occurred and a homogeneous phase existed. Once the compound had been fully dissolved, the pressure was lowered to the onset of cloud point. The volume of carbon dioxide present in the vessel at cloud point was recorded by depressurizing across micro metering valve and passing the expanded stream through a wet-test meter. For safety reasons any heavy component present in the expanded gas stream was trapped in the glass collector.

Source and Purity of Materials:
1. n-hexadene source unstated, purity unstated
2. carbon dioxide source BOC, purity 99.9%

Estimated Error:
Temperature: ± 0.1C
Pressure: ± 1.1 bar
Solubility: unstated

Figure A.77 Solubility of n-hexadene in Carbon Dioxide.

Components:	Original Measurements:
(1) dimethyl 2H-1-benzopyran2,3-dicarboxylate; A1; C₁₃H₁₂O₅; [182134-81-2]	Naader Bahramifar, Yadollah Yamini, Ali Ramazani, Nader Noshiranzadeh, J. Chem. Eng. Data, *48*,
(2) Carbon Dioxide; CO₂; [124-38-9]	1104-1108 (2003)
Variables: T/K = 308, 318 P/MPa =12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A1 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^3$
308	12.2	5.86
308	15.2	7.50
308	18.2	9.35
308	21.3	9.88
308	24.3	10.31
308	27.4	11.84
308	30.4	13.04
308	33.4	13.77
308	35.5	14.30
318	12.2	5.22
318	15.2	10.36
318	18.2	13.53
318	21.3	17.57
318	24.3	20.98
318	27.4	23.55
318	30.4	24.55
318	33.4	25.90
318	35.5	27.82

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. A1 synthesized, purity 99%m
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ± 1K
Pressure: ±0.1MPa
Solubility: ±2%

References:
1. Yamini,Y.; Bahrmifar,N, J. Chem. Eng. Data, *45*, 53-56 (2000)

Figure A.78 Solubility of A1 in Carbon Dioxide.

Components:	Original Measurements:
(1) di-tert-butyl-3H-naphtho[2,1-b]pyran-2,3-; dicarboxylate; A2; $C_{23}H_{26}O_5$; [189107-54-8]	Naader Bahramifar, Yadollah Yamini, Ali Ramazani, Nader Noshiranzadeh, J. Chem. Eng. Data, *48*,
(2) Carbon Dioxide; CO_2; [124-38-9]	1104-1108 (2003)
Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A2 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	8.98
308	18.2	12.17
308	24.3	14.91
308	30.4	16.18
308	35.5	17.32
318	12.2	6.33
318	18.2	12.20
318	24.3	16.09
318	30.4	23.07
318	35.5	26.30
328	12.2	3.30
328	18.2	10.35
328	24.3	20.82
328	30.4	30.80
328	35.5	37.31
338	12.2	2.28
338	18.2	9.62
338	24.3	29.71
338	30.4	42.62
338	35.5	59.36
348	12.2	1.99
348	18.2	11.08
348	24.3	33.07
348	30.4	67.60
348	35.5	99.19

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. A2 synthesized, purity 99%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ± 1K
Pressure: ±0.1MPa
Solubility: ±2%

References:
1. Yamini,Y.; Bahrmifar,N, J. Chem. Eng. Data, *45*, 53-56 (2000)

Figure A.79 Solubility of A2 in Carbon Dioxide.

Components:	Original Measurements:
(1) dimethyl 6-bromo-2H-1-benzopyran-2,3-dicarboxylate;A3;$C_{13}H_{11}BrO_5$;[428517-44-6] (2) CarbonDioxide;CO_2;[124-38-9]	Naader Bahramifar, Yadollah Yamini, Ali Ramazani, Nader Noshiranzadeh, J.Chem.Eng.Data, 48, 1104-1108 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A3(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	12.2	1.78
308	18.2	2.51
308	24.3	3.41
308	30.4	4.74
308	35.5	5.73
318	12.2	1.61
318	18.2	2.90
318	24.3	4.78
318	30.4	7.12
318	35.5	9.36
328	12.2	1.64
328	18.2	2.94
328	24.3	6.76
328	30.4	10.49
328	35.5	14.30
338	12.2	0.46
338	18.2	2.51
338	24.3	7.75
338	30.4	15.75
338	35.5	20.73
348	12.2	0.41
348	18.2	2.65
348	24.3	8.18
348	30.4	19.96
348	35.5	25.17

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. A3 synthesized, purity 99%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$0.1MPa
Solubility: $\pm$2%

References:
1. Yamini,Y.; Bahrmifar,N, J. Chem. Eng. Data, 45, 53-56 (2000)

Figure A.80 Solubility of A3 in Carbon Dioxide.

Components:	Original Measurements:
(1) dimethyl-3H-naphthol[2,1-b]pyran-2,3- dicarboxylate; A4; $C_{17}H_{14}O_5$; [189107-50-4] (2) Carbon Dioxide; CO_2; [124-38-9]	Naader Bahramifar, Yadollah Yamini, Ali Ramazani, Nader Noshiranzadeh, J. Chem. Eng. Data, *48*, 1104-1108 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 15.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A4(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308	15.2	2.08
308	18.2	2.42
308	24.3	3.48
308	30.4	3.89
308	35.5	4.85
318	15.2	1.85
318	18.2	2.42
318	24.3	3.80
318	30.4	5.85
318	35.5	7.97
328	15.2	1.53
328	18.2	2.29
328	24.3	4.38
328	30.4	7.20
328	35.5	10.66
338	15.2	1.57
338	18.2	2.22
338	24.3	6.14
338	30.4	10.60
338	35.5	14.96
348	15.2	1.33
348	18.2	1.53
348	24.3	6.00
348	30.4	11.50
348	35.5	17.46

Auxiliary Information

A Suprex MPS/225 system equipped with a modified
static system for solubility determination in the SFE
mode was used. A detailed description of the equipment
and operating procedures has been given previously[1].
The solid solutes were mixed well with a proper
amount of glass beads and packed into the extraction
vessel. Supercritical carbon dioxide was pressurized
and passed into the extraction vessel. After equilibrium
portion of saturated supercritical carbon dioxide was
loaded into an injection loop. Then the loop was
depressurized into the collection vial. The solubility
were calculated by absorbance measurements using
a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. A4 synthesized, purity 99%
2. carbon dioxide source Sabalan, Iran;
   purity 99.99%

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$0.1MPa
Solubility: $\pm$2%

References:
1. Yamini,Y.; Bahrmifar,N, J. Chem. Eng.
   Data, *45*, 53-56 (2000)

Figure A.81 Solubility of A4 in Carbon Dioxide.

Components:	Original Measurements:
(1) alpha – asarone; trans-1-propenyl-2,4,5-trimethoxy-benzene; $C_{12}H_{16}O_3$; [2883-98-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Xingquan Chen, Tiansheng Zhao, Wei Yu, Fluid Phase Equilibria, *211*, 11-15 (2003)
Variables: T/K = 308, 315, 322 P/MPa = 9.0 - 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of alpha - asarone(1) in Carbon Dioxide (2)

T/K	P/bar (compiler – from graph)	P/MPa	$x_1 \times 10^4$ (compiler – from graph)
308	90	9.0	100
308	120	12.0	140
308	150	15.0	190
308	180	18.0	220
315	90	9.0	20
315	120	12.0	80
315	150	15.0	150
315	180	18.0	190
322	90	9.0	10
322	120	12.0	50
322	150	15.0	95
322	180	18.0	155

Auxiliary Information

A certain amount of alpha- asarone was put in an equilibrium cell. After the whole apparatus was sealed, high pressure carbon dioxide from supply tank was continuously compressed into the equilibrium cell by a compressor. After a certain time when equilibrium was reached, alpha-asarone containing sample was taken via the sampling valve and the released carbon dioxide was measured by rotameter. The amount of alpha- asarone separated out from super – critical carbon dioxide was weighed by an automatic analysis balance.

Source and Purity of Materials:
(1) alpha- asarone obtained from alkali-melting; purity unstated.
(2) carbon dioxide source Feida Co., Shanghai; purity 98.5%

Estimated Errors:
Temperature: ±0.1C
Pressure: unstated
Solubility: ± 0.0001g

Figure A.82 Solubility of Alpha- asarone in Carbon Dioxide.

