

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

NEIC

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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, though 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

U.S. EPA Headquarters Library
Mail code 3404T
1200 Pennsylvania Avenue NW
Washington, DC 20460
202-566-0556

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 2003⁴⁴
- 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 trends⁴⁶
- Annual review; analysis of environmental samples by atomic spectroscopy; divided into analysis categories of air, water, soils, and geologic materials⁴⁷
- Review; covers developments in environmental mass spectrometry during 2000 and 2001 with emphasis on new and emerging contaminants and issues⁴⁸

Sampling, Sample Preservation, Sample Preparation

- Review; addresses changes that may occur in water samples during storage and techniques for water sample preservation⁴⁹
- Reviews factors affecting stability of inorganic mercury and methylmercury during sample storage, mechanisms for loss of mercury, and suggested treatments to decrease these losses⁵⁰
- Review; methods of sample preparation for separation techniques; includes examples of extraction and concentration of analytes from solid, liquid, and gas matrices⁵¹
- Sample pretreatment using chromatomembrane cells (CMC) for extraction and preconcentration of pollutants (polycyclic aromatic hydrocarbons and extractable organic halogens) from wastewater⁵²

- 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 repellent, 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 samples⁶⁹
- Fast, inexpensive screening method for heavy metal contamination in water samples⁷⁰
- Sample screening method for benzene, toluene, ethylbenzene, and xylenes (BTEX) in water using a headspace sampler with a mass spectrometer⁷¹

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-MS⁷⁵
- 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 samples⁷⁷
- Determination of residues of eight selected herbicides (trifluralin, butachlor, pretilachlor, metolachlor, atrazine, acetochlor, alachlor, and fluroxypyr-meptyl) in groundwater samples by SPME⁷⁸
- Comparison of six organic solvents for the GC analysis of pesticides; study emphasized the stability of selected pesticides in a given solvent⁷⁹

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 discussed⁸⁰
- Application of SPME to the analysis of BFRs in water samples; method uses GC tandem MS for quantification⁸¹
- Use of two-dimensional gas chromatography (GC x GC) to separate and identify the 209 PCBs⁸²
- Study of the effects from solvent and temperature to the various extraction methods for removing PCBs and polychlorinated dibenzodioxins (PCDDs) from fly ash⁸³
- 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 studied⁸⁴

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 highlighted⁸⁵
- 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 species⁸⁷
- Sb speciation problems and progress; table of Sb speciation from water, soil, and extracts using high performance liquid chromatography (HPLC) separation⁸⁸
- 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-MS⁹⁰
- 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 discussed⁹¹
- Review of modern sample preparation techniques used in speciation analysis that reduce waste generation and minimize environmental hazards⁹²
- 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 spectroscopy⁹³

Field-Portable Techniques & Instrumentation

- Portable capillary electrophoresis instrument prototype; basic instrument design and three electrochemical detection methods are discussed⁹⁴
- 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 results⁹⁵
- Use of SPME for sampling environmental contaminants in air, water, and soil for analysis by GC-MS in the field⁹⁶
- 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 detector⁹⁷
- 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 fuel⁹⁸
- Development of a portable laser-induced plasma spectrometer; field results on steel-scrap samples were compared to laboratory X-ray fluorescence (XRF) results⁹⁹
- 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 applications¹⁰⁰
- Enhancement of selectivity in field-portable high-speed GC analysis for organic vapors using a thicker film in the nonpolar column¹⁰¹
- Development of a battery-operated, miniature, cylindrical ion trap (CIT) mass spectrometer that maintains laboratory-scale instrument functionality and performance¹⁰²

- 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 interferences 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—¹⁹F 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 techniques²⁰¹
- 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 conditions²⁰³
- Pressurized liquid extraction of veterinary antibacterial agents from soil samples followed by SPE and LC-MS analysis²⁰⁴

