

# Waste Testing and Quality Assurance

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THIRD  
VOLUME

David Friedman, editor



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# ***Waste Testing and Quality Assurance: Third Volume***

*David Friedman, editor*

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#### **Peer Review Policy**

Each paper published in this volume was evaluated by three peer reviewers. The authors addressed all of the reviewers' comments to the satisfaction of both the technical editor(s) and the ASTM Committee on Publications.

The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of these peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution to time and effort on behalf of ASTM.

## Foreword

This publication is one in a continuing series of ASTM Special Technical Publication (STP) on one of the most diverse and far reaching branches of environmental testing—namely that dealing with waste management. The papers presented in this STP series, are an outgrowth of those presented at the annual Waste Testing and Quality Assurance symposium sponsored by the United States Environmental Protection Agency's Office of Solid Waste and Emergency Response. The editor, David Friedman of EPA, serves as symposium chairperson and is the editor of this Special Technical Publication (STP) series. This publication is the third in the series which began in 1988 with STP 999.

# Overview

This Special Technical Publication (STP) is the third in a series highlighting recent developments in the areas of waste and environmental media characterization. While the information presented in these papers focuses on problems related to hazardous waste management and contaminated site cleanup, the methodology and practices are applicable to all forms of environmental monitoring and material characterization.

## Organic Analysis

Analysis of environmental samples for organic chemical contamination likely accounts for the lion's share of the millions of dollars expended annually for environmental testing. The number and complexity of these analyses continues to grow as a result of our improved understanding of the effects these chemicals have on human health and the environment, and our ability to identify and measure a wider variety of organic chemicals.

This volume focuses on two areas related to improving the methodology used for organic analysis: techniques to improve analytical accuracy or cost-effectiveness and efforts to expand the scope of the available methods.

Because of the high cost of data gathering to the economy, a great deal of effort has been put into reducing its costs. Such efforts range from eliminating the expense, difficulty, and time involved in shipping samples to the laboratory by, whenever possible, conducting analyses in the field to development of less expensive analytical techniques. In the papers presented, we highlight progress in both areas.

The range of these efforts is illustrated by Wright et al. and Accra and Hafferty. Wright et al. evaluates Supercritical Fluid Extraction for use in the field for analyzing contaminated soils. Accra and Hafferty report on the evaluation of hand-held photoionization devices for use in screening ambient air for potential contamination. Supercritical fluid extraction is an efficient tool for extraction of organic compounds from solid materials that is under study in many organizations. In their work to adapt the techniques to field use, the authors show foresight and an appreciation of the growing importance of on-site sample processing.

The photoionization detector is a commonly employed tool for monitoring work areas to ensure worker safety and for site screening to identify areas of concern. While the technique and equipment are very popular, there has been little data published that compares instruments and demonstrates the limitations of the technique.

Improvements in analytical cost-effectiveness is by no means restricted to changing what and how we do things in the field. The methods used in the laboratory have and remain the focus of intense research. Because of the tremendous importance of this aspect of environmental analysis, we have included ten papers dealing with efforts to reduce analytical cost. Of these, nine deal with problems related to organic analysis.

The first group of papers use automation and the power of the computer to increase productivity. Several analytical methodologies exist for the analysis of volatile organic compounds in ambient air. In research reported on by Kirshen and Almasi, two automated analytical systems were developed to analyze sub part per billion levels of Volatile Organic Compounds (VOCs) in ambient air and soil gases. The authors provide a description of the

two systems, their operation, and their performance. Dewald and Koch also employed automation to improve productivity. He and his coworkers developed a software enhancement for the GC/MS system that permits feedback from quality control samples to control the autosampler. This automatically prevents analysis when the instrument is out of control and thus permits 24-h unattended operation.

One of the most important, complex, and time consuming issues facing enforcement agencies and potentially responsible parties is the determination of a suspect waste's identity. Hughes et al. describe one approach that has recently been used to answer such questions. The method employs a state-of-the-art mass spectrometric data processing system to produce visual and quantitative comparisons of chromatographic and mass spectrometric patterns in an attempt to determine a waste's origin. They demonstrate how the calculation of a Cross Correlation Coefficient is a very powerful way of comparing complex chemical data to evaluate similarities and differences between chromatograms.

Another important approach to lowering the cost of waste and environment media characterization is use of screening to eliminate samples with contaminant concentrations below levels of concern. Draper et al. and Flynn et al. show how to screen samples for contaminants of environmental concern. Draper et al. employ Electron Capture/Gas Chromatography to determine the concentration of chlorinated dibenzo-dioxins and dibenzo-furans in soils and biological samples. Looking at the other end of the volatility spectrum, Flynn employs headspace analysis to screen environmental samples for volatile organics.

