1254 *J. Chem. Eng. Data* **1999,** *44,* 1254−1258

Solubility Determination of TNT and Wax and Their Fractionation from an Explosive Material Using a Supercritical Fluid

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The solubilities of 2,4,6-trinitrotoluene (TNT) and wax have been measured in supercritical carbon dioxide (CO_2) at three pressures and temperatures under static conditions. The concentrations of each component were determined off-line via ultraviolet (TNT) and evaporative light scattering (wax) detection. The solubility of TNT was an order of magnitude higher than that of wax. Gas chromatographic assay of the wax extract revealed that only the lower molecular weight components dissolved. Fractionation of the TNT and wax from an explosive material referred to as Composition B was attempted by making incremental increases in CO_2 density. Composition B contains 59.5% cyclotrimethylene triamine (RDX), 39.5% TNT, and 1% wax. While TNT and wax could be easily isolated from nitramine RDX, attempts to separate TNT from wax were not totally successful. More specifically, the initial fractions contained the lower molecular weight wax components in addition to major amounts of TNT. Since the percentage of TNT was approximately 50 times the amount of wax, later fractions were 100% TNT although most of the TNT was removed at the lower densities.

Introduction

Fractionation of multicomponent materials by chemical class and/or molecular weight, thereby taking advantage of differences in extractability and/or solubility in supercritical fluids (SFs), is technologically attractive although not often demonstrated to date. SFs are ideal in this regard, since solvating power can be mechanically adjusted (Via et al., 1993). To aid in the fractionation process, information regarding the solubility of the respective components in SFs under various conditions is desirable. Two methods of fractionation appear to be most popular. One method which has been used on the industrial scale involves dissolution of the entire sample in SF at high pressure followed by careful fractional precipitation of the components as incremental decreases in pressure at constant temperature are carried out. The other method of fractionation is also isothermal, but the procedure initiates at low pressure. Higher molecular weight components (i.e. lower vapor pressure) are progressively solubilized and isolated by incremental increases in solvating power (i.e. SF density). The latter method appears to be the more common one in laboratories.

The most logical application of SF fractionation at the moment seems to be in the area of polymeric materials. Pratt et al. (1993) used supercritical propane, propylene, butane, 1-butene, and chlorodifluoromethane to fractionate poly(ethylene-*co*-methyl acrylate) polymers with 30, 40, 60, and 70 wt % acrylate in the backbone. Fractionation was performed at constant temperature, while pressure was increased at a constant interval. Results showed that the degree of fractionation of the polymer using propane and propylene was highly dependent on the structure of the polymer and the polymer backbone. On the other hand, fractionation of the polymer using chlorodifluoromethane was only dependent on the polymer molecular weight but not its structure. Later, these workers fractionated a poly(ethylene-*co*-acrylic acid) copolymer of 3.1 and 3.9 mol % acrylic acid using supercritical propane, butane, and dimethyl ether. They were able to fractionate the polymer first by utilizing the poor-quality solvent, propane or butane, to solubilize the nonpolar ethylene-rich oligomers and then using dimethyl ether, a stronger solvent, to dissolve the acrylic acid-rich oligomers (Pratt and McHugh, 1996). Cansell et al. (1997) used a similar method to fractionate poly(ethylene oxide) at constant temperature (150 °C) with a pressure range of 8 to 35 MPa using supercritical 1-chloro-1,1-difluoroethane. Polymers with a very narrow molecular weight distribution were obtained.

Eckert et al. (1992) used both pure and modified supercritical fluids not only to remove unreacted dodecanol from nonionic surfactant poly(oxyethlene)dodecyl ethers but also to fractionate the polymer on the basis of chain length. They used both pure and methanol-modified carbon dioxide, propane, and ammonia as supercritical solvents. At high pressures polymer solubility increased; however, selectivity for fractionation decreased.

