

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

**ENVIRONMENTAL CRIME**

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**Prepared for:**

15<sup>th</sup> International Forensic Science Symposium  
October 23-26, 2007

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## LIST OF ACRONYMS

$\mu$ -ECD – micro-electron capture detector	LLE – liquid-liquid extraction
AA – atomic absorption	LPME – liquid-phase microextraction
AAS – atomic absorption spectrometry	LVI – large volume injection
AE – atomic emission	MAE – microwave-assisted extraction
AED – atomic emission detection	MASE – membrane-assisted solvent extraction
AES – atomic emission spectrometry	MEPC – Marine Environment Protection Committee
AF – atomic fluorescence	MIPs – molecularly imprinted polymers
APCI – atmospheric pressure chemical ionization	MS – mass spectrometry
ASE – accelerated solvent extraction	MS/MS – tandem mass spectrometry
BFRs – brominated flame retardants	MSDS – Material Safety Data Sheet
BTEX – benzene, toluene, ethylbenzene, and xylene	MTBE – methyl <i>t</i> -butyl ether
CE – capillary electrophoresis	NEIC – National Enforcement Investigations Center
CFME – continuous flow microextraction	NSAIDs – nonsteroidal anti-inflammatory drugs
CGI – combustible gas indicator	OCPs – organochlorine pesticides
CID – Criminal Investigation Division	OES – optical emission spectrometry
CME – capillary microextraction	OPPs – organophosphorus pesticides
DAD – diode array detection	PAHs – polycyclic aromatic hydrocarbons
DLLME – dispersive liquid-liquid microextraction	PBDEs – polybrominated diphenyl ethers
ECD – electron capture detector	PBDDs – polybrominated dibenzo- <i>p</i> -dioxins
EDCs – endocrine disrupting compounds	PBDFs – polybrominated dibenzofurans
EDTA – ethylene diamine tetraacetic acid	PCBs – polychlorinated biphenyls
EI – electron impact ionization	PCDDs – polychlorinated dibenzo- <i>p</i> -dioxins
ENRD – Environment and Natural Resources Division	PCDFs – polychlorinated dibenzofurans
ERT – Environmental Response Team	PE – performance evaluation
ESI – electrospray ionization	PFE – pressurized fluid extraction
EU – European Union	PID – photoionization detector
FAAS – flame atomic absorption spectrometry	PLE – pressurized liquid extraction
FID – flame ionization detector	POPs – persistent organic pollutants
FLD – fluorescence detection	PPCPs – pharmaceuticals and personal care products
FPD – flame photometric detection	PPE – personal protective equipment
FRTR – Federal Remediation Technologies Roundtable	QA/QC – quality assurance/quality control
FTIR – Fourier transform infrared (spectrometry)	RP – reversed phase
GC – gas chromatography	SPSE – stir bar sorptive extraction
GC x GC – two-dimensional gas chromatography	SFE – supercritical fluid extraction
GC/MS – gas chromatography/mass spectrometry	SOPs – standard operating procedures
GPS – global positioning system	SPE – solid-phase extraction
HF – hydrofluoric acid	SPME – solid-phase microextraction
HG – hydride generation	SRMs – standard reference materials
HPLC – high performance liquid chromatography	SVOCs – semi-volatile organic compounds
HRGC – high resolution gas chromatography	TCD – thermal conductivity detector
HS – headspace	TCLP – toxicity characteristic leaching procedure
IC – ion chromatography	TLC – thin layer chromatography
ICP – inductively coupled plasma	TOF – time-of-flight
ICP-AES – inductively coupled plasma-atomic emission spectrometry	TPHs – total petroleum hydrocarbons
ICP-MS – inductively coupled plasma-mass spectrometry	TXRF – total reflection X-ray fluorescence
ICP-OES – inductively coupled plasma-optical emission spectrometry	UAE – ultrasonic assisted extraction
ILO – International Labour Organization	US – United States
IMO – International Maritime Organization	USACE – United States Army Corps of Engineers
INECE – International Network for Environmental Compliance and Enforcement	US DOJ – United States Department of Justice
IR – infrared	US EPA – United States Environmental Protection Agency
ISE – ion selective electrodes	UV – ultraviolet
LC – liquid chromatography	UV/Vis – ultraviolet/visible
LC/MS – liquid chromatography/mass spectrometry	VOCs – volatile organic compounds
	WHO – World Health Organization
	XRF – X-ray fluorescence
	XRS – X-ray spectrometry

## INTRODUCTION

This environmental crime review is a follow-up to those papers prepared for the 13<sup>th</sup> and 14<sup>th</sup> International Forensic Science Symposia. This paper contains an increased emphasis on the field work necessary in an environmental forensics investigation. Crime scenes for an environmental forensics investigation are routinely referred to as “the field” and this term is used repeatedly when referring to the sampling site. Suggested equipment, supplies, and on-line educational resources in field operations have been included. As in prior papers, several useful analytical books are described and published papers have been searched for updates, reviews, and new developments in environmental analytical chemistry.

Two special interest topics are included in this paper. The international waste “trade” of electronic waste (or e-waste) and shipbreaking creates hazards for humans and the environment and requires the attention of decision-makers worldwide. Nanotechnology developments are leading to innovative products that can assist in the protection of the environment but may also present some hazards as well.

Limitations in the presentation of material within this review paper have been described in previous papers. An environmental crime investigation requires the efforts of a large number of people from the initial and continuous work of the criminal investigative agent to the closing remarks of the prosecuting attorney for the case. Only a portion of the many aspects of investigation can be covered in this paper, but it is hoped that the references provided will aid the reader in locating answers for most questions.

Stephen P. Solow, formerly a chief of the Environmental Crimes Section in the United States Department of Justice (US DOJ) and currently a partner in a private law practice, has authored recent overviews of the “State of Environmental Crime Enforcement” in the United States. In these overviews, he briefly discusses policy and legislative changes and then summarizes the criminal cases and conclusions from the previous year (cases from 2005 and 2006).<sup>(1, 2)</sup> The US DOJ Environment and Natural Resources Division (ENRD) works with United States Environmental Protection Agency (US EPA) Criminal Investigation Division (CID) agents to prosecute violators of environmental laws in the US.<sup>(3)</sup> Summaries of environmental crime cases are provided in the press release section of the US DOJ ENRD web site. These summaries on settlements and convictions contain links to the full press releases that contain some additional case details.<sup>(4)</sup>

Solow also published a primer for preventing an environmental violation from becoming a criminal case. In this paper, he outlines the thought process of US EPA CID agents when deciding whether or not to pursue an environmental crime case. He continues with some legislative history and advice on how legal counsel can take steps to help their client avoid prosecution.<sup>(5)</sup>

Interest in environmental forensics and developing regulations to define environmental crime and pursue this type of criminal investigation has continued to increase. The European Union (EU) has penned a draft law that would require member governments to enforce serious offenses against the environment as crimes.<sup>(6, 7)</sup> The United Kingdom has added environmental forensics degrees to higher education institutions and the dilemma of how to define an environmental forensics expert or professional is under consideration.<sup>(8-10)</sup> Presentation of chemistry to a courtroom and the repercussions of Daubert continue to be points of discussion.<sup>(11-13)</sup>

# ENVIRONMENTAL CRIME SCENE INVESTIGATION

## PLANNING THE GOALS OF THE FIELD INVESTIGATION

The US Army Corps of Engineers (USACE) has a long list of engineering manuals available on-line.<sup>(14)</sup> The publication dates for the available manuals range from 1972 to 2007. Most of these manuals listed by the USACE are designed for engineers involved in various civil works projects such as building dams or levees, designing small water systems, constructing flood controls, or dealing with coastal engineering issues. The manuals that can be of use in planning and executing environmental chemistry field investigations are listed in Table 1.

The US EPA office located in the Pacific Northwest area of the United States, Region 10 (offices located in Seattle, Washington), developed an online manual detailing the points (inspection, safety, legal considerations, and evidence) in an environmental compliance inspection some years ago.<sup>(15)</sup>

In 2002, an additional training course was developed and the materials for that environmental compliance course can be located at the International Network for Environmental Compliance and Enforcement (INECE) web site. The course manual is titled “Conducting Environmental Compliance Inspections: Inspector’s Field Manual – International Edition” and is available online in English and Spanish versions.<sup>(16)</sup> The instructor’s manual titled “Environmental Compliance Inspection Training: Facilitator’s Manual” is also available on this same web page. The instructor’s manual can be downloaded as the separate course sessions (inspection planning, collecting defensible evidence, field equipment and supplies, etc.) or as the entire manual.

Supplemental resources for nearly every session in the course can be found in the “Related Materials” box on the web page. The session numbers and titles match those listed for the chapters in the instructor’s manual. Links from this box connect to a page containing separate sections from the “INECE Training Course for Multimedia Inspectors, Student Manual.”<sup>(17)</sup> Many of the links on this page connect back to the US EPA Region 10 “Conducting Environmental Compliance Inspections” web site.<sup>(15)</sup> In another part of the INECE web site, the “Training Course for Multimedia Inspectors, Student Manual” can also be accessed and downloaded.<sup>(18)</sup>

One addition to these course materials is the “Multi-Media Investigation Manual” produced in 1992 by the US EPA National Enforcement Investigations Center (NEIC) in cooperation with the US EPA regional offices.<sup>(19)</sup> This manual develops field inspection strategy, touches upon the US environmental regulations, and has many useful appendices. Appendix C lists responsibilities for the leader of the team going to the field site. Appendix D provides examples of types of information to gather about the site or the party under investigation. Appendix G is an equipment checklist. Appendix M covers sampling guidelines and includes two tables for sample collection and handling (one for liquid samples and one for non-liquid samples). Appendix M also has a list of the instruments most often used in the field.

Table 1. US Army Corps of Engineers (USACE) Engineering Manuals Available on the Web

Title	Publication #	Publication Date	Description (number of pages)
Validation of Analytical Chemistry Laboratories	EM 200-1-1	01 July 94	Guidance for determining if an analytical laboratory is following an appropriate quality assurance and quality control program. This validation process requires several steps including a review of qualifications, the analysis of performance evaluation samples, and an on-site laboratory inspection (504 pages). <sup>(20)</sup>
Technical Project Planning (TPP) Process	EM 200-1-2	31 August 98	Summarizes the systematic process involved in planning field activities when dealing with sites containing hazardous or toxic materials. The appendices in this document provide useful tables and worksheets and include a comparison of the TPP process to the US EPA version of project planning using data quality objectives (132 pages). <sup>(21)</sup>
Requirements for the Preparation of Sampling and Analysis Plans	EM 200-1-3	01 February 01	Outlines steps in preparing a plan for field sampling; lists items to be considered when making decisions prior to going into the field. Appendix B is an excellent detailed table for sampling. The table lists analytes and the recommended preservatives, holding times and containers for liquid or solid samples, as applicable, for each analyte. Appendix C covers extensive details on surface water, groundwater, potable water, sediment, soil, and air and soil gas sampling with diagrams and step-by-step directions and discussion of sampling strategies. The sampling sections contain information on containers, preservation, and sampling procedures, methods, and tools (376 pages). <sup>(22)</sup>
Risk Assessment Handbook: Volume I – Human Health Evaluation	EM 200-1-4 (Volume I)	31 January 99	Guidance for considering the possible health risks to humans when investigating a field site (187 pages). <sup>(23)</sup>
Risk Assessment Handbook: Volume II – Environmental Evaluation	EM 200-1-4 (Volume II)	30 June 96	Discusses considerations in deciding how hazardous chemical materials have damaged an ecosystem at a site and what remediation actions may be needed (409 pages). <sup>(24)</sup>
Chemical Quality Assurance for Hazardous, Toxic and Radioactive Waste Projects	EM 200-1-6	10 October 97	Document describes the use of several different tools for assessing the quality of project work including proficiency testing, field audits, and reviews of laboratory data (126 pages). <sup>(25)</sup>
Performance Evaluation (PE) Program	EM 200-1-7	01 February 01	Explains how to develop parts of a performance evaluation program. It provides good details about what to consider when designing PE samples to meet the needs of a specific project; consider variables such as matrices, challenging analytes, incompatible analytes, concentrations of analytes, stability, and verification of the PE sample (113 pages). <sup>(26)</sup>
Guidance for Evaluating Performance-Based Chemical Data	EM 200-1-10	30 June 05	Good details on quality parameters that can be used to evaluate data and ensure that only scientifically defensible data is used in future decision-making. Quality parameters described in this document include initial and continuing calibration verifications, laboratory control samples, blanks, matrix spikes, surrogates, and internal standards (129 pages). <sup>(27)</sup>
Safety and Health Requirements	EM 385-1-1	03 November 03	Large collection of health and safety information extending beyond usual needs in environmental forensic field work. Sections contain information on personal protective equipment, hazardous substances and environments, accident prevention, material handling and disposal, and machinery and mechanized equipment and also include many figures, tables, and appendices (977 pages). <sup>(28)</sup>
Radiation Protection Manual	EM 385-1-80	30 May 97	Document provides guidance when activities require handling radioactive material or devices that generate radiation (186 pages). <sup>(29)</sup>

## **PLANNING THE SAMPLING FOR THE FIELD INVESTIGATION**

The US EPA Environmental Response Team (ERT) provides a collection of free documents in the products area of their web site.<sup>(30)</sup> The best of these are the Superfund Program Representative Sampling Guidance Documents and the ERT Standard Operating Procedures (SOPs). The representative sampling documents are divided by the type of media being sampled (soil, waste, water, air, and biological). Each document includes details on sampling design, sampling equipment, sample collection and preparation, quality assurance/quality control (QA/QC) samples and evaluation, and possible sources of error in the sampling process.

The soil sampling guidance document is one document divided into three separate files, probably due to size alone. The water sampling guidance document has two separate parts divided by the type of water being sampled. The first part addresses sampling of surface water and sediment and the second part addresses the sampling of groundwater. Each part of the water sampling guidance stands alone as each has a separate table of contents and has topics specific to either surface water (migration pathways, estuaries and wetlands, etc.) or groundwater (types of pumps for sampling, geophysics, well purging, etc.). The waste sampling guidance includes details on waste sample types (grab and composite), the waste characteristics of homogeneity, chemical stability, particle size (solids), and viscosity (liquids), and field analytical screening (identifies the instrumentation needed for field screening).

Over 50 ERT SOPs are available on the web site and many of these provide guidance on sampling. The SOP list includes procedures for sampling soil, surface water, sediment, air (general air sampling, sampling for asbestos in air, and sampling for metals in air), groundwater wells, waste piles, and drums. Chip, wipe, and sweep sampling guidance is also provided in one of the procedures. Of particular interest is the SOP for sampling equipment decontamination which presents a nine-step decontamination procedure, breaks down physical decontamination into abrasive and non-abrasive cleaning methods, details the use of separate washing and spraying stations for an extensive decontamination process, and provides a brief QA/QC section about rinsate blanks.