In the search to find more efficient techniques for organic analysis, new analytical techniques are finding increased application to environmental problems. Highlighted in this volume are application of HPLC, supercritical fluid extraction, supercritical fluid chromatography, and solid phase extraction.

The report by Okamoto et al. of a new derivitization technique that permits the fast, effective HPLC technique to be used for low-level analysis for *N*-methylcarbamate pesticides in environmental samples begins this section. Following the discussion on HPLC, Lopez-Avila et al. present a report on work sponsored by the U.S. Environmental Protection Agency to evaluate use of solid phase extraction as a means of removing interferences from environmental samples. Her work is followed up by two papers dealing with use of supercritical fluids both for extracting contaminants from environmental matrices and as a chromatography mobil phase. These papers by Lopez-Avila and Pospisil et al. highlight what is one of today's most promising areas of environmental organic analysis research.

While the search to develop less expensive approaches to environmental analysis continues, research on improving the accuracy of our current techniques has not been neglected. Three papers are presented that address this area. Urban et al., looks at one of our more difficult problems, that of characterizing soils for VOCs. In their paper, the authors show how their new methodology reduces VOC loss during the sampling and transportation process and thus leads to data that are more representative of what the sample in the field actually contains.

One of the more contentious issues facing the testing community revolves around how holding a sample for an extended period of time affects sample integrity. Preanalytical holding times are currently a legal and/or contractual consideration. Bottrell et al. report on three separate studies that were designed to address the effects sample storage time and preservation condition has on sample integrity. Consideration of the information developed in these studies by those responsible for data acceptance allows for maintaining accuracy while at the same time giving the laboratory increased work scheduling flexibility.

Use of silica gel in the sorbent trap during the gas chromatographic/mass spectrometric determination of volatile organic compounds has sometimes been found to lead to water vapor buildup in the instrument vacuum system. This can result in loss of sensitivity, in-

accuracies in the data, and in some cases, automatic shutdown of the mass spectrometer. Weston et al. investigated several alternative sorbent packings and describe silica gel free packings that can maintain the needed performance, while at the same time, eliminating the water buildup problem.

While High Performance Liquid Chromatography (HPLC) has been available for more than a decade, its application to environmental problems has been limited. This has been due to a lack of detectors, which combine the selectivity needed for positive compound identification with high sensitivity. The mass spectrometer, which transformed gas chromatography, remains the most promising tool for attaining these objectives. The difficulty lies in suitably interfacing the two techniques.

While tremendous strides continue to be made in the area of instrumentation, in some cases we are limited not by analytical methodology but rather by an inability to collect and transport a valid sample to the laboratory. Solecki et al., in their evaluation of the Summa Canister for sampling of landfill off-gas demonstrate how the problems encountered were overcome. The technique presented proves to be one of the more efficient ways currently available for collecting and preserving gas samples.

## **Inorganic Analysis**

Extraction of the elements of interest from the matrix is usually the most time consuming step in analysis and the source of most analytical errors. Kimbrough and Wakakuwa looked at the problems associated with extraction of antimony, silver, and barium using Environmental Protection Agency EPA Method 3050, which is a conventional hot acid extraction. The authors describe an alternative procedure that can simultaneously solubilize not only these elements but can also solubilize other RCRA regulated elements with performance equal to or superior to EPA Method 3050. Use of the improved procedure should lead to more accurate waste concentration determinations in soils, sludges, sediments, and other solid waste materials.

One of the most promising new tools for winning elemental analyses from solid matrices is microwave assisted extraction. Procedures for using the microwave method have recently been developed and commercialized. Binstock et al. compared the microwave method to the conventional open vessel hot plate digestion and demonstrated that the new method should prove a suitable alternative to existing techniques while yielding a substantial time/cost saving.

## **Mobility Methods**

One question that continues to arise when evaluating the impact a waste or contaminated media might have on human health or the environment, is whether the toxic constituents are likely to migrate from the disposal point and reach a sensitive target. In order to give the environmental community the tools needed for making such assessments, mobility estimation has been and continues to be the subject of much study. The papers presented in this section give an overview of some approaches currently being developed to improve our ability to more accurately and cost-effectively estimate potential hazard.