Fractionation of asphalt materials into 10 fractions by supercritical cyclohexane and pentane was reported by Jemison et al. (1995). Fractionation of fat from different food matrixes has been performed. Chao et al. (1993) used pure CO_2 to extract and fractionate cholesterol from beef tallow. Results showed that at low pressure a high concentration of cholesterol and a low concentration of lipid could be extracted, while at a higher pressure of CO_2 a lower concentration of cholesterol and a higher concentration of lipid were extracted. Bhaskar et al. (1993) have used a continuous pilot-scale supercritical fluid extraction system to separate and fractionate up to 400 $g{\cdot}h^{-1}$ of anhydrous milk fat. The extracted fat was separated into five different fractions. Fatty acid analysis of each fraction showed that short chain (C_4-C_8) and medium chain ($C_{10}-C_{12}$) fatty acids increased gradually from fraction no. 1 to fraction no. 5, while the trend was opposite for the long chain and unsaturated fatty acids ($C_{14}-C_{18}$). In a similar study, Yoon et al. (1993) used a flow through extraction reactor to study supercritical fluid fractionation of anhy-

10.1021/je9901280 CCC: $18.00

drous milk fat. The milk fat was mixed with glass beads and then was packed into the reactor. A large layer of glass beads was placed on top of the packed bed, whose diameter and height were 5 cm and 20 cm, respectively. The system was then operated under slow continuous flow of supercritical fluid CO_2 (1 to 6 $L\cdot min^{-1}$ at 1 atm and 25 °C). At the lowest portions of the bed, where CO_2 was introduced into the vessel, the triglycerides were totally depleted, while the fat concentration increased at higher points in the packed bed.

In addition to polymeric materials, supercritical fluid fractionation has been applied to energetic materials. The goal here has been to reclaim various explosive and propellant ingredients. This is important, since conventional demilitarization techniques such as open detonation are being phased out due to concerns regarding their impact on the environment. In addition, conventional techniques are often wasteful, thereby resulting in the destruction of potentially valuable energetic materials. Supercritical fluids have also been used for processing and characterization of explosives and propellants. For example, the U.S. Army (Krukonis et al.), Air Force (Krukonis and Gallagher), and Navy (U.S. Department of Defense) have funded supercritical fluid processing technology research for a number of years. Analysis of crude, purified, and synthetic candelilla wax (an ingredient of propellants) using supercritical fluids has also been reported (Ashraf-Khorassani and Taylor, 1980).

The objective of our study was to perform a feasibility study to extract and fractionate using supercritical fluid technology 2,4,6-trinitrotoluene (TNT), wax, and cyclotrimethylene triamine (RDX) from Composition B by selectively applying different pressures and temperatures. For this purpose, it was first necessary to obtain an accurate estimate of the solubility of TNT and wax under supercritical conditions. Preliminary work by Morris (1997) has suggested the feasibility of removing TNT and wax from RDX, but no detailed study has been reported. Morris reported that RDX was recovered from Composition B with a purity in excess of 99% using neat CO_2. An attempt to separate the TNT from the wax, however, was not reported.

Experimental Section

TNT and wax samples were provided by the Naval Surface Warfare Center and were used as received. Figure 1 shows the schematic of the in-house constructed apparatus that was used to measure the solubility of TNT and wax in supercritical carbon dioxide (CO_2) (Ashraf-Khorassani et al., 1997; Ashraf-Khorassani and Taylor, 1999). Nine experimental conditions were studied using three pressures (13.8, 25.0, and 37.9 MPa) and three temperatures (35, 50, and 70 °C). For each solubility measurement, a 1.0 mL supercritical fluid extraction vessel was filled with approximately 0.75 g of either TNT or wax. Air was initially removed from the system by passing CO_2 gas through the vessel. The system was then pressurized to the desired pressure setting. Heating of both the vessel and the transfer lines after pressurization of the system was next accomplished. The three-way valve was next closed, and the recirculating pump was activated (Figure 1A). The function of the recirculating pump was to ensure complete mixing of the fluid and the analyte of interest. After 30 min of equilibration time, the four-port−two-position valve with a 0.5 μL internal sample loop attached was rotated to allow 0.5 μL of the supercritical solution to be transferred from the loop to the liquid chromatographic (LC) system (Figure 1B). A liquid chromatographic solvent composed of 50/50 CH_3OH/CH_2Cl_2 then transferred the analyte, which had been dissolved in supercritical CO_2, from the 0.5 μL loop through a second loop onto a packed C_8 column (Hypersil, 150 mm × 2.1 mm, dp = 5 μm). An ultraviolet detector operating at 254 nm was used for TNT chromatographic detection, while an evaporative light scattering detector (ELSD, Varex, Deerfield, IL) was employed in a flow injection mode (e.g. no column) for wax solubility determination. By employing a second valve (six-port−two-position), a stream of air could be passed through the 0.5 μL sample loop to ensure removal of the liquid chromatographic mobile phase, thus avoiding modification of the supercritical fluid composition during the subsequent extraction (Figure 1C). HPLC grade methanol and methylene chloride were purchased from EM Science (Gibbstown, NJ). The four-port−two-position valve was then rotated back to the load position, and the procedure was repeated (Figure 1D). Five replicate determinations were made on each saturated supercritical fluid solution of analyte to ensure reproducibility of the system. SFE/SFC grade CO_2 with a 2000 psi helium head was obtained from Air Products and Chemicals Inc. (Allentown, PA) and used as received.

