Characteristics and Performance of Supercritical Fluid Extraction (SFE) in the Analysis of Petroleum Hydrocarbons in Soils and Sludges

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PURPOSE AND MAJOR FINDINGS OF THE RESEARCH

This project was designed to evaluate and improve supercritical fluid extraction (SFE) methods and instrumentation for the analytical-scale extraction from soils and sludges of petroleum hydrocarbons ranging from benzene, toluene, ethylbenzene, and xylene (BTEX) to polynuclear aromatic hydrocarbons (PAHs) and heavy crudes. The primary goals of this two-year study were to:

- Determine which types of petroleum industry environmental samples and wastes can be extracted using SFE, by:

  Quantitative comparisons to standard (Soxhlet) methods;

  Qualitative descriptions on SFE performance (primarily restrictor plugging);

- Evaluate when presently-available SFE methods are viable alternatives to conventional liquid solvent extraction;
- Determine development needs for SFE extraction and collection conditions, and develop extraction conditions for a wide range of petroleum-based hydrocarbons and PAHs. This effort included:

  Development of two SFE methods that can yield quantitative extraction and recovery of gasoline- to diesel-range organics from soils, allowing BTEX and total petroleum hydrocarbons (TPH) to be determined using a single extraction method;

  Development of quantitative extraction conditions for PAHs and heavier hydrocarbons including heavy crudes and heavy resids;

- Determine hardware development needs based on problems encountered with real-world samples, and identify commercially available instrumentation to meet those needs.

Commercially available instrumentation and standard SFE approaches, such as the proposed Environmental Protection Agency (EPA) method for TPH, were used. Comparisons were made to standard liquid solvent

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extraction methods. Good comparability for gasoline- and diesel-range TPH was demonstrated between conventional Soxhlet extraction and SFE at conditions similar to the proposed EPA SFE method, using both infrared (IR) and gas chromatograph/flame ionization detector (GC/FID) quantification of extracted hydrocarbons.

For the more volatile (e.g., BTEX and light alkane) components, SFE yielded lower extraction losses and higher efficiencies than Soxhlet extraction. Compared to SFE and conventional Soxhlet extractions performed in the laboratory, field SFE gave good agreement for gasoline- and diesel-range TPH with IR determination. Commercial instruments using both solvent trapping (ISCO®) and sorbent trapping (Hewlett-Packard®) yielded quantitative recoveries (> 90%) of BTEX and gasoline- and diesel-range alkanes as volatile as C_6 (for sorbent) and C_7 (for solvent trapping), demonstrating that BTEX and TPH determinations can be performed with a single extraction.

Also developed was an on-line supercritical fluid extraction/gas chromatography (SFE/GC) method for gasoline- and diesel-range organics that allows species as volatile as n-butane to be extracted and collected at approximately 100% efficiency. In addition to allowing quantitative determinations of very volatile species, the SFE/GC method allows sensitive detection limits (e.g., < 10 ppb for benzene) for samples as small as 1 gram. However, the on-line method is more difficult to perform than the standard SFE methods, and requires modifications to existing SFE and GC instruments.

SFE methods were also developed utilizing high-temperature SFE and the addition of organic modifiers for components that were not efficiently extracted using standard SFE conditions (e.g., heavy hydrocarbons and PAHs). With the combined use of either high SFE temperatures (e.g., 150°C) and/or organic modifiers, the recoveries of heavy hydrocarbons (e.g., heavy resids > C_{40}) were higher than those achieved using Freon-

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113® Soxhlet extraction as determined by IR. In general, contaminated soils could be extracted as received without drying or other sample preparation, although soils and waste sludges contaminated with high levels of heavy hydrocarbons often caused plugging of some types of SFE flow restrictors. In nearly every case, heating the restrictor and mixing such samples with dispersants and/or drying agents eliminated restrictor plugging.

Practical advantages of SFE included typical extraction times of 30-40 minutes, compared to 4 hours or greater for Soxhlet extraction; and typical total solvent use of less than 10 mL, compared to 150 mL for Soxhlet extraction. In nearly all of the samples studied, SFE yielded efficiencies similar to or higher than Soxhlet extraction; however, elevated temperature and/or organic modifiers were often needed to obtain high extraction efficiencies for organics beyond the gasoline- and diesel-range. It should be noted that SFE instruments continue to evolve, especially in the areas of improved restrictor and collection system designs, as well as systems offering automated extraction of up to 20 samples without operator intervention. Such developments should further increase reliability and speed of SFE for petroleum hydrocarbon extractions from soils and sludges.