Components:	Original Measurements:
(1) Thioxanthone;T1; $C_{13}H_8OS$; [492-22-8] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, Ali Reza Salimi, J. Chem. Eng. Data, *48*, 1088-1091 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.1 - 35.4	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of T1 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	121	12.1	0.24
308	182	18.2	0.30
308	243	24.3	0.42
308	304	30.4	0.45
308	354	35.4	0.52
318	121	12.1	0.16
318	182	18.2	0.39
318	243	24.3	0.53
318	304	30.4	0.65
318	354	35.4	0.68
328	121	12.1	0.19
328	182	18.2	0.47
328	243	24.3	0.71
328	304	30.4	0.85
328	354	35.4	1.06
338	121	12.1	0.00
338	182	18.2	0.43
338	243	24.3	0.83
338	304	30.4	1.08
338	354	35.4	1.29
348	121	12.1	0.01
348	182	18.2	0.43
348	243	24.3	1.02
348	304	30.4	1.43
348	354	35.4	1.68

Auxiliary Information

A Suprex MPS/225 system equipped with a modified
static system for solubility determination in the SFE
mode was used. A detailed description of the equipment
and operating procedures has been given previously[1].
The solid solutes were mixed well with a proper
amount of glass beads and packed into the extraction
vessel. Supercritical carbon dioxide was pressurized
and passed into the extraction vessel. After equilibrium
portion of saturated supercritical carbon dioxide was
loaded into an injection loop. Then the loop was
depressurized into the collection vial. The solubility
were calculated by absorbance measurements using
a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. T1 synthesized, purity 99.5%
2. carbon dioxide source Sabalan, Iran;
   purity 99.99%

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$0.5bar
Solubility: $\pm$3%

References:
1. Fathi,M.R.;Yamini,Y.;Sharghi,H.;
   Shamsipur,M, J. Chem. Eng. Data, *43*,
   400-402 (1998).

Figure A.83 Solubility of T1 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxythioxanthane; T2; $C_{13}H_8O_2S$;[84682-31-5]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, Ali Reza Salimi, J. Chem.
(2) Carbon Dioxide; CO_2; [124-38-9]	Eng. Data, *48*, 1088-1091 (2003)
Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348	Dana E. Knox; Syed A. Abdullah
P/MPa = 12.1 - 35.4	

Experimental Data
Solubility of T2 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	121	12.1	0.99
308	182	18.2	1.38
308	243	24.3	1.92
308	304	30.4	2.21
308	354	35.4	2.59
318	121	12.1	0.74
318	182	18.2	1.46
318	243	24.3	2.00
318	304	30.4	2.45
318	354	35.4	2.75
328	121	12.1	0.61
328	182	18.2	1.47
328	243	24.3	2.22
328	304	30.4	2.83
328	354	35.4	3.28
338	121	12.1	0.37
338	182	18.2	1.32
338	243	24.3	2.35
338	304	30.4	3.25
338	354	35.4	3.88
348	121	12.1	0.33
348	182	18.2	1.20
348	243	24.3	2.46
348	304	30.4	3.61
348	354	35.4	4.24

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. T2 synthesized, purity 99.5%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: $\pm$ 1K
Pressure: $\pm$0.5bar
Solubility: $\pm$3%

References:
1. Fathi,M.R.;Yamini,Y.;Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998).

Figure A.84 Solubility of T2 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-hydroxy-3-methylthioxanthane;T3; $C_{14}H_{10}O_2S$;[583826-13-5]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, Ali Reza Salimi, J. Chem.
(2) Carbon Dioxide; CO_2; [124-38-9]	Eng. Data, *48*, 1088-1091 (2003)
Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348 P/MPa = 12.1 - 35.4	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of T3 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	121	12.1	0.57
308	182	18.2	0.65
308	243	24.3	0.76
308	304	30.4	0.92
308	354	35.4	1.04
318	121	12.1	0.45
318	182	18.2	0.76
318	243	24.3	1.04
318	304	30.4	1.25
318	354	35.4	1.47
328	121	12.1	0.41
328	182	18.2	0.76
328	243	24.3	1.14
328	304	30.4	1.53
328	354	35.4	1.93
338	121	12.1	0.36
338	182	18.2	0.81
338	243	24.3	1.40
338	304	30.4	1.96
338	354	35.4	2.35
348	121	12.1	0.43
348	182	18.2	0.80
348	243	24.3	1.56
348	304	30.4	2.42
348	354	35.4	3.18

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility wWere calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1.T3 synthesized, purity 99.5%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ± 1K
Pressure: ±0.5bar
Solubility: ±3%

References:
1. Fathi,M.R.;Yamini,Y.;Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998).

Figure A.85 Solubility of T3 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1,4-dihydroxy-3-methyl-thioxanthane;T4; $C_{14}H_{10}O_3S$; [583826-14-6]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, Ali Reza Salimi, J. Chem.
(2) Carbon Dioxide; CO_2; [124-38-9]	Eng. Data, *48*, 1088-1091 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.1 - 35.4	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of T4 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	x_1 X 10^5
308	121	12.1	0.35
308	182	18.2	0.38
308	243	24.3	0.79
308	304	30.4	0.94
308	354	35.4	1.20
318	121	12.1	0.42
318	182	18.2	0.93
318	243	24.3	1.19
318	304	30.4	0.44
318	354	35.4	1.55
328	121	12.1	---
328	182	18.2	0.95
328	243	24.3	1.48
328	304	30.4	2.33
328	354	35.4	2.74
338	121	12.1	---
338	182	18.2	0.81
338	243	24.3	2.07
338	304	30.4	3.08
338	354	35.4	3.67
348	121	12.1	---
348	182	18.2	1.23
348	243	24.3	1.92
348	304	30.4	3.29
348	354	35.4	4.31

Auxiliary Information

A Suprex MPS/225 system equipped with a modified static system for solubility determination in the SFE mode was used. A detailed description of the equipment and operating procedures has been given previously[1]. The solid solutes were mixed well with a proper amount of glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium portion of saturated supercritical carbon dioxide was loaded into an injection loop. Then the loop was depressurized into the collection vial. The solubility were calculated by absorbance measurements using a UV-Vis spectrophotometer.

Source and Purity of Materials:
1. T4 synthesized, purity 99.5%
2. carbon dioxide source Sabalan, Iran; purity 99.99%

Estimated Error:
Temperature: ± 1K
Pressure: ±0.5bar
Solubility: ±3%

References:
1. Fathi,M.R.;Yamini,Y.;Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998).

Figure A.86 Solubility of T4 in Carbon Dioxide.

Components:	Original Measurements:
(1) 7,8-Dihydroxyflavone; $C_{15}H_{10}O_4$; [38183-03-8] (2) Carbon Dioxide; CO_2; [124-38-9]	Kiyoshi matsuyama, Kenji Mishima, Ryugen Ohdate, Masuhiro Chidori, Huai Yang, J. Chem. Eng. Data, *48*, 1040-1043 (2003)
Variables: T/K = 308.2, 318.2 P/MPa = 9.1 - 25.3	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 7,8-Dihydroxyflavone (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
308.2	9.1	0.86
308.2	10.1	1.21
308.2	15.2	1.51
308.2	20.3	1.80
308.2	25.3	1.87
318.2	9.1	1.60
318.2	10.1	3.21
318.2	15.2	3.60
318.2	20.3	3.88

Auxiliary Information

A flow type apparatus was used to measure the solubility. A detailed description of the apparatus and operating conditions is given elsewhere[1]. The liquefied carbon dioxide leaving a gas cylinder was passed through a cooling unit to prevent vaporization of carbon dioxide from warming up. Carbon dioxide was passed through a preheating coil and then entered in two equilibrium cells containing the solute. The solid component was packed in the second cell while the first cell was used as a buffer. The supercritical fluid saturated with the solid component. They were separated in a tube. The solid collected was weighed by a direct balance reading.

Source and Purity of Materials:
1. 7,8-dihydroxyflavone source Tokyo Kasei Kogyo Co. Ltd, purity 98%
2. carbon dioxide source Fakuoka Sanso Co. Ltd, purity 99%

Estimated Errors:
Temperature:± 0.1K
Pressure: ± 0.3%
Solubility: ± 4.0%

References:
1. Uchiyama,H.; Mishima,K.; Oka,S.; Ezawa,M.; Ide,M.; Takai,T.; Park,P.W, J. Chem. Eng. Data, 42, 570-573 (1997)

Figure A.87 Solubility of 7,8-dihydroxyflavone in Carbon Dioxide.