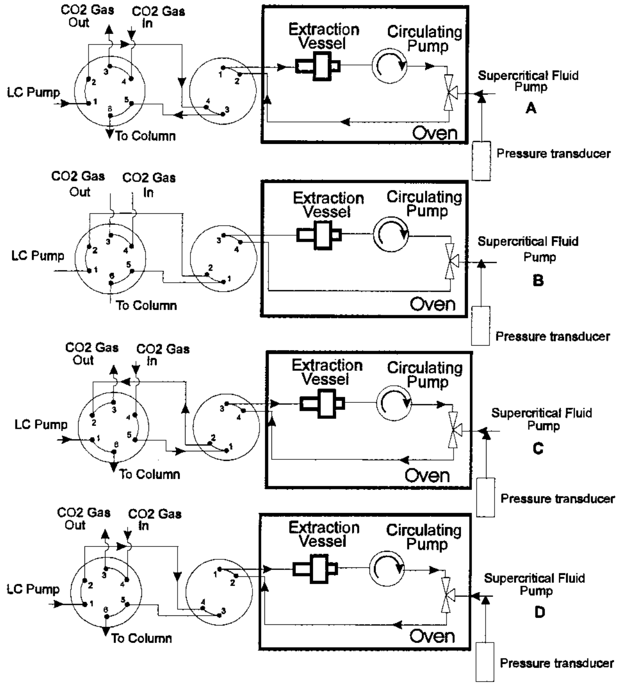

Figure 1. Schematic diagram of solubility measurement device in various modes.

All fractionations of the explosive sample were performed using an Isco-Suprex (Lincoln, NE) prepmaster. Approximately 200 mg of explosive (e.g. Composition B) which had been manually ground to a fine powder was intimately mixed with 3 g of Ottawa sand and placed in a 5 mL stainless steel extraction vessel. The sample contained approximately 1% wax, 39.5% TNT, and 59.5% nitramine RDX, which was known to have negligible solubility in supercritical CO_2. Fractionation of the sample components via sequential SFE was attempted at four pressures (15.1, 25.3, 35.4, and 45.5 MPa) and two temperatures (35 and 70 °C). After charging the vessel and heating it to a selected temperature, the initial extraction was performed at 15.1 MPa CO_2 using 25 g of CO_2 at a liquid flow rate of 2 $mL\cdot min^{-1}$. Extracted components were collected after decompression in a vial which contained 5 mL of CH_2Cl_2/CH_3OH (80/20). Extraction was resumed at 25.3 MPa with

Table 1. Solubility of TNT and Wax in Supercritical CO_2 at Different Pressures and Temperatures

	solubility $mg \cdot mL^{-1}$ (%RSD)[a]		
temp	13.8 MPa	25.3 MPa	37.9 MPa
35 °C, TNT	4.27 (2.4)	9.97 (0.9)	14.43 (0.4)
50 °C, TNT	2.84 (3.0)	11.55 (5.5)	16.51 (2.6)
70 °C, TNT	5.93 (6.6)	18.92 (0.5)	25.64 (2.5)
35 °C, wax	0.27 (3.9)	0.36 (5.0)	0.60 (4.0)
55 °C, wax	0.24 (4.7)	0.51 (3.8)	1.09 (2.6)
70 °C, wax	0.21 (3.9)	0.58 (7.3)	1.37 (2.7)

[a] %RSD for triplicate measurements.