GASOLINE-RANGE TPH, DIESEL-RANGE TPH, AND BTEX BY ON-LINE SFE/GC

On-line SFE/GC methodology was developed to allow extraction and analysis of organics as volatile as n-butane from solids at part-per-billion (ppb) detection limits (Burford *et al.*, 1994a). A solid-based calibration standard, consisting of several n-alkanes and aromatic hydrocarbons spiked onto Tenax-TA®, was successfully used to optimize the chromatographic parameters for coupled SFE/GC. A simple and reliable split SFE/GC system was developed utilizing a septumless injector installed on a split/split-

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less injection port. The high gas flow rate generated inside the injection port during the SFE step was accommodated for by using the correct split ratio, so that high (1 ml/min liquid CO_2) SFE flow rates could be used. The use of thick-film (5 μm film thickness) columns and cryogenic trapping temperatures in the GC oven as low as -50°C allowed efficient trapping of species as volatile as n-butane, acetone, and methylene chloride. The chromatograms obtained using the optimized SFE/GC technique showed good peak shapes (comparable to those obtained using a conventional split injection) and typical peak area reproducibilities of < 5% relative standard deviation.

The SFE/GC method was used for the quantitative extraction and analysis of gasoline- and diesel-range organics from real-world environmental samples (Burford *et al.* 1994b). Petroleum contaminated samples containing gasoline, kerosene, diesel or motor oil (total hydrocarbon contents of 168, 2, 26, and 10 mg/g, respectively) were quantitatively extracted by a 15 minute SFE/GC extraction using 400 atm, 60°C CO_2. The SFE/GC hydrocarbon recoveries were comparable to those obtained by sonicating the samples in methylene chloride for 14 hours. Gasoline recovery was higher by SFE/GC analysis, due to the more efficient collection of volatile analytes. Gasoline- and diesel-range organics could be quantitatively retained during the SFE step of the SFE/GC analysis using a thick-film (30-m x 0.32-mm I.D., 5-μm film thickness) DB-1® column operated at a cryogenic trapping temperature of -25°C. Using split SFE/GC operated at a high split ratio (100:1), relatively large (1 g) sample sizes could be investigated; and by using a drying agent (molecular sieve 3A), very wet (25 wt% water) samples could be analyzed without extracted water freezing in the GC column during the SFE step.

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GASOLINE-RANGE TPH, DIESEL-RANGE TPH, AND BTEX BY OFF-LINE SFE

The determination of TPH in contaminated soils by supercritical CO_2 extraction with infrared spectrometry (SFE/IR) has been compared with conventional Soxhlet extraction (Hawthorne *et al.*, 1993a). Generator-powered SFE and infrared instruments were operational only 20 min after arriving at the sampling locations, and no specialized support vehicle was required. SFE/IR analyses of gasoline-, diesel-, motor oil-, and crude-oil-contaminated soils yielded virtually identical results (less than 10% variation), whether the samples were extracted and analyzed in the field or in the laboratory. TPH concentrations by 30-min SFE and 4-hr Soxhlet extraction agreed to within 20%. Except for the motor oil-contaminated soil, SFE for 10 min yielded 70-95% of the quantity that was extracted after 30 min, indicating that short SFE extractions can be useful for rapid field surveys.

Further comparisons for gasoline- and diesel-range organics demonstrated that SFE often yields higher recoveries than Soxhlet extraction (Eckert-Tilotta *et al.*, 1993). SFE of the gasoline-contaminated sample extracted at 40 MPa CO_2 and 65°C resulted in higher TPH quantities than those obtained from Soxhlet extraction (134% SFE vs. Soxhlet), owing to more efficient collection of BTEX and other volatile components by SFE. Comparable TPH results were obtained using gas chromatography with flame ionization detection and infrared spectrometry. Quantitative reproducibility for replicate SFE extracts was good (relative standard deviation of 2-10%), and the quantity of Freon-113® solvent was reduced from 150 ml for Soxhlet to < 10 ml for SFE.