Components:	Original Measurements:
(1) 3,3',4',5,7-pentahyfroxyflavone(quercetin) $C_{15}H_{10}O7$; [117-39-5]	Kiyoshi matsuyama, Kenji Mishima, Ryugen Ohdate, Masuhiro Chidori, Huai Yang, J. Chem.
(2) Carbon Dioxide; CO_2; [124-38-9]	Eng. Data, *48*, 1040-1043 (2003)
Variables: T/K = 308.2, 318.2 P/MPa = 9.1 - 20.3	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of quercetin (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
308.2	9.1	0.61
308.2	10.1	0.95
308.2	15.2	1.05
308.2	20.3	1.20
318.2	9.1	0.54
318.2	10.1	1.88
318.2	15.2	2.14
318.2	20.3	2.20

Auxiliary Information

A flow type apparatus was used to measure the solubility. A detailed description of the apparatus and operating conditions is given elsewhere[1]. The liquefied carbon dioxide leaving a gas cylinder was passed through a cooling unit to prevent vaporization of carbon dioxide from warming up. Carbon dioxide was passed through a preheating coil and then entered in two equilibrium cells containing the solute. The solid component was packed in the second cell while the first cell was used as a buffer. The supercritical fluid saturated with the solid component. They were separated in a tube. The solid collected was weighed by a direct balance reading.

Source and Purity of Materials:
1. quercetin source Tokyo Kasei Kogyo Co. Ltd, purity 98%
2. carbon dioxide source Fakuoka Sanso Co. Ltd, purity 99%

Estimated Errors:
Temperature:± 0.1K
Pressure: ± 0.3%
Solubility: ± 4.0%

References:
1. Uchiyama,H.; Mishima,K.; Oka,S.; Ezawa,M.; Ide,M.; Takai,T.; Park,P.W, J. Chem. Eng. Data, 42, 570-573 (1997)

Figure A.88 Solubility of Quercetin in Carbon Dioxide.

Components:	Original Measurements:
(1) Poly(beta-hydroxybutyrate); (C₄H₈O₃)X; [26063-00-3] (2) Carbon Dioxide; CO₂; [124-38-9]	Kianoush Khosravi-Darani, Ebrahim Vasheghani-Farahni, Yadollah Yamini, Naader Bahramifar, J. Chem. Eng. Data, *48*, 860-863 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of poly(beta-hydroxybutyrate) (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	x_1 X 10^8
308	122	12.2	3.852
308	182	18.2	6.926
308	243	24.3	6.930
308	304	30.4	10.990
308	355	35.5	13.880
318	122	12.2	2.579
318	182	18.2	7.361
318	243	24.3	10.780
318	304	30.4	12.990
318	355	35.5	15.860
328	122	12.2	2.510
328	182	18.2	7.954
328	243	24.3	12.240
328	304	30.4	17.350
328	355	35.5	21.710
338	122	12.2	2.480
338	182	18.2	8.495
338	243	24.3	16.270
338	304	30.4	21.540
338	355	35.5	30.940
348	122	12.2	2.520
348	182	18.2	6.489
348	243	24.3	20.760
348	304	30.4	33.560
348	355	35.5	54.320

Auxiliary Information

A suprex MPS/225 system equipped with a modified static system for solubility determination in super-critical fluid extraction mode used. Detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid polymer was mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the vessel. After equilibrium was obtained, the loop was depressurized into the collection vial. To determine the polymer concentration, 2ml of solution was subjected to methanolysis in the presence of sulfuric acid according to Brandl method. Gas chromatography of the resulting methyl ester of the constituent 3-hydroxybutyrate was performed with a Philip's scientific model 44100 gas chromatograph.

Source and Purity of Materials:
1. Poly(beta-hydroxybutyrate) source Aldrich, purity unstated.
2. carbon dioxide source Sabalan, purity 99.99 %w

Estimated Errors:
Temperature: ± 1K
Pressure : ±0.5 bar
Solubility : ±5%

References:
1. Yamini,Y.; Bahramifar,N, J. Chem. Eng. Data, 45, 53-56 (2000).
Figure A.89 Solubility of Poly (beta-Hydroxybutyrate in Carbon Dioxide.

Components:	Original Measurements:
(1) Hexanoic acid;	Hassan S.Ghaziaskar, M. Nikravesh, fluid Phase
$C_6H_{12}O_2$; [142-62-1]	Equilibria, *206*, 215-221 (2003)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 313, 323	Dana E. Knox; Syed A. Abdullah
P/MPa = 7.5 - 14.0	

Experimental Data
Solubility of hexanoic acid (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^3$
313	75	7.5	0.35(± 0.03)
313	88	8.8	6.27(±0.50)
313	95	9.5	---
313	100	10.0	10.32(±0.54)
313	105	10.5	10.51(±0.61)
313	120	12.0	10.72(±0.53)
313	123	12.3	---
313	130	13.0	10.83(±0.46)
313	140	14.0	---
323	75	7.5	0.43(±0.05)
323	88	8.8	2.28(±0.24)
323	95	9.5	5.63(±0.33)
323	100	10.0	---
323	105	10.5	8.95(±0.35)
323	120	12.0	---
323	123	12.3	9.91(±0.28)
323	130	13.0	10.03(±0.61)
323	140	14.0	10.10(±0.37)

Auxiliary Information

The solubility measurements were carried out using a continuous flow apparatus. The solute was placed in an equilibrium cell. With the cell in the air thermostat, the liquid carbon dioxide passed through a cooling coil into a reciprocating HPLC pump. The equilibrium cell was fitted with glass wool to increase the contact surface. The liquid carbon dioxide was pressurized through a preheated coil in the oven. After equilibrium was reached, the saturated supercritical carbon dioxide was depressurized. The dissolved compound was trapped and collected in a vial. The trapped solute was weighed with an analytical balance.

Source and Purity of Materials:
1. hexanoic acid source Merck Chemical Co., purity 98%.
2. carbon dioxide source ZamZam Co.Ltd.(Iran), purity 99.95%

Estimated errors:
Temperature:±0.5K
Pressure:±1bar
Solubility: varies

Figure A.90 Solubility of Hexanoic acid in Carbon Dioxide.

Components:	Original Measurements:
(1) butyl acetate; $C_6H_{12}O_2$; [123-86-4] (2) Carbon Dioxide; CO_2; [124-38-9]	Hassan S.Ghaziaskar, M. Nikravesh, fluid Phase Equilibria, *206*, 215-221 (2003)
Variables: T/K = 313, 323 P/MPa = 7.5 - 14.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of butyl acetate (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^3$
313	80	8.0	3.5($\pm$0.5)
313	85	8.5	11.1($\pm$1.3)
313	90	9.0	13.5($\pm$1.7)
313	100	10.0	14.4($\pm$0.7)
313	120	12.0	10.0($\pm$1.0)
313	130	13.0	16.6($\pm$1.0)
313	140	14.0	19.6($\pm$0.5)
313	160	16.0	17.9($\pm$0.6)
313	180	18.0	22.4($\pm$2.2)
323	80	8.0	3.4($\pm$0.4)
323	95	9.5	4.3($\pm$0.3)
323	90	9.0	7.2($\pm$0.8)
323	100	10.0	15.4($\pm$1.1)
323	120	12.0	11.1($\pm$0.8)
323	130	13.0	---
323	140	14.0	12.3($\pm$0.6)
323	160	16.0	17.0($\pm$0.5)
323	180	18.0	19.5($\pm$2.0)

Auxiliary Information

The solubility measurements were carried out using a continuous flow apparatus. The solute was placed in an equilibrium cell. With the cell in the air thermostat, the liquid carbon dioxide passed through a cooling coil into a reciprocating HPLC pump. The equilibrium cell was fitted with glass wool to increase the contact surface. The liquid carbon dioxide was pressurized through a preheated coil in the oven. After equilibrium was reached, the saturated supercritical carbon dioxide was depressurized. The dissolved compound was trapped and collected in a vial The trapped solute was weighed with an analytical balance.

Source and Purity of Materials:
1. butyl acetate source Merck Chemical Co., purity 99.5%.
2. carbon dioxide source ZamZam Co.Ltd.(Iran), purity 99.95%

Estimated errors:
Temperature:$\pm$0.5K
Pressure:$\pm$1bar
Solubility: varies

Figure A.91 Solubility of Butyl Acetate in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-naphthol; $C_{10}H_8O$;[135-19-3]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase
(2) Carbon Dioxide; CO_2; [124-38-9]	Equilibria , *207*, 183-192 (2003)
Variables: T/K = 308.1, 318.1, 328.1 P/MPa =10.05 - 30.00	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 2-naphthol (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^4$
308.1	10.05	2.49
308.1	14.00	4.33
308.1	18.00	5.09
308.1	22.05	5.47
308.1	26.00	6.10
308.1	30.00	6.54
318.1	10.05	1.48
318.1	14.00	4.70
318.1	18.00	6.49
318.1	22.00	7.72
318.1	26.00	8.47
318.1	30.00	9.64
328.1	10.05	1.16
328.1	14.00	4.41
328.1	18.00	6.92
328.1	22.05	9.25
328.1	26.00	11.4
328.1	30.00	12.3

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. 2-naphthol source German firm, purity 99%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%

Estimated Errors:
Temperature: +0.1K
Pressure: +0.5bar
Solubility: ±2%

Figure A.92 Solubility of 2-naphthol in Carbon Dioxide.