Chemicals in soil can be subjected to environmental factors, which may influence their mobility. Climatic stress can play a significant role in altering soil structure, and this can change its properties relative to contaminant retention. While the effect these stresses have has been of concern for many years, research, such as that reported by Erickson et al., has only now begun to shed light on this neglected area. This paper presents the results of work in which soils from several hazardous waste sites were initially characterized using EPA



Method 1311 to determine mobility and then subjected to repetitive freeze/thaw and wet/dry cycles. The authors reported that they have detected no clear differences in the mobility of metals before and after weathering.

McNevin also studied soil leachability in order to determine how best to dispose of radium contaminated soils. While his interest revolved around the leachability of radium, the results are applicable to the mobility of other metals of interest and can be used to estimate mobility in similar situations.

While the previous authors relied on laboratory procedures to estimate leachability, Murarka et al. took a different approach and looked at a geochemical basis for predicting leachability. He and his coworkers have developed a computer algorithm to predict leachability based on thermodynamic and solubility product data and have compared the calculated leachability to actual leachate from fossil fuel combustion residue piles. Preliminary results bear out the applicability of the mechanistic approach. The approach taken should be applicable to other industries in which large volumes of inorganic residuals are generated.

The last two mobility papers deal with two very different subjects, both of which are of great importance and concern. Bone et al. report on the problem of characterizing wastes that are in the form of large pieces. The test procedure currently used in the United States for evaluating leachability of treated wastes is EPA Method 1311 (the TCLP). Method 1311 requires that, even quite strong monolithic wastes, be crushed to pass a 9.5-mm mesh before contact with the extraction fluid. In his paper, Bone et al. look at work the EPA and ASTM are doing to develop better approaches to evaluating structurally secure monolithic wastes.

As a result of the large number of spills that have taken place, petroleum products have often been found as having contaminated ground water. In his study, Davini examined the mobility of fuel oil through soil as it relates to its potential to serve as a source of ground water contamination. He and his coworkers also looked at the applicability of EPA Method 1311 to this issue and concluded that it is an inappropriate tool for predicting the mobility of oily wastes.

### **Quality Assurance and Quality Control**

While the previous papers deal with improving our measurement methods as a means of improving data quality, those in this section look at the issue from a variety of other perspectives. These perspectives include sampling, data reporting, and data evaluation.

The quality of our field and laboratory measurements can never be better than the quality of the sample taken for analysis. Unless the sample is representative of the material to be evaluated, the data cannot yield an accurate characterization.

In their paper, Eatmon and Marcus give us a behind the scenes look at how a major hazardous waste management firm has attacked the problem of developing sampling protocols for their own hazardous waste characterization program. They share with us the approach Chemical Waste Management, Inc. (CWM) has used to ensure that the myriad of decisions that one has to make in managing hazardous waste are based on data of known and documented quality.

While the previous paper looked at how one develops and implements sampling protocols for a wide variety of situations, Kontopanos and Williams look at the issue of field sampling quality control. The authors examine three parts of a field sampling effort: (1) the preparation of the site's specific sampling plans; (2) the quality control samples that should be collected in order to have data of known quality; and (3) the post sampling documentation needed for data defensibility.

Kontopanos in the previous paper introduced the issue of post sampling documentation as an integral part of the data collection process. Expanding on this critical area of data



collection, Pink et al. take a more in depth look at this area. They report on a comprehensive approach that they have developed for environmental program documentation. They present a mechanism for assessing, documenting, and reporting on the quality of the data, and consequently its usability.

One aspect of waste characterization that often goes unrecognized relates to use of historical information. By looking at information that is already available, one can often eliminate a great deal of sampling and analysis work and identify potential problems. However, in order to effectively and efficiently use historical data, one needs to have a means of indexing the information in a manner that facilitates later comparisons. Pospisil and Marcus have looked at this problem and have developed a system for classifying the wide variety of materials found to be of interest to those involved in hazardous waste management. The system will be of use to any organization faced with organizing sampling and analysis data covering a wide variety of material types.

Completing our coverage of issues dealing with data evaluation is the paper by Peak, which concerns a new and increasingly important means of evaluating monitoring data; the use of computers to automate the evaluation of quality control data. In his paper, Peak shows how computers can be used to give an early warning of potential problems.

They report on a newly developed automated review system that evaluates laboratory quality control data. The review serves to supply the laboratory manager with an early warning when laboratory control limits are in danger of being exceeded.

