UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT AND COMPLIANCE ASSURANCE
OFFICE OF CRIMINAL ENFORCEMENT, FORENSICS AND TRAINING

ENVIRONMENTAL CRIME

Jennifer A. Suggs, MS
Kenna R. Yarbrough, BS

Library assistance provided by:

Dorothy E. Biggs, NEIC
Barbara L. Wagner, Contractor (Vistronix)
Nancy B. Greer, Contractor (Vistronix)

Prepared for:

14th International Forensic Science Symposium

Author to whom correspondence should be addressed:
Jennifer A. Suggs
EPA-NEIC
Bldg. 25, Denver Federal Center
Denver, CO 80225

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
Diana A. Love, Director
Denver, Colorado

CONTENTS

LIST OF ACRONYMS

μ-AED – micro-atomic emission detector
μ-ECD – micro-electron capture detector
AA – atomic absorption
AAS – atomic absorption spectrometry
AE – atomic emission
AED – atomic emission detection
AES – atomic emission spectrometry
AF – atomic fluorescence
ANSI – American National Standards Institute
AOCD – 9-(2-acridone)oxyethylcarbonylimidazole
APCI – atmospheric pressure chemical ionization (mode)
APEO – alkylphenol ethoxylate
ASE – accelerated solvent extraction
ATR – attenuated total reflectance
BFRs – brominated flame retardants
BTEX – benzene, toluene, ethylbenzene, and xylene
CAA – Clean Air Act
CAS – Chemical Abstracts Service (as in CAS #)
CDFMS – compact double-focusing mass spectrometer
CE – capillary electrophoresis
CERCLA – Comprehensive Environmental Response, Compensation, and Liability Act
CFR – US Code of Federal Regulations
CIT – cylindrical ion trap
CLU-IN – USEPA Technology Innovation Program. Hazardous Waste Clean-Up Information
CMC – chromatomembrane cells
CRM – certified reference material
CWA – Clean Water Act
DFG – Deutsche Forschungsgemeinschaft (German Research Foundation)
ECD – electron capture detector
EDCs – endocrine disrupting compounds or endocrine disrupting chemicals
EDSP – Endocrine Disruptor Screening Program
EDXRF – energy dispersive X-ray fluorescence
EPCRA – Emergency Planning and Community Right-To-Know Act
ES – electrospray
ESA – Endangered Species Act
ESI – electrospray ionization (mode)
ETAAS – electrothermal atomic absorption spectrometry
ETV – electrothermal vaporization
FA – flow analysis
FAAS – flame atomic absorption spectrometry
FEM – USEPA Forum on Environmental Measurement
FFDCA –Federal Food, Drug and Cosmetic Act
FI – flow injection
FID – flame ionization detector
FIFRA – Federal Insecticide, Fungicide, and Rodenticide Act
FIMS – fiber introduction mass spectrometry
FLD – fluorescence detection
FM – focused-microwave (irradiation)
FMASE – focused-microwave assisted Soxhlet extraction

FMOC – 9-fluorenylmethyl chloroformate
FPC – focal plane camera
FPD – flame photometric detection
FSL – fused-silica-lined
FT – Fourier transform
FTIR – Fourier transform infrared (spectrometry)
GC – gas chromatography
GC x GC – two-dimensional gas chromatography
GD – glow discharge
GPO – US Government Printing Office
HFs – hydraulic fluids
HG – hydride generation
HPLC – high performance liquid chromatography
HS – headspace
IC – ion chromatography
ICP – inductively coupled plasma
ICR – ion cyclotron resonance
IR – infrared
ISO – International Organization for Standardization
LC – liquid chromatography
LIF – laser induced fluorescence
LLLME – liquid-liquid-liquid microextraction
LPME – liquid-phase microextraction
LVI – large-volume injection
MAE – microwave assisted extraction
MC – multicollector
MEC – microwave enhanced chemistry
MIP – molecularly imprinted polymer
MS – mass spectrometry
MTBE – methyl *t*-butyl ether
MWFs – metalworking fluids
NCI – negative chemical ionization
NEIC – National Enforcement Investigations Center
NEPA – National Environmental Policy Act
NI – negative ion (mode)
NICI – negative ion chemical ionization
NIST – National Institute of Standards and Technology
NMR – nuclear magnetic resonance
NSAIDs – nonsteroidal anti-inflammatory drugs
NTIS – National Technical Information Service
NTTAA – National Technology Transfer and Advancement Act
OCPs – organochlorine pesticides
OERR – USEPA Office of Emergency and Remedial Response
OES – optical emission spectrometry
OMB – US Office of Management and Budget
OPA – Oil Pollution Act
ORD – USEPA Office of Research and Development
OSWER – USEPA Office of Solid Waste and Emergency Response
OVAs – organic vapor analyzers
PAHs – polycyclic aromatic hydrocarbons or polynuclear aromatic hydrocarbons
PBDEs – polybrominated diphenyl ethers

PCBs – polychlorinated biphenyls
PCDDs – polychlorinated dibenzodioxins or polychlorinated dibenzo-*p*-dioxins
PCDFs – polychlorinated dibenzofurans
PFBBR – pentafluorobenzyl bromide
PFE – pressurized fluid extraction
PFPA – pentafluoropropionic acid
PI – positive ion (mode)
PID – photoionization detector
PIF – photochemically induced fluorimetry
PLE – pressurized liquid extraction
PM – particulate matter
PPA – Pollution Prevention Act
PPCPs – pharmaceuticals and personal care products
PXRF – portable X-ray fluorescence
PyGC – flash pyrolysis gas chromatography
RCRA – Resource Conservation and Recovery Act
RP – reversed-phase
SARA – Superfund Amendments and Reauthorization Act
SBSE – stir bar sorptive extraction
SCCPs – short-chain chlorinated paraffins
SDWA – Safe Drinking Water Act
SES – sequential extraction schemes
SF – sector field
SIA – sequential injection analysis
SIM – selected ion monitoring
SPE – solid phase extraction
SPME – solid-phase microextraction
SRMs – standard reference materials
SSPE – sequential solid-phase extraction
SVOCs – semi-volatile organic compounds
SW-846 – *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods*
TBA – *t*-butyl alcohol
TBT – tributyltin
THMs – trihalomethanes
TIC – total ion chromatogram
TLC – thin-layer chromatography
TMA – trimethylamine
TMSD – trimethylsilyldiazomethane
TMSI – *N*-trimethylsilylimidazole
TOF – time-of-flight
TSCA – Toxic Substances Control Act
TXRF – total reflection X-ray fluorescence
UE – ultrasonic extraction
US – United States
USEPA – United States Environmental Protection Agency
USGS – United States Geological Survey
VOCs – volatile organic compounds
WRI – USGS Water-Resources Investigations Report
WWTP – wastewater treatment plant
XRF – X-ray fluorescence
XRS – X-ray spectrometry

INTRODUCTION

OVERVIEW

This environmental crime review is a follow-up to the 2001 review paper prepared for the 13th International Forensic Science Symposium. [1] Some information presented earlier will be repeated such as resources for sampling methods and the annual or biannual instrumentation reviews regularly featured in some journals. Other topics within the paper are new or in an expanded presentation such as consensus standards, environmental measurements, sampling guidance, and endocrine disrupting compounds.

The paper will proceed in an order roughly resembling that taken during an environmental crime investigation, as the previous review paper did. Information regarding the fieldwork or work at the actual crime scene will be presented first then followed by a section on analytical methods developed and published during the past 3 years. Reference information has been gathered from books, websites, and journals.

AUTHORS' PERSPECTIVE

The United States Environmental Protection Agency (USEPA) was established to protect human health and the environment in the United States (US). Several environmental laws provide the guidance for this mission (a list of major environmental laws is provided in Appendix A). The National Enforcement Investigations Center (NEIC) is part of the USEPA. [2] The NEIC investigates, develops, and assists in prosecuting environmental crimes.

The Code of Federal Regulations (CFR) contains the guidance, requirements, and boundaries for environmental crime investigations. [3] The USEPA is bound by these regulations for the prosecution of environmental crime cases. Many specific sampling and testing methods are required by law for the prosecution of an environmental crime. Much of the information presented here (for example, alternate methodologies) is not being used in the prosecution of environmental crimes in the United States, but it is our hope that it may be useful to other environmental protection entities.

