

Environmental Analysis

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This review covers developments in applied environmental analytical chemistry from November 1996 to the end of October 1998, as found in the Chemical Abstracts Service *CA Selects* for gas chromatography, mass spectrometry, inorganic analytical chemistry, and pollution monitoring. Because of other reviews that appear in this issue, we have excluded most references to the following topics: industrial hygiene, general air and water quality parameters, greenhouse gases, guidelines and regulations, risk assessment, human levels, modeling, commercial products, and food. We include coverage of pesticides and herbicides this

year, because a separate review on that subject was not prepared. Most coverage of field analytical chemistry was eliminated because of the review on that area that appears in this issue. The coverage of water is somewhat different this year, as we have coordinated the preparation of this review directly with the separate water analysis review that appears in this issue, to eliminate overlaps in coverage. We emphasize the determination of trace organics, trace metals, and organometallics in real environmental samples. Therefore, many method development studies that have only been applied to artificial samples or reference materials have also been excluded. As always, the headings and subheadings show some changes from those used in the previous review in this series (A1), to reflect the ever-changing field of environmental analysis.

The number of relevant citations this year was much greater than for any previous review in this series, and a greater effort was made to cite only those studies of greatest interest to the environmental analytical community. However, additional coverage of relevant information that can be found through the Internet is included here for the first time. The authors believe that any who are interested in this review will find the Internet sites listed in Table 1 to be of significant value. We do not claim to be exhaustive in our coverage, but all sites listed contain information directly applicable to the environmental analytical field. No sites are included that are of an exclusively commercial nature. The authors are interested in knowing about any additional sites to include in future reviews in this series. Please e-mail any such information, or comments about this review, to Ray Clement (clemenra@ene.gov.on.ca).

GENERAL REVIEWS

Many reviews concerning most aspects of environmental analysis were published in the past two years. In addition to the general reviews referred to here, more narrowly focused reviews on specific aspects of environmental analysis appear in the following sections of this review. Reviews and books on field analytical chemistry (A2), environmental chemical analysis (A3), and chemometrics in environmental analysis (A5) have been published. Patnaik edited a handbook of environmental analysis (A4). Reviews of waste sampling and analysis (A6), soil analysis (A7), and chemical characterization of environmental and industrial particulate samples (A8) have also appeared.

Various issues related to sample extraction and preparation for instrumental analysis have been reviewed by various authors (A9–A23). Microwave-assisted sample preparation is increasing in importance (A9–A11). Barwick noted that the use of some

Table 1. Useful Internet Links for Environmental Analytical Chemists**A. Methods and Technical Literature**

- 1 <http://www.epa.gov/epahome/Standards.html>
EPA environmental test methods and guidelines
- 2 <http://www.epa.gov/region01/oarm/links.html>
links to sources of EPA test methods on the Internet
- 3 <http://www.epa.gov/ttn/amtic>
EPA's Ambient Monitoring Technology Information Center (AMTIC): contains details on monitoring methods and related information
- 4 <http://www.epa.gov/oar/>
home page for EPA Office of Air and Radiation
- 5 <http://www.epa.gov/OGWDW/methods/methods.html>
EPA Office of Ground Water and Drinking Water: analytical methods for drinking water
- 6 <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>
EPA Office of Solid Waste: contains SW-846 on-line test methods for evaluating solid waste physical/chemical methods
- 7 <http://www.wpi.org/wpi/prodser/chemmethods/chemmeth.asp>
detailed summaries and comparison of critical parameters of EPA methods; useful for method selection, method modification, and data comparability
- 8 <http://www.sampleprep.duq.edu>
Sample Prep Web: contains information and advice regarding analytical sample preparation, speciated analysis, trace analysis and microwave chemistry
- 9 <http://www.sigma-aldrich.com>
from menu choose Supelco and then Supelco Technical Library: ~100 environmental bulletins and application notes can be downloaded and printed

B. Certified Reference Materials (CRMs/SRMs)

- 1 <https://ois.nist.gov/SRMcatalog/>
technical and ordering information for National Institute of Standards and Technology (NIST) SRMs
- 2 <http://www.ems.nrc.ca/ems1.htm>
CRMs of the Canadian National Research Council
- 3 http://www.pss.aus.net/products/ref_mat.html
U.K. organization that acts as a central supply for CRMs from around the world
- 4 <http://www.jrc.org/irmm/index.asp>
Institute for Reference Materials and Measurements: promotes European harmonization and standardization in analytical measurements, quality control in preparation of CRMs