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 methods²⁰⁵
- 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 interest²⁰⁶
- 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-MS²⁰⁸
- Comparison of SPE materials for the removal of polar compounds from spiked water samples; phenolic compounds and organochlorine pesticides were studied²⁰⁹

Solid-Phase Microextraction (SPME)

- General overview of SPME technique²¹⁰
- Comparison of the extraction efficiencies of five different SPME fiber coatings in the analysis of 52 pesticides and PCBs²¹¹
- 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 tetrapropylborate²¹²
- Rapid procedure for determining butyltin species in sediments by SPME; analysis was performed on a certified reference material (CRM-462) and coastal sediment samples²¹³
- 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 tool²¹⁴
- 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 water²¹⁵
- Determination of tributyltin (TBT) in water and sediment by automated in-tube SPME and HPLC coupled to a quadrupole mass spectrometer with electrospray ionization²¹⁶

- 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 pharmaceuticals 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 Deutsche 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 therapy²³⁶
- Review of pharmaceutical antibiotic compounds in soils with an extensive section on fate studies²³⁷

- 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 fluoroquinolones 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*-trimethylsilylimidazole (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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ANACOSTIA REBIRTH

Restoration for
the River

Risk Reduction
for the
Community



The waters that give rise to the Anacostia River begin in the northern most reaches of Montgomery and Prince George's Counties. The Northwest and Northeast branches are free-flowing streams that join to form the tidal Anacostia River near the historic port of Bladensburg, Maryland. The tidal river flows 8.4 miles to its confluence with the Potomac River near the southern tip of the District of Columbia. Along its course, a number of smaller streams contribute directly to the tidal river, although most of these streams are now enclosed in storm sewers.

The Anacostia watershed was once a thriving center of Indian culture set amidst the Piedmont and Coastal Plain provinces in the early 17th century. The Nanchotank Indians, a semiagricultural tribe, lived at the confluence of the Anacostia and Potomac Rivers in what is now Washington, DC. Containing healthy populations of American and hickory shad, white and yellow perch, red-breasted sunfish, striped bass, catfish, and herring, the fish of the Anacostia River provided the Nanchotank Indians and others living in the surrounding region with a seemingly limitless food source. Lush forests and abundant wildlife complemented the crystal clear river that flowed into the Potomac and ultimately the Chesapeake Bay.

During the eighteenth and nineteenth centuries, large portions of the watershed were cleared for tobacco, corn, and cotton farming. Severe erosion resulted and sediment accumulated in the Anacostia. By the mid-nineteenth century, ships could no longer navigate up river to the once thriving port of Bladensburg.

Since the late nineteenth century, ecological problems in the Anacostia watershed have largely resulted from an expanding human population and the associated changes in land use and land cover. Today more than 800,000 people live in the Anacostia watershed—an average population density of 4,570 people per square mile. The ongoing loss of forest and wetland habitat, alteration of stream flow, increases in nonpoint source pollution, and discharges of combined sewer overflows and industrial waste have all contributed to the decline in the ecological health of the watershed.

A study completed in 1990 by the U.S. Army Corps of Engineers determined that the main causes of the ecological degradation of the Anacostia River were (1) lack of environmental controls during the extensive urbanization of the watershed in the twentieth century and (2) past activities of the Corps of Engineers in flood control, channelization, navigation implementation, debris removal, and aquatic-vegetation control. Between 1902 and 1960, those activities destroyed approximately 2,600 acres of wetlands, 99,000 linear feet of aquatic habitat, and 700 acres of bottomland hardwood forest in the Anacostia watershed.