Components:	Original Measurements:
(1) anthracene;	Qunsheng Li, Zeting Zhang, Chongli Zhong,
$C_{14}H_{10}$;[120-12-7]	Yancheng Liu, Qingrong Zhou, Fluid Phase
(2) Carbon Dioxide; CO_2;[124-38-9]	Equilibria , 207, 183-192 (2003)

Variables:	Prepared By:
T/K = 308.1, 318.1, 328.1	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.00 - 30.00	

Experimental Data
Solubility of anthracene (1) in Carbon Dioxide (2)

T/K	P/MPa	x_1 X 10^4
308.1	10.00	0.416
308.1	15.00	0.508
308.1	20.00	0.696
308.1	25.00	0.759
308.1	30.00	0.783
318.1	10.00	0.205
318.1	15.00	0.539
318.1	20.00	0.745
318.1	25.00	0.851
318.1	30.00	0.940
328.1	10.00	0.138
328.1	15.00	0.573
328.1	20.00	0.807
328.1	25.00	1.020
328.1	30.00	1.113

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. anthracene source Xuxing Chemical Factory, purity 99.8%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%

Estimated Errors:
Temperature: $\pm$0.1K
Pressure:$\pm$0.5bar
Solubility:$\pm$2%

Figure A.93 Solubility of Anthracene in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-naphthol; $C_{10}H_8O$;[135-19-3]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase
(2) Carbon Dioxide; CO_2; [124-38-9], with ethanol; C_2H_5OH; [64-17-5]	Equilibria , *207*, 183-192 (2003)

Variables:	Prepared By:
T/K = 308.1, 318.1, 328.1	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.05 - 30.00	

Experimental Data
Solubility of 2-naphthol (1) in Carbon Dioxide with 3.6% ethanol (2)

T/K	P/MPa	$x_1 \times 10^4$
308.1	10.05	18.2
308.1	14.00	21.7
308.1	18.00	23.6
308.1	22.05	25.3
308.1	26.00	26.8
308.1	30.00	27.7
318.1	10.05	11.5
318.1	14.00	20.6
318.1	18.00	23.6
318.1	22.00	25.7
318.1	26.00	27.5
318.1	30.00	30.2
328.1	10.05	5.53
328.1	14.00	14.9
328.1	18.00	21.6
328.1	22.05	24.3
328.1	26.00	29.3
328.1	30.00	32.7

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. 2-naphthol source German firm, purity 99%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%; ethanol Source Beijing Chem. Factory, purity 99.5%

Estimated Errors:
Temperature: ±0.1K
Pressure:±0.5bar
Solubility:±2%

Figure A.94 Solubility of 2-naphthol in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) 2-naphthol; $C_{10}H_8O$; [135-19-3]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase
(2) Carbon Dioxide; CO_2; [124-38-9], with acetone; CH_3COCH_3; [67-64-1]	Equilibria , *207*, 183-192 (2003)
Variables: T/K = 308.1, 318.1, 328.1 P/MPa = 10.05 - 30.00	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of 2-naphthol (1) in Carbon Dioxide with 3.6% acetone (2)

T/K	P/MPa	$x_1 \times 10^4$
308.1	10.05	4.83
308.1	14.00	5.85
308.1	18.00	6.35
308.1	22.05	7.06
308.1	26.00	8.16
308.1	30.00	9.16
318.1	10.05	4.78
318.1	14.00	7.66
318.1	18.00	9.77
318.1	22.00	11.0
318.1	26.00	12.6
318.1	30.00	15.0
328.1	10.05	3.79
328.1	14.00	10.7
328.1	18.00	15.0
328.1	22.05	18.7
328.1	26.00	22.3
328.1	30.00	19.0

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. 2-naphthol source German firm, purity 99%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%; acetone Source Beijing Alc. Factory, purity 99.5%

Estimated Errors:
Temperature: +0.1K
Pressure:+0.5bar
Solubility:+2%

Figure A.95 Solubility of 2-naphthol in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-naphthol; C₁₀H₈O;[135-19-3] (2) Carbon Dioxide; CO₂; [124-38-9], with cyclohexane; C₆H₁₂; [110-82-7]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase Equilibria , *207*, 183-192 (2003)

Variables:	Prepared By:
T/K = 308.1, 318.1, 328.1 P/MPa = 10.05 - 30.00	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 2-naphthol (1) in Carbon Dioxide with 3.6% cyclohexane (2)

T/K	P/MPa	$x_1 \times 10^4$
308.1	10.05	7.33
308.1	14.00	8.84
308.1	18.00	9.74
308.1	22.05	10.6
308.1	26.00	11.4
308.1	30.00	12.4
318.1	10.05	5.80
318.1	14.00	8.51
318.1	18.00	10.7
318.1	22.00	13.3
318.1	26.00	13.8
318.1	30.00	15.5
328.1	10.05	3.13
328.1	14.00	8.93
328.1	18.00	12.2
328.1	22.05	14.6
328.1	26.00	17.8
328.1	30.00	18.9

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. 2-naphthol source German firm, purity 99%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%; cyclohexane Source Beijing Chemical Factory, purity 99%

Estimated Errors:
Temperature: ±0.1K
Pressure: ±0.5bar
Solubility: ±2%

Figure A.96 Solubility of 2-naphthol in Carbon Dioxide with Cyclohexane.

Components:	Original Measurements:

(1) anthracene;
    $C_{14}H_{10}$;[120-12-7]
(2) Carbon Dioxide; CO_2; [124-38-9]
    acetone; CH_3COCH_3; [67-64-1]
	Qunsheng Li, Zeting Zhang, Chongli Zhong,
	Yancheng Liu, Qingrong Zhou, Fluid Phase
	Equilibria , 207, 183-192 (2003)

Variables: T/K = 308.1, 318.1, 328.1 P/MPa = 10.00 - 30.00	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of anthracene (1) in Carbon Dioxide with 4% acetone (2)

T/K	P/MPa	x_1 X 10^5
308.1	10.00	6.66
308.1	15.00	7.68
308.1	20.00	8.59
308.1	25.00	9.16
308.1	30.00	9.56
318.1	10.00	5.57
318.1	15.00	9.39
318.1	20.00	10.3
318.1	25.00	11.2
318.1	30.00	11.9
328.1	10.00	3.68
328.1	15.00	8.68
328.1	20.00	10.6
328.1	25.00	12.8
328.1	30.00	13.5

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. anthracene source Xuxing Chemical Factory, purity 99.8%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%;acetone Source Beijing Alc. Factory, purity 99.5%

Estimated Errors:
Temperature: +0.1K
Pressure: +0.5bar
Solubility: ±2%

Figure A.97 Solubility of Anthracene in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) anthracene; C$_{14}$H$_{10}$;[120-12-7] (2) Carbon Dioxide; CO$_2$; [124-38-9] ethanol; C$_2$H$_5$OH; [64-17-5]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase Equilibria , *207*, 183-192 (2003)
Variables: T/K = 308.1, 318.1, 328.1 P/MPa = 10.00 - 30.00	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of anthracene (1) in Carbon Dioxide with 4% ethanol (2)

T/K	P/MPa	x$_1$ X 10^5
308.1	10.00	8.51
308.1	15.00	9.92
308.1	20.00	10.9
308.1	25.00	11.6
308.1	30.00	12.5
318.1	10.00	7.78
318.1	15.00	9.93
318.1	20.00	11.5
318.1	25.00	12.7
318.1	30.00	13.7
328.1	10.00	5.22
328.1	15.00	9.88
328.1	20.00	12.7
328.1	25.00	14.4
328.1	30.00	15.4

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. anthracene source Xuxing Chemical Factory, purity 99.8%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%;ethanol Source Beijing Chem. Factory, purity 99.5%

Estimated Errors:
Temperature: +0.1K
Pressure: +0.5bar
Solubility: ±2%

Figure A.98 Solubility of Anthracene in carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) anthracene; $C_{14}H_{10}$;[120-12-7] (2) Carbon Dioxide; CO_2; [124-38-9] cyclohexane; C_6H_{12}; [110-82-7]	Qunsheng Li, Zeting Zhang, Chongli Zhong, Yancheng Liu, Qingrong Zhou, Fluid Phase Equilibria , 207, 183-192 (2003)
Variables: T/K = 308.1, 318.1, 328.1 P/MPa = 10.00 - 30.00	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data

Solubility of anthracene (1) in Carbon Dioxide with 4% cyclohexane (2)

T/K	P/MPa	$x_1 \times 10^5$
308.1	10.00	9.22
308.1	15.00	11.10
308.1	20.00	13.00
308.1	25.00	14.20
308.1	30.00	15.10
318.1	10.00	7.54
318.1	15.00	10.90
318.1	20.00	13.30
318.1	25.00	15.00
318.1	30.00	15.80
328.1	10.00	5.60
328.1	15.00	11.10
328.1	20.00	14.30
328.1	25.00	16.40
328.1	30.00	17.80

Auxiliary Information

The apparatus consists of an equilibrium cell packed with solid solute and each end was plugged with glass wool. Carbon dioxide and if any co-solvents were compressed into the mixer and finally they were put into the equilibrium cell. In the cell equilibrium was reached through mass transfer. The fluid phase which reached equilibrium flowed from the top of the cell through a decompress valve into two U-tubes. The solid solute was settled and weighed up by an analysis scale after drying. The volume was measured by the wet gas meter.