Procedures for some equipment and instrumentation are also included in the ERT SOPs list. Included in these are procedures for the operation of X-ray fluorescence detectors, gas chromatographs, photoionization detectors (PID) or HNU meters, and the Geoprobe sampling device.

## **SAMPLE CHARACTERIZATION AND ANALYSIS IN THE FIELD**

Instrumentation and techniques useful for a field kit for characterizing and analyzing samples in the field are listed in Table 2. Many laboratory instruments have been reduced down to useful and desirable field-portable instrumentation. For some time now, pH meters have been small and sturdy enough to easily transport and use in the field and now these instruments can be accompanied by portable X-ray fluorescence (XRF) spectrometers, gas chromatographs (GC), and gas chromatograph/mass spectrometers (GC/MS).

Table 2. Basic Field Characterization & Analysis Kit

Technique or Instrument	Analyte(s)	Description	Vendors <sup>†</sup>
Chemical Identification Test Kits	atrazine and simazine in water arsenic in water, cyanide in water	pesticide, arsenic, and cyanide test kits with simple test steps (add kit reagents to the sample, shake, dip test strips, and match test results to a color chart)	Industrial Test Systems, Inc.
	pH (pH test paper)	quick dip and compare test for narrowing of the pH range for an aqueous sample	EMD, pHydriion
	various, for solid or liquid samples (multiple tests to narrow range of possible analytes in sample)	general chemical identification kit that characterizes waste according to chemical and physical properties; a chart is provided to follow the positive or negative results of the characterization tests from the kit	Haztech Systems, Inc.
Gas Detector Tubes	over 300 gases	indicator tubes provide a rapid visual identification of gases and gas concentrations by a visual color comparison; extensive list of gases that can be detected including acid gases, basic gases, nitrous gases, petroleum hydrocarbons, chlorinated hydrocarbons, and sulfur dioxide	Sensidyne Dräger
pH Meter	pH	determine acidity or basicity of aqueous samples with accuracy	Mettler Toledo, Thermo Scientific
Ultraviolet (UV) Fluorescence Analyzer	hydrocarbons (several hydrocarbon test kits are available)	simple sample preparation for solids or liquids; calibrate with a test kit such as polycyclic aromatic hydrocarbons (PAHs), total petroleum hydrocarbons (TPHs), gasoline range organics (GRO), and diesel range organics (EDRO)	Sitelab
XRF	metals (Cd, Cr, As, Pb, Hg, Se, Ba, Cu, Zn, etc.)	rapid, multi-element screening and semi-quantitative analysis with no sample preparation needed; high sample throughput for low cost	Innov-X Systems Thermo Scientific
GC/MS	volatile organic compounds (VOCs)	rapid, high sample throughput analysis for VOCs; can add an interface for headspace analysis and analyze air, water, and soil samples	INFICON Constellation Technology
other additions to consider			
GC	organic compounds and some inorganic gases; can be specific to halogens, cyano and nitro groups	choice of detector determines compounds that can be identified, the linear range, and the sensitivity; common detectors are flame ionization (FID), photoionization (PID), electron capture (ECD), and thermal conductivity (TCD)	Quadrex Corp. Photovac, Inc.
Raman Spectrometer	organic and inorganic compounds and metals	rapid, non-destructive identification of sample using vibrational spectroscopy; no sample preparation required for solid or liquid samples	DeltaNu JASCO International
Multi-gas Sensor	many gas-specific sensors available (ex. HCN, Cl <sub>2</sub> , CO, PH <sub>3</sub> , and H <sub>2</sub> S)	handheld monitors can combine up to five sensors and data logging to a portable computer	RAE Systems Gray Wolf Sensing Solutions
Ion Selective Electrodes (ISE)	analytes include halides, perchlorate, fluoroborate, sodium, cyanide, cadmium, and copper	ISEs identify and provide concentration of an analyte in solution but can be affected by interference from other ions; many pH meters have an ISE interface, so both pH electrodes and ISEs can be used with the same meter	Hanna Instruments Omega Engineering, Inc.

<sup>†</sup> Vendors, brand names or products listed are not endorsements or recommendations and are not an exhaustive list of suppliers of the materials or instrumentation listed.

The Federal Remediation Technologies Roundtable has two useful matrices on its web site providing information on sampling in the field and instrumentation and techniques needed for sample analyses.<sup>(31)</sup> The sample analysis tools matrix is called the “analysis matrix” and the field sampling and collection techniques matrix is called the “sampling matrix.”<sup>(32, 33)</sup>

Each matrix has active links to summary pages for items (instruments, techniques, or tools) listed on the vertical axis of the matrix. The vertical axis for the analysis matrix has the list of analytical instrumentation or techniques useful in analyses for VOCs, semi-volatile organic compounds (SVOCs), pesticides, polychlorinated biphenyls (PCBs), TPHs, metals, radionuclides, and explosives. The specific information provided for each analysis technique includes the basic description, media type, analytes, detection limits, selectivity and potential for interference, quantitation capability, turnaround time per sample, relative cost per sample, limitations of the technique, and published standards or methods for the technology. The vertical axis of the sampling matrix lists access tools (drilling methods and samplers), collection tools (hand-held tools, pumps, gas collection devices, etc.), and extraction methods (solvent, thermal, membrane, etc.). These tools and techniques have summary information including the basic description and use, analytes, sample media, speed of use, sample volume, relative cost per sample, limitations of the tool or technique, and any published standards for the technique.

### **PUTTING A FIELD KIT TO WORK**

A checklist of items that are useful in field investigations is presented in Table 3. It is not an exhaustive list and should be compared with other available resources and common-sense ideas (for example, having an extra set of vehicle keys when out in the field). The list has been divided into essential categories of supplies, health and safety items that will be needed to some degree any time chemicals are involved, and some optional items that might be added into the field kit if the sampling plan or investigation indicates that a particular item would be necessary.

If samples are being taken for later analysis at a laboratory, then some sort of container is needed. Containers should be free of any contamination that may interfere with the laboratory analysis. Recommended containers are those that are provided by the manufacturer as certified clean and with Teflon-lined lids. Containers should be chosen to match the type of analysis being performed. Plastic jars or bottles will not introduce interferents for metals analysis, but the phthalates in the plastic could leach out into a sample that has been designated for organics analysis. Glass jars or bottles can be used to contain samples taken for either organics or metals analysis, but should be avoided if the samples are suspected to have a high concentration of hydrofluoric acid (HF). HF in the sample could etch and weaken the glass jar and create an even greater hazard for field and laboratory personnel.

Table 3. Checklist for Field Kit Essentials, Health & Safety Items, and Optional Extras<sup>†</sup>

Item	Description / Use / Value in the Field Kit
	Essentials
Sample Containers	<ul style="list-style-type: none"> <li>• glass jars or bottles – combination of clear and brown glass, depending on the potential light sensitivity of analytes in the sample; glass is required for samples being tested for organic chemicals, but can also be used for samples collected for metals analysis</li> <li>• plastic jars or bottles – can be used for samples being analyzed for metals but must be used for samples suspected of containing hydrofluoric acid (HF)</li> <li>• stainless steel canisters for air and gas sampling (Summa canisters)</li> </ul>
Sampling Devices	<ul style="list-style-type: none"> <li>• scoops and trowels – plastic (for samples undergoing analysis for metals) and stainless steel (for samples undergoing analysis for organics)</li> <li>• spatulas and spoons – stainless steel (organics analysis), plastic (metals analysis), and wooden (metals or organics)</li> <li>• bailers – for liquid sampling of wells or tanks (liquid that is deep or a distance from the sampler)</li> <li>• Coliwesas (composite liquid waste samplers) or glass thieving tubes – for representative vertical sampling of liquids, can maintain the separate layers of liquids during sampling</li> <li>• soil auger and core sampler – for soil samples below the ground surface</li> <li>• shovel – to aid in removing surface soil, if necessary, but it can also be used as a large sampling scoop</li> <li>• bacon bomb/sample thief – for liquid sampling at the bottom of storage tanks, tank cars, drums</li> <li>• siphoning equipment or pumps – if needed for liquid sampling</li> </ul>
In-Field Sample Preparation	<ul style="list-style-type: none"> <li>• filters – for removing suspended material that is not part of the sample to be analyzed in the lab; to filter sample prior to adding a preservative</li> <li>• sample preservatives (acids, sodium hydroxide, zinc acetate, etc.)</li> </ul>
Labeling Materials	<ul style="list-style-type: none"> <li>• labels – with good adhesive and sizes to fit various sample containers and still provide sufficient writing space</li> <li>• tags and rubber bands – the heavy-duty paper tags have strings through a grommet in the paper, plenty of writing space to fully identify the sample, and can be attached securely to a sampling jar just below the lid by knotting the string; use a rubber band to hold the tag against the jar, if desired, during transport</li> <li>• forms – chain-of-custody forms to log the samples taken, where the samples were taken (sampling station), what type or types of containers were used for each sampling station, the name of the person or persons taking the samples, what day and time the samples were taken</li> <li>• custody seals – tape, stickers, or heavily sealed bags to secure samples from any potential tampering during transit between the sampling site and the laboratory</li> <li>• permanent ink pens – for example, Sharpie-type markers with wide and narrow tips to mark on jars, lids, and labels, and ball-point pens or extra-fine tip markers for filling out chain-of-custody forms and sample tags</li> </ul>
Packing Materials	<ul style="list-style-type: none"> <li>• zipper-lock bags – have plenty of these in all sizes, use to seal up each sample container or to double-bag sample containers with liquids, can provide some protection against cross-contamination between samples and samples being contaminated by other means</li> <li>• lockable, insulated cooler (ice chest) for shipping (or ease of transport) – or a similar large container that will seal completely to ensure that no fluids leak from the large container, the container has some temperature stability to maintain low temperatures when samples must be packed in ice, and with some locking mechanism to secure the container from tampering during transit</li> <li>• absorbent packing material – can be loose material or packaged in porous bags; if samples need to be cooled with ice as well, take care to separate the absorbent material from the melting ice</li> <li>• cardboard structure packing material – secure samples from moving loose within shipping cooler</li> <li>• ice or reusable frozen packs – if samples require cooling in transit to the laboratory</li> </ul>

Item	Description / Use / Value in the Field Kit
Packing Materials (continued)	<ul style="list-style-type: none"> <li>• thermometer – can be used to log the temperature of the inside of a sample cooler prior to transport to the laboratory</li> <li>• bubble-wrap or sealed air pillows – good cushioning material to keep samples from moving loose (tipping over, rolling around) within the shipping cooler; plastic cushioning material (unlike cardboard packing material) can be used in combination with ice and samples that might leak out of sample containers</li> <li>• packing tape or fiber tape – can be used to help secure samples inside coolers (tape packing materials around samples or groups of samples) or an additional measure for keeping the sample cooler closed if the latch breaks</li> </ul>
Camera & Film	<ul style="list-style-type: none"> <li>• camera and film – photos provide excellent documentation of work performed in the field and provide evidence of waste piles or illegal activity to present in a judicial setting; an instant-photo developing type of camera (Polaroid-type) may be used; if 35mm film is used, consider any chain-of-custody issues if film is developed outside of your control</li> <li>• digital camera, memory cards, and batteries – digital cameras provide flexibility (photos can be easily incorporated into a report or presentation) and some long-term cost savings (no photo developing fees)</li> <li>• rulers, measuring tapes – provide scale for the sample or sampling site when photos are being taken</li> </ul>
General Cleaning and Decontamination Materials	<ul style="list-style-type: none"> <li>• tap water, purified water (distilled or deionized), and non-phosphate detergent</li> <li>• decontamination solutions: 10% nitric acid (if metals are a contaminant), acetone or hexane (if organics are a contaminant)</li> <li>• drop cloth or plastic sheeting to prevent ground contamination during cleaning of materials</li> <li>• long and short handled brushes, tubs or buckets, sprayers for decontamination solutions</li> <li>• containers for storage of decontamination solutions and for the collection and disposal of rinse waste (used decontamination solutions)</li> <li>• large trash bags – to collect contaminated waste items (used wipes) for proper disposal or to gather scoops or tools that must be decontaminated at a later time</li> <li>• purified water (distilled or deionized) – good for cleaning (or rinsing) equipment when the same tool must be used for multiple samples; the rinsate from cleaning the piece of equipment can be taken to the analytical lab and analyzed as a “rinse blank” to determine if any cross-contamination occurred during sampling</li> <li>• Kimwipes or paper towels – disposable wipes of varying sizes to clean tools, clean the exterior of sample containers, clean parts of instrumentation, etc.</li> <li>• soap, waterless hand cleaner, towels and clean water for personal hand cleaning</li> </ul>
Miscellaneous Items	<ul style="list-style-type: none"> <li>• field log books, any additional forms or records specific to or required by your organization, and pens or pencils that are fade and water resistant</li> <li>• specific shipping regulations and requirements as needed (for example, if the samples are being shipped across state or country boundaries) and any required hazard labels for the sample cooler or other shipping container</li> <li>• absorbent material (in addition to any needed for packing samples) to aid in cleaning up any spills during sampling or clean-up</li> <li>• crescent wrenches, bung openers, or other devices to open drums, if drum sampling will be necessary</li> <li>• two-way radios and chargers or batteries – if the site is large, this is useful to keep in touch with other team members working at different locations on the site</li> <li>• various office supplies – additional pens or pencils, paper clips, staples and a stapler, a calculator, file folders; anything that might be needed (other than field log books) to document and organize the activities in the field</li> <li>• various tools and tool kits for adjusting or repairing instruments, equipment, or vehicles in the field</li> <li>• drinking water and electrolyte-enhanced water – this is essential for field personnel comfort and an absolute necessity if any field personnel must don protective suits in the course of the investigation</li> </ul>