Progress and planning under the

Anacostia Ecosystem Initiative



A Program of the
U.S. Environmental
Protection Agency, Region III



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LOCAL RESTORATION EFFORTS

By 1990, when the United States Army Corps of Engineers released its findings on the degradation of the Anacostia River (summarized in the text on the cover), the process of restoring the watershed was already under way. The Anacostia Watershed Restoration Committee was formed in 1987 under the authority of the Anacostia Watershed Restoration Agreement signed by the District of Columbia, Montgomery County, Prince George's County, the State of Maryland, and the U.S. Army Corps of Engineers. In 1991, the Committee adopted a plan entitled *A Commitment to Restore Our Home River: A Six Point Action Plan to Restore the Anacostia River*. That restoration initiative established six scientifically based goals for restoring and protecting the Anacostia River watershed. It emphasized the need for interagency and intercommunity cooperation in achieving the goals.

Since 1991, the Anacostia River watershed has experienced certain improvements in water quality and its ability to sustain critical habitat and aquatic life. More importantly, there has been a groundswell of environmental, public and civic group activism to clean-up the River and protect the watershed from further degradation. The Anacostia is no longer the "forgotten River." It is clearly undergoing a rebirth with local groups leading the way.

FEDERAL INVOLVEMENT

Small waterways like the Anacostia River do not often command the direct or targeted attention of Federal environmental protection programs. Their welfare is left to local initiatives, exemplified by the work of the Anacostia Watershed Restoration Committee and the public-interest groups, private citizens, and local officials that have supported it. These special problem areas often escape Federal attention when the impacted communities do not muster sufficient political clout. The combined influence of those two factors is demonstrated by the disparate attention paid at the Federal level to pollution of Washington's two main waterways prior to 1990. The millions of dollars spent on restoring and protecting the large, high-profile Potomac River and the successes achieved there stand in sharp contrast to the modest Federal resources applied to cleaning up the Anacostia River.

Eventually, the extent of the Anacostia's plight and the consequences of neglect—to the capital city's image, to the water quality of the Potomac River and the Chesapeake Bay, and to the quality of life in the communities that line the river's banks—were spotlighted in a Report to Congress prepared by the U.S. Environmental Protection Agency in 1992. Based on the recommendations in that document, the Anacostia watershed was designated by the President of the United States as one of seven ecosystems in the nation requiring priority restorative attention. Recommendations from this White House Ecosystem Management Task Force included a stronger and more coordinated Federal role in the restoration. EPA began to work in earnest with the Army Corps of Engineers and others to bring the full benefit of Federal involvement to the Anacostia watershed restoration.

In July 1994, the *Agreement of Federal Agencies on Ecosystem Management in the Chesapeake Bay* (developed by the Chesapeake Bay Program of the U.S. Environmental Protection Agency, Region III) was signed by 23 Federal agencies and departments. A line item in that document pledged the signers' full support of a Federal workplan to clean up the Anacostia in cooperation with the Anacostia Watershed Restoration Committee. That item reflected the fact that the Anacostia River is linked to the Chesapeake Bay via its outflow into the Potomac River, which in turn flows into the Bay.

Three months later, in September 1994, the Environmental Protection Agency, Region III, introduced its *Anacostia Ecosystem Initiative*. This was the culmination of some three years of work from the time the Agency first espoused the restoration of the Anacostia River watershed in the early 1990s. The Initiative would allow the Agency to sign on as a full partner in the local restoration effort.

ANACOSTIA ECOSYSTEM

U.S. Environmental Protection

Beginning with the Special Report to Congress in 1992, prepared by the Chesapeake Bay Program, the EPA began to apply its expertise in ecosystem management principles directly to the effort to strengthen the Federal presence in restoration activities. Federal participation allows for the establishment of an Urban Watershed Model that can be used in other urban areas throughout the country.

In September 1994, the EPA Region III announced the *Anacostia Ecosystem Initiative*. The four main features of the Initiative are: watershed restoration; multi-media risk reduction; environmental justice; and public education and involvement.

Activities under the Initiative can be broken down as follows:

- To identify and target compliance and enforcement activities at Federal facilities in the watershed.
- To assist in coordinating restoration activities at Federal facilities in the watershed.
- To seek greater controls over combined sewer and storm water discharges.
- To ensure equal protection from environmental hazards of the watershed community regardless of race, ethnicity, or economic status.
- To provide a full-time liaison to the Anacostia community to seek out and report local concerns and interests.