Source and Purity of Materials:
1. anthracene source Xuxing Chemical Factory, purity 99.8%
2. carbon dioxide source Beijing Chemical Experimental factory, purity 99%;cyclohexane Source Beijing Chem. Factory, purity 99%

Estimated Errors:
Temperature: +0.1K
Pressure: +0.5bar
Solubility: ±2%

Figure A.99 Solubility of Anthracene in Carbon Dioxide with Cyclohexane.

Components:	Original Measurements:
(1) Amical-48(diiodomethyl p-tolyl sulfone) $C_8H_8I_2O_2S$; [20018-09-1] (2) Carbon Dioxide; CO_2; [124-38-9]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)
Variables: T/K = 318, 328, 338 P/MPa = 10.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Amical 48 (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
318	10.0	1.22
318	15.0	---
318	20.0	3.90
318	25.0	6.20
318	30.0	7.26
328	10.0	---
328	15.0	---
328	20.0	6.38
328	25.0	8.98
328	30.0	8.66
338	10.0	0.63
338	15.0	3.67
338	20.0	8.45
338	25.0	9.63
338	30.0	11.5

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. Amical 48 source Sigma Chemical Co., purity 96%.
2. carbon dioxide source unstated, purity unstated.

Estimated Errors:
Temperature: $\pm 0.1C$
Pressure: $\pm 0.1MPa$
Solubility: $\pm 1.3\%$

Figure A.100 Solubility of Amical 48 in Carbon Dioxide.

Components:	Original Measurements:
(1) Chlorothalonil (tetrachloroisophthalonitrile) $C_8Cl_4N_2$; [1897-45-6] (2) Carbon Dioxide; CO_2; [124-38-9]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)
Variables: T/K = 318, 328, 338 P/MPa = 10.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Chlorothalonil (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
318	10.0	1.32
318	15.0	4.81
318	20.0	13.2
318	25.0	13.2
318	30.0	15.6
328	10.0	0.53
328	15.0	6.09
328	20.0	9.93
328	25.0	14.1
328	30.0	18.0
338	10.0	0.64
338	15.0	2.17
338	20.0	13.7
338	25.0	19.2
338	30.0	26.8

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. Chlorothalonil source Sigma Chemical Co., purity 95%.
2. carbon dioxide source unstated, purity unstated.

Estimated Errors:
Temperature: ±0.1C
Pressure: ±0.1 MPa
Solubility: ±1.3%

Figure A.101 Solubility of Chlorothalonil in Carbon Dioxide.

Components:	Original Measurements:
(1) TCMTB(2-(thiocyanomethylthio)benzothiazole $C_9H_6N_2S_3$; [1897-45-6] (2) Carbon Dioxide; CO_2; [124-38-9]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)
Variables: T/K = 323, 338 P/MPa = 10.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of TCMTB(1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
323	10.0	0.8
323	11.0	3.8
323	12.5	10.7
323	15.0	25.6
323	20.0	59.0
323	30.0	111.5
338	10.0	0.2
338	11.0	0.6
338	12.5	2.3
338	15.0	11.1
338	20.0	62.1
338	30.0	139.6

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1.TCMTB source Buckman Laboratories purity 99%.
2. carbon dioxide source unstated, purity unstated.

Estimated Errors:
Temperature: $\pm 0.1C$
Pressure: $\pm 0.1MPa$
Solubility: $\pm 1.3\%$

Figure A.102 Solubility of TCMTB in Carbon Dioxide.

Components:	Original Measurements:
(1) tebuconazole(alpha-[2-(4-chlorophenyl)ethyl]-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol); $C_{16}H_{22}ClN_3O$; [107534-96-3] (2) Carbon Dioxide; CO_2; [124-38-9]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)
Variables: T/K = 323, 338 P/MPa = 10.0-30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of tebuconazole (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^5$
323	10.0	0.63
323	11.0	1.34
323	12.5	4.78
323	15.0	16.50
323	20.0	33.43
323	30.0	64.21
338	10.0	0.35
338	11.0	0.57
338	12.5	1.80
338	15.0	8.08
338	20.0	43.38
338	30.0	185.71

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. tebuconazole source Mobay Corp. purity 95%.
2. carbon dioxide source unstated, purity unstated.

Estimated Errors:
Temperature: ± 0.1C
Pressure: ± 0.1MPa
Solubility: ± 1.3%

Figure A.103 Solubility of Tebuconazole in Carbon Dioxide.

Components:	Original Measurements:
(1) tebuconazole(alpha-[2-(4-chlorophenyl)ethyl]-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol); $C_{16}H_{22}ClN_3O$; [107534-96-3] (2) Carbon Dioxide; CO_2; [124-38-9], with methanol; CH_3OH; [67-56-1]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)

Variables:	Prepared By:
T/K = 323, 338 P/MPa = 10.0 - 30.0	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of tebuconazole (1) in Carbon Dioxide with 3%methanol (2)

T/K	P/MPa	$x_1 \times 10^5$
323	10.0	0.72
323	11.0	3.17
323	12.5	15.83
323	15.0	57.21
323	20.0	115.32
323	30.0	292.8
338	10.0	0.35
338	11.0	0.73
338	12.5	2.44
338	15.0	14.71
338	20.0	93.84
338	30.0	370.71

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. tebuconazole source Mobay Corp. purity 95%.
2. carbon dioxide and methanol source unstated, purity unstated.

Estimated Errors:
Temperature: ±0.1C
Pressure: ±0.1MPa
Solubility: ±1.3%

Figure A.104 Solubility of Tebuconazole in Carbon Dioxide with Methanol.

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Components:	Original Measurements:
(1) tebuconazole(alpha-[2-(4-chlorophenyl)ethyl]-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol); $C_{16}H_{22}ClN_3O$; [107534-96-3]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data, *48*, 541-547 (2003)
(2) Carbon Dioxide; CO_2; [124-38-9], with acetone; CH_3COCH_3; [67-64-1]	

Variables:	Prepared By:
T/K = 323, 338 P/MPa = 10.0 - 30.0	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of tebuconazole (1) in Carbon Dioxide with 3%acetone (2)

T/K	P/MPa	$x_1 \times 10^5$
323	10.0	1.94
323	11.0	9.59
323	12.5	17.86
323	15.0	44.83
323	20.0	88.56
323	30.0	169.10
338	10.0	0.86
338	11.0	1.68
338	12.5	4.55
338	15.0	19.33
338	20.0	91.07
338	30.0	246.89

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. tebuconazole source Mobay Corp. purity 95%.
2. carbon dioxide and acetone source unstated, purity unstated.

Estimated Errors:
Temperature: ±0.1C
Pressure: ±0.1MPa
Solubility: ±1.3%

Figure A.105 Solubility of Tebuconazole in Carbon Dioxide with Acetone.

Components:	Original Measurements:
(1) TCMTB(2-(thiocyanomethylthio)benzothiazole C$_9$H$_6$N$_2$S$_3$; [1897-45-6].	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J. Chem. Eng. Data,
(2) Carbon Dioxide; CO$_2$; [124-38-9], with methanol; CH$_3$OH; [67-56-1]	*48*, 541-547 (2003)
Variables: T/K = 323, 338 P/MPa = 10.0 - 30.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of TCMTB(1) in Carbon Dioxide with 3%methanol (2)

T/K	P/MPa	x$_1$ X 10^5
323	10.0	0.63
323	12.5	9.63
323	15.0	25.69
323	20.0	69.81
323	30.0	128.58
338	10.0	1.49
338	12.5	---
338	15.0	9.02
338	20.0	59.7
338	30.0	132.5

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. TCMTB source Buckman Laboratories purity 99%.
2. carbon dioxide and methanol source unstated, purity unstated.