While the USEPA NEIC is limited to enforcement, other sections of the agency are involved in the research of new environmental issues, the development of methods for identification and quantitation of hazardous substances, and the development of improved ways of sampling and assessing sites, as well as improving the treatment or recovery of these same sites. The Office of Research and Development (ORD) is the scientific research arm of the USEPA. ORD has eight priority areas for scientific research: air, drinking water, ecosystem assessment and restoration, global change, human health protection, water quality, pollution prevention and new technologies, and endocrine disrupting chemicals (EDCs). [4] Internationally, endocrine disrupting chemicals (or endocrine disrupting compounds) and the affects of these chemicals on human and environmental health are developing areas of concern. A brief section on EDCs and analytical techniques for the identification and quantitation of EDCs has been included after the laboratory methods section.

ENVIRONMENTAL CRIME SCENE

Decisions made by investigators regarding an environmental investigation must be supported by facts obtained about the scene. If samples are taken and if laboratory analysis occurs, all individuals gathering information and evidence need to maintain certain high standards of care that will enable results to be accurate and reproducible. Several organizations are involved in developing consistent, quality standards for sampling and field measurements. The general agreements on what should be included in the quality standards become consensus standards.

VOLUNTARY CONSENSUS STANDARDS AND METHOD STANDARDIZATION

Voluntary consensus standards are standards developed or adopted by voluntary consensus standards bodies. These bodies must agree to make the standards available on a non-discriminatory, royalty-free or reasonable royalty basis to all interested parties. Voluntary standards are distinguished from mandatory standards, which are published standards that are part of a code, rule or regulation and there is an obligation by certain parties to conform to the standard. Some voluntary standards become de-facto mandatory standards when they are referenced or required by a code, rule, or regulation.

In February 1998, the revised circular No. A-119 was published by the Office of Management and Budget (OMB) concerning federal participation in the development and use of voluntary consensus standards. [5] The circular directs US federal agencies to use voluntary consensus standards in lieu of government-unique standards except where inconsistent with law or otherwise impractical. This circular was developed due to the approval of the 1995 National Technology Transfer and Advancement Act (NTTAA) which emphasized the use by federal agencies of standards developed by private, consensus organizations. [6] A recent article in *ASTM Standardization News* makes the point that regulators may be forced to change regulations to incorporate provisions of relevant consensus standards. [7]

The American National Standards Institute (ANSI) is a private, non-profit organization that administers and coordinates the US voluntary standardization system. [8] It is the official US representative to the world's leading standards bodies, including the International Organization for Standardization (ISO). ANSI has a membership of approximately 1000 companies, organizations, government agencies and others. Although ANSI doesn't develop standards, it provides interested parties a setting to come together, working toward common agreements. A 1996 National Institute of Standards and Technology (NIST) document reports that there are almost 700 standards producing bodies in the United States, with more than 93,000 standards produced. [9] The NSSN is a product of ANSI, and serves as a comprehensive data network on developing and approved national/international standards and regulatory documents on the World Wide Web. [10] The NSSN routes different users to both commercially available and regulatory technical documents that they may need. NSSN has many helpful lists concerning consensus standards and the bodies that develop them, for example, the acronym directory of US standards developers. [11] This list is updated regularly.

Environmental measurement related voluntary consensus standards are developed from many different national and international organizations. The ISO produces the largest number of international standards. [12] ISO has standards concerning environmental measurements and sampling, particularly wastewater sampling, and these standards are available for purchase on the organization's webpage. [13] ASTM International is also one of the largest voluntary consensus standard development organizations in the world. This organization has many different

committees and subcommittees on environmental measurements. [14] The ASTM committee on *Waste Sampling* is designated as D-34. This committee focuses on the promotion of knowledge, stimulation of research and development of test methods, practices, etc. relating to the management of wastes, especially as they relate to sampling of waste streams. Other ASTM International committees concerning environmental measurements and sampling are D-18, *Soil and Rock*; D-19, *Water*; and D-22, *Sampling and Analysis of Atmospheres*. Of additional interest is Committee E-30, *Forensic Sciences.* The scope of this committee focuses on the promotion of knowledge and development of standards for methods and standard reference materials for the collection, preservation, scientific examination, preparation and reports relating to physical evidence for forensic purposes and the general practice of forensic science. Additional information concerning these committees and the standards available for purchase is located on the organization's webpage. [15]

The USEPA has created a Forum on Environmental Measurement (FEM). [16] This committee of senior USEPA managers was formed in order to enhance measurement programs by agreeing on basic principles to promote consistency and consensus within the agency on measurement issues. Some Agency-wide measurement issues that are being worked on include:

- Validation of new USEPA measurement methods
- Identifying and correcting problems with existing measurement methods
- Ensuring and demonstrating the competency of USEPA laboratories
- Consolidation USEPA supported measurement science conferences and
- Use of performance approach in USEPA-mandated monitoring programs

The work being performed concerning validation of new USEPA measurement methods is to help the FEM develop guidelines as to what represents the minimum level of method validation and peer review before methods are issued by the Agency.

SAMPLING GUIDANCE

The USEPA publication, SW-846, titled *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* is the USEPA's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with the USEPA's waste regulations (Resource Conservation and Recovery Act or RCRA). [17] This guidance publication has acceptable, through not required, methods for the regulated/regulating communities to use for RCRA-related sampling and analysis requirements. Chapters 9 and 10 of SW-846 concern developing sampling plans and sample methodologies. The multi-volume publication changes over time as new information and data are available. The current version is available on-line or an official printed copy of SW-846 and most of its updates can be purchased from either the US Government Printing Office (GPO) or the National Technical Information Service (NTIS). [18, 19] In October 2002, the USEPA made available a new draft guidance document for public comment entitled "RCRA Waste Sampling Draft Technical Guidance." This new RCRA waste sampling guidance updates the information provided in Chapter 9 of SW-846, which was last published in 1986, and is also available on-line.

Methods and Guidance for Analysis of Water is the USEPA's primary resource for sampling and analyses of water. This resource was most recently updated in 1999. [20] The USEPA Office of Solid Waste and Emergency Response (OSWER) published a groundwater forum issue paper in 2002 with the most up-to-date groundwater sampling issues and guidelines. [21] Most air emission sampling and measurement methods for the US EPA have been proposed or promulgated in the Federal Register and codified in the Code of Federal Regulations (CFR). The

methods are directly cited by specific regulations for determining compliance under the air regulations in 40 CFR Parts 60, 61, and 63. [3]

Within the USEPA Office of Emergency and Remedial Response (OERR) Superfund program for cleaning up abandoned waste sites, there are several representative sampling guidances for air, water, sediment/soil, biota, and waste. [22-26] These and other sampling and environmental technologies are available through the USEPA Technology Innovation program's Hazardous Waste Clean-Up Information (CLU-IN) internet site. [27] The USEPA's Information Sources webpage lists hundreds of published reports and test methods/sampling procedures and many are available for direct downloading. [28] Also, the USEPA's most comprehensive list of sampling, measurement, and analytical test methods was developed by the Region 1 library staff. [29] The list is continually updated, currently contains about 1600 methods, and the majority of sources listed have live web links to the actual method.

LABORATORY ANALYSIS OF ENVIRONMENTAL SAMPLES

Environmental analysis continues to be a growing field and while not all current areas of study can be applied directly to environmental crime investigations, the research can provide insights into the latent potential of new developments in instrumentation and techniques.