GOALS OF THE INITIATIVE

Watershed restoration

Multi-media risk reduction

Environmental justice

Public education/involvement

STEM INITIATIVE

Agency, Region III

PROGRESS TO DATE

COMMUNITY LIAISON APPOINTED

In May 1995, EPA Region III hired a community liaison for the Anacostia River restoration project. The liaison has established a base at the Kramer Middle School in Anacostia, and divides time between that location and home base at EPA Region III's Chesapeake Bay Program office in Annapolis. The liaison has regular contact with citizens, community leaders, and interest groups to maintain a two-way flow of information and ideas associated with the day-to-day and long-term aspects of the restoration project. The liaison also assists in delivering environmental science education to the District of Columbia's school children.

EPA REGION III BECOMES A FORMAL PARTNER

In May 1996, the relationship between EPA Region III and the local groups working on the Anacostia River's restoration was formalized in a *Memorandum of Understanding* signed by the Region III Administrator of the U.S. Environmental Protection Agency and the Chairman of the Anacostia Watershed Restoration Committee. The two parties to the Agreement pledged to cooperate in continuing the restoration of the Anacostia River and in providing diligent outreach and environmental services to the river's bordering communities. As a result of the agreement, EPA Region III became a non-voting member of the Anacostia Watershed Restoration Committee.

KEY STRATEGIES FORMALIZED

Two achievements in 1996 stemmed directly from the *1994 Agreement of Federal Agencies on Ecosystem Management in the Chesapeake Bay*. Key strategies were developed with considerable Federal leadership and participation:

- *Special Tributary Strategy for Federal Lands in the District of Columbia.*
- *Biennial Federal Workplan for the Anacostia River Watershed*

In addition, a third strategy was completed in October 1996 with extensive public participation:

- *Regional Action Plan for Toxics*

Special Tributary Strategy for Federal Lands: On March 25, 1996, the *Special Tributary Strategy for Federal Lands in the District of Columbia* was signed by 18 high-level Federal officials at the U.S. National Arboretum on the banks of the Anacostia River. The voluntary agreement calls for improved control of stormwater runoff on all Federal lands in the District of Columbia. It also supplements the District's strategy to meet the 40% nutrient reduction goal of the Chesapeake Bay Program.

Biennial Federal Workplan: On November 22, 1996, a draft *Biennial Federal Workplan for the Anacostia River Watershed* was introduced by EPA Region III's Chesapeake Bay Program and the U.S. Army Corps of Engineers (Baltimore District) in cooperation with the Anacostia Watershed Restoration Committee.

The workplan translates the goals of the Anacostia Watershed Restoration Committee's *Six-Point Action Plan* into specific actions to be undertaken by Federal agencies and facilities that impact water quality in the Anacostia and watershed. The workplan emphasizes that the Federal agencies cannot singlehandedly restore and protect the Anacostia River watershed. Success will depend on the continuation of efforts by state and local agencies, private landholders, and citizens organizations.

The primary purpose of the workplan is to provide guidance necessary to ensure that Federal actions are:

- Coordinated and collaborative in their approach;
- Compatible with and supportive of actions of other Federal and non-Federal participating groups;
- Designed to address effectively the restoration priorities defined in the *Six-Point Action Plan* and others that may subsequently arise;
- Able to be routinely tracked and documented in terms of cost and progress.

Success will depend on

the continuation of efforts

by state and local agencies,

private landholders, and

citizens' groups.

Regional Action Plan for Toxics: In October 1996, the District of Columbia completed a *Regional Action Plan for Toxics*. The plan defines a series of steps that organizations may take to minimize toxic loading into the Anacostia River and to rectify past damage to the river. The plan incorporates inputs from local citizens and interest groups that were obtained during interviews and public forums. The work was supported by grants from the Chesapeake Bay Program.