C. Environmental/Analytical Conferences and Organizations

- 1 <http://www.acs-envchem.duq.edu/>
home page of the American Chemical Society, Division of Environmental Chemistry
- 2 <http://www.carleton.ca/~rburk/ea2000/title.htm>
information concerning the biennial conference EnviroAnalysis: devoted to all aspects of environmental pollution analysis and monitoring
- 3 <http://www.wpi.org/wtqa/>
information concerning the (EPA sponsored) annual Waste Testing and Quality Assurance Symposium (WTQA)

D. Links to Relevant Environmental Information

- 1 <http://pw1.netcom.com/~qaa/links.html>
links to sources of relevant environmental chemistry information
- 2 <http://www.liv.ac.uk/Chemistry/Links/refanal.html>
contains many links to analytical chemistry sources of information
- 3 <http://www.fasor.com/~iso25/>
ISO/IEC Guide 25 page: contains extensive links to international accreditation bodies and to standards organizations

solvents for sample extraction is becoming difficult because of increasing legislative restrictions, and she presents a review designed to help analysts select alternative solvents (A12). Sample digestion methods for solid samples were reviewed by Ostapczuk (A13) and De Castro (A14). Sample preparation for X-ray fluorescence (A15), elemental analysis (A16), and metal preconcentration and speciation (A17) were also reviewed. Reviews were published concerning supercritical fluid extraction of metal species (A18), solid-phase extraction (A19), solid-phase microextraction (A20–A22), and Soxhlet extraction (A23).

A number of reviews concerning developments in chemical sensors have appeared (A24–A28). Janata covered four years of chemical sensor developments and critically assessed new trends and features in sensor development (A24), and in addition reviewed sensors specifically developed for environmental applications (A25). Other reviews covered developments in optical sensors for the determination of heavy metal ions (A26), fiber-optic sensors for environmental applications (A27), and screen-printed electrochemical sensors for biomedical, environmental, and industrial analyses (A28).

Several reviews of various instrumental methods of analysis were published (A29–A39). Dean reviewed environmental applications of atomic spectrometry with over 800 references (A29). A book on applications of atomic spectrometry to regulatory compliance monitoring (A30) and a review of environmental

analysis with electrothermal vaporization-ICPMS (A31) have also appeared. Specific reviews on environmental instrumental analysis included the use of neutron activation analysis for aquatic sediments analysis (A32), X-ray spectroscopy (A33), flow injection systems for elemental soil analysis (A34), thin-layer chromatography applied to toxic metals determination (A35), capillary electrophoresis (A36), and proton-induced X-ray emission (A37–A39).

Environmental analyte speciation is a topic of increasing importance, as illustrated by the many reviews on this topic that were published (A40–A54). Soon, environmental legislation will force many more laboratories to carry out speciation analyses (A40). One author suggested that the speciation field has reached a point of stagnation in terms of research originality, but is poised for a breakthrough (A41). Sources of error in speciation analysis were discussed (A42). Specific reviews on speciation analysis of trace metals (A43, A44, A47–A52), selenium compounds (A45), and organometallic and organohalogen compounds (A46) have appeared. Reviews on the use of ICPMS coupled with separation techniques for speciation (A53) and on speciation analysis as the future direction of atomic absorption spectroscopy (A54) were also published.

The environmental determination of specific analytes was reviewed by several authors (A55–A68). Specific reviews covered chemical pollutants in soils (A55), metals and organic pollutants

in solid wastes and contaminated soils (A56), inorganic pollutants by capillary electrophoresis (A57), trace metals in intertidal sediments (A58), quality control for environmental mercury determination (A59), organotin determination (A60–A62), sampling and analysis for in situ hydrocarbon remediation (A63), hydrocarbons in air, soil, and water (A64), EDTA and DTPA (A65), platinum in biological and environmental materials (A66), alkylphenol ethoxylates (A67), and persistent halogenated chiral contaminants (A68).