Estimated Errors:
Temperature: ±0.1C
Pressure: ±0.1MPa
Solubility: ±1.3%

Figure A.106 Solubility of TCMTB in Carbon Dioxide with 3% Methanol.

Components:	Original Measurements:
(1) TCMTB(2-(thiocyanomethylthio)benzothiazole $C_9H_6N_2S_3$; [1897-45-6]	E. Sahle- Demessie, U.R.Pillai, S.Junsophonsri, K.L.Levien, J.Chem.Eng.Data,
(2) Carbon Dioxide; CO_2; [124-38-9], with acetone; CH_3COCH_3; [67-64-1]	*48*, 541-547 (2003)

Variables:	Prepared By:
T/K = 323, 338	Dana E. Knox; Syed A. Abdullah
P/MPa =10.0 - 30.0	

Experimental Data

Solubility of TCMTB(1) in Carbon Dioxide with 1.68%acetone (2)

T/K	P/MPa	$x_1 \times 10^5$
323	10.0	0.48
323	12.5	---
323	15.0	25.39
323	20.0	87.73
323	30.0	162.30
338	10.0	0.06
338	12.5	0.89
338	15.0	---
338	20.0	44.61
338	30.0	115.50

Auxiliary Information

The equipment used to measure solubility was a modified dual-pump system. The solute was ground and charged to the saturator with glass beads. Liquid carbon dioxide was drawn through a dip tube from a supply cylinder by a syringe pump. The system was purged with carbon dioxide at low pressure before being brought to the required temperature and pressure. It was then allowed to equilibrate thermally. The amount of solute dissolved was determined by two methods, either gravimetric or by chromatography. A precision balance was used for weighing.

Source and Purity of Materials:
1. TCMTB source Buckman Laboratories purity 99%.
2. carbon dioxide and acetone source unstated, purity unstated.

Estimated Errors:
Temperature: ±0.1C
Pressure: ±0.1MPa
Solubility: ±1.3%

Figure A.107 Solubility of TCMTB in Carbon Dioxide with 1.68% acetone.

Components:	Original Measurements:
(1) Impramine HCl; $C_{19}H_{24}N_2.HCl$; [113-52-0] (2) Carbon Dioxide; CO_2; [124-38-9]	Eliana Jara-Morante, David Suleiman, Antonio Estevez, Ind. Eng. Chem. *42*,1821-1823 (2003)
Variables: T/K = 313, 323 P/MPa = 30 - 50	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of Impramine.HCl (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^6$
313	30	6.4
313	35	7.6
313	40	9.0
313	45	9.9
313	50	5.1
323	30	6.1
323	35	8.8
323	40	8.2
323	45	8.0
323	50	6.4

Auxiliary Information

The experimental apparatus, a commercial extraction equipment, ISCO SFX 2-10, modified for this application. A typical experimental run was carried out by placing a measured amount of solids in the equilibrium cell and allowing the supercritical solvent to circulate through the cell slowly enough to ensure equilibrium. The amount of solute collected in the trap or reception tube was measured in two ways : gravimetrically and by dissolving the solute in methanol and measuring the concentration spectrophotometrically.

Source and Purity of Materials:
1. Impramine.HCl source Sigma Chemical Co, purity 99.99%
2. carbon dioxide source Scott Specialty Gases, purity 99%

Estimated Errors:
Temperature: unstated
Pressure: unstated
Solubility : $\pm 30\%$

Figure A.108 Solubility of Impramine HCl in Carbon Dioxide.

Components:	Original Measurements:
(1) Artemisinin;	Huabin Xing, Yiwen Yang, Baogen Su, Mei Huang,
$C_{15}H_{22}O_5$; [63968-64-9]	Qilong Ren, J. Chem. Eng. Data,
(2) Carbon Dioxide; CO_2; [124-38-9]	48, 330-332 (2003)
Variables:	Prepared By:
T/K = 310.1, 318.1, 328.1, 338.1	Dana E. Knox; Syed A. Abdullah
P/MPa = 10.0 - 27.0	

Experimental Data
Solubility of Artemisnin (1) in Carbon Dioxide (2)

T/K	P/MPa	$x_1 \times 10^3$
310.1	10.0	0.663
310.1	12.0	0.903
310.1	16.0	1.346
310.1	20.0	1.734
310.1	27.0	2.125
318.1	10.0	0.342
318.1	12.0	0.768
318.1	16.0	1.337
318.1	20.0	1.799
318.1	26.0	2.226
328.1	10.0	0.099
328.1	12.0	0.502
328.1	16.0	1.242
328.1	20.0	1.893
328.1	26.0	2.519
338.1	10.0	---
338.1	12.0	0.307
338.1	16.0	0.990
338.1	20.0	1.868
338.1	26.0	2.659

Auxiliary Information

A modification of the previous applied flow-type apparatus was used[1]. Pressurized carbon dioxide was introduced into the saturation cell. A commercially available empty HPLC column was used as the saturation cell, which was filled with Artemisinin mixed with clean sands. equilibrium was reached in within 5 to 7 minutes. A high pressure UV detector was used to detect solute concentration in supercritical carbon dioxide.

Source and Purity of Materials:
1. Artemisinin source Yunnan Phytopharma Co. LTD, China; purity 99%
2. carbon dioxide source unknown, purity 99.9%

Estimated Error :
Temperature: ± 0.1K
Pressure: ± 0.1MPa
Solubility: $\pm 4 \times 10^{-5}$

References:
1. Ren, Q.L.; Su,B.G.; Huang,M.; Wu, P.D, J. Chem. Eng. Data, 45, 464-466 (2000)

Figure A.109 Solubility of Artemisinin in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-Ethyl-1-hexanol; $C_8H_{18}O_6$; [104-76-7]	Hassan S. Ghaziaskar, Habibollah Eskandari, Ali Daneshfar, J. Chem. Eng. Data
(2) Carbon Dioxide; CO_2; [124-38-9]	*48*, 226-240 (2003)
Variables: T/K = 313, 323 P/MPa = 6.8 - 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 2-Ethyl-1-Hexanol (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	x_1 X 10^2
313	68	6.8	0.06
313	81	8.1	0.16
313	99	9.9	3.79
313	120	12.0	4.34
313	135	13.5	4.49
313	180	18.0	4.58
323	68	6.8	0.08
323	81	8.1	0.14
323	99	9.9	---
323	120	12.0	4.46
323	135	13.5	6.66
323	180	18.0	6.87

Auxiliary Information

The liquid carbon dioxide was transferred from a cylinder and an HPLC pump was used. The solute was placed in the equilibrium cell before it was placed into the oven to start the measurement. Some glass wool was placed to prevent physical entrapment of the non dissolved liquid. Under a constant flow rate of liquid carbon dioxide , the solubilized compounds were collected in a vial. The collected pure solutes were weighed and a packed column gas chromatograph was used to analyze the sample.

Source and Purity of Materials:
1. 2-ethyl-1-hexanol source Merck Chemical Co., purity 99%.
2. carbon dioxide source Zamzam Co. Ltd, purity 99.99%

Estimated Error:
Temperature: ±0.1K
Pressure: ±1bar
Solubility: ±10%

Figure A.110 Solubility of 2-ethyl-1-hexanol in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-Ethyl hexanoic acid; $C_8H_{16}O_2$; [149-57-5]	Hassan S. Ghaziaskar, Habibollah Eskandari, Ali Daneshfar, J. Chem. Eng. Data
(2) Carbon Dioxide; CO_2; [124-38-9]	*48*, 226-240 (2003)
Variables: T/K = 313, 323 P/MPa = 6.8 - 18.0	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of 2-Ethylhexanoic acid (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^2$
313	68	6.8	0.07
313	81	8.1	---
313	99	9.9	1.81
313	120	12.0	3.36
313	135	13.5	3.59
313	180	18.0	4.13
323	68	6.8	0.09
323	81	8.1	---
323	99	9.9	0.67
323	120	12.0	2.54
323	135	13.5	4.83
323	180	18.0	6.21

Auxiliary Information

The liquid carbon dioxide was transferred from a cylinder and an HPLC pump was used. The solute was placed in the equilibrium cell before it was placed into the oven to start the measurement. Some glass wool was placed to prevent physical entrapment of the non dissolved liquid. Under a constant flow rate of liquid carbon dioxide, the solubilized compounds were collected in a vial. The collected pure solutes were weighed and a packed column gas chromatograph was used to analyze the sample.

Source and Purity of Materials:
1. 2-ethylhexanoic acid source Merck Chemical Co purity 99%.
2. carbon dioxide source Zamzam Co. Ltd, purity 99.99%

Estimated Error:
Temperature: ± 0.1K
Pressure: ± 1bar
Solubility: $\pm 10\%$

Figure A.111 Solubility of 2-ethylhexanoic acid in Carbon Dioxide.