Ideally, the SFE system should be able to extract and collect BTEX and all other gasoline and diesel components so that a single extraction can be used for both BTEX and TPH determinations. Commercially available SFE systems employing sorbent (Hewlett-Packard®) and solvent (ISCO®) traps

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were used for extracting TPH from real-world soil samples contaminated with gasoline- or diesel-range hydrocarbons (Yang *et al.*, 1994a). Quantitative extractions using both SFE systems were performed at 80°C and 340 atm with a flow rate of 1.5 mL/min. Both sorbent and solvent trapping systems effectively collected BTEX (> 90%). Sorbent trapping yielded quantitative collections (≥ 88%) of n-alkanes as volatile as n-hexane, while the solvent trapping effectively collected n-alkanes as volatile as n-heptane (pressurized trapping system) and n-octane (normal trapping system). The quantification of BTEX, TPH, and individual species from contaminated soils obtained by both SFE systems agreed well. However, because of the greater losses of BTEX and the volatile n-alkanes, Soxhlet extraction yielded significantly less BTEX, TPH, and compound-specific analytes than both SFE systems. This study demonstrated that commercially available SFE instrumentation can be used to determine BTEX and TPH levels using a single extraction.

HEAVY HYDROCARBON DETERMINATIONS BY SFE

Heavy hydrocarbons are not extracted as readily as gasoline- and diesel-range organics using pure CO_2 at conventional temperatures (e.g., 50°C) and pressures (e.g., 340 to 400 atm). Therefore, both elevated temperature and the addition of organic modifiers to supercritical CO_2 have been evaluated. SFE with CO_2 was used for the determination of TPH in real-world fuel-spill soil samples containing heavy fuel oil, diesel fuel, and light crude oil (TPH contents of 150, 15, 15 mg/g, respectively) (Eckert-Tilotta *et al.*, 1993). Quantitative extraction by SFE was accomplished at 400 atm CO_2 and 150°C extractor temperature, and TPH results were comparable (within standard deviations) with those obtained by Freon-113® Soxhlet extraction (4 hr) for all samples. Comparable TPH results for the soil extracts were obtained from analytes using gas chromatography with flame ioniza-

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tion detection and infrared spectrometry. Quantitative reproducibility for replicate SFE extracts was good (relative standard deviation of 2-10%), and the quantity of Freon-113® solvent was reduced from 150 ml for Soxhlet to < 10 ml for SFE.

Supercritical fluid extraction at high temperature (150°C), and with an infrared-clear organic solvent as a modifier, quantitatively extracted even heavier hydrocarbons from soil (Hawthorne *et al.*, 1994a). SFE with pure CO_2 at 65°C yielded good recovery for light crude oil components (smaller than approximately C_{25} alkanes), but did not efficiently extract heavier crude oil components. While raising the temperature during SFE to 150°C increased the recovery of the heavier hydrocarbons, the best recoveries were achieved when extractions were performed at 150°C after a single addition of perchloroethylene as a modifier. With these conditions, SFE (15 minutes static followed by 15 minutes dynamic extraction) yielded 5 to 45% higher recoveries than four hours of Soxhlet extraction for soils contaminated with light to heavy crude oils, motor oil, and a heavy residual oil. Based on silica absorption of the extracted polar compounds, both polar organics and non-polar organics were more efficiently extracted by SFE. Since the modifier is added directly to the soil sample, the method does not require either dual pumps or pre-mixed fluids.

PAH DETERMINATIONS BY SFE

SFE with pure CO_2 at conventional temperatures (e.g., 50°C) often cannot quantitatively extract PAHs from well-aged samples. In an effort to increase recoveries, supercritical fluid extractions using eight different CO_2 + organic modifier mixtures and one ternary mixture (CO_2 + methanol/toluene) at two different concentrations (1 and 10% v/v) were performed on two certified reference materials, including polychlorinated biphenyls (PCBs) from

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river sediment and PAHs from urban air particulate matter (Langenfeld *et al.*, 1994). Modifier selection was more important than modifier concentration in increasing extraction efficiencies. Acidic/basic modifiers including methanol, acetic acid, and aniline greatly enhanced the extraction of PCBs. Low molecular weight PAHs were best extracted with modifiers including aniline, acetic acid, acetonitrile, methanol/toluene, hexane, and diethylamine. In contrast, modifiers capable of dipole-induced and p-p interactions, such as toluene, diethylamine, and methylene chloride, were the best modifiers for SFE of high molecular weight PAHs from air particulates. In general, increasing the modifier concentration from 1 to 10% (v/v) had little effect on PCB and low molecular weight PAH recoveries, although the recoveries of high molecular weight PAHs from urban air particulate matter were enhanced significantly at the higher modifier concentration. Although there is no definite theory that explains modifier selection for SFE, it appears that modifiers should be selected on the basis of matrix characteristics and the target analytes.