Item	Description / Use / Value in the Field Kit
	Health & Safety
Personal Protective Equipment (PPE)	<ul style="list-style-type: none"> <li>gloves – have a variety of protective materials available and sizes</li> <li>safety glasses, goggles, and face shields – eye protection against splashes, particles in air</li> <li>Tyvex suits – complete protective cover for body, varying protective materials</li> <li>face masks, respirators, and extra gas cartridges – half-face protection against breathing dusts, masks to supply air using air tanks</li> <li>self-contained breathing apparatus and air tanks – if situation requires supplied air for samplers</li> <li>ear plugs and hard hats – necessary when working around heavy equipment or in factories or warehouses</li> <li>rubber boots or disposable plastic boot coverings</li> <li>heavy tape or duct tape – to seal gaps and securely attach protective suits and gloves and suits and boot coverings</li> </ul>
First Aid	<ul style="list-style-type: none"> <li>first aid kit with scissors or pocket knife</li> <li>emergency eye wash</li> <li>substance-specific first aid information (Material Safety Data Sheets or MSDS) for any compounds suspected of being on the site</li> <li>emergency telephone numbers, locations of and maps to nearby hospitals</li> <li>fire extinguisher</li> </ul>
Photoionization Detector (PID)	<ul style="list-style-type: none"> <li>also known as H-NU or HNu meter– used for both personal protection of samplers and to provide some characterization of materials, the detector can indicate the presence of volatile organic compounds and potential explosion hazards but cannot detect methane (for methane, use a flame ionization detector or FID)</li> </ul>
Combustible Gas Indicator (CGI) and Oxygen Meter	<ul style="list-style-type: none"> <li>CGI/oxygen meters measure the concentration of flammable vapors or gases in the air and the level of oxygen; this is essential for determining if an explosive situation is present or developing and is crucial for confined space or similarly restrictive areas. Some meters include compound-specific detectors.</li> </ul>
Flame Ionization Detector (FID)	<ul style="list-style-type: none"> <li>measures the level of total organic compounds in atmosphere; best as an ethane and methane detector</li> </ul>
Geiger Counter	<ul style="list-style-type: none"> <li>radiation screening of sampling area and samples (with a probe that can detect a combination of forms of radiation); vendors include Ludlum Measurements and SE International</li> </ul>
	Optional Extras
Dyes and Fluorometer	<ul style="list-style-type: none"> <li>colored dyes – if it is necessary to trace the source of a discharge into a waste stream or local waters; different colors of dyes can be used for multiple possible discharge sources</li> <li>fluorometer – to measure very small quantities of the dye, if trail of dye is not completely obvious to the eye; one vendor is Turner Designs</li> </ul>
Automatic Water Sampler	<ul style="list-style-type: none"> <li>good for sampling over a period of time or sampling that begins when certain conditions are detected by the sampling device (for example, when water pH drops below a set level)</li> <li>good for covert sampling – place in sampling location, set sampler working, return at a later time to retrieve samples and replace with additional sampling bottles, as necessary</li> <li>some automatic samplers can be controlled by remote devices or can maintain temperature control for the samples; one vendor of automatic samplers is Teledyne Isco</li> </ul>
Global Positioning System (GPS) Receiver	<ul style="list-style-type: none"> <li>GPS can be used to precisely record the exact location for a sampling site</li> </ul>
Metal Detector	<ul style="list-style-type: none"> <li>useful when there is a suspicion of buried drums; the metal detector can save time in locating where the drums are buried</li> </ul>
Microscope	<ul style="list-style-type: none"> <li>may be used in the field for asbestos fiber identification</li> </ul>

† Vendors, brand names or products listed are not endorsements or recommendations and are not an exhaustive list of suppliers of the materials or instrumentation listed.

Sampling tools vary widely and the decision of which tool or combination of tools to use depends on the answers to several questions.

- What type of sample media is involved (water, soil, waste, etc.)?
- Where is the sample located on the field site (in a stream, field, empty lot, drum, waste pile, dumpster, etc.)?
- How deep is the sample located? For example, is the sample located on the soil surface? Are samples needed from one or more of the liquid layers in a drum or tank car? Is the sample located underground in a buried drum or waste pile? Does the sampling strategy require a cored soil sample or samples from groundwater or a well?
- How much sample must be taken? What amount does the laboratory need for the analytical procedures planned for the samples? Are duplicate samples needed for quality control or for testing at an alternate laboratory (some companies under investigation request duplicate samples for their own testing)? Is there a need for more sample material for additional types of testing or statistical studies? Will an untouched (not affected by laboratory analysis) sample be needed as a visual item in evidence for a courtroom trial?
- What type of sampling strategy is needed for the samples? Does the sampling plan indicate grab samples only (a sample from one discrete sampling location)? Or will a composite sampling strategy be used (two or more equal aliquots collected from several distinct points and combined to form one sample)?

Surface sampling may only require simple scoops, but an underground sampling location would necessitate some means of relocating the surface soil in order to access the sample (shovels, heavy machinery, augers or drills, and corers). In sampling liquids, the presence of different liquid layers will require a thieving device (Coliwasa or “bacon bomb”) to allow sampling of a discrete layer. Water samples from groundwater or wells may require the use of a pump.

The sampling scoop can be as elementary as using the sample jar itself in a grab sample or as complex as a grid with composite sampling to provide as representative a sample as possible. Plastic scoops may be economical enough to use once and then dispose of properly; it may be considered wasteful, but it can be a cheaper and less time-consuming way of avoiding cross-contamination during sampling.

At times, the laboratory may request that field samplers add preservative to some samples immediately after sampling. The US EPA Region 9 laboratory has a list of requirements for samples arriving at their laboratory.<sup>(34)</sup> Field personnel should discuss the sampling and analysis strategy with the laboratory prior to going into the field. (What is the investigation attempting to demonstrate from certain samples and analyses?) Once the purpose behind each sample and analysis is understood, the laboratory can make recommendations on the use or avoidance of sample preservatives in the field.

Log books are an important tool in the field investigation. A log book should be bound and have consecutively numbered pages. If multiple log books are used on the site, it should be clearly indicated on each log book that it is one of a set of log books. Personnel in the field should log information about locations chosen as discrete sampling sites, details on samples taken, information about the field site (description or conditions, sketches or drawings, etc.), and interviews of anyone on the site (name and position or title of the person and what the person

said when interviewed). In addition to general information about the site or investigation, personnel should detail any unusual conditions, problems, or incidents that occur on the field site. If the field site is at a company facility and if records are collected from the facility, then the details about the records should be written down in the log book (what documents, how many pages, provided by what person at the facility).

Photographs are an excellent way to document what the field investigators observed on the site. Photographs of the conditions on the site, the locations where samples were taken, and the samples as labeled prior to packaging and transport are useful as the criminal case progresses. If digital cameras are used, consider copying the photos from the camera media cards to a CD or DVD and retaining the original media card in a secure location until the case has been successfully prosecuted. Commercially available software packages can be used to store and organize digital photos.

Samples must be well labeled to provide the correct information to the laboratory. At a minimum, the discrete sampling location (or sampling station), the numerical increment for this sample at that station (original, duplicate, or triplicate) if applicable, the name of the person taking the sample, the date and time that the sample was taken, and the analyses requested from the laboratory for that specific sample, should be indicated on a sample tag or sample label. As in other forensic science disciplines, the chain-of-custody for evidence is crucial and paperwork from the field to the laboratory should indicate what member of the investigative team was in possession of the samples (the evidence) at any time during the investigation.

Insulated coolers or ice chests are highly recommended for storage, transport, and shipping of samples taken in the field. Obviously ideal for samples that need cooling with ice while in transit, the coolers also offer extra protection for samplers and transporters on the occasion that a sample jar cracks or breaks causing material to leak out. Hasps can be attached to coolers in order to accommodate locks; an insulated cooler can then be secured and help maintain the sample integrity and chain-of-custody during transit to the laboratory.

If a specific sampling tool will be used for taking samples from more than one sampling location or station, then a means of thoroughly cleaning or decontaminating the tool will be necessary. Cross-contamination of samples can be prevented by first removing any visible particulate matter and then using some combination of rinsing or washing and scrubbing or wiping to remove what cannot be seen. The wash or rinse for decontamination should be chosen based on the suspicion of what compounds or elements are present in the sample. A rinse blank, taken from the final rinse of a sampling tool, should be labeled as a sample for laboratory analysis. This quality control step will allow the investigative team to show that sampling tools were clean prior to another use or it may show that a contaminant was not completely removed and may be present in a later sample.

The safety of personnel involved in the field investigation is important and a safety plan (in addition to any investigative or sampling plans) can be useful. Research into chemicals that may be on-site and possible exposures can help determine the extent of personal protective equipment (PPE) needed at the site. Detectors should be used prior to entering a site to check for levels of radiation, oxygen (too low or too high), and flammable gases if knowledge about the area indicates that any of these hazards may be present. First aid kits can be stocked with gels or creams that specifically address certain chemical exposures (for example, calcium gluconate should be included in the first aid kit if hydrofluoric acid may be present on site). A list of emergency phone numbers and locations of nearby hospitals should be included in the safety plan. Local weather conditions such as extremes in temperature and humidity should also be

considered when planning for any health and safety issues. Thicker gloves, additional layers of clothing, and some instant hot packs may be needed when temperatures drop. Frequent rest breaks, additional disposable gloves, and extra drinking water will be needed when the temperature and humidity climb.

# LABORATORY ANALYSIS OF ENVIRONMENTAL SAMPLES

## BOOKS ON ENVIRONMENTAL ANALYSIS

A good overview of environmental analysis is found in *Practical Environmental Analysis* by Radojević and Bashkin.<sup>(35)</sup> Designed for classroom use, each chapter includes questions and problems and project suggestions relating to the topics within that chapter or chapter section. Chapters are divided by the type of media undergoing analysis (air, water, rainwater, soil/sediment/sludge/dust, and plants). The authors address sampling issues and the analytical techniques involved for each media type and then follow up with details on specific analytes of interest. References for further reading on topics are prolific throughout the entire book.

The appendices included for this book contain some real gems that help make this book an essential handbook. Appendix B, Laboratory Practice, is a great reminder of all those bits of information covered in undergraduate general chemistry laboratories. This includes many practical items for people working in the field including reagent classification, properties and uses of specific acids, and a review of spectroscopic methods of analysis. Appendix C contains 13 pages of tables listing environmental standards on pollutants as set by the World Health Organization (WHO), the European Union (EU), and regulatory bodies in several countries (United Kingdom, Netherlands, Germany, Russia, and the United States). Other appendices include statistical tables, safety reminders, lists of environmental and analytical journals, and websites with helpful information on environmental analysis, pollution, or analytical chemistry.

Brian Murphy and Robert Morrison recently released the second edition of their essential guide to the forensic tools available in environmental investigations titled *Introduction to Environmental Forensics*.<sup>(36)</sup> The second edition adds chapters on laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) and scanning electron microscope (SEM) techniques. Chapters on sampling techniques and statistical methods have been expanded and the last chapter presents some emerging forensic techniques.

Steven Drielak has written a detailed guide about forensic evidence collection protocols in *Hot Zone Forensics: Chemical, Biological, and Radiological Evidence Collection*.<sup>(37)</sup> In this book, Drielak presents the procedures used and equipment needed at the crime scene, the sampling plan and the instruments needed to locate the source of contamination, and the preparation and protocols needed for evidence collection. The three types of evidence listed in the book title, chemical, biological, and radiological, are divided into separate chapters for in-depth coverage of the sampling techniques for each. An earlier book co-authored by Drielak, *Weapons of Mass Destruction: Response and Investigation*, expands the perspective on the crime scene.<sup>(38)</sup> The book begins with the initial recognition of an incident (first responder actions, personal protective equipment, handling victims, scene management) and then expands into the planning, procedures, and sampling for evidence within the restricted area. A final chapter presents the planning and execution of a search of a suspect facility.

*Practical Environmental Forensics: Process and Case Histories* is a thought-provoking and interesting review of some of the legal cases and relevant topics in the environmental forensics arena.<sup>(39)</sup> As expected, this book includes a review of the regulatory environment and of the frequently examined issue of a judge as the gatekeeper for expert opinions being accepted into evidence. The authors include advice on aspects of case preparation such as dealing with paper trails, risk assessments, and courtroom visuals and include overviews on contaminant transport models and chemical fingerprinting. The fascinating presentations of actual cases

comprise the latter third of the book and the authors review these cases with wit and clarity. Cases are briefly summarized at the beginning and arguments on both (or multiple) sides are presented along with the data on the costs of clean-up. Regulatory information and its application to the case are blended into the story along with tales of lawyer and expert witness antics. The conclusion to the case is given and a brief summary of “lessons learned” wraps up each case presentation.

If information centered on monitoring issues is needed, the *Environmental Instrumentation and Analysis Handbook* may be useful.<sup>(40)</sup> Major topics in this book include wastewater monitoring, air monitoring, and groundwater monitoring. The first part of this book gives an overview of some instrumentation and techniques used in environmental analysis.

Editors Reemtsma and Jekel present a rapid overview of polar compounds in groundwaters and surface waters in *Organic Pollutants in the Water Cycle*.<sup>(41)</sup> Each brief chapter of the book provides a large number of references for further reading. Pollutants covered in this book include human and veterinary pharmaceuticals, iodinated X-ray contrast media, herbicides, and disinfection by-products.

The presentation of a survey into enantioselective trace analysis and its potential as a new field of analytical research is the goal of *Chiral Environmental Pollutants: Trace Analysis and Ecotoxicology* by Kallenborn and Hühnerfuss.<sup>(42)</sup> After a brief discussion of chirality, enantioselective chromatographic methods including high performance liquid chromatography (HPLC), high resolution gas chromatography (HRGC), and capillary gas chromatography are covered. The survey continues with chapters on chiral xenobiotics in the environment and enantioselective toxic effects of drugs and other chiral environmental pollutants. The book concludes with perspectives and regulations regarding chiral drugs in the United States, in the European community, and in Japan and other countries. The authors have included extensive lists of references to support topics covered within the chapters.

Speciation of elements and compounds in environmental samples are reviewed and referenced in *Chemical Speciation in the Environment*.<sup>(43)</sup> Separation strategies, methods of speciation, and quality control considerations are discussed in the first section of the book. Environmental sample types presented include soils, sediments, fresh water, and seawater.

Methods used for the determination of endocrine disruptors in water samples are presented in several tables within *Endocrine Disruptors in Wastewater and Sludge Treatment Processes*.<sup>(44)</sup> Methods are listed for the determination of steroid hormones, alkylphenols, and hydrophobic xenoestrogens.

A variety of topics in speciation analysis is covered in *Trace Element Speciation for Environment, Food and Health*.<sup>(45)</sup> Topics reviewed in the book include sample treatment and storage, arsenic, mercury, organotins, rapid sample screening tests, quality control issues, and health issues.

*Environmental Sampling and Analysis for Metals* contains chapters on specific spectrometry and spectroscopy techniques, quality control, sample collection, sample preparation, and reporting of data for metals analysis.<sup>(46)</sup> Analytical techniques covered include ultraviolet/visible (UV/Vis) and infrared (IR) spectrophotometry, atomic absorption (AA) spectrometry (flame, graphite furnace, cold-vapor, and hydride-generation), and inductively coupled plasma atomic emission spectroscopy (ICP-AES). Selected flame atomic absorption

spectroscopy (FAAS) and graphite furnace atomic absorption spectrometry (GrAAS) methods are presented by element in one of the chapters.

Overviews of specific contaminants in the environment are presented in another book by Morrison and Murphy titled *Environmental Forensics: Contaminant Specific Guide*.<sup>(47)</sup> Arsenic, dioxins and furans, pesticides, PCBs, chromium, lead, and polycyclic aromatic hydrocarbons (PAHs) are some of the contaminants covered within this book. Chapters on each contaminant or group of contaminants end with large lists of references for further reading.

General instrument information and the applications related to environmental analysis are compiled in the chapters in *Atomic Spectroscopy in Elemental Analysis*.<sup>(48)</sup> Some techniques covered include inductively coupled plasma mass spectrometry (ICP-MS), X-ray fluorescence analysis (XRF), and atomic absorption (AA) spectroscopy.