INSPECTIONS CONDUCTED AT FEDERAL FACILITIES

In 1995, EPA Region III conducted multi-media inspections to determine compliance under various environmental statutes at the Washington Navy Yard, the Southeast Federal Center, and the Bureau of Engraving and Printing. Subsequently, there were high-level meetings with Navy personnel



to address the outstanding compliance issues at the facility. Section 7003 of the Resource Conservation and Recovery Act was used as the basis for an Order addressing hazardous-waste contamination of soil, ground water, and surface water. In June 1996, the Navy agreed to contract for immediate clean up of contaminated sediments in three outfall locations and to perform additional clean up in 1997.

Follow up of compliance issues involving the Southeast Federal Center continues. A special Federal appropriation of \$15 million was directed to environmental remediation at this location.

Two other Notices of Violation under the Resource Conservation and Recovery Act were issued to the Navy in June 1996 and are pending resolution. They concern violations at the Washington Navy Yard and the Anacostia Naval Station.

Assessments conducted at the Washington Navy Yard site indicate that the contamination there in the presence of a subsistence fishery and other sensitive environments would qualify the site for inclusion on the Superfund National Priorities List. In July 1996, EPA Region III requested District of Columbia concurrence with the listing and subsequently received it in October 1996. Meanwhile, progress toward clean up continues under the Resource Conservation and Recovery Act action.

The Bureau of Engraving and Printing is presently in compliance.

CONTROL OF COMBINED SEWER OVERFLOW IMPROVED

A new operating permit for the Blue Plains sewage-treatment plant is now strengthening the monitoring, public notification, and control of combined sewer overflows in the District of Columbia. The new permit was issued by the Environmental Protection Agency in January 1997, following certification by the District of Columbia. It incorporates controls on combined sewer overflow and nutrients in the Anacostia that are in line with national policy. The permit also defines Biological Nitrogen Removal pilot plant requirements that will further reduce nutrient pollution in the Potomac River basin and, consequently, in the Anacostia River, since the basin is a tidal system.

FINANCIAL SUPPORT TO COMMUNITY PROJECTS

Since the start of its *Anacostia Ecosystem Initiative* in 1994, EPA Region III has channeled over \$1 million to projects designed to generate community involvement and awareness in the issues associated with the Anacostia's pollution and restoration. Examples include support for the *Toxics Action Plan* and *Biennial Federal Workplan* described previously. Further examples include fish advisory signs along the river, educational canoe trips for school children, science fairs and festivals, comparative risk studies on overall health risks, and environmental justice projects.

In addition to those individual, relatively small grants, EPA Region III, through its Chesapeake Bay Program and Non-Point Source Program, has channeled more than \$2.5 million to the District of Columbia and Maryland for environmental protection work.

**DISTRICT OF COLUMBIA
FISHING NOTICE
REGULACIONES PARA LA PESCA**

➤ A District of Columbia fishing license is required to fish in these waters.
 Para pescar en el Distrito de Columbia se necesita una licencia.

➤ District of Columbia fishing regulations are in effect for these waters.
 Están vigentes las regulaciones de pesca en el Distrito de Columbia.

HEALTH ADVISORY/PRECAUCIONES PARA SU SALUD

WARNING

➤ Fish from these waters contain PCBs and other chemical contaminants. Los peces de esta zona contienen PCBs y otros químicos contaminantes.

➤ Do not eat catfish, carp, or eel from these waters. No se pueden comer: "catfish," "carp," ni anguila ("carp") de estas aguas.

➤ You may eat 1/4 pound per month of largemouth bass or 1/2 pound per week of sunfish or other fish. Se pueden comer: 1/4 libra por mes de robalo ("largemouth bass"), 1/2 libra por semana de "sunfish" u otros peces.