A similar organic modifier method for PAHs, in which three soil samples were extracted with SFE and compared to standard sonication, was employed in a mini-round robin study (Lopez-Avila *et al.*, 1994). Interlaboratory reproducibility was good. The recoveries were typically > 80% for samples contaminated at 1 mg/kg or higher, while recoveries typically ranged from 50 to 60% for samples contaminated at lower levels. A possible defect of the specified method was that it did not provide a static time to allow the modifier to contact the sample. Extensive work with other samples suggests that had the static time been provided, the recoveries would have been much higher for these samples.

Three other studies have demonstrated that raising the temperature of the SFE step to 200°C greatly enhances the extraction of PAHs and other organics, and high temperature SFE typically yields quantitative recoveries of PAHs without the need for organic modifiers (Langenfeld *et al.*, 1993;

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Hawthorne *et al.*, 1994b). For particularly difficult samples, combined high temperature and modifiers yield the highest recoveries (Yang *et al.*, 1994b).

PRACTICAL ASPECTS AND THEORETICAL FACTORS CONTROLLING THE APPLICATION OF SFE

Efficient methods development using SFE is greatly enhanced by an understanding of the chemical and instrumental parameters and their effect on recoveries. The development of quantitative SFE methods for the recovery of organic pollutants from environmental samples requires three steps: quantitative partitioning of the analytes from the sample into the extraction fluid, quantitative removal from the extraction vessel, and quantitative collection of the extracted analytes (Hawthorne *et al.*, 1993b). While spike recovery studies are an excellent method to develop the final two steps, they are often not valid for determining extraction efficiencies from complex real-world samples such as soils and sediments, exhaust particulates, and sludges. SFE conditions that yield quantitative recoveries of spiked analytes may recover < 10% of the same analytes from real-world samples, because spiked pollutants are not exposed to the same active sites as the native pollutants. Because of the heterogeneous nature of environmental samples, the partitioning step may be controlled by analyte solubility in the extraction fluid, kinetic limitations, and/or the ability of the extraction fluid to interrupt matrix-analyte interactions. While the interactions that control SFE rates from heterogeneous environmental samples are not well understood, a generalized scheme for developing quantitative SFE methods is proposed based on interactive considerations of the collection efficiencies after SFE, fluid flow parameters in the extraction cell, analyte solubility, extraction kinetics, and analyte-matrix-extraction fluid interactions. The proposed development scheme includes increasing SFE

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extraction rates by the use of more polar fluids than CO_2, such as chlorodifluoromethane (Freon-22®), the addition of organic modifiers to CO_2, and the use of high temperature extractions with pure CO_2. Validation of quantitative extractions based on multiple extraction methods (SFE followed by liquid solvent extractions) is also described.

Spiked analytes are typically used to determine extraction efficiencies; however, since well-aged analytes may be more strongly bound to sample matrices, spike recoveries may not be valid. To investigate this possibility, the relative extraction rates of native PAHs ranging from naphthalene (M = 128) to benzo(b)fluoranthene (M = 252) and those of spiked deuterated PAHs (d-PAHs) from heterogeneous environmental samples including petroleum waste sludge, urban air particulate matter (SRM 1649), and railroad bed soil were compared using sequential extractions with pure supercritical CO_2 or modified (10% v/v methanol) supercritical CO_2 and using sonication with methylene chloride (Burford *et al.*, 1993a). Regardless of the spiking method (injection of the spike or suspension of the sample in the spiking solution) or aging time (up to 14 hr), the extraction rates of most of the spiked d-PAHs were substantially higher (up to 10 times) those of the same native PAHs. Differences in extraction rates of the spiked and native PAHs were most dramatic for the lower molecular weight PAHs, indicating that relatively volatile species such as naphthalene must be tightly bound in order to remain associated with a real-world sample. In most cases, 30-min extractions with pure CO_2 quantitatively recovered (> 90%) the spiked deuterated-PAHs, but only extracted approximately 25-80% of the native PAHs. Similar differences were observed using conventional methylene chloride sonication, demonstrating that spike recovery studies are not valid for developing quantitative extraction methods for heterogenous environmental samples.