Table 2. Measured Solubilities in Supercritical CO_2 for Other Propellant-Related Materials

compound	solubility $mg \cdot mL^{-1}$	temp °C	pressure/ MPa	density/ $g \cdot mL^{-1}$
2-nitrodiphenylamine	1.97	40	16.0	0.82
	4.07	60	26.3	0.80
diphenylamine	16.00	40	16.0	0.82
	19.26	60	26.3	0.80
ethyl centralite	25.65	31	15.2	0.86
	290.72	60	30.3	0.83

a new vial containing fresh solvent to collect new analyte. The same CO_2 mass and liquid CO_2 flow rate were employed. In analogous fashion, fractionations were carried out sequentially at 35.4 and 45.5 MPa. A second temperature was then selected, and fractionation of a fresh sample in a fashion similar to that described above was conducted.

Extract solutions were analyzed with a Hewlett-Packard (Wilmington, DE) 5890 Series II GC/FID. Separations were obtained with a DB-1 column (530 μm × 5 m, d_f 0.25 μm). The temperature program started at 50 °C for 3 min, and the temperature was then increased to 285 °C at $10 °C \cdot min^{-1}$. The final temperature was held for 15 min.

Results and Discussion

The study was two-fold: (a) solubility determination of wax and TNT separately in supercritical CO_2 at various pressures and temperatures and (b) attempted fractionation of wax and TNT via SFE with CO_2 from Composition B. As stated previously, the explosive formulation contained approximately 59.5% RDX, which is known to not be soluble in supercritical CO_2 (Morris, 1998).

Information on solubilities in supercritical fluids is perhaps the most important thermophysical property that must be determined and modeled in order to efficiently design large scale extraction procedures based on supercritical fluids. Experimental data on the solubility of energetic materials in supercritical fluids are limited due to the experimental difficulty and inherent safety considerations associated with high-pressure measurements. However, if a limited amount of highly reliable experimental solubility data can be obtained and fit to a suitable equation of state, solubility predictions for these energetic materials at conditions for which there are no experimental data become feasible.

To measure the solubility of wax and TNT, an external calibration curve was constructed. It should be noted that while TNT was a single component, the wax was basically an homologous series. The measured wax solubility therefore will be an average solubility of all wax components, wherein, the low-molecular-weight component may be truly miscible but the higher molecular weight oligomers may have marginal solubility. Using the raw chromatographic ultraviolet peak areas (TNT) and the flow injection light scattering responses (wax) in conjunction with the appropriate calibration data, the solubilities of the wax and TNT in supercritical CO_2 at three pressures and two temperatures were determined (Table 1). The solubility ($mg \cdot mL^{-1}$) of TNT in supercritical CO_2 was 10 to 50 times higher than the solubility of wax in the same medium. The solubility dramatically increased with increasing CO_2 pressure for both the TNT and wax to about the same extent. The increase in solubility was more pronounced with an increase in pressure in both cases at the highest temperature. This behavior can be readily explained by the increase in CO_2 density with an increase in pressure. Classical solubility behavior was observed at fixed pressure and varying temperature. At 13.8 MPa, the solubility of TNT was lowest at 50 °C rather than at 35 °C or 70 °C. These data suggest that not only a CO_2 density effect but also a temperature effect are important factors. The decrease in density going from 35 to 50 °C at 13.8 MPa reflects a significant loss in density for which the increase in temperature is not able to compensate. At 25.3 and 37.9 MPa, the decrease in CO_2 density with increase in temperature is not nearly as great. Therefore, TNT solubility steadily increased with temperature. Although not quite as apparent, the trend with wax solubility was similar at varying pressures and temperatures. In other words, solubility was a function of CO_2 density and analyte vapor pressure. There is one slight difference in wax solubility with temperature at 13.8 MPa in that solubility decreases (or remains constant) as temperature increases from 35 °C to 70 °C. This behavior probably reflects the lower vapor pressure of the wax components. Regardless of the supercritical conditions, the TNT solubility was at least an order of magnitude higher than the wax solubility.

Our solubility data are somewhat at odds with a previous study by Teipel et al. (1998) concerning TNT−CO_2 solubility. These workers used a variable volume view cell. They based their solubility measurement solely on the temperature and pressure at which the TNT dissolved during heat up of the apparatus. No stirring was indicated. In contrast to our data, these workers reported that the equilibrium concentration of TNT dissolved in compressed CO_2 increased linearly up to a pressure of 24.4 MPa and was independent of temperature. It appears conceivable that equilibrium conditions may not have been achieved in this study.

Prior to this work, we have measured the solubility of other propellant ingredients in supercritical CO_2. Even though these components do not appear in the material that we have attempted to fractionate, we report their solubility data here for comparison in Table 2.