RECENT BOOKS

General Environmental Chemistry Books

A few books published during the past three years provide introductions for the newcomer into environmental chemistry. The *Fundamentals of Environmental Chemistry* textbook has a fast, basic overview of environmental chemistry. The first third of the book consists of a general chemistry primer with a touch of biochemistry. The rest of the book provides a quick, clear foundation for learning about the environmental chemistry and pollution of water, soil and the atmosphere. Topics in this section progress to include industrial ecology, hazardous wastes, toxicological chemistry, analytical chemistry, and environmental analysis. [30]

Environmental Organic Chemistry is a textbook that progresses from the basic makeup of organic compounds to the interaction of these compounds in environmental systems. Major sections include the introduction to basic concepts in organic chemistry, the equilibrium between the gas, liquid and solid phases, transformation processes of organic compounds (chemical, photochemical and biological), transport phenomena (diffusion and interaction between boundaries, for example, the air-water interface) and environmental case studies combining the concepts presented throughout the book. One clear benefit to the user of this book is the extensive bibliography allowing further follow-up for the subjects covered in the book. [31]

Soil and Environmental Analysis is an updated book on analytical techniques for the analysis of environmental samples—primarily soil, but topics do include some water and gas analyses. Each chapter provides a general background on techniques and addresses several technique-specific concerns like sample preparation, sample introduction systems and specific applications of the technique within environmental analysis. [32]

Introduction to Environmental Forensics walks the reader through the early steps of an environmental forensic investigation (witness statements, historical documents, and photographic records) into the analytical measurements taken for forensic use. The majority of the book deals with the measurements and includes topics such as chemical fingerprinting of hydrocarbons, air dispersion modeling, particulate pattern recognition and statistical methods. Each chapter provides a list of references or additional reading sources at the end and the appendices contain detailed chlorinated solvent information (history, chemical names, commercial synonyms, and chemical properties). [33]

General Instrument Technique Books

A history of mass spectrometry (MS) and its developments in sample introduction and detection is given by de Laeter in the first portion of his book, *Applications of Inorganic Mass Spectrometry*. The majority of the book contains an overview of the large number of applications for the MS technique including a chapter on environmental science. de Laeter's book also has extensive references at the end of each chapter. [34]

X-Ray Spectrometry: Recent Technological Advances is an up-to-date review of progress in X-ray spectrometry (XRS); most of the references within this book are less than eight years old. It does not revisit the fundamentals of analytical chemistry techniques based on X-rays, but does include references to handbooks and text books that provide the basic information. Early chapters include details on advances and improvements in X-ray sources, optics and detector technology. Other sections are dedicated to special XRS configurations, computerized advances and developments in specific fields. [35]

According to one of the editors, R. Van Grieken, there has been a steady increase in the amount of work published on the environmental applications of XRS analysis. A section worth highlighting is the one titled "Portable Equipment for X-ray Fluorescence Analysis." The authors describe the form and function of energy-dispersive X-ray fluorescence (EDXRF) equipment and the advances of internal construction—the miniatures and lightweight solutions for X-ray tubes and detectors. The portable EDXRF systems rival laboratory-sized spectrometers in analytical capability and have the advantage of providing results "on the scene" for immediate decision-making.

Budde in *Analytical Mass Spectrometry: Strategies for Environmental and Related Applications* presents gas chromatography coupled with mass spectrometry (GC/MS) analyses as the "killer application" of mass spectrometry and the bulk of the book is dedicated to GC/MS methods. The author limits his scope to those methods used by the USEPA and the chapters separate into organic and semi-volatile organic compounds amenable to gas chromatography (separate chapters), strategies to improve analytical results, and compounds that are not amenable to GC. A brief mention of ICP/MS is near the end of the book. [36]

Method and Technique Specific Books

The "Air Analysis" group of the Deutsche Forschungsgemeinschaft (DFG), the German Research Foundation, published a tidy, well-organized source of methods for the analysis of hazardous materials in the air in several volumes. The most recent volume is #8 and among the analytical methods included are ones for atrazine (herbicide), polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and lacquer aerosols. A brief method summary with a description of the substance (CAS #, structure, physical properties and synonyms) and the data quality results (accuracy, recovery and limit of quantitation) begins each method. Methods include extensive details on equipment, chemicals and solutions, sample collection, operating conditions, sources of error and references. At the back of the book, there is a list of the methods contained in volumes #1-8. The list is in numerical order by the CAS # of the substance. [37]

The US Geological Survey (USGS) National Water Quality Laboratory has published several methods of analysis for contaminants in water samples or sediment samples in the Water-Resources Investigations Report (WRI) series. Methods include both organic and inorganic analyses. A list of the available methods and information on how to obtain these reports is provided in Appendix B.

Crompton wrote an exceptional reference book on preconcentration techniques for the analysis of organics, organometallics and inorganics (divided into cations and anions) for water samples. Each major section ends with one or more summary tables for preconcentration of specific types of compounds (hydrocarbons, PCBs, insecticides and herbicides, etc.) or within certain matrices (non-saline water, sea water, surface water, wastewater, etc.). Crompton provides references for every method and has organized this book to be a master source of information on preconcentration techniques. [38]

Substance Specific Books

Organometallic Compounds in the Environment provides analytical methods and environmental details (toxicology, pathways, fate, etc.) for organometallic compounds containing Hg, Sn, Pb, As, Sb, Si, Se, Tl, Bi, Cd, Te, Mn, Ge or Co. [39]

A hefty two-volume set titled *Handbook of Residue Analytical Methods for Agrochemicals* contains sections of compound classes and the analytical methods for each class. Some method information is specific to one compound within the particular class. In addition to the compound class section, volume two has "best practices in the generation and analyses of residues in environmental samples" including water, soil, sediment, and air samples. [40, 41]

If an environmental release of a petroleum product must be investigated, *Analytical Advances for Hydrocarbon Research* contains a variety of techniques including gas chromatography (GC), gas chromatography mass spectrometry (GC-MS), liquid chromatography mass spectrometry (LC-MS), thin-layer chromatography (TLC), and X-ray spectrometry (XRS) for the characterization of petroleum and fossil fuel samples. [42] ASTM International has published tables of test methods for products like aviation fuel, automotive gasoline, fuel oxygenates, crude oils, and more. A brief quality assurance section and a detailed sampling section (with diagrams) are included. [43]

INSTRUMENTATION AND TECHNIQUES BY TOPIC

Environmental Analysis

- Biennial review; developments in mass spectrometry as applied to environmental analysis; primarily includes work published during 2002 and 2003 [44]
- Annual review of atomic spectrometry as applied to environmental analysis; major sections are divided by the matrix (air, water, soil and plant material, and geologic materials) [45]
- Biennial review; developments in environmental analytical chemistry from 2001 and 2002; contents include sampling, extraction methods, detection techniques, analytes of interest and emerging trends [46]
- Annual review; analysis of environmental samples by atomic spectroscopy; divided into analysis categories of air, water, soils, and geologic materials [47]
- Review; covers developments in environmental mass spectrometry during 2000 and 2001 with emphasis on new and emerging contaminants and issues [48]

Sampling, Sample Preservation, Sample Preparation

- Review; addresses changes that may occur in water samples during storage and techniques for water sample preservation [49]
- Reviews factors affecting stability of inorganic mercury and methylmercury during sample storage, mechanisms for loss of mercury, and suggested treatments to decrease these losses [50]
- Review; methods of sample preparation for separation techniques; includes examples of extraction and concentration of analytes from solid, liquid, and gas matrices [51]
- Sample pretreatment using chromatomembrane cells (CMC) for extraction and preconcentration of pollutants (polycyclic aromatic hydrocarbons and extractable organic halogens) from wastewater [52]

- Addresses sample handling and analysis for resin acids in water samples; provides tables of analytical techniques (primarily gas chromatography and liquid chromatography, with various detectors) [53]

Extraction

- Comprehensive review of sequential extraction schemes (SES) for metal fractionation of environmental samples; massive applications tables of SES based on types of samples (soils, sediments, sewage sludges, etc.) [54]
- Comparison of leaching tests on various soils and sediments for the extraction of Cd, Cr, Cu, Ni, Pb, and Zn [55]
- Use of an ultrasound accelerated sequential extraction method for rapid metal partitioning profiles; study compares results from conventional extraction and ultrasound extraction on compost samples [56]
- Ultrasonic extraction (UE) as sample preparation technique for elemental analysis; extracted standard reference materials and analyzed by inductively coupled plasma atomic emission spectrometry (ICP-AES) [57]
- Comparison of ultrasound-assisted extraction to other techniques (Soxhlet, microwave-assisted, and supercritical fluid extraction) [58]
- Chelating polymeric sorbent for metal ion extraction (Mn, Pb, Ni, Co, Cu, Cd and Zn); method applied to sea water, well water, and tap water samples [59]
- Methods of extraction for isolating and preconcentrating organic analytes from environmental matrices [60]