The *Chromium (VI) Handbook* provides a review of the background and characteristics of chromium (VI) in the environment and very little analytical information.<sup>(49)</sup> One chapter includes a summary table on analytical methods for the analysis of chromium. Most methods referenced in this table are US EPA analytical methods.

Elemental speciation is the topic of a large two volume set published in 2003 (volume one) and 2005 (volume two). *Handbook of Elemental Speciation: Techniques and Methodology* includes chapters on sampling, sample preparation, separation techniques, and methods of detection.<sup>(50)</sup> *Handbook of Elemental Speciation II: Species in the Environment, Food, Medicine and Occupational Health* divides speciation information by each element.<sup>(51)</sup>

## **INSTRUMENTATION AND TECHNIQUES BY TOPIC**

For this review paper, the section summarizing recently published work or articles on developments in environmental analysis has been compiled in a table. Table 4 contains brief descriptions of recent work categorized by the matrix being analyzed, the contaminants being investigated, or the technique or instrument used for the sample analysis.

Many publications reference the use of multiple techniques such as combinations of sample separation or sample concentration techniques and analytical instrumentation for the determination of a particular environmental contaminant (for example, the combination of solid-phase microextraction with gas chromatography for the analysis of explosives in soil samples). If possible, papers are categorized by the technique receiving greater emphasis and the other techniques used are mentioned briefly in the summary of that publication. Papers that emphasize the analysis of a particular contaminant instead of the development of a specific technique are grouped in the category for that element or compound.

The Instrumental Criteria Sub-Committee of the Analytical Methods Committee of the Royal Society of Chemistry is publishing helpful evaluations of analytical instrumentation. The Sub-Committee examines the parts (hardware, software, training, etc.) that need to be considered when selecting an instrument and list the fine details of features, importance of the particular features, and reasons for having the features in tabular form.<sup>(52-55)</sup>

Table 4. Instrumentation and Techniques by Topic

Category	Description
Environmental Analysis	<p>Review of developments in environmental analytical chemistry during the years of 2003 and 2004; review includes sections on sampling and extraction methods, separation techniques, and current and developing detection techniques; also includes a table of emerging environmental contaminants with analysis techniques and references <sup>(56)</sup></p> <p>Review with tables of standard reference materials for the determination of organic contaminants in environmental samples <sup>(57)</sup></p> <p>Collection of tables listing certified reference materials for the analysis of inorganic and organic compounds in environmental samples <sup>(58)</sup></p> <p>Use of biosensors as tools in environmental analysis and monitoring; includes a summary table listing organic and inorganic analytes to be detected <sup>(59)</sup></p> <p>Study discusses the different degradation pathways for selected persistent organic pollutants (POPs) under varying conditions including a list of analytical procedures for the determination of POPs and their degradation products <sup>(60)</sup></p> <p>Brief note on types of immunoassays developed for environmental analysis <sup>(61)</sup></p> <p>Review of the analysis of environmental samples (eighth biennial review) covering developments during 2004 and 2005; tables include lists of analytes, methods, and instrumentation and sections include water and wastewater analysis and soil and sediment analysis; in Chinese <sup>(62)</sup></p>
Air Analysis	<p>Review of techniques for monitoring air pollutants including details on sampling and sample handling <sup>(63)</sup></p> <p>Two review papers on the determination of complex mixtures of volatile organic compounds (VOCs) in ambient air; the first paper presents an overview with sections on emissions, monitoring, sampling methods, and analytical methods; the second paper presents canister methodology and details, with many illustrations, canister cleaning, sampling, sample preparation, and sample analysis by gas chromatography/mass spectrometry (GC/MS) <sup>(64, 65)</sup></p> <p>Review in the use of Fourier transform infrared (FTIR) spectrometry for trace gas analysis in the atmosphere; most of the paper discusses passive remote sensing <sup>(66)</sup></p> <p>Chemical characterization of odorous gases emitted from a municipal landfill using GC/MS <sup>(67)</sup></p> <p>Evaluation of a low-pressure gas chromatography ion trap mass spectrometry method for determining polycyclic aromatic hydrocarbons (PAHs) in air samples; the method was faster than conventional GC/MS, but with reduced separation power <sup>(68)</sup></p>
Water Analysis	<p>Investigation of the best internal standards to use for the analysis of volatile pollutants in water; five compounds were found to be suitable for 49 pollutants <sup>(69)</sup></p> <p>Review of the history of methyl <i>tert</i>-butyl ether (MTBE) in rivers, lakes, and groundwater including a table referencing analytical methods for the determination of MTBE in natural surface waters, groundwater, wastewater, drinking water, sea water, and runoff waters <sup>(70)</sup></p>

Category	Description
Water Analysis (continued)	<p data-bbox="415 228 1942 289">Determination of 1,4-dioxane and tetrahydrofuran in groundwater using a solid-phase extraction (SPE) method based on activated carbon disks with analysis by gas chromatography/tandem mass spectrometry (GC/MS/MS) <sup>(71)</sup></p> <p data-bbox="415 321 1911 381">Extensive review of analytical methods for the determination of metals in sea water; includes many tables listing methods specific to certain analytes including Cr, Cu, Fe, Hg, Cd, Mo, and mixtures of metals; over 500 references included in this review <sup>(72)</sup></p> <p data-bbox="415 414 1942 474">Analysis of groundwater samples for five benzoylureas (diflubenzuron, triflumuron, hexaflumuron, lufenuron, and flufenoxuron) using on-line preconcentration and liquid chromatography-fluorescence detection <sup>(73)</sup></p> <p data-bbox="415 506 1843 566">Brief review of procedures used to determine PAHs and PCBs in aqueous samples including a table of analytical problems that can be encountered during analysis <sup>(74)</sup></p> <p data-bbox="415 599 1974 659">Determination of degradation products of nonylphenol polyethoxylate surfactants (NPEOs) in river water samples from 18 major rivers in Taiwan <sup>(75)</sup></p> <p data-bbox="415 691 1921 751">Review of methods and instruments for the microanalysis of volatile organic compounds (VOCs) in water samples; includes an overview of liquid, solid, and membrane extraction techniques and a table with advantages and disadvantages of techniques for isolation or enrichment <sup>(76)</sup></p> <p data-bbox="415 784 1911 844">Overview of industrial effluent analysis focusing on methods and techniques most frequently used and categorized by physical and chemical properties, inorganic metals analysis, inorganic non-metallic analysis, and organic analysis <sup>(77)</sup></p> <p data-bbox="415 876 1900 937">Presents an approach for determining detection limits for the analysis of metals in environmental water samples; As, Ca, Cr, and Cu are the analytes used to illustrate the procedure <sup>(78)</sup></p> <p data-bbox="415 969 1932 1029">Determination of phthalates in water samples using liquid-liquid extraction (LLE) and large volume injection (LVI) gas chromatography/mass spectrometry (GC/MS); discusses the problems and offers ideas for reducing contamination <sup>(79)</sup></p> <p data-bbox="415 1062 1942 1122">Use of an electronic nose to detect and discriminate between primary alcohols and phenol in water samples to provide a gross approximation of the concentrations present in the sample <sup>(80)</sup></p> <p data-bbox="415 1154 1963 1230">Biennial review into emerging contaminants and current issues in water analysis including pharmaceuticals, hormones, and endocrine disruptors, disinfection byproducts, pesticides and pesticide degradation products, and perfluorinated compounds; review of developments includes work from 2005 through early 2007 <sup>(81)</sup></p> <p data-bbox="415 1263 1963 1323">Analysis for bromate, a disinfection by-product, in groundwater and wastewater samples; three high performance liquid chromatography (HPLC) techniques, two ion chromatography (IC) techniques, and one spectrophotometry technique are compared <sup>(82)</sup></p> <p data-bbox="415 1356 1732 1388">Sampling and analysis for VOCs in wastewater streams from an industrial park and a nearby wastewater treatment plant <sup>(83)</sup></p>

Category	Description
Water Analysis (continued)	<p>Biennial review of developments in water analysis during 2003 and 2004 including some significant references from early 2005; review focuses on emerging contaminants and environmental issues driving current research <sup>(84)</sup></p> <p>Testing a spectrophotometric technique to measure pH in-situ; has potential to provide vertical (depth) pH measurements and spatial resolution <sup>(85)</sup></p>
Field-Portable Techniques & Instrumentation	<p>Comparison of analyses for lead on air filter samples using portable X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectroscopy (ICP-OES); most of the portable XRF results agreed within 25% of the ICP-OES results <sup>(86)</sup></p> <p>Advantages in using a passive, in situ sampler for the sampling of a stream receiving agricultural, municipal, and industrial wastewaters for the detection of 96 organic wastewater-related contaminants; results from the analyses of samples taken by the polar organic chemical integrative sampler (POCIS) were compared to results from samples taken using standard water-column sampling methodology <sup>(87)</sup></p> <p>Field analysis for BTEX (benzene, toluene, ethylbenzene, and xylene) in water by portable GC with micro flame ionization detection (GC-<math>\mu</math>FID) combined with headspace solid-phase microextraction (HS-SPME) <sup>(88)</sup></p> <p>Presents the development of a field-portable GC/MS consisting of a “concentrator-thermodesorber” unit, a multiple module GC system, and a compact magnetic sector mass spectrometer with a two-stage vacuum system and multicollector ion detector; the advantage of high speed screening of toxic substances in air and extracts is described <sup>(89)</sup></p> <p>Development of a field-deployable, high-resolution, time-of-flight aerosol mass spectrometer is presented <sup>(90)</sup></p> <p>Review of arsenic analysis in the environment with emphasis on field measurements <sup>(91)</sup></p> <p>Evaluation of two arsenic field test kits for water analysis <sup>(92)</sup></p> <p>Describes resistive column heating with smaller mass spectrometry systems specific for field use as a way to design a field-portable GC/MS system with improved performance <sup>(93)</sup></p> <p>Compact, shoebox-sized, handheld mass spectrometer has been developed and its capabilities are tested; continuous monitoring of air and solutions was used to demonstrate its utility <sup>(94)</sup></p> <p>A miniature, battery-operated flame ionization detector (FID) for hydrocarbon detection in portable gas chromatography (GC) is described <sup>(95)</sup></p> <p>Evaluation in the laboratory and in the field of an underwater mass spectrometer using a membrane introduction mass spectrometer (MIMS) for the detection of chemical plumes <sup>(96)</sup></p> <p>Developments for a portable mass spectrometer using an atmospheric pressure inlet and atmospheric pressure chemical ionization (APCI) for real-time monitoring of toxic compounds in air; benzene, toluene, pyridine, and vinyl acetate were the toxic industrial compounds tested and the detection of arsine and dimethyl methylphosphonate (DMMP), a Sarin simulant, were also tested <sup>(97)</sup></p>

Category	Description
<p>Sampling, Sample Preservation, Sample Preparation</p>	<p>Review of theory and techniques for sample preparation (isolation/enrichment) of organic analytes <sup>(98)</sup></p> <p>Methodology for determining if samples are representative with questions to help make assessments about samples taken in the field <sup>(99)</sup></p> <p>Extensive review on the use of tetramethylammonium hydroxide, water-soluble tertiary amines, and strong bases in sample preparation prior to elemental analysis; three highly detailed tables (one for each of the three reagent categories) present sample types, analytes, procedures, analysis techniques, additional remarks and literature references <sup>(100)</sup></p> <p>Tutorial on the theory of sampling involved in acquiring representative samples for reliable data analysis; several terms are defined with examples and numerous illustrations <sup>(101)</sup></p> <p>Miniaturizing sample preparation using fiber-packed capillaries in a technique called fiber-in-tube solid-phase extraction <sup>(102)</sup></p> <p>Sampling of water, soil, and sediment for the analysis of organic pollutants; describes field sampling techniques and provides details on sampling devices including illustrations <sup>(103)</sup></p> <p>Test for better humidity control with an air sample using a Tedlar bag with two layers of film; no major loss of VOCs in using the new design <sup>(104)</sup></p> <p>Presentation of advanced oxidation processes for sample treatment in atomic spectrometry including UV photo-oxidation, ozonation, and ultrasound energy <sup>(105)</sup></p> <p>Discussion of representative samples, sampling theory, and a strategy in forming a laboratory subsampling plan <sup>(106)</sup></p> <p>Example of the effects of poor laboratory subsampling in the study of contaminants in heterogeneous solid waste samples <sup>(107)</sup></p> <p>Stresses the importance of planning ahead to form a sampling design that yields data representative of the site where the sampling occurs; includes a table of different types of sampling designs with references <sup>(108)</sup></p> <p>Briefly covers sample representativeness according to the US EPA data quality objectives perspective; includes section of the macroscale and microscale definitions of representativeness <sup>(109)</sup></p> <p>Preliminary investigation into the proper sampling of biosolids, composts and soils to which biosolids have been applied; presented within the regulatory framework set by the European Commission <sup>(110)</sup></p>
<p>Extractions and Separations</p>	<p>Capillary microextraction (CME) is used for the extraction of semivolatile organic compounds (SVOCs) in water samples with follow-up analysis using GC/MS <sup>(111)</sup></p> <p>Concise biennial review of extraction techniques including advances in membrane-based procedures, molecularly imprinted polymers (MIPs) and several reduced solvent methods including supercritical fluid extraction (SFE), pressurized-liquid extraction (PLE), microwave-assisted extraction (MAE), solid-phase extraction (SPE), and solid-phase microextraction (SPME) <sup>(112)</sup></p>