Components:	Original Measurements:
(1) 2-Ethyl-hexyl-2-ethyl $C_{16}H_{32}O_2$; [7425-14-1]	Hassan S. Ghaziaskar, A. Daneshfar, J. of Supercritical Fluids, *25*, 1-6 (2003)
(2) Carbon Dioxide; CO_2; [124-38-9]	

Variables:	Prepared By:
T/K = 313	Dana E. Knox; Syed A. Abdullah
P/MPa= 13.0 - 25.3	

Experimental Data

Solubility of 2-Ethyl-hexyl-2-ethyl (1) in Carbon Dioxide (2) at 250ml/min flow rate

T/K	P/bar	P/MPa	$x_1 \times 10^2$
313	130	13.0	0.57
313	151	15.1	1.30
313	170	17.0	1.60
313	202	20.2	1.70
313	253	25.3	2.00
333	170	17.0	0.95
353	170	17.0	0.87

Auxiliary Information

The solubility measurements were carried out using a continuous flow apparatus. The solute was placed in an equilibrium cell which was filled with glass Co. Ltd beads. The liquid carbon dioxide was pressurized through a preheated coil in the oven, and enabled carbon dioxide to reach oven temperature before it enters the cell. The saturated supercritical carbon dioxide was depressurized through a back-pressure regulator. The trapped 2E2E was dried and weighed with an analytical balance. The trapped mixtures were analyzed with a GC-TCD with a packed SE-30 column.

Source and Purity of Materials:
1. 2E2E synthesized , purity 99%.
2. carbon dioxide source ZamZam

Iran; purity 99.99%

Estimated Errors:
Temperature: ±0.1C
Pressure: unstated
Solubility: ± 10%

Figure A.112 Solubility of 2E2E in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-amino-2-methyl-9,10-anthraquinone	Mojtaba Shamsipur, Ali Reza Karami, Yadollah,
A1; $C_{15}H_{11}NO_2$; [82-28-0]	Yamini, Hashem Sharghi, J.Chem.Eng.Data,
(2) Carbon Dioxide; CO_2; [124-38-9]	*48*, 71-74 (2003)

Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348	Dana E. Knox; Syed A. Abdullah
P/MPa = 12.2 - 35.5	

Experimental Data
Solubility of A1 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308	122	12.2	1.90
308	182	18.2	3.08
308	243	24.3	4.17
308	304	30.4	4.62
308	355	35.5	5.51
318	122	12.2	1.48
318	182	18.2	3.06
318	243	24.3	4.72
318	304	30.4	5.38
318	355	35.5	6.12
328	122	12.2	0.80
328	182	18.2	2.76
328	243	24.3	4.97
328	304	30.4	7.93
328	355	35.5	9.07
338	122	12.2	0.46
338	182	18.2	2.49
338	243	24.3	4.99
338	304	30.4	6.68
338	355	35.5	---
348	122	12.2	0.48
348	182	18.2	3.05
348	243	24.3	5.49
348	304	30.4	9.52
348	355	35.5	10.96

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrofluorimeter.

Source and Purity of Materials:
1. A1 synthesized, purity 99.9%
2. carbon dioxide source Sabalan Gas Co., Iran; purity 99.99%

Estimated Errors:
Temperature: unstated
Pressure: unstated
Solubility: ±5%

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.113 Solubility of A1 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-amino-2-ethyl-9,10-anthraquinone A2; $C_{16}H_{13}NO_2$; [6937-81-1]. (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah, Yamini Hashem Sharghi, J. Chem. Eng. Data, *48*, 71-74 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A2 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308	122	12.2	1.58
308	182	18.2	2.55
308	243	24.3	3.35
308	304	30.4	3.61
308	355	35.5	3.91
318	122	12.2	1.29
318	182	18.2	2.75
318	243	24.3	3.63
318	304	30.4	4.03
318	355	35.5	4.41
328	122	12.2	0.89
328	182	18.2	2.75
328	243	24.3	4.48
328	304	30.4	5.70
328	355	35.5	6.07
338	122	12.2	0.26
338	182	18.2	2.97
338	243	24.3	5.00
338	304	30.4	5.10
338	355	35.5	4.36
348	122	12.2	0.13
348	182	18.2	3.09
348	243	24.3	5.99
348	304	30.4	7.78
348	355	35.5	---

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrofluorimeter.

Source and Purity of Materials:
1. A2 synthesized, purity 99.9%
2. carbon dioxide source Sabalan Gas Co.,Iran; purity 99.99%

Estimated Errors:
Temperature: unstated
Pressure: unstated
Solubility: ±5%

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.114 Solubility of A2 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-amino-2,3-di-methyl-9,10-anthraquinone A3; $C_{16}H_{13}NO_2$; [3225-89-6]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, J. Chem. Eng. Data,
(2) Carbon Dioxide; CO_2; [124-38-9]	*48*, 71-74 (2003)
Variables:	Prepared By:
T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A3 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308	122	12.2	0.51
308	182	18.2	0.34
308	243	24.3	0.89
308	304	30.4	1.08
308	355	35.5	1.18
318	122	12.2	---
318	182	18.2	1.30
318	243	24.3	1.81
318	304	30.4	2.20
318	355	35.5	2.10
328	122	12.2	0.54
328	182	18.2	1.56
328	243	24.3	2.33
328	304	30.4	3.02
328	355	35.5	3.66
338	122	12.2	---
338	182	18.2	1.14
338	243	24.3	2.11
338	304	30.4	3.16
338	355	35.5	3.79
348	122	12.2	---
348	182	18.2	0.46
348	243	24.3	2.52
348	304	30.4	3.92
348	355	35.5	4.60

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrofluorimeter

Source and Purity of Materials:
1. A3 synthesized, purity 99.9%
2. carbon dioxide source Sabalan Gas Co.,Iran; purity 99.99%

Estimated Errors:
Temperature: unstated
Pressure: unstated
Solibility: ±5%

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.115 Solubility of A3 in Carbon Dioxide.

Components:	Original Measurements:
(1) 1-amino-2.4-di-methyl-9,10-anthraquinone A4; $C_{16}H_{13}NO_2$; [3225-89-6] (2) Carbon Dioxide; CO_2; [124-38-9]	Mojtaba Shamsipur, Ali Reza Karami, Yadollah Yamini, Hashem Sharghi, J. Chem. Eng. Data, *48*, 71-74 (2003)
Variables: T/K = 308, 328, 338, 348, 358 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of A4 (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^5$
308	122	12.2	2.07
308	182	18.2	3.62
308	243	24.3	5.62
308	304	30.4	6.55
308	355	35.5	7.02
328	122	12.2	0.02
328	182	18.2	3.73
328	243	24.3	6.07
328	304	30.4	7.77
328	355	35.5	9.74
338	122	12.2	0.02
338	182	18.2	3.59
338	243	24.3	7.25
338	304	30.4	9.59
338	355	35.5	11.97
348	122	12.2	0.02
348	182	18.2	0.03
348	243	24.3	10.19
348	304	30.4	20.59
348	355	35.5	25.98
358	122	12.2	0.03
358	182	18.2	2.68
358	243	24.3	15.12
358	304	30.4	22.35
358	355	35.5	24.70

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrofluorimeter.

Source and Purity of Materials:
1. A4 synthesized, purity 99.9%
2. carbon dioxide source Sabalan Gas Co.,Iran; purity 99.99%

Estimated Errors:
Temperature: unstated
Pressure: unstated
Solibility: ±5%

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.116 Solubility of A4 in Carbon Dioxide.

Components:	Original Measurements:
(1) methylparaben; $C_8H_8O_3$; [99-76-3]	Mehdi Asghari-Khiavi, Yadollah Yamini, J. Chem. Eng. Data, *48*, 61-65 (2003)
(2) Carbon Dioxide; CO_2; [124-38-9]	
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of methylparaben (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	122	12.2	1.13
308	182	18.2	1.60
308	243	24.3	1.94
308	304	30.4	2.33
308	355	35.5	2.56
318	122	12.2	1.13
318	182	18.2	2.00
318	243	24.3	2.62
318	304	30.4	3.45
318	355	35.5	3.96
328	122	12.2	1.08
328	182	18.2	2.37
328	243	24.3	3.51
328	304	30.4	4.22
328	355	35.5	4.93
338	122	12.2	1.18
338	182	18.2	2.95
338	243	24.3	4.51
338	304	30.4	6.41
338	355	35.5	7.19
348	122	12.2	1.35
348	182	18.2	3.45
348	243	24.3	6.02
348	304	30.4	9.60
348	355	35.5	12.13

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrophotometer.