Rounding out the papers presented in this volume are those by Gibbons et al., which examine new approaches to evaluate analytical method sensitivity, and by Brown et al., which looks at quality assurance from the perspective of the person who has to make decisions based on the data. They look at the problem of qualified data and examine how qualified data can often be used to increase one's confidence in the ultimate decision.

*David Friedman, Editor*

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# **Organic Analysis**



Bob W. Wright<sup>1</sup>, Cherylyn W. Wright<sup>1</sup>, and Jonathan S. Fruchter<sup>2</sup>

EVALUATION OF A FIELD-PORTABLE SUPERCRITICAL FLUID EXTRACTION  
APPARATUS FOR RAPID CHARACTERIZATION OF CONTAMINATED SOILS

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**REFERENCE:** Wright, B. W., Wright, C. W., and Fruchter, J. S., "Evaluation of a Field-Portable Supercritical Fluid Extraction Apparatus for Rapid Characterization of Contaminated Soils," Waste Testing and Quality Assurance: Third Volume, ASTM STP 1075, D. Friedman, Ed., American Society for Testing and Materials, Philadelphia, 1991

**ABSTRACT:** Rapid, field-portable methods for measuring the concentration of semivolatile organic pollutants are desirable for on-site characterization of contaminated sediments and soils. In addition to providing efficient site assessment capability, rapid characterization methods would also be highly useful at sites undergoing remediation treatments. As an alternative to current methods, supercritical fluid extraction (SFE) has been evaluated for in-the-field coal and petroleum gasification waste site characterization. The gas-like mass transfer properties of supercritical fluids allow more rapid extraction rates and more efficient penetration of the matrix than conventional solvent extractions. The method has proved to be rapid, reproducible, and successful in several field trips.

**KEYWORDS:** supercritical fluids, polycyclic aromatic hydrocarbons, field extraction, field screening, contaminated soils

Several modern analytical techniques (e.g., capillary gas chromatography, high performance liquid chromatography, mass spectrometry) are available for the identification and quantification of a range of organic compounds found in contaminated soils and sediments. However, some type of extraction method is generally required to remove the analytes from the environmental matrix. The most commonly employed extraction method (Environmental Protection Agency SW 846 method 3540) requires long extraction times (up to 24 h for Soxhlet methods), large quantities of ultrapure solvents, and lengthy solvent concentration procedures. Because of these constraints, it is not generally considered to be suitable for field-portable applications. In order to implement remedial measures and to carry out cost-effective site assessments, more rapid and field-adaptable sample extraction methods are desirable. Supercritical fluid extraction (SFE) techniques provide a viable alternative with promising advantages over the current liquid extraction methods [1-4]. SFE

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equipment has the capability of being field-portable; the SFE method is rapid (approximately 30 min), and it is highly reproducible for various contaminated or potentially contaminated soils and sediments.

The advantages of SFE accrue from the properties of a solvent at temperatures and pressures above its critical point. The liquid-like solvating power and rapid mass-transfer properties of a supercritical fluid provide the potential for more rapid extraction rates and more efficient extraction due to better penetration of the matrix than is feasible with liquids. The properties of a supercritical fluid are intermediate between those of the gas and those of the liquid phases. The compressibility of a supercritical fluid is large just above the critical temperature, and small changes in pressure result in large changes in the density of the fluid. The density of a supercritical fluid is typically  $10^2$  to  $10^3$  times greater than that of the gas. Molecular interactions increase at these higher densities because of shorter intermolecular distances, and solvating characteristics of the supercritical fluid approach those of a liquid. However, the diffusion coefficients and viscosity of the fluid remain intermediate between those of the gas and liquid phases, thus allowing rapid mass transfer of solutes compared to a liquid. The properties of a supercritical fluid are dependent on the fluid composition, pressure, and temperature. Many fluids have comparatively low critical temperatures that allow extractions to be conducted at relatively mild temperatures. For example, the critical temperature of carbon dioxide is only  $31^\circ\text{C}$ . Density or solvating power of a supercritical fluid can be controlled by fluid pressure and/or temperature. In addition, various different fluids or fluid mixtures that exhibit different specific chemical interactions can be used to obtain the desired solvent strength and selectivity.

The present work describes the characterization of two contaminated soil samples by SFE followed by analysis by high resolution gas chromatography. One soil sample was from a coal gasification waste site, while the other was from a petroleum gasification waste site. The coal gasification soil was extracted in the laboratory using replicate SFE and Soxhlet extractions to compare the extraction efficiency and reproducibility of the SFE method versus the conventional Soxhlet method. The petroleum gasification soil was extracted in the field as one sample of ten that were extracted during a recent field trip. The SFE method and apparatus that have been used for several in-the-field studies will be discussed.