The second part of our study concerned the feasibility of using supercritical CO_2 to fractionate sequentially TNT and wax from an explosive materials (Comp B). The actual Comp B in the study was manufactured during World War II. Previously, it had been shown that both TNT and wax could be separated from the nitramine RDX with supercritical CO_2 (Morris, 1997). On the basis of measured solubility data, one would predict removal of TNT first at low density followed by removal of wax components at higher density. RDX naturally would not be extracted with CO_2 regardless of the conditions. Preliminary to this study, a gas chromatographic (GC) separation of a standard mixture of wax and TNT dissolved in (80/20) CH_2Cl_2/CH_3OH was developed in order to follow the fate of the fractionation. Figure 2 describes the GC trace with flame ionization detection. TNT eluted at 8.06 min, while the wax components eluted over the range 10 to 50 min. The peak appearing at approximately 7 min was not identified. The wax appears to be a mixture of two homologous series in a

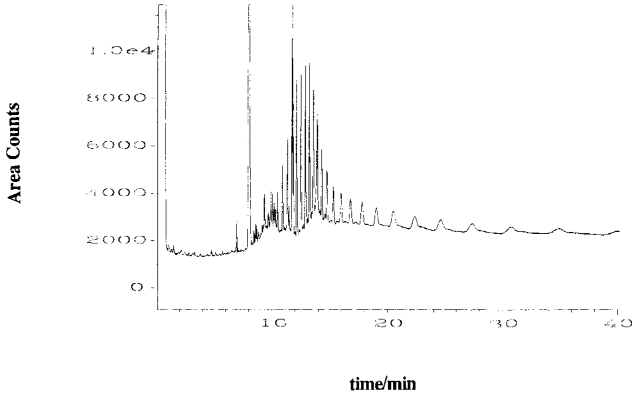

Figure 2. GC/FID of standard mixture of wax and TNT. DB-1 column (530 μm × 5 m, d_f 0.25 μm). Oven temperature started at 50 °C for 3 min and was then heated to 285 °C at a rate of 10 °C·min^{-1} and then held for 15 min at 285 °C.

ratio of about 1 to 3. No doubt all components of the wax did not exhibit solubility in supercritical CO_2 at the tested conditions.

Fractionation of TNT and wax from Composition B was first attempted with 25 g of CO_2 at 70 °C and 15.2 MPa. Decompression of the CO_2 with extracted analytes was into 5 mL of (80/20) CH_2Cl_2/CH_3OH. The GC/FID trace of the resulting extract solution revealed that a large amount of TNT was extracted along with a small quantity of wax (Figure 3A). Surprisingly, all evidence for one of the two proposed wax homologous series had disappeared. No doubt the higher homologue, while soluble in a mixture of CH_2Cl_2/CH_3OH, was not soluble in supercritical CO_2 at this density. While some highly volatile unknown components eluted just after TNT, only one homologous series was apparent in the extract. The wax used for the so-called "standard" was not the same as the wax in the Comp B sample. Re-extraction of the raffeinate with another 25 g of CO_2 at 25.3 MPa and 70 °C revealed that the initial extraction did not completely remove all of the TNT. Figure 3B shows the resulting GC/FID trace of the second extract solution. Under these higher density conditions, slightly higher molecular weight wax components were extracted, as evidenced by peaks appearing out to 18 min. Results for the extraction of the same raffeinate at higher pressure (35.4 MPa) showed the total absence of wax components; however, TNT continued to appear in the liquid solvent trap (Figure 3C). This may not be too surprising, since TNT was approximately 40% of Comp B. Extraction at 45.5 MPa gave similar results to those at 35.4 MPa, such that both fractions 3 and 4 were void of wax components. Therefore, one concludes that fractions highly enriched with TNT can be readily obtained via supercritical fluid fractionation. Unfortunately, more than 95% of the extractable TNT was removed in the first two fractions at 15.2 and 25.3 MPa.