Source and Purity of Materials:
1. methylparaben source Food and Drug Control Lab, Iran; purity 99.5%
2. Carbon dioxide source Sabalan; purity 99.99%

Estimated Errors:
Temperature: $\pm 1K$
Pressure: $\pm 0.5bar$
Solubility: $\pm 3\%$

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)

Figure A.117 Solubility of Methylparaben in Carbon Dioxide.

Components:	Original Measurements:
(1) bisacodyl; $C_{22}H_{19}NO_4$; [603-50-9] (2) Carbon Dioxide; CO_2; [124-38-9]	Mehdi Asghari-Khiavi, Yadollah Yamini, J. Chem. Eng. Data, *48*, 61-65 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of bisacodyl (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	122	12.2	0.36
308	182	18.2	0.71
308	243	24.3	1.00
308	304	30.4	1.20
308	355	35.5	1.39
318	122	12.2	0.27
318	182	18.2	0.71
318	243	24.3	1.13
318	304	30.4	1.58
318	355	35.5	2.03
328	122	12.2	0.22
328	182	18.2	0.63
328	243	24.3	1.45
328	304	30.4	2.27
328	355	35.5	2.97
338	122	12.2	0.09
338	182	18.2	0.58
338	243	24.3	1.79
338	304	30.4	3.09
338	355	35.5	4.43
348	122	12.2	---
348	182	18.2	0.54
348	243	24.3	2.05
348	304	30.4	4.06
348	355	35.5	5.83

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere. The solid solutes were mixed well with glass beads and packed into the extraction vessel. supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility was measured by spectrometer.

Source and Purity of Materials:
1. bisacodyl source Food and Drug Control Lab, Iran; purity 99.5%
2. Carbon dioxide source Sabalan; purity 99.99%

Estimated Errors:
Temperature: $\pm 1K$
Pressure: $\pm 0.5bar$
Solubility: $\pm 3\%$

References:
Figure A.118 Solubility of Bisacodyl in Carbon Dioxide.

Components: (1) methimazole; $C_4H_6N_2S$; [60-56-0] (2) Carbon Dioxide; CO_2; [124-38-9]	Original Measurements: Mehdi Asghari-Khiavi, Yadollah Yamini, J. Chem. Eng. Data, *48*, 61-65 (2003)
Variables: T/K = 308, 318, 328, 338, 348 P/MPa = 12.2 - 35.5	Prepared By: Dana E. Knox; Syed A. Abdullah

Experimental Data
Solubility of methimazole (1) in Carbon Dioxide (2)

T/K	P/bar	P/MPa	$x_1 \times 10^4$
308	122	12.2	0.22
308	182	18.2	0.29
308	243	24.3	0.45
308	304	30.4	0.53
308	355	35.5	0.60
318	122	12.2	0.21
318	182	18.2	0.32
318	243	24.3	0.55
318	304	30.4	0.70
318	355	35.5	0.83
328	122	12.2	0.19
328	182	18.2	0.35
328	243	24.3	0.64
328	304	30.4	0.92
328	355	35.5	1.18
338	122	12.2	0.15
338	182	18.2	0.35
338	243	24.3	0.77
338	304	30.4	1.16
338	355	35.5	1.49
348	122	12.2	---
348	182	18.2	0.30
348	243	24.3	0.85
348	304	30.4	1.38
348	355	35.5	1.90

Auxiliary Information

A suprex MPS/225 integrated SFE/SFC system equipped with a modified static system for the solubility determination in the SFE mode was used. A detailed description of the apparatus and operating procedure is given elsewhere[1]. The solid solutes were mixed well with glass beads and packed into the extraction vessel. Supercritical carbon dioxide was pressurized and passed into the extraction vessel. After equilibrium was reached saturated supercritical carbon dioxide was pressurized and passed into the injection loop. Next the loop was depressurized into the collection vial. The solubility were calculated by fluorescence measurements using a spectrophotometer.

Source and Purity of Materials:
1. methimazole source Food and Drug Control Lab, Iran; purity 99.5%
2. Carbon dioxide source Sabalan; purity 99.99%

Estimated Errors:
Temperature: $\pm 1K$
Pressure: ± 0.5bar
Solibility: $\pm 3\%$

References:
1. Fathi,M.R.; Yamini,Y.; Sharghi,H.; Shamsipur,M, J. Chem. Eng. Data, *43*, 400-402 (1998)
Figure A.119 Solubility of Methmazole in Carbon Dioxide.

Components:	Original Measurements:
(1) Eflucimibe; $C_{29}H_{43}NO_2S$; [202340-45-2]	M.Sauceau,J.-J.Letourneau,B.Freiss,D.Richon,
(2) 99.95%Carbon Dioxide; CO_2; [124-38-9], with .05% ethanol; C_2H_5OH; [64-17-5]	J.Fages, J. of Supercritical Fluids, *31*, 133-140 (2005)

Variables:	Prepared By:
T/K = 318.15 P/MPa = 9.80 - 30.36	Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of eflucimibe(1) in 99.95% carbon dioxide with .05%ethanol(2)

T/K	P/MPa	$x_1 \times 10^7$
318.15	9.80	11.09
318.15	9.95	12.03
318.15	12.25	20.43
318.15	12.27	20.87
318.15	14.80	29.31
318.15	14.90	30.98
318.15	17.45	48.37
318.15	22.40	72.81
318.15	27.17	88.20
318.15	26.69	90.82
318.15	30.36	90.75

Auxiliary Information

Equilibrium solubility data have been obtained by using a continuous flow apparatus described previously[1].The liquid solvents are compressed by two high pressure syringe pumps working at constant flow rates depending on the desired composition. The homogeneous high pressure liquid then passes through a heater used to rapidly heat the solvent to temperatures above its critical temperature. The supercritical fluid then enters an oven in which the solubility cell is located. The total volume of gaseous carbon-dioxide is measured by means of a gas volumeter and the concentration of the solid in the recovery liquid phase is obtained by analysis. The solubility of the solid in supercritical fluid can then be calculated from these two quantities and the total volume of the recovery liquid.

Source and Purity of Materials:
1. Eflucimibe source Institut de Recherche Pierre Fabre(IRPF), purity 99%
2. Carbon dioxide, ethanol source unknown, purity known.

Estimated Errors:
Temperature: $\pm$0.05K
Pressure: $\pm$0.7%
Solubility: $\pm$5%

References:
1.. M.Sauceau, J.Fages, J.J Letourneau, D.Richon, Ind. Eng. Chem. Res., *39*, 4609-4614 (2000).

Figure A.120 Solubility of Eflucimibe in Carbon Dioxide with Ethanol.

Components:	Original Measurements:
(1) Eflucimibe; $C_{29}H_{43}NO_2S$; [202340-45-2] (2) 99.98%Carbon Dioxide; CO_2; [124-38-9], with .02%Dimethylsulphoxide; DMSO; C_2H_6OS; [67-68-5]	M.Sauceau, J.-J.Letourneau, B.Freiss, D.Richon, J.Fages, J. of Supercritical Fluids, *31*, 133-140 (2005)

Variables:	Prepared By:
T/K = 318.15 P/MPa = 12.25 - 29.11	Dana E. Knox, Syed A. Abdullah

Experimental Data
Solubility of eflucimibe(1) in 99.98% carbon dioxide with .02%DMSO(2)

T/K	P/MPa	$x_1 \times 10^7$
318.15	12.25	32.84
318.15	14.97	72.25
318.15	17.17	113.84
318.15	20.10	152.34
318.15	26.76	282.59
318.15	29.11	365.37

Auxiliary Information

Equilibrium solubility data have been obtained by using a continuous flow apparatus described previously[1].The liquid solvents are compressed by two high pressure syringe pumps working at constant flow rates depending on the desired composition. The homogeneous high pressure liquid then passes through a heater used to rapidly heat the solvent to temperatures above its critical temperature. The supercritical fluid then enters an oven in which the solubility cell is located. The total volume of gaseous carbon-dioxide is measured by means of a gas volumeter and the concentration of the solid in the recovery liquid phase is obtained by analysis. The solubility of the solid in supercritical fluid can then be calculated from these two quantities and the total volume of the recovery liquid.

Source and Purity of Materials:
1. Eflucimibe source Institut de Recherche Pierre Fabre(IRPF), purity 99%
2. Carbon dioxide, DMSO source unknown, purity known.

Estimated Errors:
Temperature: ±0.05K
Pressure: ±0.7%
Solubility: ±5%

References:
1. .M.Sauceau, J.Fages,J. J Letourneau, D.Richon, Ind. Eng. Chem. Res.,*39*, 4609-4614 (2000)

Figure A.121 Solubility of Eflucimibe in Carbon Dioxide with DMSO.

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