## EXPERIMENTAL

### Supercritical Fluid Extraction (SFE)

A schematic diagram of the SFE apparatus is shown in Figure 1. A similar apparatus used in "proof-of-concept" and contaminated soil studies has been previously described [1-4]. Soil samples of approximately 2 g each (weighed to within  $\pm 0.005$  g) were sealed inside the stainless steel extraction cell, which was then placed inside an oven heated to  $100^\circ\text{C}$ . The extraction cells were made from approximately 3-in. (7.6-cm) lengths of 1/4-in. (0.6-cm) stainless steel tubing capped with Swagelok stainless steel zero-volume 1/4-in. to 1/16-in. (0.6-cm to 0.15-cm) column end fittings (SS-400-6-12V) containing 1.0  $\mu\text{m}$  pore size sintered stainless steel frits. All samples were

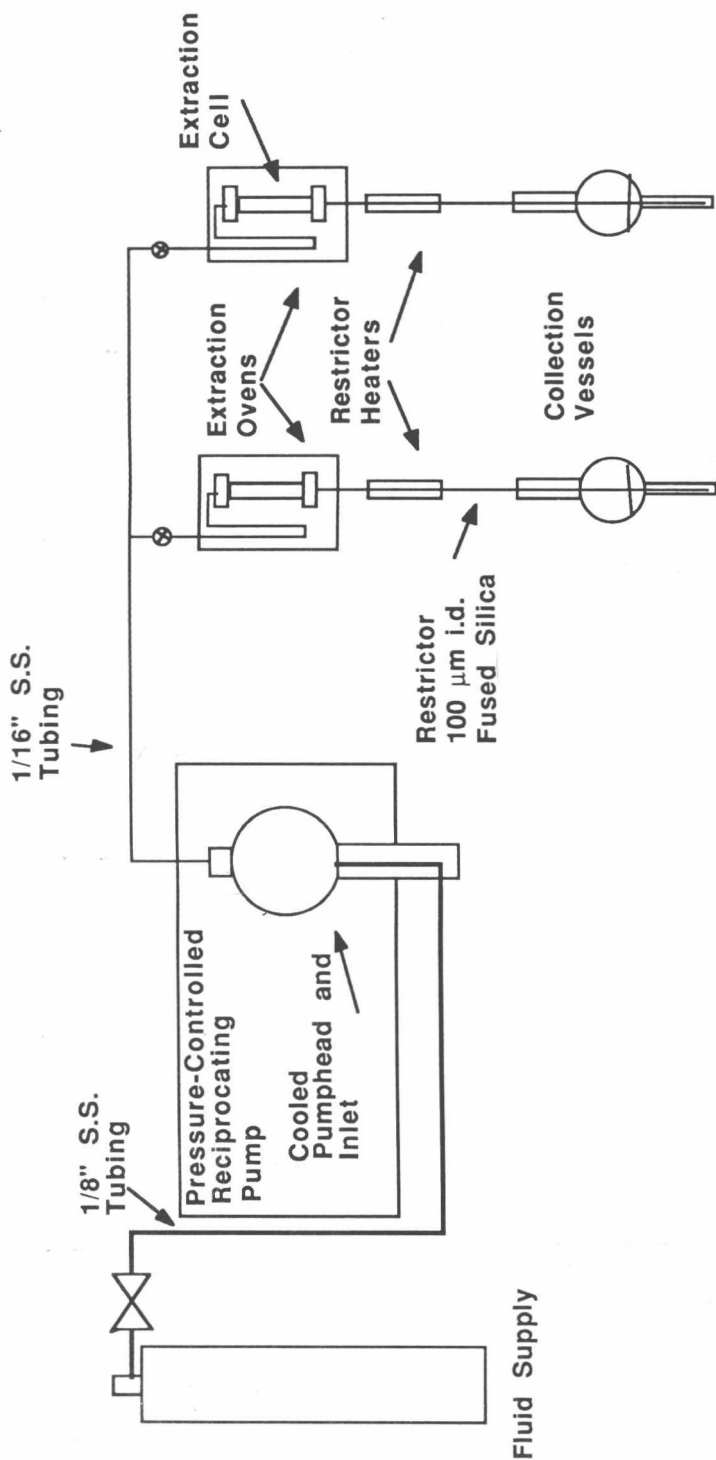


Figure 1. Schematic Diagram of the Field-Portable Supercritical Fluid Extraction Apparatus.