Category	Description
Extractions and Separations (continued)	<p data-bbox="415 228 1978 321">Dispersive liquid-liquid microextraction (DLLME) was investigated as a new method for the extraction of organophosphorus pesticides (OPPs) from water samples with analysis by gas chromatography-flame photometric detection (GC-FPD); DLLME was found to be simple and rapid and only required a low sample volume; the performance is compared to SPME and single drop microextraction <sup>(113)</sup></p> <p data-bbox="415 350 1978 410">Use of water as an extractant in sample preparations; presents high-temperature water extraction, microwave-assisted water extraction, and ultrasound-assisted water extraction <sup>(114)</sup></p> <p data-bbox="415 440 1978 500">Method using membrane-assisted solvent extraction (MASE) for the extraction of chlorinated and non-chlorinated phosphates from wastewater samples prior to analysis by liquid chromatography tandem mass spectrometry (LC/MS/MS); MASE is compared to SPE <sup>(115)</sup></p> <p data-bbox="415 529 1978 621">Determination of four volatile halocarbons in water by headspace liquid-phase microextraction (HS-LPME) with gas chromatography using a micro electron capture detector (GC-<math>\mu</math>ECD); parameters such as the type and amount of extraction solvent, headspace volume, stirring rate, and others are investigated and optimized <sup>(116)</sup></p> <p data-bbox="415 651 1978 711">Use of liquid-phase microextraction (LPME) coupled with GC for the determination of phenolic compounds in wastewater; extraction parameters are investigated and optimized <sup>(117)</sup></p> <p data-bbox="415 740 1978 800">Comparison of three extraction techniques (ultrasonic, pressurized liquid, and microwave-assisted) for PCBs in sludge; three GC techniques were used in the subsequent analysis of PCBs <sup>(118)</sup></p> <p data-bbox="415 829 1978 889">Review of developments and applications in chiral separations as published from January 2004 to January 2006 including capillary electrophoresis (CE), GC, and HPLC <sup>(119)</sup></p> <p data-bbox="415 919 1978 979">Use of molecularly imprinted polymers (MIPs) as the materials in molecularly imprinted solid-phase extraction in the extraction of selected analytes from environmental samples <sup>(120)</sup></p> <p data-bbox="415 1008 1978 1068">Membrane-assisted solvent extraction using miniaturized membrane bags were applied to the extraction of seven volatile organic compounds (VOCs) from aqueous samples; analysis was performed using gas chromatography (GC) <sup>(121)</sup></p> <p data-bbox="415 1097 1978 1157">Comparison of three extraction techniques (Soxtec, Soxhlet, and pressurized liquid extraction) in the determination of 16 organochlorine pesticides (OCPs) in sewage sludges using GC-ion trap-MS/MS <sup>(122)</sup></p> <p data-bbox="415 1187 1978 1247">A rapid leaching procedure using pressurized liquid extraction (PLE) with acetic acid to extract As, Cd, Cr, Ni, and Pb from marine sediments and soil samples <sup>(123)</sup></p> <p data-bbox="415 1276 1978 1336">Inaugural biennial review of extraction techniques; the author includes articles from 2002 and 2003 and divides the review into the major categories of fluid-phase partitioning methods (SFE, PLE, and MAE) and sorptive extractions (SPE, SPME, and others) <sup>(124)</sup></p>

Category	Description
Extractions and Separations (continued)	<p>Ultrasound-assisted extraction (UAE) as a sample preparation method for inductively coupled plasma optical emission spectrometry (ICP-OES) for the analysis of eight heavy metals (Cd, Co, Cr, Cu, Mn, Ni, Pb, and Zn) in sewage sludge; UAE was found to be less hazardous and cheaper than microwave digestions <sup>(125)</sup></p> <p>Comparison of solid-phase extraction (SPE), pressurized liquid extraction (PLE) and Soxhlet extraction in the simultaneous extraction and determination of the most frequently used anionic surfactants (linear alkylbenzene sulfonates, alkyl ethoxysulfates, and alkyl sulfates) <sup>(126)</sup></p> <p>Application of continuous flow microextraction (CFME) to the analysis of five pesticides (simazine, fensulfothion, etridiazole, mepronil, and bensulide) in water samples by HPLC-UV; includes information on the effects of different organic solvents on the CFME efficiency <sup>(127)</sup></p> <p>Pressurized liquid extraction (PLE) method developed for extracting PAHs and the PAH ketones and PAH quinones (oxygenated PAHs or oxy-PAHs) from soil samples with results similar to Soxhlet extraction but with the advantage of speed and lower solvent use <sup>(128)</sup></p> <p>Use of a solvent extraction method to preconcentrate heavy metal ions in wastewater (Cu, Pb, Mn, Zn, Co, and Ni) for analysis by FAAS <sup>(129)</sup></p> <p>Quick overview of SFE including a chart comparing SFE with Soxhlet extraction, MAE, and accelerated solvent extraction (ASE) <sup>(130)</sup></p> <p>Review of membrane extractions for the preparation of environmental and biomedical liquid samples; includes a table of membrane extraction techniques and analytes for liquid environmental samples <sup>(131)</sup></p> <p>Biennial review of developments and applications of chiral separations; looks at work published from January 2002 to January 2004 <sup>(132)</sup></p> <p>Concise review focusing on methods of extraction of As from soils and sediments for the purpose of achieving a total As concentration; includes a table on sequential extraction schemes for As and the references for each <sup>(133)</sup></p> <p>Determination of tributyltin compounds in water and solid samples using single-drop microextraction (SDME) coupled with gas chromatography/mass spectrometry (GC/MS); results obtained with SDME were compared with results obtained using solid-phase microextraction (SPME) and liquid-liquid extraction (LLE) <sup>(134)</sup></p> <p>Use of supported liquid membrane extraction (SLM) for speciation studies of metal ions in environmental and biological samples <sup>(135)</sup></p> <p>Discussion of past and current use of ionic liquids as extraction solvents <sup>(136)</sup></p>
Pesticides: Herbicides, Insecticides, and Fungicides	<p>Extensive overview comparing the capabilities of gas chromatography mass spectrometry (GC/MS) with electron impact ionization (EI) and liquid chromatography with tandem mass spectrometers (LC/MS/MS) using electrospray ionization (ESI) for the determination of pesticides <sup>(137)</sup></p> <p>Review of the analysis of pesticides in water using LC/MS/MS; the authors include an informative summary table of the methods reported in the literature with details on the sample type, clean-up methods and specifications for the LC/MS/MS analysis <sup>(138)</sup></p>

Category	Description
Pesticides: Herbicides, Insecticides, and Fungicides (continued)	<p data-bbox="415 228 1978 318">Determination of acidic herbicides in aqueous samples using a combination of ion-pair hollow fiber-protected liquid-phase microextraction (LPME) and injection port derivatization before analysis with GC/MS; parameters involved in the LPME and derivatizations steps were evaluated and optimized <sup>(139)</sup></p> <p data-bbox="415 350 1978 410">Use of thin-layer chromatography (TLC) and reversed-phase high performance liquid chromatography (RP-HPLC) in a water stability study for a selected group of sulfonylurea herbicides <sup>(140)</sup></p> <p data-bbox="415 443 1978 472">Analysis for atrazine using an electrochemical magnetoimmunosensing strategy; potential for a simple, fast, on-site analytical method <sup>(141)</sup></p> <p data-bbox="415 505 1978 565">Method for the simultaneous determination of four herbicide families (carbamate, atrazines, dinitroaniline, and chloroacetamide) by solid-phase extraction (SPE) gas chromatography mass spectrometry (GC/MS) <sup>(142)</sup></p> <p data-bbox="415 597 1978 657">Determination of five diphenylether herbicides (aclonifen, bifenoxy, fluoroglyphen-ethyl, oxyfluorfen and lactofen) in water samples using SPME coupled to LC <sup>(143)</sup></p> <p data-bbox="415 690 1978 750">Coupling of capillary electrophoresis (CE) to inductively coupled plasma mass spectrometry (ICP-MS) for the analysis of organophosphorus pesticides; method was tested on river water samples <sup>(144)</sup></p> <p data-bbox="415 782 1978 842">Optimization and evaluation of a method to determine organophosphorus pesticides in spiked river water samples using SPME; the procedure was evaluated on a GC with electron impact ionization mass spectrometry (EI-MS) and on a GC coupled to ICP-MS <sup>(145)</sup></p> <p data-bbox="415 875 1978 904">A microwave-assisted extraction and partitioning method with GC analysis for the determination of pesticides in agricultural soils <sup>(146)</sup></p> <p data-bbox="415 937 1978 997">Development of US EPA Method 535 for the analysis of chloroacetanilide and other acetamide herbicide degradation products in drinking water using SPE and liquid chromatography/tandem mass spectrometry (LC/MS/MS) <sup>(147)</sup></p> <p data-bbox="415 1029 1978 1089">Use of hollow fiber liquid phase microextraction as a sample pre-treatment technique for determining trace concentrations of insecticides in aqueous samples; analysis by gas chromatography flame thermionic detection (GC-FTD) followed the extraction procedure <sup>(148)</sup></p> <p data-bbox="415 1122 1978 1211">Comparison of five inlet liner designs in splitless and pulsed splitless injection modes to optimize pesticide analysis and reduce detection limits; a test mixture of 15 pesticides was analyzed by gas chromatography with electron capture detection (GC-ECD); combinations of liner types and changes to injection parameters were investigated <sup>(149)</sup></p> <p data-bbox="415 1243 1978 1304">Determination of five carbamate pesticides (promecarb, propham, carbaryl, methiocarb, and chlorpropham) in water using derivatization and GC/MS <sup>(150)</sup></p> <p data-bbox="415 1336 1978 1396">Method developed for the identification and quantification of four nicotinoid insecticides (acetamiprid, imidacloprid, thiacloprid, and thiamethoxam) in drinking water using SPE and LC/MS <sup>(151)</sup></p>

Category	Description
Pesticides: Herbicides, Insecticides, and Fungicides (continued)	<p data-bbox="415 224 1806 256">Application of SPME to the rapid screening for seven organophosphate pesticides in drinking water treatment facility samples <sup>(152)</sup></p> <p data-bbox="415 289 1459 321">Review of the advances in the detection of pesticides in a liquid phase by chemiluminescence <sup>(153)</sup></p> <p data-bbox="415 354 1932 410">Design of an on-line sample preparation system to couple with liquid chromatography/time-of-flight mass spectrometry (LC/TOF-MS) and its application to the simultaneous analysis of 21 pesticides in river water <sup>(154)</sup></p> <p data-bbox="415 443 1780 475">Extraction and preconcentration of 10 organophosphorus insecticides from water samples using single-drop microextraction <sup>(155)</sup></p> <p data-bbox="415 508 1932 565">Analysis for nine herbicides and degradation products in water using SPME coupled with GC and ion-trap MS/MS; a method validation study was performed using water from the Seine <sup>(156)</sup></p> <p data-bbox="415 597 1869 654">Details on aspects of US EPA Method 527 including sample preservation and storage; this method uses SPE with analysis by GC/MS to determine pesticides and flame retardants <sup>(157)</sup></p> <p data-bbox="415 686 1953 743">Method for chiral speciation of the pesticide ruelene in different spiked samples including river water; the SPME-enantioselective GC procedure was evaluated using ECD and a phosphorus-specific detector with ICP-MS <sup>(158)</sup></p> <p data-bbox="415 776 1906 833">Use of solid-phase extraction coupled with large volume injection/gas chromatography/mass spectrometry (SPE-LVI-GC/MS) to determine organochlorine pesticides in water <sup>(159)</sup></p> <p data-bbox="415 865 1890 898">Microwave-assisted extraction (MAE) was investigated for the extraction of organochlorine pesticides (OCPs) from sediment samples <sup>(160)</sup></p> <p data-bbox="415 930 1864 987">Ultrasonic solvent extraction of organochlorine pesticides (OCPs) from soil was investigated; variables of solvent type, solvent amount, sonication time, and number of steps were optimized <sup>(161)</sup></p> <p data-bbox="415 1019 1948 1076">Review of applications of thin-layer chromatography (TLC) and high-performance thin-layer chromatography (HP-TLC) for the separation and analysis of a variety of insecticides, herbicides, and fungicides <sup>(162)</sup></p> <p data-bbox="415 1109 1974 1166">A GC/MS method for the determination of 85 compounds (13 fungicides, 43 herbicides, and 29 insecticides) in sediment; the pesticides were first extracted from the sediment using ultrasonically assisted extraction (UAE) <sup>(163)</sup></p> <p data-bbox="415 1198 1894 1255">Evaluation of methods used to determine pesticides and degradation products in soil samples including comparisons of sample preparation methods <sup>(164)</sup></p> <p data-bbox="415 1287 1948 1385">Method for simultaneous determination of phenyl- and sulfonyleurea herbicides and a degradation product by either liquid chromatography with diode array ultraviolet detection (LC/DAD-UV) or liquid chromatography with positive ion electrospray mass spectrometry (LC/ESI-MS) in spiked river water samples <sup>(165)</sup></p>

Category	Description
<p>Polybrominated &amp; Polychlorinated Compounds</p>	<p>Comparison of chromatographic separations and mass spectrometric detection techniques in the detection of 49 polybrominated diphenylethers (PBDEs) <sup>(166)</sup></p> <p>A method for the determination of 25 polychlorinated biphenyls (PCBs) from water is optimized using stir bar sorptive extraction (SBSE); analysis was made using thermodesorption-gas chromatography/mass spectrometry <sup>(167)</sup></p> <p>Use of headspace solid-phase microextraction gas chromatography tandem mass spectrometry (HS-SPME-GC/MS/MS) for the determination of brominated flame retardants in sediment and soil samples <sup>(168)</sup></p> <p>Advances in analytical techniques for the determination of polychlorinated dibenzo-<i>p</i>-dioxins (PCDD), polychlorinated dibenzofurans (PCDF), and dioxin-like PCBs; includes sample extraction methods, extract clean-up, gas chromatography columns and methods, and mass spectrometry detection methods <sup>(169)</sup></p> <p>Discussion of the advantages and disadvantages of the variety of analytical techniques used for the analysis of polychlorinated <i>n</i>-alkanes (PCAs) in environmental samples <sup>(170)</sup></p> <p>Review of analysis of PCAs in environmental samples; includes a table of pre-treatment and extraction methods and a table of mass spectrometry analysis methods <sup>(171)</sup></p> <p>Two-step separation procedure to extract PBDEs, PCBs, and PCDD/Fs in one extraction solution prior to analysis by GC/MS <sup>(172)</sup></p> <p>Study on the use of comprehensive two-dimensional gas chromatography coupled to time-of-flight mass spectrometry (GC x GC/TOF-MS) in the separation of the 209 PCB congeners; four column combinations were investigated <sup>(173)</sup></p> <p>Presentation of results from international interlaboratory studies in the analysis of brominated flame retardants (BFRs); problems in analysis and possible solutions to those problems are discussed <sup>(174)</sup></p> <p>Sample preparation techniques are discussed for the analysis of BFRs in sewage sludge samples <sup>(175)</sup></p> <p>SPME method for the analysis of brominated phenolic flame retardants in water samples <sup>(176)</sup></p>
<p>Polycyclic Aromatic Hydrocarbons (PAHs)</p>	<p>Determination of 16 PAHs in aqueous (wastewaters, industrial discharges, and treated effluents) and solid samples (suspended solids and sludges) from a wastewater treatment plant using SPE coupled to HPLC with UV-diode array detection (UV-DAD) and fluorescence detection (FLD) <sup>(177)</sup></p> <p>Comparison of the performance of two chromatographic techniques (GC/MS and HPLC-FLD-UV) and three extraction techniques (SPE using cartridges, SPE using discs, and LLE) for the analysis of PAHs in drinking water <sup>(178)</sup></p> <p>Extraction of aliphatic diesel range organics (DROs) and PAHs from soil using pressurized solvent extraction (PSE) and ultrasound-assisted pressurized solvent extraction; the analytes were determined using GC/MS <sup>(179)</sup></p>