Air Analysis

- Study of recoveries and stabilities of volatile organic compounds (VOCs) stored in fused-silica-lined (FSL) and SUMMA polished canisters; tables of recoveries under various humidified conditions and over various time intervals at a set relative humidity [61]
- System for generating gas mixtures of volatile and semi-volatile organic compounds (VOCs and SVOCs) for use as calibration standards prior to air sampling and analysis [62]
- Development of a solid-phase microextraction (SPME) technique for the analysis of odorous gases (triethylamine, propionic and butyric acids, and sulfur compounds) [63]
- Method for the sampling and analysis of airborne particulate matter (PM) from an inhaler-administered drug, spray insect repellant, and tailpipe diesel exhaust by SPME fibers and a needle trap device; the designed devices proved useful for simple and inexpensive screening and were robust enough for field sampling [64]
- Use of microwave heating for the preparation of gas standards containing mixtures of VOCs and SVOCs; method uses a domestic microwave oven and 1 liter gas-sampling bulbs [65]

Water Analysis

- Biennial review; developments in water analysis during 2001 and 2002; contents focus on new, emerging contaminants and environmental issues [66]
- Methods of analysis of methyl *tert*-butyl ether (MTBE) and *tert*-butyl alcohol (TBA) in ground and surface water samples; summarizes issues with direct aqueous injection, headspace analysis, purge-and-trap, and solid-phase microextraction [67]
- Review; analysis for organic contaminants in sea water samples; includes table of analytes, pre-treatment, analysis technique, and limit of detection [68]

- Brief review of methods for the determination of organochlorine compounds in wastewater discharge samples [69]
- Fast, inexpensive screening method for heavy metal contamination in water samples [70]
- Sample screening method for benzene, toluene, ethylbenzene, and xylenes (BTEX) in water using a headspace sampler with a mass spectrometer [71]

Pesticides, Herbicides, Insecticides

- Method for determining pesticides in environmental ground and surface water using solid-phase extraction (SPE) with liquid chromatography (LC) electrospray (ESI) tandem mass spectrometry (MS-MS) [72]
- Methods and instrumentation used to detect substituted urea compounds used as herbicides and insecticides; techniques include gas chromatography, liquid chromatography (and coupling of both to mass spectrometry detectors) and capillary electrophoresis (CE) [73]
- Solid-phase extraction of sulfonylurea herbicides in water and soil samples using a molecularly imprinted polymer (MIP) [74]
- Study of pyrethroid insecticide tralomethrin and its transformation product, deltamethrin, in gas chromatographic (GC) pesticide residue analysis; separation and identification of compounds were possible using LC-MS [75]
- Determination of organophosphorus pesticides in aqueous samples by SPME and GC with flame photometric detection (FPD) [76]
- Comparison of different SPME fiber coatings for the extraction and analysis of organochlorine pesticides in groundwater; optimized methods for each fiber type were applied to the analysis of polluted groundwater samples [77]
- Determination of residues of eight selected herbicides (trifluralin, butachlor, pretilachlor, metolachlor, atrazine, acetochlor, alachlor, and fluroxypyr-meptyl) in groundwater samples by SPME [78]
- Comparison of six organic solvents for the GC analysis of pesticides; study emphasized the stability of selected pesticides in a given solvent [79]

Polychlorinated & Polybrominated Compounds

- Review of analytical methods for the determination of brominated flame retardants (BFRs) with an emphasis on polybrominated diphenyl ethers (PBDEs); sample pre-treatment, extraction, cleanup, chromatographic separation, detection methods, and quality control are among the topics discussed [80]
- Application of SPME to the analysis of BFRs in water samples; method uses GC tandem MS for quantification [81]
- Use of two-dimensional gas chromatography (GC x GC) to separate and identify the 209 PCBs [82]
- Study of the effects from solvent and temperature to the various extraction methods for removing PCBs and polychlorinated dibenzodioxins (PCDDs) from fly ash [83]
- Method for the determination of polychlorinated dibenzo-p-dioxins (PCDDs), dibenzofurans (PCDFs), and biphenyls (PCBs) using GC-MS; the use of different reagent gases is studied [84]

Speciation

- Application of liquid chromatography (LC) coupled with inductively coupled plasma-mass spectrometry (ICP-MS) in field of elemental speciation; a few key elements for speciation (As, Se, Cd) are highlighted [85]
- Review of applications and instrumentation for speciation analysis; focus is on ICP-MS and electrospray ionization mass spectrometry (ESI-MS) [86]
- As speciation using classical ion-exchange column chromatography; presents strategies for separation and pre-concentration of arsenic species [87]
- Sb speciation problems and progress; table of Sb speciation from water, soil, and extracts using high performance liquid chromatography (HPLC) separation [88]
- Review; trace metal speciation in environmental studies using sector field inductively coupled plasma mass spectrometry (ICP-SFMS) and multicollector inductively coupled plasma mass spectrometry (MC-ICP-MS) [89]
- Review of As speciation analysis using HPLC coupled to ICP-MS [90]
- Review of developments in GC-plasma interfaces for use in elemental speciation; advantages and disadvantages of GC-plasma interfaces, types of plasmas and mass spectrometers, and variants of plasma sources are discussed [91]
- Review of modern sample preparation techniques used in speciation analysis that reduce waste generation and minimize environmental hazards [92]
- Comparison of extraction methods for arsenic speciation analysis; river sediment, sewage sludge, and agricultural soil reference materials were used in the study; analyses were completed by high-performance liquid chromatography-hydride generation-atomic fluorescence spectroscopy [93]

Field-Portable Techniques & Instrumentation

- Portable capillary electrophoresis instrument prototype; basic instrument design and three electrochemical detection methods are discussed [94]
- Use of portable X-ray fluorescence (PXRF) instrumentation in analysis of environmental samples; includes tables comparing PXRF analysis of certified reference materials and comparing performance of PXRF and laboratory ICP-AES results [95]
- Use of SPME for sampling environmental contaminants in air, water, and soil for analysis by GC-MS in the field [96]
- Design test for the next generation of field-portable and low-cost GC-MS; the design combined a small gas chromatograph with a compact double-focusing mass spectrometer (CDFMS) as the detector [97]
- Comparison of organic vapor analyzers (OVAs) with either a flame ionization detector (FID) or a photoionization detector (PID) to the analysis of soils contaminated with diesel fuel [98]
- Development of a portable laser-induced plasma spectrometer; field results on steel-scrap samples were compared to laboratory X-ray fluorescence (XRF) results [99]
- Report on prototype portable GC for use in the determination of complex vapor mixtures; analysis was performed on a 30-vapor mixture and performance compared to indoor air quality monitoring applications [100]
- Enhancement of selectivity in field-portable high-speed GC analysis for organic vapors using a thicker film in the nonpolar column [101]
- Development of a battery-operated, miniature, cylindrical ion trap (CIT) mass spectrometer that maintains laboratory-scale instrument functionality and performance [102]

- Use of a portable in-situ spectrophotometric analysis system for the measurement of copper in coastal waters [103]
- Overview of field-portable GC-MS analytical systems and applications to environmental and forensic analyses [104]
- Development of a fast gas chromatograph coupled to a time-of-flight mass spectrometer (GC-TOFMS) for portable field use [105]

Atomic Spectrometry

- Annual review; atomic spectrometry update on chemical analysis of environmental samples; extensive table summarizes applications by analyte, matrices (soils, plants, sediments, etc.), and technique [106]
- Annual review; new developments in atomic emission (AE), atomic absorption (AA), atomic fluorescence (AF), and related techniques [107]
- Annual review of novel developments and trends in atomic emission, atomic absorption, and atomic fluorescence spectrometry [108]
- Annual review of trends and developments in atomic emission, absorption, and fluorescence spectrometry [109]
- Review of advances in atomic emission, absorption, and fluorescence spectrometry and related techniques [110]
- Biennial review of atomic spectroscopy developments published from January 2002 to December 2003; includes AA, AF, AE, ICP-MS, and glow discharge (GD) [111]
- Biennial review of new developments in atomic absorption, atomic fluorescence, atomic emission, glow discharge atomic spectrometry and inductively coupled plasma-mass spectrometry from October 1999 to October 2001; primarily new applications and methodology [112]
- Review of recent advances in the hydride-generation technique for analysis by atomic emission spectrometry (AES) [113]

Atomic Absorption Spectrometry (AAS)

- Comparison of analytical performance of atom trapping systems and atomization techniques for flame atomic absorption spectrometry (FAAS) [114]
- Review; developments and trends in sample pretreatment for electrothermal atomic absorption spectrometry (ETAAS); coupling of flow injection (FI) for analyte separation and preconcentration [115]
- Procedure for determining Cu, Ca, and Cr in interferents using pulsed peristaltic pumps and Fourier transforms [116]

Capillary Electrophoresis (CE)