AIR ANALYSIS APPLICATIONS

General Comments. This review focuses on the development of analytical methods and their applications in the analysis of toxic and hazardous air pollutants. Toxic and hazardous pollutants are organic compounds and inorganic metals or ions with defined toxicity, carcinogenicity, and/or mutagenicity. In addition to toxic and hazardous air pollutants, new analytical methods and their applications in the analysis of pesticides are included in this review. Articles related to nonmetal gases, acid gases, and criteria gaseous pollutants are reviewed in Air Pollution Reviews in this issue of *Analytical Chemistry*.

The organization of this review is the same as the previous one with some minor adjustments to reflect current trends in air analysis. For example, there are limited method development and application activities in the area of mobile sources. There are, however, increasing number of publications in the area of optical spectroscopy. In view of the potential of optical spectroscopy monitoring of mobile sources and the requirement of using extensive data processing to assist in the data interpretation, the Optical Spectroscopy and Chemometric subsection under the Ambient Air section has been merged with the Mobile Sources section.

The Monitoring/Source Apportionment subsection of the Fixed Sources section was also moved to the Mobile Source section because of the inherent data processing nature of these applications. Similarly, due to limited publications in the area of air emissions from waste and waste sites, the section Air Emissions from Waste and Waste Sites was moved and became a subsection of the Fixed Source section. As a result, there is a total of eight instead of nine sections in this review of air analysis applications.

Three trends have been observed in applied environmental air analysis since the 1997 review. In comparison to polychlorinated dioxins/dibenzofurans (PCDD/PCDF), polycyclic aromatic hydrocarbons (PAHs) have a relatively low toxicity but a very high abundance in the environment. There are many sources, including domestic activities, for the generation and the release of PAHs into the environment and this, probably, is the reason 18 of the 249 references cited in the air applications are related to the PAH and only 3 are related to PCDD/PCDF analysis and/or characterization. Second, 2,4-dinitrophenylhydrazine (DNPH) cartridge sampling, in situ derivatization, high-pressure liquid chromatography (HPLC) separation, and ultraviolet detection has been the method of choice for the analysis of airborne oxygenated volatile organic compounds (VOCs) and semi-volatile VOCs (SVOCs). This has been a reliable method, but the use of HPLC can be cumbersome and a UV detector lacks the specificity offered by a mass detector. In view of these requirements and taking advantage

of the state-of-the-art benchtop gas chromatography/mass spectrometry (GC/MS) systems, new sampling and sample preparation methods have been developed by using fluorinated chemicals such as pentafluorobenzyl bromide or *o*-2,3,4,5,6-pentafluorobenzyl-hydroxyamine hydrochloride as derivatizing agent, and GC/MS analysis of this class of compounds. Finally, with the increasing requirements of the PM_{2.5} monitoring, there were many publications on the speciation of particulate-associated organic and inorganic compounds, especially PAHs, pesticides, and heavy metals. In fact, there were more than 50 publications related to the analytical method development, characterization, and interpretation of airborne and deposition-borne particulates throughout this review.

Reviews. There were 26 reviews published in environmental air analysis. They cover concerns and issues in the following six categories: sampling, monitoring methods and results, indoor and personal exposure, biogenic emissions, atmospheric deposition and transport, and miscellaneous. A review on the use of the denudation technique for sampling and measurement of atmospheric trace contaminants was done for both organic and inorganic species (B1). Baron reviewed commercially available personal aerosol samplers in terms of their possible problems and potential solutions (B2). Monitoring results on the urban air pollution in the United Kingdom, including monitoring networks, urban pollution episodes, trend in emissions, chemistry, and air management (B3), were reported along with rural air pollution (B4) for sulfur, NO_x, and photochemical oxidants. The possibility of using light detection and ranging (LIDAR) for the ambient air detection of formaldehyde was reviewed (B5). Reviews on the particulate-associated PAH (B6) and PCDD/PCDF and polychlorinated biphenyls (PCBs) (B7) were done with 188 and 65 references. The structural heterogeneity of airborne particles (B8) and speciation techniques for fine atmospheric aerosols (B9) were also reviewed. Dehollander reviewed the quantitative analysis, control, and modeling of gaseous emissions from wastewater treatment facilities (B10). Methodologies used by the U.S. Environmental Protection Agency (EPA) for assessing direct and indirect exposure to air contaminants emitted from combustion sources, including air, soil, surface water, and food were summarized in a review (B11). Schroeder and Munthe reviewed the physical, chemical, and toxicological properties of mercury and its environmentally significant species in the atmosphere (B12). The inorganic composition of atmospheric aerosols, especially in areas of natural and anthropogenic sources of metals, size distributions, analysis, and gas-to-particle conversion, was reviewed with 217 references (B13).