Category	Description
Pharmaceuticals, Personal Care Products (PPCPs), and Endocrine Disrupting Compounds (EDCs)	<p>Review of analytical methods for the determination of synthetic organic chemicals in personal care products; describes procedures for the analysis of five classes (synthetic musk fragrances, antimicrobials, ultraviolet filters, insect repellents, and parabens) in many sample types including water, sediment, sewage sludge, and air <sup>(180)</sup></p> <p>Review of LC/MS/MS methods for the analysis of pharmaceuticals (including antibiotics, non-steroidal anti-inflammatory drugs, <math>\beta</math>-blockers, lipid regulating agents, and psychiatric drugs) in environmental samples (soil and natural and wastewaters) <sup>(181)</sup></p> <p>A comprehensive method for the analysis of 58 potential endocrine-disrupting compounds (EDCs) in water matrices using SPE and GC/MS/MS and LC/MS/MS <sup>(182)</sup></p> <p>Comparison of SPME and SPE methods for the extraction of 10 sulfonamide antibiotics from wastewater effluent and influent; sample extracts were analyzed using LC/MS/MS <sup>(183)</sup></p> <p>Automated method using direct immersion SPME and headspace on-fiber silylation followed by GC/MS analysis for simultaneous determination of endocrine disrupting compounds and steroid hormones in river water <sup>(184)</sup></p> <p>Analytical and Bioanalytical Chemistry published an issue (volume 387, number 4) primarily dedicated to this topic in February 2007; the issue contains information and analyses on pharmaceuticals and personal care products in the environment and includes many review papers (selected papers in bulleted list below)</p> <ul style="list-style-type: none"> <li>• determination of pharmaceuticals from different therapeutic classes in effluent samples from sewage-treatment plants using reversed-phase liquid chromatography-tandem mass spectrometry <sup>(185)</sup></li> <li>• use of a silicone rod for the extraction and enrichment of selected polar pharmaceuticals (antipyrine, carbamazepine, diclofenac, ibuprofen, and bezafibrate) in spiked water <sup>(186)</sup></li> <li>• review of GC/MS and LC/MS methods for determining non-steroidal anti-inflammatory drugs (NSAIDs) in natural waters and wastewaters <sup>(187)</sup></li> <li>• determination of NSAIDs (ibuprofen, naproxen, ketoprofen, and diclofenac) in river sediment samples by GC/MS; five extraction techniques were investigated to determine the optimal extraction conditions (ultrasonic extraction, Soxhlet extraction, pressurized liquid extraction, supercritical fluid extraction, and microwave-assisted extraction) <sup>(188)</sup></li> <li>• review of chromatographic and electrophoretic techniques for the analysis of NSAIDs in primarily natural waters and wastewaters <sup>(189)</sup></li> <li>• analysis for three antibiotic groups (tetracyclines, sulfonamides, and macrolides) and a feed additive (ionophore polyethers) using SPE-HPLC/MS/MS in water and sediment samples <sup>(190)</sup></li> <li>• review of developments in the determination of fluoroquinolones (antibacterial agents in human and veterinary medicine) in soil <sup>(191)</sup></li> <li>• development of a GC/MS method for the analysis of five major steroid estrogens in river sediments <sup>(192)</sup></li> <li>• review of LC/MS and LC/MS/MS analyses for pharmaceutically active compounds in water systems <sup>(193)</sup></li> <li>• review of LC/MS analysis of lipid-regulating agents, “fibrate” and “statin” classes, and some of the metabolites (clofibric acid and fenofibric acid) in environmental samples (surface and wastewaters, sludge, and river sediments) <sup>(194)</sup></li> <li>• analysis for ultraviolet filters and two common anti-microbial agents (clorophene and triclosan) in recreational waters (seawater, lake water, river water, and pool water) and in wastewaters <sup>(195)</sup></li> </ul>

Category	Description
Pharmaceuticals, Personal Care Products (PPCPs), and Endocrine Disrupting Compounds (EDCs) (continued)	<p>Quantification of 16 antibiotics (10 quinolones and 6 penicillins) in ground and surface water using solid-phase extraction coupled on-line to liquid chromatography/electrospray tandem mass spectrometry (SPE-LC-ESI-MS/MS) <sup>(196)</sup></p> <p>Application of SPE-HPLC to the determination of important veterinary pharmaceuticals from different classes (three sulfonamides, a sulfonamide synergist, a tetracycline, a fluoroquinolone, and a <math>\beta</math>-lactam) in a complex wastewater matrix <sup>(197)</sup></p> <p>Analysis for five betablockers (oxprenolol, metoprolol, propranolol, bisoprolol, and betaxolol) in wastewater treatment plant effluents from the Lyon, France area; analysis was performed using SPE and GC/MS after derivatization <sup>(198)</sup></p> <p>Review of methods of analysis of bisphenol A and its derivatives; includes summary tables with GC and HPLC methods, matrices, sample preparation details, chromatographic details, and detection limits <sup>(199)</sup></p> <p>Determination of pharmaceuticals (antiphlogistics, lipid regulators, the antiepileptic carbamazepine, cytostatic agents, and the psychiatric drug diazepam), iodinated contrast media, and two major polycyclic musk fragrances in activated and digested sludge; the procedure consisted of ultrasonic solvent extraction or pressurized liquid extraction followed by clean-up and LC/MS/MS and GC/MS analysis <sup>(200)</sup></p> <p>Simultaneous determination of psychoactive drugs and metabolites (including codeine, hydrocodone, methadone, morphine, oxycodone, diazepam, oxazepam, carbamazepine, primidone, doxepin, and verapamil) in raw and treated wastewater, surface water, groundwater, and drinking water using LC/MS/MS <sup>(201)</sup></p> <p>Method for the analysis of six anti-infectives (sulfamethoxazole, trimethoprim, ciprofloxacin, levofloxacin, clarithromycin, and azithromycin) in raw sewage and wastewater plant effluents using SPE and LC/MS/MS <sup>(202)</sup></p> <p>Use of CE and electrospray ionization mass spectrometric detection (ESI-MS) for the detection of antidepressants in river water and municipal sewage treatment plant effluent <sup>(203)</sup></p> <p>Screening of more than 60 EDCs in water samples using stir bar sorptive extraction followed by liquid desorption and large volume injection with capillary gas chromatography-mass spectrometry <sup>(204)</sup></p> <p>Application of hollow fiber liquid-phase microextraction (LPME) for the extraction and enrichment of acidic pharmaceuticals (including naproxen, clofibric acid, ibuprofen, and diclofenac) in treated and untreated wastewater samples and analyzed by LC-ESI-MS/MS <sup>(205)</sup></p> <p>Determination of estrogenic steroids (17-ethynylestradiol, 17-estradiol, and estrone) in surface water and wastewater (sewage influent and effluent) using automated SPE and LC/MS/MS <sup>(206)</sup></p> <p>Rapid and simultaneous analysis of 15 stimulatory drugs including nicotine, caffeine, amphetamine, cocaine, lysergic acid diethylamide (LSD), and phencyclidine (PCP) in wastewater and surface river water using ultra-performance liquid chromatography coupled to electrospray tandem mass spectrometry <sup>(207)</sup></p>

Category	Description
Pharmaceuticals, PPCPs, and EDCs (continued)	<p data-bbox="415 228 1896 289">Analysis for triclocarban and two co-contaminants in surface water samples affected by wastewater treatment plant effluent and in primary sludges from municipal wastewater treatment plants using LC/MS/MS <sup>(208)</sup></p> <p data-bbox="415 315 1602 347">Measurement of illicit drugs and metabolites in urban wastewater (untreated and treated) using LC/MS/MS <sup>(209)</sup></p>
Speciation	<p data-bbox="415 354 1940 443">Comparative study of three different techniques, capillary electrophoresis-ultraviolet (CE-UV) detection, hydride generation-atomic absorption spectrometry (HG-AAS), and liquid chromatography-inductively coupled plasma mass spectrometry (LC-ICP-MS), for the speciation of As in groundwater samples; As<sup>III</sup>, As<sup>V</sup>, and dimethyl arsenic acid (DMA) were studied <sup>(210)</sup></p> <p data-bbox="415 472 1398 505">Review of the speciation methods of alkylated metals and metalloids in the environment <sup>(211)</sup></p> <p data-bbox="415 532 1566 565">On-line speciation and determination of Cr(III) and Cr(VI) in aqueous solutions using HPLC and FAAS <sup>(212)</sup></p> <p data-bbox="415 592 1646 625">The analytical performance of two ion-exchange columns are compared in the determination of Cr(VI) in water <sup>(213)</sup></p> <p data-bbox="415 652 1913 716">Investigation of the addition of ethylene diamine tetraacetic acid (EDTA) and acetic acid to drinking water samples as a means of preserving As(III) and As(V) and preventing the formation of iron-arsenic precipitates <sup>(214)</sup></p> <p data-bbox="415 743 1969 833">Separation of Cr(VI) from Cr(III) by coprecipitation of a Cr(VI) ethyl xanate complex onto naphthalene; the Cr(VI) complex and naphthalene were dissolved in dimethyl formamide and analyzed by flame atomic absorption spectrometry (FAAS); the method was tested on tannery effluent samples <sup>(215)</sup></p> <p data-bbox="415 860 1503 893">Review of Al speciation in environmental samples; lists techniques and methods for Al speciation <sup>(216)</sup></p> <p data-bbox="415 920 1885 984">Sequential determination of arsenic species in water by varying the pH and using a borohydride reduction, reacting arsine with ozone, and measuring the resulting chemiluminescence <sup>(217)</sup></p> <p data-bbox="415 1011 1247 1044">Review article on As and its speciation analysis using HPLC and ICP-MS <sup>(218)</sup></p> <p data-bbox="415 1071 1913 1136">Study of mobile phases in high-performance thin-layer chromatography (HP-TLC) for the separation of Cr<sup>6+</sup> and Cr<sup>3+</sup> and other heavy metal cations <sup>(219)</sup></p> <p data-bbox="415 1164 1969 1227">Review on analysis and speciation of As by voltammetry; includes large table of methods with many environmental sample matrices (soil, mining waters, industrial effluents, sewage sludge, sediment, polluted river water, groundwater, etc.) <sup>(220)</sup></p>
Mercury	<p data-bbox="415 1268 1896 1328">Overview of mercury in environmental samples; includes speciation and general overview of sampling, extraction, separation, and analysis methods <sup>(221)</sup></p> <p data-bbox="415 1356 1583 1388">Direct determination of mercury(II) in hazardous wastes using ion-selective polymeric membrane sensors <sup>(222)</sup></p>

Category	Description
Atomic Spectrometry	<p data-bbox="415 232 1978 289">Overview of gas-phase sample introduction techniques for analytical atomic spectrometry including hydride generation, cold vapor generation of Hg, and analyte volatilization reactions <sup>(223)</sup></p> <p data-bbox="415 321 1978 378">Annual review of atomic spectrometry as applied to environmental analysis; sections are categorized by media type being analyzed (air, water, soils and plants, and geologic materials); this update includes work from 2005 and 2006 <sup>(224)</sup></p> <p data-bbox="415 410 1978 467">Annual review of advances in atomic emission, absorption, and fluorescence spectrometry including a good glossary of abbreviations and over 400 references <sup>(225)</sup></p> <p data-bbox="415 500 1978 532">Biennial review with emphasis on innovations in atomic absorption (AA), fluorescence (AF), and emission spectrometry (AES) <sup>(226)</sup></p> <p data-bbox="415 565 1978 621">Annual review of the use of atomic spectrometry in the chemical analysis of environmental samples divided by media type; references from work published in 2004 and 2005 are included in this update <sup>(227)</sup></p> <p data-bbox="415 654 1978 711">Annual review on new developments in atomic spectrometry; solid-phase extraction for preconcentration and matrix separation prior to atomic spectrometry analysis was the most popular topic in the literature <sup>(228)</sup></p> <p data-bbox="415 743 1978 800">Annual review of publications addressing the application of atomic spectrometry to environmental analysis; categories are provided by the type of media in the analysis; references from publications in 2003 and 2004 <sup>(229)</sup></p> <p data-bbox="415 833 1978 889">Annual review of advances throughout atomic spectrometry (atomic emission, atomic absorption, atomic fluorescence, etc.) during 2004; includes a large section on hydride generation reactions for the analysis and determination of many metals of interest in environmental analysis <sup>(230)</sup></p> <p data-bbox="415 922 1978 954">Review on developments in microwave-induced plasmas and including coupling of MIP to mass spectrometry as an alternative to ICP-MS <sup>(231)</sup></p>
Atomic Absorption Spectrometry (AAS)	<p data-bbox="415 967 1978 1024">Comparison of open vessel digestion and microwave-assisted digestion on soil samples; analysis for heavy metals was performed using flame atomic absorption spectroscopy <sup>(232)</sup></p> <p data-bbox="415 1057 1978 1114">Developments in electrothermal atomization (ETA) atomic absorption spectrometry with references to ETA-AAS application including the analysis of environmental samples <sup>(233)</sup></p>
Capillary Electrophoresis (CE)	<p data-bbox="415 1122 1978 1154">Review of the current status for the analysis of inorganic and charged small organic species by CE <sup>(234)</sup></p> <p data-bbox="415 1187 1978 1243">Discussion of precision problems in CE; presents the factors behind nonreproducibility in CE methods (buffers, capillary, injection modes, and voltage) and suggestions to improve reproducibility <sup>(235)</sup></p> <p data-bbox="415 1276 1978 1333">Use of microporous membrane liquid-liquid extraction (MMLLE) and capillary electrophoresis for the simultaneous analysis of eight aromatic amines in water samples <sup>(236)</sup></p> <p data-bbox="415 1365 1978 1422">Microchip CE with electrochemical detection analysis for separating and detecting environmental pollutants is reviewed including recent advances and the discussion of portable devices <sup>(237)</sup></p>