- Review of applications of capillary electrophoresis for detection and determination of pesticides in formulations, environmental samples, and for chiral separations; includes comparison table of detection limits for different detectors [117]
- Review for determining pesticide residues in environmental matrices; provides overview of current developments and emerging techniques (CE-MS); includes analyte and sample preparation methods table and provides comparison of CE to gas chromatography (GC) and liquid chromatography (LC) pesticide-residue analysis techniques [118]
- Review; use of CE in the speciation of metal ions; includes table of metal ions and analysis conditions [119]

- Developments in sample preparation for capillary electrophoresis including clean-up and concentration methods [120]
- Reviews sensitivity enhancements for CE and gives several examples of water sample analysis for both inorganic and organic analytes [121]

Gas Chromatography (GC)

- Biennial review of developments in gas chromatography including published work from 2002 and 2003, primarily; sections are divided into reviews and general interest, column principles and technology, high-speed and portable GC, and detectors [122]
- Biennial review of developments in gas chromatography (articles from 2000 and 2001); contents include high-speed and portable GC, detectors, column technology, and general interest publications [123]
- Review of the use of GC in identification and quantification of environmental pollutants; pollutant groups covered include volatile organic compounds (VOCs), polycyclic aromatic hydrocarbons (PAHs), pesticides, and halogenated compounds [124]
- Two-dimensional gas chromatography (GC x GC) for separation and identification of organic compounds in environmental samples [125]
- Two-dimensional gas chromatography (GC x GC) and recommended column combinations for separating polychlorinated biphenyls (PCBs) [82]
- Use of two-dimensional GC coupled to time-of-flight mass spectrometry for the improved analysis of polychlorinated biphenyl (PCB) congeners [126]
- Overview of the two-dimensional gas chromatographic technique [127]
- Data interpretation for two-dimensional gas chromatographic separations [128]
- Use of pressurized liquid extraction (PLE) and two-dimensional gas chromatography as a broad screening method for PAHs in soil [129]
- Overview of fast GC with table of “speeding up” options and the practical aspects related to the reduction in analysis time [130]
- Review; developments in coupled-column GC for the analysis of environmental toxins such as dioxin, PCBs, and PAHs [131]

Gas Chromatography-Atomic Emission Detection (GC-AED)

- Brief overview of GC-AED technique including applications, performance issues, and extended hyphenation options [132]
- Simultaneous determination of polychlorinated organic compounds using GC with micro-electron capture detector (μ-ECD) and micro-atomic emission detector (μ-AED); mixtures of organochlorine pesticides and PCBs were analyzed [133]
- Analysis of river sediments and sewage sludges by flash pyrolysis coupled with gas chromatography and atomic emission detection (PyGC-AED); results from comparison analyses made using flash pyrolysis coupled with gas chromatography and mass spectrometry (PyGC-MS) are provided; river sediments were collected from rivers known to be polluted by agricultural and industrial activities and sewage sludges were taken from industrial and urban wastewater treatment plants and a lagoonal purification system [134]
- Use of GC-AED with a microwave-induced helium plasma for speciation of Hg, Sn, and Pb compounds; includes large summary of GC-AES methods for determining inorganic mercury and organomercury species in a variety of sample matrices [135]

Gas Chromatography-Inductively Coupled Plasma-Mass Spectrometry (GC-ICP-MS)

- Review article; advances in gas chromatography (GC) coupled with inductively coupled plasma mass spectrometry (ICP-MS) [136]
- Application note about the use of GC-ICP-MS for metal speciation in environmental samples [137]

Gas Chromatography-Mass Spectrometry (GC-MS)

- Simultaneous determination of semi-volatile organic compounds (SVOCs) in air samples by GC-MS; two combined disk-type filters used as adsorbents for air sampling [138]
- Analysis for PAHs and heavy metals in storm water and sediments using GC-MS [139]
- Study combining large-volume injection (LVI) to fast GC-MS in the analysis of 15 organochlorine pesticides (OCPs) in aqueous solutions; all analytes separated in less than 8 minutes [140]
- Review of analyses for metalworking fluids (MWFs) and hydraulic fluids (HFs) in oily wastewater using GC/MS with "soft" ionization and two-dimensional GC techniques [141]

High Performance Liquid Chromatography (HPLC), Liquid Chromatography (LC)

- Review of developments in liquid chromatography equipment and instrumentation from January 2000 through December 2001; topics include instrumentation, columns, detectors (elemental, optical, luminescent, electrochemical, mass spectrometry, and others), and computation [142]
- Glossary of terms used in HPLC [143]
- Determination of trimethylamine (TMA) in water samples by LC; the analyte was derivatized in a precolumn using 9-fluorenylmethyl chloroformate (FMOC) [144]
- Analysis for phenols (from river and coke plant water) and herbicides (from soil samples) by solid-phase extraction (SPE) and HPLC [145]
- Review of analysis for non-metals (carbon, sulfur, phosphorus, and halogens) using high performance liquid chromatography coupled with inductively coupled plasma (HPLC-ICP) [146]
- Precolumn derivatization method for the determination of free amines in real water samples (wastewater from different sampling sites) by HPLC with fluorescence detection; the amide intermediate, 9-(2-acridone)oxyethylcarbonylimidazole (AOCD), formed from the reaction of 9-(2-hydroxyethyl)acridone and N,N'-carbonyldiimidazole, reacts with free amines in the presence of a base catalyst, 4-(dimethylamino)pyridine [147]
- Review of the use of HPLC in metal speciation (As, Se, Pb, Hg, Sn, and Cr); includes sections on sample handling and different chromatographic modes [148]

Ion Chromatography (IC)

- Overview of use of ion chromatography (IC) for the determination of inorganic ions in drinking water; includes table of regulatory methods and analytes [149]
- Basic principles, methods, and applications of ion chromatography [150]
- Comparison of IC detection techniques; includes table rating characteristics of each technique [151]
- Approaches to sample pretreatment to overcome analysis problems from difficult matrices [152]

- Coupling ion chromatography with ICP-AES and ICP-MS; highlights advantages and applications in sample preconcentration, elimination of interferences, and speciation analysis [153]

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES), Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

- Review on matrix effects during ICP-AES sample analysis; addresses the effects from easily ionized elements to the plasma and the analytical signal; includes a section on correction methods [154]
- Preconcentration and determination of Cd, Hg, Ag, Ni, Co, Cu and Zn in water and sediment samples using a chelating resin and ICP-AES [155]
- Determination of total metals with discrimination of chemical forms present in a sea water matrix; use of solid-phase extraction for separation and preconcentration prior to ICP-AES sample analysis [156]
- Method for studying long-term stability in ICP-AES [157]

Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

- Biennial review; developments in ICP-MS from October 2001 to October 2003; topics include sample introduction, spectroscopic interferences, and isotope ratios [158]
- Biennial review; developments in ICP-MS from October 1999 to September 2001; several tables with selected methods such as chemical vaporization, electrothermal vaporization (ETV), and speciation are provided [159]
- Use of a modified Burgener parallel path nebulizer for vapor generation of noble and transition metal species through reaction with tetrahydroborate [160]
- Comparison of on-line preconcentration columns in sequential injection analysis (SIA) for the determination of metals in sea water (Al, As, Co, Cu, Mn, Mo, Ni, Pb and V) by ICP-MS [161]
- Trace metal determination (Cr, Mn, Fe, Co, Ni and Cu) in saline water using a flow injection on-line precipitation system coupled to ICP-MS [162]
- Overview of “practical aspects” of environmental analysis (measurement parameters and analytical parameters) by ICP-MS [163]
- Determination of hexavalent chromium in ambient air with analysis using ICP-MS [164]
- Analysis for Sn in soil samples using potassium hydroxide fusion followed by continuous hydride generation coupled to ICP-MS [165]
- Overview of the electrothermal vaporization (ETV) technique for ICP-MS [166]

Infrared (IR) Spectrometry

- Analytical technique for the simultaneous determination of Sb, As, and Sn in aqueous samples; coupled flow analysis (FA) hydride generation (HG) and Fourier transform infrared (FTIR) spectrometry system [167]
- Quantitative determination of various alkaline and alkaline earth metals in aqueous solutions using Mid-FTIR spectroscopy [168]
- Quantitative method for the determination of ion concentrations of aqueous polyatomic anions using attenuated total reflectance (ATR) FTIR spectroscopy [169]