Indoor chemical reactions between VOCs and oxidants, e.g., ozone and NO_x, were reviewed as they can form irritants which may be responsible for the reported sick building syndrome symptoms (B14). Burton reviewed, with 139 references, the characterization of indoor VOCs such as pesticides, environmental tobacco smokes, combustion gases, human bioeffluents, and personal care products (B15). Problems associated with using pets as sentinels of environmental cancer risk for the investigation of the carcinogenicity of indoor air pollutants, especially on the maximum tolerated source in rodent bioassay of exposure, were reviewed with 112 references (B16). Contributions of organic solvents common to the indoor environment (B17) and indoor

air pollution due to the presence of pesticides (B18) were reviewed with 77 and 57 references, respectively. Crump reviewed the main types of sources of indoor pollutants and their health effects (B19).

Volatile carbonyl compounds of biogenic origin in the atmosphere, including their vegetation sources, sampling and analytical methods, reactivities, and concentration were reviewed (B20). The emission of alcohols, esters, ethers, and higher aldehydes from terrestrial plants, including qualitative and quantitative studies of ambient concentrations, production pathways, and the use of the data in modeling (B21), was reported. A review on the atmospheric measurement of alkenes and acetylene, including emissions from oceans, soils, wetlands, and terrestrial plants, concluded that there was no conclusive evidence of emissions of acetylene from terrestrial plants. There are, however, low levels of acetylene emissions from oceanic sources, and ethylene is probably the most important nonisoprenoid alkene emitted from vegetation (B22). The interaction of lichens with metals and lichens' ability to accumulate high levels of potentially toxic metals led to their widespread use as biomonitors of atmospheric depositions and was reviewed with 42 references (B23).

A review with 123 references concerning atmospheric removal processes of PCBs and PCDD/DF, including gas/particle partitioning, physical removal via wet and/or dry deposition, and chemical transformation was presented (B24). A review on the atmospheric transport and deposition of toxic compounds of heavy metals and persistent organic pollutants within air masses was published with 52 references (B25).

A review on the current status of environmental exposure to benzene, including air, food, and blood media, that enhanced our knowledge of its environmental occurrence and personal exposure in the indoor and outdoor environment appeared (B26). Use of molecular biomarkers as indicators of early response to environmental pollutants, in relation to risk assessment and public health, was also discussed (B27). A review with 27 references covered the experience gained by INCO Ontario in diesel emission control in mining, including the implementation and use of modern engines, improved fuel consumption, and air pollution control measures (B28).

Standards and QA and QC. All environmental measurements require quality control and quality assurance (QC and QA) to support the usability of analytical results. Effective and comprehensive QC procedures are critical for doing accurate toxic air pollutants analysis and minimizing error in environmental measurements. A general overview of the QC requirements defined by EPA methodologies, including presampling preparation, facility preparation, sample collection, laboratory analysis, and data handling, was documented (C1). QC guidance for performing good ambient air and source emission VOC measurements, including checks on field sampling and laboratory analysis such as the use of certified standards, surrogates, and internal standards, was discussed (C2). Clearly, any analytical measurement would be associated with an uncertainty that affected the accuracy of the measurement. The importance of assessing uncertainty in the evaluation of air quality data, including field sampling parameters, was discussed (C3). The role of the EPA Environmental Response Team in auditing an on-site, perimeter air-monitoring system, especially those used in hazardous waste

sites to ensure that fugitive emissions from the site were within prescribed limits, was described (C4).