➤ Choose to eat younger and smaller fish of legal size. Always clean fish, trim away fat, and cook fish as far from skin as possible. Escoge para comer los peces más jóvenes y pequeños que puedas. Siempre limpia los peces, quita la grasa y cocínalos lo más lejos de la piel que puedas.

The practice of catch-and-release is encouraged.
Fomentamos la práctica de pescar y elevar el pez al agua.

SWIMMING BAN/PROHIBIDO NADAR

Swimming is prohibited in these waters due to high levels of bacteria. Se prohíbe nadar en esta zona por los niveles altos de bacterias en el agua.

For more information, call: Fisheries Management Branch: (202) 644-6306
 Para más información comuníquese con la división del programa de pesca al teléfono (202) 644-6306.

Department of Consumer and Regulatory Affairs
 Environmental Regulatory Division
 Water Resource Management Branch
 Fisheries Management Group
 1910 Mada Lutfi Way, N.W., S.E. Office Bldg.
 Washington, D.C. 20003

See the back panel for *Future Plans* ➤

How to find out more about the ANACOSTIA ECOSYSTEM INITIATIVE



The staff of the U.S. Environmental Protection Agency, Region III, will be happy to respond to questions and comments about the *Anacostia Ecosystem Initiative*—the restoration project itself and related community involvement. Below is a list of contacts and their areas of expertise.

REGION III CONTACTS

Jon M. Capacasa, *Anacostia Ecosystem Initiative Manager*
(215) 566-5422

Eric Ashton, *Federal Facilities Coordinator*
(215) 566-2713

Bill Arguto, *Remedial Project Manager/Superfund*
(215) 566-3367

Jim McCreary, *Hazardous Waste Sites Study*
(215) 566-3243

Claudette Reed, *Enforcement Coordination/Compliance Snapshot*
(215) 566-2997

Elaine Harbold, *Blue Plains Permit/CSOs*
(215) 566-5744

Reginald Harris, *Environmental Justice*
(215) 566-2988

Written correspondence to these individuals should be addressed to:
U.S. Environmental Protection Agency, Region III
841 Chestnut Building
Philadelphia, PA 19107

LOCAL AREA CONTACT

Bev Baker, *Community Liaison*
U.S. Environmental Protection Agency
Chesapeake Bay Program
410 Severn Avenue, Suite 109
Annapolis, MD 21403
1 800 YOUR BAY, ext. 772



U.S. EPA Headquarters Library
Mail code 3201
1200 Pennsylvania Avenue NW
Washington, DC 20460

Visit the Initiative's web site at
<http://www.epa.gov/chesapeake/anacostia/>

FUTURE PLANS

In November 1996, EPA Region III issued an updated workplan for the *Anacostia Ecosystem Initiative*. The overall goal to restore the Anacostia River and its watershed remains the same, with planning for the following objectives:

- To target U.S. Environmental Protection Agency programs to the benefit of the project.
- To minimize pollution from combined-sewer-overflows.
- To provide public outreach and education.
- To assist and coordinate restoration activities at Federal installations in the watershed.
- To pursue reduction of environmental risk to the public from air, soil, and water contamination.

TARGETING ENVIRONMENTAL PROTECTION AGENCY PROGRAMS

The Anacostia Working Group within EPA Region III will continue to coordinate activities across all programs and divisions. The Group will identify issues that EPA Region III can help to resolve and opportunities for channeling resources for the benefit of the Anacostia watershed restoration.

The comprehensive assessment of historical hazardous waste sites, begun in July 1996, will continue aggressively, resulting in targeted investigations of dozens of sites that have attracted the concern of the citizens of the Anacostia community and the Environmental Protection Agency. Periodic status reports will be delivered to the community by meetings and facts sheets. Brownfields will be assessed for their redevelopment potential and clean-up or stabilization needs.