Liquid Chromatography-Mass Spectrometry (LC-MS)

- Review of applications of liquid chromatography coupled with mass spectrometry (LC-MS) using atmospheric pressure ionization in water analysis [170]
- Summary of problems using LC-MS for qualitative and quantitative analysis of organic compounds in water [171]
- Determination of trace amounts of specific antibiotics in natural waters and wastewater using SPE combined with high performance liquid chromatography-ion trap tandem mass spectrometry (LC-MS-MS) [172]
- Method for the determination of halogenated and non-halogenated nonylphenols and nonylphenol carboxylates using liquid chromatography tandem mass spectrometry in water and sludge samples from a drinking water treatment plant [173]
- Summarizes sources of error in HPLC-MS analyses and suggests solutions [174]
- Analysis for emerging environmental contaminants using LC/TOF/MS [175]
- Analysis for fluorinated surfactants in spiked sludge samples using LC-MS-MS after extraction and separation [176]

Liquid-Phase Microextraction (LPME)

- Developments in liquid-phase microextraction (LPME) based on disposable hollow fibers; the technique principles, set-up, applications, and trends for the future are discussed [177]
- Use of LPME coupled to GC-MS to analyze for pesticides in soil samples [178]
- Developments in liquid-phase microextraction; includes details on some environmental applications (polycyclic aromatic hydrocarbons, herbicides, pesticides, etc.) [179]
- Determination of trihalomethanes (THMs) such as chloroform, dichlorobromomethane, chlorodibromomethane, and bromoform in drinking water samples (tap water and well water) using headspace liquid-phase microextraction (HS-LPME) with gas chromatography-electron capture detection (GC-ECD) [180]

Microwave-Assisted Extraction (MAE) & Microwave-Enhanced Chemistry (MEC)

- Overview of microwave-based devices used in solid sample pretreatment; includes both closed and open, multi-mode and focused-microwave systems [181]
- Extraction and determination of selected endocrine disrupting chemicals (EDCs) in river sediments by microwave-assisted extraction followed by gas chromatography mass spectrometry (GC-MS) [182]
- Study of a microwave digestion technique for the analysis of metals (Ca, Mg, Fe, Mn, Zn, Cr, Cd, Cu, Pb, and V) in sediments and soils; samples analyzed included standard reference materials (SRMs) of sewage sludge, marine sediment, urban particulate matter, and coal carbonization site soil [183]
- Review of sample extraction and digestion procedures using focused-microwave radiation; includes tables of focused-microwave-assisted extractions of organic, inorganic, and organometallic compounds and of focused-microwave-assisted digestions of organic and inorganic samples [184]
- Use of focused-microwave (FM) irradiation to improve upon conventional Soxhlet extraction; includes section on the use of FM-assisted Soxhlet extraction (FMASE) in the analysis of environmental pollutants (polycyclic aromatic hydrocarbons, alkanes, herbicides, metals, etc.) [185]

- Review of extraction and digestion procedures assisted by focused-microwave radiation; applications presented include inorganic, organic, and organometallic analyses in a variety of sample types [184]
- Comparison of microwave-assisted total digestion to aqua regia and nitric acid digestions for the determination of heavy metal content in environmental samples (sediments, soils, sludges, and plant materials) [186]
- Study of three microwave digestions for the determination of metals in sludge, soil, and sediment samples; metals analysis performed by ICP-AES [187]
- Determination of 17 chlorophenolic compounds in ash samples obtained from different incineration plants; a microwave system with closed extraction vessels was used in the sample preparation [188]
- Digestion of organic samples using microwave-assisted sample combustion; cadmium and copper were determined in the samples using electrothermal atomic absorption spectrometry [189]
- Study of the thermal degradation of five carbamates (propoxur, thiuran, propham, methiocarb, and chlorpropham) in microwave-assisted extraction followed by HPLC analysis [190]

Mass Spectrometry (MS)

- Annual review of atomic mass spectrometry; review focuses on significant developments in instrumentation and methodology and an improved understanding of the fundamental phenomena of MS [191]
- Annual review of atomic mass spectrometry; developments in instrumentation and methodology or fundamental principles of MS [192]
- Annual review of developments in atomic mass spectrometry [193]
- Structural and chemical characterization of compounds in complex environmental mixtures using electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry (ESI FT-ICR MS) [194]
- Brief comparison of GC-MS and LC-MS [195]
- Development of a mass spectrometer with simultaneous detection using a Faraday cup-based detector called the focal plane camera (FPC) [196]
- Direct coupling of SPME with mass spectrometry to make a new analytical technique, fiber introduction mass spectrometry (FIMS); analysis of VOCs, SVOCs, and two herbicides using the new technique is discussed [197]

Nuclear Magnetic Resonance Spectroscopy (NMR)

- Use of high performance liquid chromatography coupled with nuclear magnetic resonance spectroscopy (HPLC-NMR) in environmental analysis as a method of identifying components of complex mixtures; references work using HPLC-NMR for the analysis of environmental contaminants [198]
- Study of two techniques used for the analysis of perfluorinated surfactants in water samples–^{19}F NMR and LC-MS-MS; techniques complimented each other for sample analysis and the methodology is applicable to other sample matrices [199]
- Use of NMR in hyphenated techniques with HPLC and MS with potential of separating complex mixtures of pharmaceuticals or drug metabolites [200]

Pressurized Fluid Extraction (PFE), Pressurized Liquid Extraction (PLE), Accelerated Solvent Extraction (ASE)

- Overview of the accelerated solvent extraction (ASE) technique for the extraction of environmental pollutants from solid samples; presents basics of technique and comparison to other extraction techniques [201]
- One-step cleanup and extraction method for the analysis of polycyclic aromatic hydrocarbons (PAHs) using pressurized liquid extraction (PLE) [202]
- Study of efficiency of a pressurized liquid extraction (PLE) system for the isolation of organic pesticides and polychlorinated biphenyls (PCBs) under varying pressure and temperature conditions [203]
- Pressurized liquid extraction of veterinary antibacterial agents from soil samples followed by SPE and LC-MS analysis [204]

Solid-Phase Extraction (SPE)

- Reviews use of solid-phase extraction for separation and sensitive determination of metal ions (primarily in water samples); includes tables of on-line and off-line SPE applications with details on matrix, trace elements, experimental conditions and methods [205]
- Innovative development of molecularly imprinted polymers (MIPs) for use in solid-phase extraction (SPE) to selectively extract analytes from complicated matrices; addresses analytes of environmental interest [206]
- Comparison of extraction techniques–solid-phase extraction (SPE) and solid-phase microextraction (SPME)–in the analysis for short-chain chlorinated paraffins (SCCPs) in tap and river water samples using GC coupled to negative chemical ionization mass spectrometry (GC-NCI-MS) [207]
- Sample clean-up and separation by sequential solid-phase extraction (SSPE) for analysis of polar pollutants in water samples; analyses of samples from wastewater treatment plants (WWTPs), rivers, and streams were performed using LC-ESI-MS [208]
- Comparison of SPE materials for the removal of polar compounds from spiked water samples; phenolic compounds and organochlorine pesticides were studied [209]

Solid-Phase Microextraction (SPME)

- General overview of SPME technique [210]
- Comparison of the extraction efficiencies of five different SPME fiber coatings in the analysis of 52 pesticides and PCBs [211]
- Derivatization and SPME of organotin and organolead compounds in aqueous samples with analysis by gas chromatography atomic emission detection (GC-AED); derivatization is carried out *in situ* using sodium tetrapropylborate [212]
- Rapid procedure for determining butyltin species in sediments by SPME; analysis was performed on a certified reference material (CRM-462) and coastal sediment samples [213]
- Direct headspace SPME method for the determination of polynuclear aromatic hydrocarbons (PAHs) in atmospheric particulate matter; results showed potential for using method as a screening tool [214]
- Simple and fast procedure for the analysis of aldehydes in water using SPME technique with on-fiber derivatization; quality of results were evaluated using spiked bidistilled water, chlorinated tap water, and well water [215]
- Determination of tributyltin (TBT) in water and sediment by automated in-tube SPME and HPLC coupled to a quadropole mass spectrometer with electrospray ionization [216]