Certified reference materials (CRM) and standard reference materials (SRM) have been used to improve the data quality in environmental air analysis and to acquire results with higher precision and accuracy. For example, bias existed in the analysis of air particulates on filter using analytical techniques such as instrumental neutron activation analysis (NAA), proton-induced X-ray emission (PIXE), and energy-dispersive X-ray fluorescence (XRF). With the development of a universal SRM, one could calibrate these analytical systems to obtain results with good agreement (C5). The certification of a CRM for the monitoring of aldehydes in air by DNPH cartridge sampling, in situ derivatization, and subsequent analysis by HPLC was described (C6). A procedure for the preparation of airborne particulate SRM on a Teflon membrane filter for laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) analysis was developed. Results were compared to those obtained from XRF analysis and ICPMS after sample digestion in a high-pressure bomb. It was demonstrated that LA-ICPMS could be a sensitive and effective approach for the analysis of elements in airborne particulates (C7). The preparation of SRM could also lead to new challenges and/or opportunities for further development work. This was illustrated in the preparation of a rainwater SRM for trimethyllead analysis. A comprehensive study, including the homogeneity, the certification of the concentration, and the stability of the standard was carried out. This SRM was deemed to be not suitable for the purpose because of concerns on the long-term stability of the sample (C8).

Using a scanning proton microprobe (SPM) technique, the homogeneity of an International Atomic Energy Agency (IAEA) urban dust SRM was examined within $100 \times 200 \mu\text{m}^2$ areas of spatial resolution. It was validated that the IAEA-396A/M Vienna urban dust is homogeneous enough for an SRM (C9). The ability to generate simulated airborne particulate matters (APM) on filters and the possibility of producing large batches of filters for a future PM_{2.5} SRM have been developed using particulate collected from an urban industrial area. Homogeneity between filter samples was evaluated using PIXE, NAA, and XRF (C10). An interlaboratory comparison of air particulate monitoring data using ion chromatography (IC), PIXE, and XRF was carried out. It was demonstrated that XRF and PIXE data showed good agreement but the IC data was ~20% lower than that obtained by PIXE and XRF (C11). NAA was used to validate four kinds of environmental SRM, including APM, coal fly ash, soil, and pine needle, obtained from NIST and IAEA, for 40 trace and toxic elements. The results were demonstrated to have a relative standard deviation of 15% or less for more than 75% of the target compounds under optimized operating conditions (C12). An interlaboratory study was carried out to evaluate analytical techniques such as NAA, synchrotron XRF, and optical emission spectroscopy (OES) for the multielemental analysis of aerosol samples on filters with an attempt to evaluate the effect of aerosols on the air quality, atmospheric process, and climate changes (C13).

Applications of QC and QA to instrumental analysis were also investigated. For example, the precision and accuracy of on-line monitoring of elemental pollutants in flue gases are dependent on the water content of the gases and the generation of standard

aerosols. A procedure used to generate standard aerosols and control of moisture content in flue gas was described. With this procedure, it was demonstrated that inductively coupled plasma optical emission spectroscopy (ICP-OES) could be used for the on-line monitoring of elemental pollutants in flue gas (C14). Solid-phase microextraction (SPME) followed by GC analysis of airborne VOCs has been a task because of the lack of a good calibration procedure. Using a permeation tube-based VOC standard preparation apparatus and a sampling device, it was demonstrated with five VOCs that reproducible instrumental response could be obtained for calibration purposes (C15). Gatz evaluated several literature methods and applied them to the blank correction of a toxic air pollutants data set of PCB congeners, total PCB, and organochlorine pesticides (C16).

Fixed Sources. Fixed sources of airborne pollutants include both industrial and natural sources. There were minimal publications in the Air Emissions from Waste and Waste Sites section; therefore, it is now accommodated under this section as a subsection. The Nuclear Plants subsection was deleted as no publication came up from our literature search. The Source Monitoring/Source Apportionment Methods subsection, due to its chemometric nature, has been merged with the Optical Spectroscopy and Chemometric subsection and moved to the Mobile Sources section.

(a) Industrial and Fugitive Emissions. Using tandem impinger sampling (two impingers), in situ derivatization with *o*-2,3,4,5,6-pentafluorobenzylhydroxyamine hydrochloride (PFB-HA), and GC separation followed by electron capture detection (GC-ECD), a method was developed for the monitoring of carbonyl compounds from stationary source emissions. Typical method recovery of this new method was validated at >80% for carbonyl compounds and <70% for butanal and hexanal compounds (D1). Ambient concentrations of benzo[*a*]pyrene (B[*a*]P) around an aluminum smelter plant at Schwinigan, Quebec, have been monitored since 1983. Monitoring data showed an overall reduction of 57 and 41% for the total PAH and B[*a*]P, respectively, during a 15-year period (D2). A study on the emission factor for the estimation of vinyl chloride emissions from the actual weight of extruded poly(vinyl chloride) (PVC) tubes was done. The new emission factor was found to be a more realistic one and was ~1000 times lower than the emission factor published by the EPA in 1978 (D3). VOC emissions from a recycled paper mill situated at Maysville, KY, was also studied using dilution probe and canister sampling, followed by GC separation and flame ionization detection (GC-FID) with comparable results (D4).