While spikes should not be used to determine extraction efficiencies, they are very good to determine collection efficiencies. The collection of petro-

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leum hydrocarbons as volatile as benzene and butane was discussed above and described in detail in Burford *et al.*, 1994a; Burford *et al.*, 1994b; and Yang *et al.*, 1994a.

Since some samples encountered in this study required that drying/dispersing agents be added to avoid restrictor plugging during SFE, the use of 21 potential drying agents was investigated (Burford *et al.*, 1993b). Five (anhydrous and monohydrated magnesium sulfate, molecular sieves 3A and 5A and Hydromatrix®) were able to prevent restrictor plugging by water during off-line supercritical fluid extraction (e.g., 400 atm CO_2 at 60°C) by retaining the majority of the water (but generally not the analytes of interest) in the extraction cell. Increasing the extraction temperature (e.g., to 150°C) or adding a polar modifier (10% (v/v) methanol) to the CO_2 extraction fluid greatly reduced the amount of water the drying agents retained. However, when 10% (v/v) toluene was used for the extraction, the drying agents were able to retain the majority of the water (approximately 80% w/w). Polar and non-polar pollutants were quantitatively extracted from the wet drying agents, but nearly all of the drying agents selectively retained at least one of the polar analytes if used dry, thus demonstrating the need for a spike recovery study to determine the potential for analyte loss. The successful drying agents eliminated restrictor plugging when used with moderately wet (approximately 20% (w/w) water at a 1:1 reagent-to-sample ratio) and very wet (approximately 90% (w/w) water at 4:1 reagent-to-sample ratio) samples without the need to heat the restrictor or the collection solvent.

Fused-silica restrictors used for off-line SFE frequently break when extractions are performed with polar supercritical fluids (e.g., Freon-22®) or CO_2 containing polar modifiers (e.g., methanol)) (Burford *et al.*, 1993c). Securing the fused-silica restrictor inside a 1/16 in. (1.6 mm) O.D. stainless-steel tube with an epoxy resin eliminated the restrictor breakage and allowed restrictors to be connected to the extraction cell with conventional stainless-steel fittings. The stainless-steel clad fused-silica restrictor was

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simple and inexpensive to construct, physically robust, and proved ideal for SFE applications, since no artifacts from the clad restrictor were detected in the collection solvent. A simple correlation to predict the flow using linear restrictors was also developed. The correlation accounts for pressure, temperature, restrictor i.d., and restrictor length (Yang *et al.*, 1994c). The correlation allows the proper size of restrictor to be selected for the desired flow rate under different extraction conditions.

RECOMMENDATIONS

The results of this study clearly demonstrate that analytical-scale SFE can successfully compete with conventional (e.g., Soxhlet) extraction for the extraction of hydrocarbons from soils and sludges. However, it must be remembered that SFE is not yet as simple to perform as a Soxhlet extraction and, therefore, it is likely that a more highly-trained analyst will be required to obtain good results. SFE methods for gasoline- and diesel-range organics (e.g., the proposed EPA method) are well-developed and easily implemented using commercial SFE instrumentation. The only reason for slow adoption of SFE for routine TPH determinations appears to be based on the slow promulgation of the EPA method. For higher boiling species, SFE conditions utilizing elevated temperatures (e.g., 150°C) and/or the addition of organic modifiers are often required to obtain quantitative recoveries (e.g., for alkanes > C_{30} and PAHs), but such techniques are relatively simple to perform with most commercial instrumentation (and properly-designed "home-built" systems).

On the negative side, some problems still exist with commercial instrumentation, particularly in the areas of restrictor plugging (with particularly "dirty" matrices, e.g., wet sludges with very high extractable organic content) and collection efficiencies of more volatile analytes. The primary dif-

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ferences in commercial instruments occur in these two areas, i.e., the method used to control the CO_2 flow rate (the restrictor system), and the method used to collect extracted analytes. Often, seemingly insignificant differences in commercial instrumentation can greatly affect whether a complex sample can be extracted (e.g., whether the restrictor plugs) and whether a particular analyte can be efficiently trapped. While it is unfair to require an instrument supplier to develop a method for a particular application, investigators who wish to evaluate various SFE instrumentation should request two test evaluations. First, the restrictor system should be capable of controlling flow and not plugging while extracting the most complex (e.g., highest water content and highest extractable matrix content) samples that are expected to be encountered by the purchaser. Second, the trapping system (e.g., sorbent, solvent, or cryogenic trapping) should be demonstrated to quantitatively collect the analytes of interest (particularly the more volatile species) before purchase should be considered. In addition, the majority (but not all) of analytical SFE instruments use sample sizes of 10-mL or less, because of increased reliability in high pressure systems.