Category	Description
Gas Chromatography (GC)	<p data-bbox="415 228 1913 285">Review of developments in gas chromatography (GC) published in 2004 and 2005 with emphasis on significant advances in GC; the review includes sections on high-speed and portable GC and on multidimensional GC <sup>(238)</sup></p> <p data-bbox="415 321 1919 378">Discussion of the problem of selecting the right injector for the capillary GC analysis of semi-volatile organic contaminants in environmental samples; this review examines pulsed splitless injection, cold on-column injection, and programmable temperature vaporizer injectors <sup>(239)</sup></p> <p data-bbox="415 414 1898 470">Analysis of semi-volatile halogeno-organic compounds in sewage samples using headspace or solid-phase extraction methods prior to final analysis by gas chromatography-electron capture detection (GC-ECD) <sup>(240)</sup></p> <p data-bbox="415 506 1881 592">Quantification of chemical substances found in leachates from 11 different types of industrial wastes using a gas chromatograph-flame photometric detector (GC-FPD), a gas chromatograph-flame thermionic detector (GC-FTD), and a gas chromatograph/mass spectrometer (GC/MS); alkanes, PAHs, and phthalates were the substances detected in the highest concentrations in the wastes <sup>(241)</sup></p> <p data-bbox="415 628 1971 652">Review of the use of enantiomeric GC for chiral separations; references provided for the separation and analysis of organochlorines and PCBs <sup>(242)</sup></p> <p data-bbox="415 688 1602 712">Using synchronized dual-valve injection as a tool in achieving high-speed gas chromatographic separations <sup>(243)</sup></p> <p data-bbox="415 748 1919 805">Analysis of epichlorohydrin in water and sewage samples using direct aqueous injection into the GC chromatographic column; four detectors were used for the analysis and five different extraction methods were evaluated <sup>(244)</sup></p> <p data-bbox="415 841 1486 865">Review of modern GC techniques used for the determination of PAHs in environmental samples <sup>(245)</sup></p> <p data-bbox="415 901 1581 925">Evaluation of peak shapes to help select optimum analytical conditions for efficient and rapid separations <sup>(246)</sup></p> <p data-bbox="415 961 1864 1018">Study of two-dimensional gas chromatography for the separation of organohalogenated compounds; five combinations of columns were investigated and the methodology was tested on sediment and dust extracts <sup>(247)</sup></p> <p data-bbox="415 1053 1913 1140">Review series on developments in two-dimensional gas chromatography (GC x GC) including lists of GC x GC papers for the analysis of organohalogens in the third paper and for the analysis of pollutants in environmental samples (soil, sediment, water, air, and aerosols) in the fourth paper; references are listed at the end of the first paper in the series <sup>(248-250)</sup></p> <p data-bbox="415 1175 1514 1200">History of GC x GC including a review of theory, high resolution GC x GC and operational modes <sup>(251)</sup></p> <p data-bbox="415 1235 1377 1260">Concise review of comprehensive GC x GC in environmental analysis and monitoring <sup>(252)</sup></p> <p data-bbox="415 1295 1499 1320">Review of advances in GC instrumentation divided into hardware advances and column advances <sup>(253)</sup></p> <p data-bbox="415 1356 1885 1412">Determination of total PCB content in industrial oil samples using gas chromatography atomic emission detection (GC-AED); PCBs were extracted using dimethylsulfoxide and water and concentrated using headspace solid-phase microextraction (HS-SPME) <sup>(254)</sup></p>

Category	Description
Gas Chromatography/Mass Spectrometry (GC/MS)	<p>Study of the analysis of polycyclic aromatic compounds in the extracts from highly complex environmental samples such as soils from gasworks, coke production sites, areas where wood preservatives were used, antiskid sand, dust from heavy traffic areas, and ash and soot from municipal solid waste incinerators <sup>(255)</sup></p> <p>A method for the analysis of phenols in water by GC/MS is presented; the method involves derivatization of the phenols, liquid-liquid extraction (LLE) of the phenyl acetate esters and then analysis by GC/MS <sup>(256)</sup></p> <p>Use of GC/MS to identify inorganic anions after derivatization using pentafluorobenzyl <i>p</i>-toluenesulphonate (PFB-Tos) <sup>(257)</sup></p> <p>Simultaneous determination of MTBE, its main degradation products, and other gasoline additives in soil samples using closed-system purge-and-trap GC/MS <sup>(258)</sup></p> <p>Identification of an artifact in gas chromatography caused by the leaching of the substance from rubber septa used in sample vial caps <sup>(259)</sup></p> <p>SPME coupled to GC/MS for the determination of pesticide residues (31 pesticides of different chemical groups) in environmental water samples <sup>(260)</sup></p> <p>Development of a mist chamber for the analysis of atmospheric acrolein concentrations <sup>(261)</sup></p> <p>Analysis for organic contaminants in the atmosphere over a 12-month period of time using SPE and GC/MS <sup>(262)</sup></p> <p>Enhanced detectability of PBDEs in environmental matrices using gas chromatography high resolution time-of-flight mass spectrometry (GC/TOF-MS) with negative ion chemical ionization <sup>(263)</sup></p> <p>Seasonal sampling and analysis for VOCs in industrial sewage, rivers, and treated water samples; 53 VOCs were analyzed using headspace analysis with GC/MS <sup>(264)</sup></p>
High Performance Liquid Chromatography (HPLC)	<p>Review of strategies used for mercury species analysis in environmental samples using liquid chromatography; includes discussions of reverse phase and ion pair chromatography <sup>(265)</sup></p> <p>Determination of seven pesticides (amidosulfuron, azimsulfuron, nicosulfuron, rimsulfuron, thifensulfuron methyl, tribenuron methyl, and azoxystrobin) in surface waters using HPLC with UV and MS detection <sup>(266)</sup></p>
Liquid Chromatography/Mass Spectrometry (LC/MS)	<p>Brief overview of progress of LC/MS and LC-tandem MS (LC/MS/MS) in environmental analysis <sup>(267)</sup></p> <p>Analytical method for the identification of 13 phosphoric acid mono- and diesters in aqueous samples using solid-phase extraction (SPE) and ion-pair liquid chromatography coupled to electrospray ionization tandem mass spectrometry <sup>(268)</sup></p> <p>Review of matrix effect problems experienced in LC/MS; the quantitative analysis of pesticides in water, other environmental matrices, food (residue analysis), and biologicals is discussed <sup>(269)</sup></p>

Category	Description
Liquid Chromatography/ Mass Spectrometry (LC/MS) (continued)	<p>Quantitative determination of perfluorochemicals in sediments and sludge using HPLC coupled to a tandem mass spectrometer (LC/MS/MS) <sup>(270)</sup></p> <p>Review of the analysis of fluorinated alkyl compounds using LC/MS/MS; includes a tabular overview with lists of analytes and method details <sup>(271)</sup></p> <p>Brief summary of LC/MS in the analysis of emerging environmental contaminants with a list of LC/MS techniques <sup>(272)</sup></p> <p>Determination of chlorophenols in surface water using a solid-phase extraction (SPE) procedure to concentrate the samples prior to analysis with a hybrid quadrupole time-of-flight spectrometer <sup>(273)</sup></p> <p>Developments in atmospheric pressure photoionization (APPI) as a complement to LC/MS including references to analysis of environmental samples <sup>(274)</sup></p>
Ion Chromatography (IC)	<p>Review of ion chromatography developments with an emphasis on environmental analysis <sup>(275)</sup></p> <p>Use of macrocycle-based ion chromatography for the determination of perchlorate in drinking water <sup>(276)</sup></p> <p>Review of the application of IC to the determination of inorganic anions and cations in water and wastewater; provides characteristics of the International Organization for Standardization (ISO) standard methods and US EPA methods, tables of the methods with listings of analytes, columns, eluents, detection modes, sample matrices, and references <sup>(277)</sup></p> <p>Analyses for the synthetic chelating agents ethylenediamino tetraacetate (EDTA) and diethylenetriamino pentaacetate (DTPA) in surface and wastewater using ion chromatography-mass spectrometry (IC-MS) <sup>(278)</sup></p> <p>Use of high-speed ion chromatography in the separation of inorganic anions and cations; covers recent developments and the advantages over normal liquid chromatography <sup>(279)</sup></p>
Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) or -Optical Emission Spectrometry (ICP-OES)	<p>Study of both instrument measurement precision (repeatability) and overall method precision (reproducibility) in the elemental analysis of soils and certified reference materials by both ICP-AES and ICP-MS <sup>(280)</sup></p> <p>On-line preconcentration with mono-segmented elution for the determination of Cd, Cu, Ni, and Zn in soil samples by ICP-OES <sup>(281)</sup></p> <p>Review of sample introduction systems for plasma spectrometry for liquid microsamples (liquid consumption rate is less than 100 <math>\mu</math>L/min); applications table includes examples of metals analysis in environmental samples <sup>(282)</sup></p> <p>Use of a microwave-assisted sample introduction system for organic solvent solutions (up to 10% by weight) into ICP-AES <sup>(283)</sup></p> <p>Addresses spectral interferences by Al, Ca, Mg, Fe, and Ti in the ICP-AES radial-view analysis for As, Hg, Se, Tl, Sn, and Bi in environmental materials <sup>(284)</sup></p> <p>Simultaneous measurement of As, Sb, Se, Sn, and Ge using an electrochemical hydride generator prior to analysis by ICP-AES <sup>(285)</sup></p>

Category	Description
Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)	<p>Review of papers published for inductively coupled plasma mass spectrometry (ICP-MS) during 2003 through 2005; the author observed that many published papers are only of marginal significance <sup>(286)</sup></p> <p>Simultaneous multi-element determination by ICP-MS using a new dual mode sample introduction system and a sodium borohydride reaction <sup>(287)</sup></p> <p>Use of an on-line preconcentration system for ICP-MS in the determination of 10 metals in aqueous solutions and seawater <sup>(288)</sup></p> <p>Discusses the changes in analytical performance of double focusing sector field inductively coupled plasma mass spectrometry (ICP-SFMS) when methane gas is added to the argon gas ICP; results from the analysis of isotopes from 70 elements were studied <sup>(289)</sup></p> <p>Review of the use of ICP-MS for elemental speciation; presenting work coupling GC, LC, CE, and field flow fractionation with ICP-MS <sup>(290)</sup></p>
Microwave-Assisted Extraction (MAE)	<p>Review of the application of microwave techniques to environmental analytical chemistry including sample digestion, sample drying, and applications for the determination of pesticides, PAHs, PCBs, phenols, and organometallic compounds <sup>(291)</sup></p> <p>Investigation of microwave-assisted solvent extraction of persistent organochlorine pesticides (POPs) in marine sediment; variables of microwave power, extraction time and temperature, amount of sample, solvent volume and sample moisture were studied; extracts were analyzed using GC/MS/MS <sup>(292)</sup></p> <p>Review of microwave-assisted processes for environmental samples; presents the history and fundamentals with a quick review of the literature from 2000 to 2006 <sup>(293)</sup></p> <p>The results and advantages of the MAE technique for the determination of PCBs and OCPs in marine sediment samples is presented; MAE is compared to other extraction techniques including pressurized fluid extraction (PFE) and Soxhlet extraction <sup>(294)</sup></p> <p>Use of MAE to extract 7 organochlorine pesticides (OCPs), 11 polychlorinated biphenyls (PCBs) and 13 polycyclic aromatic hydrocarbons (PAHs) from semi-permeable membrane devices (SPMDs) used to accumulate the hydrophobic chemicals; yields from the MAE were deemed to be comparable to those obtained using dialysis on the SPMDs <sup>(295)</sup></p> <p>Method for the simultaneous extraction of PAHs, PCB, phthalate esters, nonylphenols and nonylphenol mono- and diethoxylates from sediment samples using MAE and acetone as the common solvent <sup>(296)</sup></p> <p>Validation of a MAE method for the extraction of eight phenols from a reference soil sample with a comparison to Soxhlet extraction <sup>(297)</sup></p> <p>Study of the effects of matrix, moisture content, aging (sample holding time), and solvent on focused microwave-assisted extractions (FMAE) of PAHs in spiked soils <sup>(298)</sup></p>
Mass Spectrometry (MS)	<p>Annual atomic mass spectrometry update focused on significant developments in instrumentation and methodology or in the understanding of the mass spectrometry process; applications of atomic mass spectrometry are not covered in the review of 2005 publications <sup>(299)</sup></p>

Category	Description
Mass Spectrometry (MS) (continued)	<p>Annual update of developments in atomic mass spectrometry; sections are divided into different types of mass spectrometry (inductively coupled plasma mass spectrometry, secondary ion mass spectrometry, glow discharge mass spectrometry, electrospray mass spectrometry, etc.); the work referenced in this review is from 2004 publications; applications of mass spectrometry are not covered in the review <sup>(300)</sup></p> <p>Biennial mass spectrometry review focused on environmental analysis—current research issues and emerging contaminants; review covers literature published during 2004 and 2005 with a few references from early 2006 <sup>(301)</sup></p> <p>Testing of atmospheric pressure ionization mass spectrometry (API-MS) for the analysis of nine toxic industrial compounds and two chemical warfare agent (CWA) simulants; parts-per-billion (ppb) and parts-per-trillion (ppt) detection limits and low relative standard deviations were achieved from the analyses of all compounds even in the presence of interferents <sup>(302)</sup></p> <p>New methodology for the measurement of benzene, toluene, ethylbenzene, and p-xylene (BTEX) in drinking waters using headspace-mass spectrometry (HS-MS); narrowing the data acquisition time window and using a lower scan rate improved the HS-MS results <sup>(303)</sup></p> <p>Annual review of atomic mass spectrometry with the focus on developments in instrumentation and methodology; speciation papers included in the review were limited to the study of natural systems; applications of atomic mass spectrometry were not covered in this review <sup>(304)</sup></p>
Solid-Phase Extraction (SPE)	<p>Comparison of five different SPE sorbents for the simultaneous extraction and analysis (by HPLC) of 16 pesticides <sup>(305)</sup></p> <p>Use of polyurethane foam as a sorbent in solid-phase extraction for trace metals preconcentration; lists analytes, sample types, preconcentration methods, and analysis techniques in tabular form <sup>(306)</sup></p> <p>Combination of SPE and “stacking with matrix removal” (SWMR) and capillary electrophoresis-ultraviolet (CE-UV) analysis to determine herbicides in water <sup>(307)</sup></p> <p>Determination of Pb, Fe, Mn, and Zn in sea water samples after a solid-phase extraction preconcentration step using Chromosorb 105 prior to analysis by AAS; excellent recoveries of the metals and low relative standard deviations were achieved <sup>(308)</sup></p> <p>Examination of new polymer sorbents for the extraction of polar compounds; recoveries from tests of analyte mixtures are reported <sup>(309)</sup></p>
Solid-Phase Microextraction (SPME)	<p>Review of current applications and predictions for future developments in analytical microextractions using SPME and liquid-phase microextraction (LPME); provides basic theory and calculations for SPME, results from a round robin study on pesticide analysis by SPME, and a brief discussion on switching from more traditional extraction methods to SPME <sup>(310)</sup></p> <p>Use of a needle capillary adsorption trap for headspace sampling of BTEX compounds in water samples; comparison of results to SPME <sup>(311)</sup></p> <p>Review of the applications of solid-phase microextraction-high performance liquid chromatography (SPME-HPLC) in the analysis of toxic metals species of As, Cr, Pb, Hg, and Se in complex environmental matrices (wastewater and industrial effluents) <sup>(312)</sup></p> <p>Analysis for explosives in soil using SPME and GC/MS; the temperature control and the time of the SPME extraction period are emphasized as key factors in achieving quantitative information <sup>(313)</sup></p>