Next, fractionation of wax, TNT, and RDX in Comp B was performed at 35 °C and various pressures. Again, most of the low-molecular-weight hydrocarbons were extracted at 15.2 and 25.3 MPa, while at 35.4 and 45.5 MPa only TNT residue was observed in the GC/FID trace of the respective extracts. A comparison of the raw area trace of TNT in each extract suggested that the rate of TNT extraction is measureably slower at 35 °C than at 70 °C, although the density is higher at 35 °C than at 70 °C. It is important to note here that most of the high-molecular-weight hydrocarbons present in our wax standard chromatogram (t_R > 20 min) seemingly were never extracted from the Comp B matrix. Either the wax in the standard does not match the wax in Comp B, or the higher molecular weight components do not dissolve in high-density, pure, supercritical CO_2. Unfortunately, it was not possible to obtain the same wax used in Comp B.

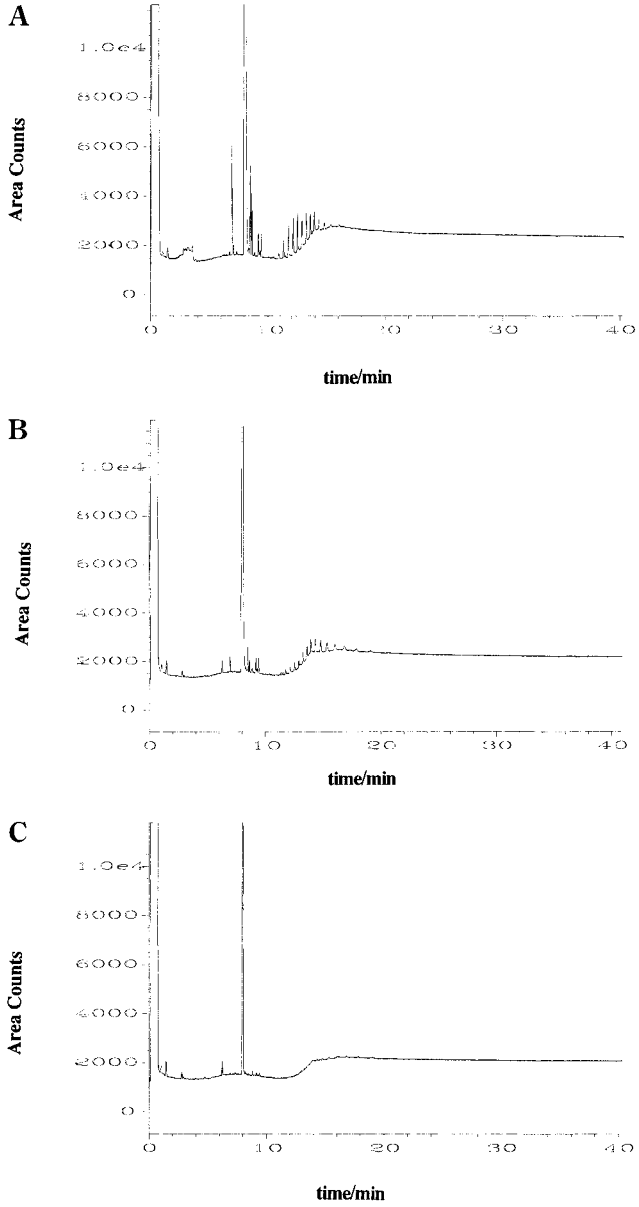

Figure 3. GC/FID trace of explosive material successively fractionated using supercritical fluid under different extraction conditions: (A) 15.2 MPa, 70 °C, 25 g of CO_2; (B) 25.3 MPa, 70 °C, 25 g of CO_2; (C) 35.4 MPa, 70 °C, 25 g of CO_2. GC conditions are shown in the caption for Figure 2.

In summary, we have shown that TNT and wax can be semiquantitatively removed from RDX in Composition B explosive with 100% supercritical CO_2. Some high-molecular-weight wax components, however, may remain with the RDX. Efforts to isolate the much more soluble TNT away from the wax via supercritical fluid fractionation were not totally successful, since even at low CO_2 density some wax components were extracted along with lots of TNT. While the wax exhibited overall low solubility, it seems likely that there were some components of the wax that exhibited considerably higher solubility, which may account for our failure to fractionate completely.

Acknowledgment

The technical assistance afforded by Jeffrey Morris and George Nauflett is greatly appreciated. We also wish to thank both Isco-Suprex, Inc., for loan of their Prepmaster SFE instrument and Air Products and Chemicals Inc. for providing SFE/SFC grade CO_2.

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Received for review May 7, 1999. Accepted July 20, 1999. This research was supported in part by the U.S. Department of Defense, through the Strategic Environmental Research and Development Program (SERDP).

JE9901280