It should be noted that, since analytical SFE instrumentation has been commercially available for only a few years, and since substantial developmental efforts (particularly in automated operation) are just coming to fruition, this report will not attempt to offer purchasing advice. However, the investigator who wishes to utilize the methods described in this report should consider the following results that relate to instrumentation:

1. Both solvent trapping and sorbent trapping were successful with species as volatile as benzene if properly performed. However, both methods can yield very poor collection efficiencies if not properly performed (thus the importance of the collection efficiency evaluation for different vendors' instruments as discussed above).

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2. A single addition of modifier to the sample was generally sufficient to increase recoveries of analytes in these investigations where pure CO_2 was not successful. Therefore, an SFE instrument should be able to perform extractions in the static (non-flowing) followed by dynamic (flowing) mode.
3. Since a single addition of modifier directly to the sample was generally sufficient (using static followed by dynamic extraction), dual pump systems (which add to instrument costs) to add modifiers were not necessary. However, since future applications may require constant addition of modifier, an instrument should have provisions to add a modifier pump at a future date.
4. We were able to extract from all of the sludge and soil matrices, but approximately 1/3 of the samples caused plugging of the restrictors severe enough necessitate premixing the sample with a dispersant (e.g., 40 µm "Empore" glass beads). This problem was most severe with samples containing high concentrations of heavy hydrocarbons, and samples with high concentrations of elemental sulfur. In general, soils contaminated with gasoline- and diesel-range organics showed no significant restrictor plugging, even though water contents were as high as 20 wt%. Therefore, an SFE system should be evaluated with the range of "real-world" samples that will be extracted.
5. The type of restrictor selected also dictates the CO_2 flow rates (and degree of control) that the analyst can select, though precise flow control had no apparent value in this study. For typical SFE sample sizes (i.e., < 10 mL), flows in the range of 1 to 2 mL/min of compressed CO_2 are usually adequate, and there appears to be no disadvantage to flows that vary by approximately ± 30% from the set-point.
6. For methods development, simpler (and less expensive) SFE instrumentation is often as good as (or better) than more complex (and higher cost) systems. A cost-effective approach to investigating SFE may be to first utilize a simpler system for methods devel-

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opment, and later purchase a more sophisticated automated extraction system when routine methods are implemented.

7. For the samples used in this study, the effect of SFE temperature was more dramatic than pressure. Commercial SFE instruments generally have reasonable upper pressure limits (typically approximately 340 to 600 atm), however, temperature limits vary more widely. Based on the results of these studies, upper temperature limits of approximately 150°C (or greater) are desirable.

The results of these investigations also demonstrate that well-aged pollutants can be extracted at different rates and conditions than recently spiked pollutants. While spiked samples may be useful in estimating the collection efficiency of an SFE trapping system, unspiked samples should be used in the development and testing of the SFE method, to ensure that the aged pollutants are extracted. When the goal is to develop an SFE method that yields recoveries similar to an accepted method (e.g., Soxhlet extraction), the recoveries of the SFE method should be compared to Soxhlet extraction on replicate identical unspiked samples. When the goal is to develop a method that yields the highest recoveries possible (thus best reflecting the true concentrations), the SFE method should be validated by performing sequential liquid solvent extraction on the sample residue after SFE extraction. If the liquid solvent extraction of the SFE residue contains no significant additional analytes, the SFE method can be considered to yield quantitative recovery.

Historically, sample extraction has often been performed by the least experienced and least trained personnel in the laboratory. During the method development stage, SFE must be considered to be more complex than Soxhlet extraction; and experienced personnel will be required to successfully develop quantitative methods. Further developments in automated instruments should allow routine SFE methods to be performed by personnel with minimal training.

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It should be noted that SFE methods can be relatively fast, since extractions used to evaluate various SFE parameters typically require < 30 minutes to perform, even with simple manual SFE systems. Finally, the demonstrated ability of SFE to reduce liquid solvent usage to less than 15 mL, depending on the specific collection system used, makes SFE an attractive approach to solving regulatory pressures on liquid solvent use and disposal.

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