In March 1997, EPA Region III will launch the "Compliance Snapshot" report for the Anacostia. This public report will tally the facility-by-facility status of compliance with various environmental laws. By making this readily accessible to the public, EPA is providing an additional Right to Know database.

In cooperation with leaders and river restoration personnel in the Anacostia community, EPA Region III will continue its development of environmental indicators and measures of progress that was initiated under this *Anacostia Ecosystem Initiative*.

The feasibility of addressing sediment remediation in the Anacostia River as part of the process of nominating deserving sites for inclusion in the Superfund National Priorities List will be investigated. Prior compliance/enforcement proceedings will be followed up and targets for new pollution inspections will be sought as resources allow.

Enforcement lawyers and managers will be provided ready access to a current Supplemental Environmental Projects lists of Anacostia River projects for use in their settlement negotiations.

The automotive service and repair facilities will be assisted and pollution-prevention workshops will be conducted in the District of Columbia to reduce toxics and oil pollution in the Anacostia River. Specifically, the sources of oil/grease pollution to Hickey Run will be investigated by sewer tracing in conjunction with the Metropolitan Washington Council of Governments and the District of Columbia. Compliance/enforcement procedures will be initiated as warranted.

COMBINED-SEWER-OVERFLOW CONTROL

EPA will closely monitor newly issued discharge permits for the watershed. Monitoring of combined-sewer-overflow loads into the Anacostia River will be enhanced and information on the discharges will be disseminated to foster scientific study in the area. Technical-exchange meetings among the several interested agencies and groups will be convened to review long-term control strategies. Funding,

monitoring, and other issues pertinent to long-term resolution of the problem will be explored with all the interested parties.

COMMUNITY OUTREACH AND EDUCATION

EPA Region III will continue to fund and support the liaison for the communities that border the Anacostia River. The community liaison will continue to establish regular contact with citizens, community leaders, and interest groups to maintain a two-way flow of information and ideas associated with the day-to-day and long-term aspects of the Anacostia River restoration project. Special emphasis will be placed on bringing the story and the science of the Anacostia River restoration project to the school children of the area.

At least two major public meetings/forums per year will be conducted in the Anacostia community, and program and project managers will be encouraged to conduct similar outreach sessions. The meetings will be oriented toward enhancing feedback to EPA Region III concerning its programs and to building consensus on the assignment of priorities to the community's perceived needs.

Fact sheets, newsletters, news releases, etc., will be issued and the Internet site will be used to deliver current news and information to the public about the Anacostia River restoration project and the current condition of the river.

Staff members will dedicate time to supporting the Science in Education program in the District of Columbia's schools and launching an environmental science curriculum at Kramer Middle School in Anacostia.

COOPERATION FROM FEDERAL INSTALLATIONS

EPA Region III will assist in the finalization of the *Biennial Federal Workplan for the Anacostia River Watershed* in April 1997, to ensure that it will serve effectively as a needs and assessment master plan for environmental restoration work at Federal installations in the Anacostia watershed.

RISK REDUCTION FOR THE COMMUNITY

The *District of Columbia Environmental Characterization Report* will be distributed to interested parties and the public by April 1997, following completion of peer review and local-community review. The report will be used as a benchmark for future risk assessment in the District of Columbia.

A working session attended by representatives of EPA Region III, the U.S. Army Corps of Engineers, and the Metropolitan Washington Council of Governments is scheduled for early 1997 to discuss implementation of the *Regional Action Plan for Toxics*, which was completed in October 1996. EPA Region III will press for the document to be expanded by a "Phase Two" effort that would provide a parallel plan for the Maryland portion of the watershed. Inclusion of that information would make the document a truly comprehensive action plan for the entire Anacostia watershed.

EPA will continue to explore, along with the various partners under this Initiative, various means to officially designate the Anacostia as a National Urban Watershed Restoration Model in legislation and policy directives. This will promote the exchange of successful techniques and lessons learned with other urban river restoration efforts across the United States.