- Use of SPME and GC-MS to characterize odorant emissions from a landfill; demonstrated use as an on-site analysis tool [217]
- Comparison of SPME with stir bar sorptive extraction (SBSE) in the extraction of semi-volatile compounds (polycyclic aromatic hydrocarbons, organochlorines) in water; post-extraction analysis was performed using GC-MS [218]
- Analysis for BTEX in water samples using headspace solid-phase microextraction (HS-SPME) with GC-FID [219]

X-ray Spectrometry (XRS)

- Review of developments and improvements in X-ray spectrometry published during 2002 and 2003 [220]
- Annual review of X-ray fluorescence (XRF); developments during 2002 and 2003 in instrumentation and detectors, trends in analysis, and a range of applications including environmental [221]
- Annual review of developments in instrumentation and applications of X-ray fluorescence including environmental applications [222]
- Annual review; developments during 2000 and 2001 in X-ray fluorescence; contents include instrumentation and detectors, optics, portable and mobile XRF, and applications of XRF [223]
- Biennial review of developments in XRS and related fields (from late 1999 to the end of 2001); topics include those of detection, instrumentation and optics, and several applications [224]
- On-line preconcentration flow system for the simultaneous determination of Co, Cu, Mn, Ni, Pb and Zn by total reflection X-ray fluorescence (TXRF) spectrometry; improvements in detection limits; system was applied to river water samples [225]

SPECIAL TOPIC – ENDOCRINE DISRUPTING COMPOUNDS (EDCS)

Although not a consideration for the environmental crime field at this time, concern over endocrine disrupting compounds (EDCs–also known as "endocrine disrupting chemicals") in the environment has been increasing. Endocrine disruptors have been defined as "exogenous agents that interfere with the production, release, transport, metabolism, binding, action, or elimination of the natural hormones in the body responsible for the maintenance of homeostasis and the regulation of developmental processes." [226] Research to identify compounds that may interfere with hormonally-controlled body systems has been increasing for both governmental and private research entities.

Currently, the USEPA is developing methods for the screening and testing of thousands of contaminants with the potential to disrupt the endocrine system. [227] USEPA, as tasked by the Federal Food, Drug and Cosmetic Act (FFDCA), developed the Endocrine Disruptor Screening Program (EDSP) to "screen pesticide chemicals and environmental contaminants for their potential to affect the endocrine systems of humans and wildlife." [228] When complete, the EDSP will provide validated methods for screening drinking water sources for EDCs.

Several other countries have taken steps to define the EDC problem and pursue solutions. The United Kingdom Environment Agency has presented a position on phamaceuticals in watercourses (prioritizing some of these pharmaceuticals for monitoring) and a strategy paper on EDCs in the environment. [229, 230] Environment Canada also has a national strategy regarding endocrine disruptors in the environment. [231]

The Australian Government Department of the Environment and Heritage has made available an EDC information paper wherein parties "agreed on the need to investigate, in depth, the human, environmental and ecotoxicological aspects of endocrine disrupting substances." [232] Deustche Forschungsgemeinschaft (DFG) has established research training group 546 for the "elimination of endocrine-disrupting substances from waste water." [233]

The Japan Environment Agency has summarized on-line the plans for investigations, research, testing, and method development for SPEED '98–Strategic Programs on Environmental Endocrine Disruptors '98. [234] The Danish Environmental Protection Agency has provided on-line an "Evaluation of Analytical Chemical Methods for the Detection of Estrogens in the Environment." This document provides environmental and chemical properties of estrogens, analytical methods and applications to estrogens, and recommendations. The document also contains active links to tables and numerous references. [235]

METHODS FOR THE IDENTIFICATION OF ENDOCRINE DISRUPTORS

Pharmaceutically Active Compounds

(antibiotics, anti-inflammatories, antibacterial agents, etc.)

- Investigates the presence of sulfadimidine (sulfamethazine), an antibiotic used for animals, in liquid manure and soil samples; surface water samples from river banks were extracted and analyzed by LC–MS/MS for more than 20 antibiotics used in both animal and human therapy [236]
- Review of pharmaceutical antibiotic compounds in soils with an extensive section on fate studies [237]

- Analysis for the nonsteroidal anti-inflammatory drugs (NSAIDs) ibuprofen and 2-(4-chlorophenoxy)-2-methylpropionic acid in wastewater; method used reversed-phase high performance liquid chromatography (RP–HPLC) combined with a simple, fast, and inexpensive two-step liquid-liquid-liquid microextraction (LLLME) [238]
- Analysis for NSAIDs in water samples using solid phase microextraction (SPME), on-fiber silylation (derivatization), and GC/MS determination; the SPME method was compared to the use of solid phase extraction (SPE) for analysis of ibuprofen and naproxen in the influent and effluent from a sewage water treatment plant [239]
- Method for the determination of the fluoroquinolines ciprofloxacin and norfloxacin (antibacterial agents) in sewage sludge and sludge-treated soil samples; samples were extracted using accelerated solvent extraction (ASE); extracts were cleaned using SPE and analyzed by liquid chromatography fluorescence detection (LC-FLD) [240]
- Extracts from sewage treatment plant wastewater and from surface water were analyzed for carbamazepine (a drug used in the treatment of epilepsy, schizophrenia, bipolar disorders and more) and five metabolites of carbamazepine using electrospray LC-MS/MS with selected reaction monitoring [241]
- Determination of clofibric acid using acid and base/neutral liquid-liquid extraction, derivatization with trimethylsilyldiazomethane (TMSD) and analysis by GC/MS [242]
- Analysis for polar pharmaceuticals by LC-MS without derivatization; samples of river sediment were spiked with standards and prepared by ultrasonicated solvent extraction and solid phase extraction (SPE) prior to analysis [243]
- Simultaneous determination of acidic and neutral pharmaceuticals in wastewater using high-performance liquid chromatography photochemically induced fluorimetry (HPLC-PIF) providing a clean, fast and inexpensive on-line post-column photoderivatization procedure; SPE clean-up was used on the sewage water sample [244]
- Analysis for nine neutral pharmaceuticals in river water and wastewater samples (influent and effluent from municipal sewage treatment plants) by liquid chromatography electrospray tandem mass spectrometry (LC–ES–MS–MS) with SPE enrichment [245]
- Details six different analytical methods that (when totaled) allow for the analysis of nearly 80 pharmaceutical compounds and phenolic antiseptics [246]
- Brief report on proficiency test results for pharmaceuticals in river water and wastewater; both GC/MS and LC-MS/MS methods were used by participants for the analyses [247]

Pharmaceutical and Personal Care Products (PPCPs)

- Study of the removal of selected pharmaceutical and personal care products (PPCPs) through wastewater treatment processes; includes results from analyses of sewage treatment works influent and effluent samples and the methodology used in analyzing for five different groups of PPCPs [248]
- Method for detection of PPCPs that have a structure conducive to fluorescence using capillary zone electrophoresis - laser induced fluorescence (CE-LIF); the determination of salicylic acid in sewage effluent is presented [249]
- Review of investigations of musk residues in sewage, sewage sludge, surface water, aquatic sediment, and biota [250]

Steroid Hormones

- Analysis for steroid hormones (gestagens, androgens, and estrogens) in drinking water and groundwater; samples were extracted (SPE) and derivatized prior to GC/MS analysis in selected ion monitoring (SIM) mode [251]

- Quantitation of estrogens in groundwater samples and swine lagoon samples; extracts were derivatized with pentafluorobenzyl bromide (PFBBR) and *N*-trimethyl-silylimidazole (TMSI) then analyzed using negative ion chemical ionization (NICI) gas chromatography tandem mass spectrometry (GC-MS-MS) [252]
- Analysis for estrogens in surface water and wastewater treatment plant samples; samples were extracted using SPE, derivatized using PFPA (pentafluoropropionic acid anhydride), and analyzed by GC/MS [253]
- SPE-GC-MS method for the analysis of steroids and phenolic compounds in water samples; changes in the method parameters were made to test the effects on the recovery of the endocrine disrupting compounds (EDCs); parameters included different types of SPE cartridges, elution solvent, salt concentration, pH, and humic acid concentration [254]
- Analysis of sewage treatment plant influent, effluent and river water for selected estrogens, phytoestrogens, mycoestrogens and alkylphenols by LC-MS-MS; optimization of instrument conditions for the EDC groups is detailed including results from electrospray ionization (ESI) and atmospheric pressure chemical ionization (APCI) modes, negative ion (NI) and positive ion (PI) modes, and mobile phase compositions [255]
- Determination of chlorinated and brominated derivatives of alkylphenol ethoxylate (APEO) surfactants (industrial cleaning agents that mimic endogenous hormones) in sludges, waters and sediments; extracts from samples were analyzed using reversed phase LC/MS with a comparison of APCI and ESI interfaces [256]
- Analysis for steroid hormones in wastewater treatment plant (WWTP) effluent using liquid chromatography tandem mass spectrometry (LC-MS-MS); samples were extracted by SPE and cleaned with a liquid-liquid separation followed by a Florisil cartridge [257]
- Presents several methodologies for separation and analysis of steroid estrogens in sludge, sediment, sewage effluent and surface water; details on the methods are summarized in a flowchart and table; biological methods for determining the endocrine-disrupting activity of a chemical are also discussed [258]