Category	Description
Solid-Phase Microextraction (SPME) (continued)	<p>Review of advances in the use of SPME in environmental analysis including devices developed for on-site sampling <sup>(314)</sup></p> <p>Use of direct fiber cooling and freezing of analyte solution to improve HS-SPME for the analysis of VOCs <sup>(315)</sup></p> <p>Extraction and analysis of 1,4-dioxane using SPME with heated headspace (HS) or direct immersion and followed by analysis by either GC/FID or GC/MS <sup>(316)</sup></p> <p>Review of developments in SPME coating and techniques: includes seven fiber coating procedures and the single-drop, liquid-phase, stir-bar sorptive, and thin-film microextraction techniques <sup>(317)</sup></p> <p>Combination of SPME to a variety of analytical and detection techniques for the determination of elements (Sn, Hg, As, Sb, Cr, Se, and Pb) in a wide variety of matrices is reviewed <sup>(318)</sup></p> <p>The coupling of SPME and CE and the problems with SPME-CE coupling are discussed; analytes used in testing included PAHs and proteins <sup>(319)</sup></p> <p>Review of SPME-HPLC for the analysis of pesticides in water, soil, and biological samples; types of fibers and the application to different pesticides is discussed <sup>(320)</sup></p> <p>Evaluation of two SPME fibers for the determination of organochlorine pesticides (OCPs) in water samples; analysis was completed using HPLC with UV-diode array detection (UV-DAD) <sup>(321)</sup></p> <p>Determination of five volatile organochlorine compounds (VOX) in landfill leachates by GC/MS; static headspace (HS) and solid-phase microextraction sampling of headspace (HS-SPME) are the two extraction and preconcentration techniques evaluated; both techniques had specific strengths and both provided good recoveries and analytical precision <sup>(322)</sup></p> <p>Technique for the on-fiber standardization of solid-coated solid-phase microextraction (SPME) as applied to the analysis of pesticides in river water and white wine and for drug analysis in plasma and whole blood samples <sup>(323)</sup></p> <p>Use of activated carbon fiber (ACF) for extraction of chlorohydrocarbons in water; paper focuses on the adsorption capacity of the ACF and provides a comparison to other fiber types <sup>(324)</sup></p>
Stir Bar Sorptive Extraction (SBSE)	<p>Review of stir bar sorptive extraction (SBSE) for the extraction and enrichment of organic compounds from liquid samples; the technique has been applied to the trace analysis of environmental and biological samples <sup>(325)</sup></p> <p>Fast pesticide screening in aqueous samples using dual SBSE (two stir bars) prior to thermal desorption-low thermal mass gas chromatography-mass spectrometry; 82 pesticides were studied <sup>(326)</sup></p> <p>SBSE followed by liquid desorption and large-volume injection capillary gas chromatography mass spectrometry (LVI-GC/MS) for the determination of eight pyrethroid pesticides in water samples <sup>(327)</sup></p>

Category	Description
Stir Bar Sorptive Extraction (SBSE) (continued)	Use of a polydimethyl siloxane (PDMS) stir bar as an extraction medium for derivatized phenols in a method to determine phenols in natural water samples; analytes are thermally desorbed from the stir bar and are analyzed by GC/MS <sup>(328)</sup>
X-ray Spectrometry (XRS)	<p>Evaluation of a low power total reflection X-ray fluorescence (TXRF) spectrometer for the on-site analysis of soils and sediments contaminated with heavy metals; tables present target concentration ranges for 13 elements, detection limits, and comparison to laboratory reference values <sup>(329)</sup></p> <p>Review of the work showing significant and essential progress in X-ray spectrometry that was published during 2004 and 2005 <sup>(330)</sup></p> <p>Annual review of X-ray fluorescence (XRF) developments during 2005 and 2006; developments covered include instrumentation and detectors, trends in analysis, and a survey of applications including environmental <sup>(331)</sup></p> <p>Annual review of XRF developments over the period of 2004 to 2005 that includes instrumentation, detectors, and a survey of applications including environmental <sup>(332)</sup></p> <p>Tutorial in the basic concepts for the mathematical correction for matrix effects in XRF analysis <sup>(333)</sup></p>

## SPECIAL TOPICS

### WASTE “TRADE”

The Basel Convention on the Transboundary Movement of Hazardous Wastes and Their Disposal is an international agreement to regulate the export of hazardous wastes between countries. In order to protect human health and the environment, the agreement requires that receiving countries agree to take the waste and ensure that the materials are disposed of in an environmentally sound manner. Wastes that are exported unknowingly to the receiving country or accepted by the receiving country by deception (claiming the materials are not hazardous or are less hazardous than in actuality) or exported for the purpose of dumping (avoiding proper disposal) constitute illegal wastes by the agreement.<sup>(334)</sup>

The European Union has a web page within its Environment Section specifically dedicated to issues dealing with the shipment of waste and a new EU waste shipment regulation came into effect on 12 July, 2007.<sup>(335, 336)</sup>

Electronic waste, the after-effects of the computer and cell phone generation, and ship-breaking waste, the results of disassembly of sea vessels, are two significant hazardous waste issues that are generating international concern and forcing nations into decisions of legality for the generation and treatment of these waste streams.

#### Electronic Waste

Electronic waste or e-waste consists of used or end-of-life electronics including computers, monitors, cellular phones, printers, photocopy machines, and television sets. E-waste might be recycled, salvaged for parts, or disposed of in multiple ways, but regardless of the next destination for the electronic item, the toxicity potential for that electronic item is high.

E-waste is estimated by the Basel Convention to be growing at a rate of 3 to 5 percent each year.<sup>(337)</sup> Estimates presented on Basel Convention pamphlets state that 500 million computers will become obsolete in the US alone between 1997 and 2007 and that 610 million cellular phones will be discarded in Japan by 2010.<sup>(338)</sup> Beyond the sheer quantity of e-waste that develops from year-to-year, electronic items contain heavy metals (including Pb, Cd, Be, and Hg), polycyclic aromatic hydrocarbons (PAHs), and polychlorinated and polybrominated organic chemicals such as brominated flame retardants (BFRs) and polychlorinated biphenyls (PCBs).

Switzerland is highly regarded as the international leader in e-waste recycling with an established formal system to manage the waste.<sup>(339)</sup> This is in contrast to the e-waste that ends up in landfills, on waste piles, in unpermitted dumps, as filler material in swamps, and at sites where rudimentary and hazardous recycling processes take place. E-wastes are exported in large quantities to recycling sites in China, Africa, Pakistan, and India, but the most well-known site is Guiyu in the Quandong Province of China.<sup>(340, 341)</sup> Workers break apart, crush, strip, burn or otherwise separate the electronic parts to find reusable material. This process can expose workers to much of the toxic materials in the electronic waste.<sup>(342)</sup>

Leaching studies, atmospheric studies, and analysis for metals, PAHs, and chlorinated and brominated compounds have been performed on samples from e-waste disassembly sites or on samples designed by researchers to mimic what can be found at those sites.

Several leaching studies use or modify the method of the US EPA toxicity characteristic leaching procedure (TCLP). Samples used in these studies were disassembled computer central processing units (CPUs), cell phones (without including the batteries), motherboards from the printed wire boards from computer CPUs, cathode ray tubes (CRTs) from televisions (TVs) and computer monitors, and a combination of miscellaneous electronics wastes (including computer CPUs, video cassette recorders, computer printers, remote controls, CRTs from computers and TVs, and liquid crystal displays).<sup>(343-346)</sup>

One study on total suspended particulate (TSP) in the atmosphere was performed using samples collected at the Guiyu electronic waste recycling site. High concentrations of both PAHs and heavy metals were found to be present.<sup>(347)</sup> Another atmospheric study simulated the open, uncontrolled combustion of e-waste (such as circuit boards and wire) in order to reduce these parts into metals only (for example, burning to remove plastics such as insulated covers on wire). At an e-waste site, the metals would be separated from the ash by water floatation. This study sampled and analyzed the air emissions and fly ash from the simulated burn for elemental composition and polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/Fs).<sup>(348)</sup>

Several papers describe analyses for chlorinated and brominated compounds from samples taken at e-waste recycling sites. Soil samples from the Taizhou city (Zhejiang province in China) recycling site were analyzed to determine the concentration of polybrominated diphenyl ethers (PBDEs).<sup>(349)</sup> Water, soil, sediment, and combusted residues from the Guiyu recycling site have been analyzed for metals, PBDEs, polybrominated dibenzo-*p*-dioxins and dibenzofurans (PBDD/Fs), and PCDD/Fs.<sup>(350-354)</sup>

## Shipbreaking

As ships age, repairs or upgrades will be needed in order for the ships to maintain some utility for the ship owner. When this expense exceeds the reasonable value of the aging ship, then the means of disposal of the ship comes into consideration and this may include the dismantling of the ship.

Ship dismantling, scrapping, or breaking can be defined as “any breaking of a vessel’s structure for the purpose of scrapping the vessel, including the removal of gear, equipment, or any component of a vessel.”<sup>(355)</sup> While some materials from older ships can be reused, the dismantling process cannot be considered trivial due to the vast amount of hazardous materials present as part of the ship. Ships contain hazards such as waste fuel and oil, asbestos, PCBs, and heavy metals such as mercury, lead, cadmium, and chromium. Waste disposal regulations in many Western nations make the disposal of ships very expensive and some ship owners may choose to have their vessels dismantled in countries with fewer or no restrictions for health and safety.

Organizations internationally are concerned about the ship breaking hazards to humans and the environment and many are involved in forming guidelines and regulations for the shipping industry. The Marine Environment Protection Committee (MEPC) of the International Maritime Organization (IMO) has set a goal of April 2009 for the adoption of the International Convention for the Safe and Environmentally Sound Recycling of Ships.<sup>(356)</sup> IMO Library

Services has prepared an extensive list of information resources about the recycling of ships. This list includes publications, videos, links to web sites, press articles and more.<sup>(357)</sup> The Basel Convention has a “Dismantling of Ships” page on its web site.<sup>(358)</sup> This web page provides links to international, industry, and non-governmental organizations, and country websites on ship scrapping.

A background paper prepared for the International Labour Organization (ILO) describes the concerns for worker health and safety during ship breaking in its examination of several ship breaking sites in India.<sup>(359)</sup> Explosions, fires, exposures to hazardous chemicals, and heavy materials (steel plates) falling on workers are just a few of the hazards faced by workers who may face the loss of their job if they are injured or develop an illness from on-site conditions. Equipment is often not tested for safety and personal protective equipment is mostly unheard of and many employees do not even have shoes. Ship breaking yards are located in many countries including India, Bangladesh, Pakistan, and China.<sup>(360)</sup>

In 2000, the US EPA published an environmental compliance guide on ship scrapping with information on proper removal and disposal of asbestos, paint, PCBs, bilge and ballast water, oil and fuel, metals, and ship machinery.<sup>(355)</sup>

A few papers have been published on the analyses of material collected at the world’s largest ship scrapping yard, Alang-Sosiya. The Alang-Sosiya yard has been in operation in the Gujarat state of India (north of Mumbai) since 1983.<sup>(361)</sup> A general overview of the type of wastes collected and the amount was presented by Reddy, et al., in 2003.<sup>(362)</sup> The industry produces a large amount of waste including wood, rubber, plastics, textiles, and metals. Small plastics debris have been analyzed using FTIR spectroscopy and polyurethane, nylon, polystyrene, polyester, and glass wool have been identified in the materials sampled.<sup>(363)</sup>

The effect of seasonal changes such as high tides and monsoons have been discussed in a study on the seasonal distribution and contamination levels of petroleum hydrocarbons, PCBs, and heavy metals in the yard.<sup>(364)</sup> Levels for all the contaminants studied peak during the winter months. Heavy metals (Pb, Cd, Co, Ni, Cr, Mn, Fe, Cu, and Zn) in samples from the Alang-Sosiya yard are studied in two other papers. Coastal sediment samples are digested using a microwave technique and analyzed using flame atomic absorption spectrometry in one study.<sup>(365)</sup> Trace metals in suspended particulate matter are studied in the other published work.<sup>(366)</sup>

## **NANOTECHNOLOGY**

Nanomaterials are structures, devices, or systems with a shape or size designed on the nano scale and for the purpose of studying or manipulating materials at an atomic, molecular, and macromolecular scale. The study of nanotechnology is leading to new products in many fields including immunology, electronics, optics, mechanics, and magnetics.<sup>(367)</sup>

Using nanoscience in environmental applications could lead to improvements in air and water quality monitoring, reductions in industrial emissions, and the creation of more lightweight and portable in-field analytical techniques. But there is concern about the unknown toxicity of nanomaterials and questions that lead many to wonder if the risks to human health and the environment outweigh any potential benefits.<sup>(368)</sup> Moore reviews studies on the interactions of nanoscale particles on biological systems and the potential consequences and then discusses questions about the effect of nanomaterials on aquatic systems.<sup>(369)</sup> In an article on the potential for nanomedicine to improve human health, Chan presents some of the nanotoxicology issues

that must be considered in order for industry to produce nanomaterials with minimal risks.<sup>(370)</sup> *Nanomaterials – Toxicity, Health and Environmental Issues*, the latest addition in the “Nanotechnologies for the Life Sciences” book series by Wiley-VCH, includes discussions on toxicity of nanomaterials along with applications to the environment. Sections are divided into toxicity, health, and environment and the chapters within address topics such as biotoxicity, ecotoxicity, metallic nanoparticles, carbon nanotubes, environmental remediation, removal of endocrine-disrupting chemicals in water, and air pollution.<sup>(371)</sup>

Despite questions of the risks of nanotechnology, researchers continue to produce new ideas, and the environmental forensics field is receiving some of the benefits of the new developments. Vaseashta, et al., have presented their studies into nanotechnology-based gas sensors. The portable sensor units provided instant concentrations and could be linked to computer to provide a real-time pollution data analysis. Additionally, the group has studied nanomaterial-based sorbents in the removal of cadmium and arsenic from water streams.<sup>(372)</sup> Wang, et al., have applied organic fluorescence nanoparticles to the selective quantification of Cr(VI) in wastewater.<sup>(373)</sup> Carbon nanotubes have been developed for use in several analytical applications such as sorbent material for solid-phase extraction, as filters and membranes for separations, and in sensors.<sup>(374)</sup> Multiwalled carbon nanotubes (MWCNT) have been used in a micro-solid-phase extraction as a fast, accurate, and cost-effective pretreatment method for the analysis of sewage sludge samples. The multiwalled carbon nanotubes were used to extract organophosphorus pesticides from the sludge samples prior to analysis by GC/MS.<sup>(375)</sup> Solid-phase extraction (SPE) with MWCNT has also been used to extract cyanazine, chlorotoluron, and chlorbenzuron successfully from groundwater and sewage water.<sup>(376)</sup>

## ACKNOWLEDGEMENTS

This paper would not have been possible without the assistance of several people, so it is with great gratitude that I list them here and thank them for their contributions.

Nancy Greer – Nancy provided all of the assistance that I received from the NEIC library by gathering publications and books for review and answering queries about available publications and reference formatting concerns.

Illa Schipporeit – Illa reviewed versions of the paper and provided ready answers to any questions regarding formatting or standard practices for NEIC reporting. Illa was not properly acknowledged in the two prior environmental crime papers (in 2001, for the 13<sup>th</sup> IFSS and in 2004, for the 14<sup>th</sup> IFSS) but her skills have been essential parts of the production of all of these papers.

Elizabeth (Beth) Mishalanie – Beth provided the definitive reviews and recommended changes for this paper.

Phoebe MacLeish – Phoebe managed the contract for the library staffing during the preparation of this paper and helped ensure that library support was available.

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