General EDC Analytical Methods

- Analysis of samples from 139 streams for pharmaceuticals, hormones, and other organic wastewater contaminants using targeted methods and selected ion monitoring (SIM) for improved sensitivity [259]
- Use of a liquid chromatography tandem mass spectrometry (LC-MS/MS) method for analysis of pharmaceuticals, steroids, and personal care products; method used one solid-phase extraction (SPE) procedure and ESI (in positive and negative modes) and APCI (in positive mode) [260]
- Comparison of separatory funnel liquid-liquid extraction to on-line continuous liquid-liquid extraction in the analysis of several EDCs in water samples; data was collected in both total ion chromatogram (TIC) mode and selective ion monitoring (SIM) mode of GC/MS [261]
- Evaluation of matrix effects on 35 endocrine disrupting chemicals for the analysis of environmental water samples by LC-ESI-MS/MS; efficient sample clean-up and the use of internal standards showed reduced matrix effects regardless of the type of environmental water sample (surface water, rain water, groundwater, channel water, wastewater treatment plant effluents and industrial effluents) [262]

Appendix A

MAJOR ENVIRONMENTAL LAWS OF THE UNITED STATES ENVIRONMENTAL PROTECTION AGENCY (USEPA)

Environmental Law	Summary
Federal Food, Drug, and Cosmetic Act (FFDCA)	Set safety and quality requirements for food, cosmetics, drugs and therapeutic devices. Allows EPA to establish safe tolerance levels for pesticide chemical residues present in raw or processed foods. [263]
Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA)	Provided federal control of pesticide distribution, sale, and use. EPA was given authority to study the results of pesticide use and to require users to register when purchasing pesticides. [264,265]
Federal Water Pollution Control Act a.k.a. Clean Water Act (CWA)	Established the basic structure for regulating discharges of pollutants into the waters of the United States and set water quality standards for all contaminants in surface waters. Gave EPA the authority to implement pollution control programs. [266, 267]
Clean Air Act (CAA)	The comprehensive federal law that regulates air emissions from area, stationary, and mobile sources. EPA was authorized to establish National Ambient Air Quality Standards to protect public health and the environment. [268, 269]
National Environmental Policy Act (NEPA)	Early environmental law that established a broad national framework for protecting our environment by requiring that all branches of government consider the environmental impact prior to undertaking any major federal action that might significantly affect the environment. Environmental Assessments and Environmental Impact Statements are required when airports, buildings, military complexes, highways, park land purchases, and other federal activities are proposed. [270, 271]
Safe Drinking Water Act (SDWA)	Established to protect the quality of drinking water and focused on all waters actually or potentially designed for drinking use (above ground or underground sources). [272]
Toxic Substances Control Act (TSCA)	Gave EPA the ability to track the 75,000 industrial chemicals currently produced or imported into the United States. EPA repeatedly screens these chemicals, can require reporting or testing of potentially hazardous chemicals and can ban the manufacture and import of chemicals that pose an unreasonable risk. Mechanisms also exist to track newly developed chemicals with unknown or dangerous characteristics. [273-275]
Resource Conservation and Recovery Act (RCRA)	RCRA (pronounced "RICK-rah") gave EPA the authority to control hazardous waste from the "cradle-to-grave" and included the generation, transportation, treatment, storage, and disposal of hazardous waste. The tracking of hazardous waste through this act only involves active and future facilities. A framework for the management of non-hazardous wastes was also established with this act. [276, 277]
Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund)	CERCLA (pronounced “SIR-cla”) created a tax on chemical and petroleum industries and provided broad Federal authority to respond directly to releases or threatened releases of hazardous substances to the environment. The tax went to a trust fund, the “Superfund,” for paying the costs of cleaning up abandoned or uncontrolled hazardous waste sites as well as accidents, spills and emergency releases of pollutants. Through CERCLA, EPA was authorized to identify parties responsible for any type of release and enjoin their participation in the cleanup or recover costs involved in the cleanup. [278-280]

Environmental Law	Summary
Superfund Amendments and Reauthorization Act (SARA)	Amended CERCLA to increase State involvement, increase the size of the trust fund, increase focus on human health problems and revise the Hazard Ranking System to accurately assess the relative degree of risk to human health and the environment from uncontrolled hazardous waste sites. [280-282]
Emergency Planning and Community Right-To-Know Act (EPCRA)	As part of SARA, EPCRA was enacted to help local communities protect public health, safety, and the environment from chemical hazards. Each state was required to appoint a State Emergency Response Commission, divide the states into Emergency Planning Districts and name a Local Emergency Planning Committee for each district. [283, 284]
Pollution Prevention Act (PPA)	Focused industry, government, and public attention on reducing pollution through cost-effective changes in production, operation, and raw materials use. Efforts to reduce pollution prior to the need for treatment or disposal are referred to as "source reduction." Pollution prevention also includes conservation and practices that increase efficiency in the use of energy, water, or other natural resources. [285, 286]
Oil Pollution Act (OPA)	Required oil storage facilities and vessels to submit plans detailing how they will respond to large discharges, established a tax on oil to finance a trust fund for spill cleanup costs, and required the development of contingency plans for oil spill response. EPA published regulations dealing with above ground oil storage facilities. [287, 288]
Endangered Species Act (ESA)	Provided a program for the conservation of threatened and endangered plants and animals and their habitats. EPA approval to register a pesticide is based in part on the risk of adverse effects on endangered species and habitats. Under FIFRA, EPA can issue emergency suspensions of certain pesticides (cancel or restrict the use) if an endangered species will be adversely affected. [289, 290]

Appendix B

USGS NATIONAL WATER QUALITY LABORATORY METHODS OF ANALYSIS

Water-Resources Investigations (WRI) Report Number	Title
2003-4293	Determination of Organochlorine Pesticides and Polychlorinated Biphenyls in Bottom and Suspended Sediment by Gas Chromatography with Electron-Capture Detection [291]
2003-4174	Evaluation of Alkaline Persulfate Digestion as an Alternative to Kjeldahl Digestion for Determination of Total and Dissolved Nitrogen and Phosphorus in Water [292]
2003-4139	Determination of Organophosphate Pesticides in Whole Water by Continuous Liquid-Liquid Extraction and Capillary-Column Gas Chromatography with Flame Photometric Detection [293]
2003-4079	Determination of Gasoline Oxygenates, Selected Degradates, and BTEX in Water by Heated Purge and Trap/Gas Chromatography/Mass Spectrometry [294]
2002-4222	Determination of Organophosphate Pesticides in Bottom Sediment by Gas Chromatography with Flame Photometric Detection [295]
2002-4144	Arsenic Speciation in Natural-Water Samples Using Laboratory and Field Methods [296]
2002-4071	Determination of Organophosphate Pesticides in Filtered Water by Gas Chromatography with Flame Photometric Detection [297]
2001-4186	Determination of Wastewater Compounds by Polystyrene-Divinylbenzene Solid-Phase Extraction and Capillary-Column Gas Chromatography/Mass Spectrometry [298]
2001-4134	Determination of Pesticides in Water by Graphitized Carbon-Based Solid-Phase Extraction and High-Performance Liquid Chromatography/Mass Spectrometry [299]
2001-4132	Determination of Organic Plus Inorganic Mercury in Filtered and Unfiltered Natural Water with Cold Vapor–Atomic Fluorescence Spectrometry [300]
2001-4098	Determination of Moderate-Use Pesticides and Selected Degradates in Water by C-18 Solid-Phase Extraction and Gas Chromatography/Mass Spectrometry [301]

Methods can be purchased from the USGS through the "Publications Warehouse" on the USGS website. [302]

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