Speciation of airborne dust from a nickel-refining operation (D5) and uranium and thorium fallout from the neighborhood of a phosphate fertilizer plant (D6) was carried out using scanning electron microprobe (SEM) and X-ray diffraction, and optical microscopy, respectively. Heavy metal outputs from a cement kiln cofired with hazardous waste fuels were monitored and compared to the background value for concentrations of arsenic, beryllium, cadmium, chromium, and lead. It was found that concentrations of these elements at the highest intended hazardous waste feed rate are 68, 10, 72, 18, and 68 times higher than their respective background values (D7).

(b) Incinerators, Stacks, and Residues. The effect of hydrochloric acid on the XAD-2 resin sampling and analysis of

SVOCs such as chlorobenzene (CB) and PCB, especially its use with the MM-5 sampling train, was reported (D8). Zhang reported the VOC emissions from seven large-scale incineration plants in Sweden (D9). Using cartridge sampling, in situ DNPH derivatization, micellar electrokinetic chromatography (MEKC) separation, and ultraviolet (UV) detection, a method was developed for the determination of lower aliphatic carbonyl VOCs in stack gas (D10). Real-time, on-line analysis of aromatic pollutants in waste incinerator flue gases using a mobile resonance-enhanced multiphoton ionization (REMPI) time-of-flight mass spectrometer (TOF-MS) was demonstrated with a time resolution of 5 Hz (D11). Using GC/MS, 97 oxygenated PAHs were qualitatively identified from municipal solid waste incinerator (MSWI) fly ash (D12). Results obtained from a 5-year study on the variability of concentrations of metals and PCDDs in stack emissions of a MSWI was used to determine the effect of the feed and different technologies such as electrostatic precipitator, furnaces with a spray dryer/absorbers, and dry-lime sorbent injection (D13). The status of emerging technologies, for example, laser spark spectrometry (LASS) and ICP applications for continuous emission monitoring (CEM) and on-line sampling by hazardous element sampling train (HEST) followed by XRF analysis, were described (D14).

(c) Natural Sources. Model evaluation of biogenic isoprene emission in southeastern U.S. bottomland deciduous forests were compared with emission factors, light and temperature response, leaf area, and canopy level isoprene emissions (D15). Monoterpene and isoprene emissions in Norway spruce forests were evaluated and reported. In 1991, 88% of monoterpenes were emitted in July and August; the contributions of the trunk compartment and soil to the total forest emission were estimated to be from 1 to 64% and 3%, respectively (D16). Isoprene and monoterpene emissions from a eucalyptus plantation in Portugal were measured from a mature and an immature tree using a branch enclosure sampling system and GC/MS analysis. It was found isoprene was the dominant compound emitted and accounted for more than 90% of the total VOCs. It was also found that under standard temperature (30 °C) and light (1000 $\mu\text{mol}^2/\text{ms}$) conditions, VOC emissions from a 1-year-old tree were ~5 times more than that of a 7-year-old tree (D17). Ambient biogenic hydrocarbons and isoprene emission from a mixed deciduous forest in southern Ontario in 1993 were also reported (D18). Isoprenoid emission of oak species of Mediterranean and typical VOC emissions from 18 Mediterranean plant species were studied and reported (D19, D20). Seasonal emissions of isoprene and other reactive hydrocarbon gases from the ocean were correlated with the chlorophyll content of the water from the North Sea and southern ocean and estimated to have a seasonally averaged flux of isoprene to the atmosphere of 1.7×10^7 molecules/($\text{cm}^2 \text{ s}$) (D21).

(d) Air Emissions from Waste and Waste Sites. A method encompassing an airborne and a solution-phase VOC and oxygenated VOC analytical method for the monitoring of emissions and wastes from a swine production facility was developed. Using this method, 40 VOCs were identified in liquid and ambient air samples from the neighborhood of the facility. A total of 27 of the 40 VOCs were confirmed to contribute to decreased air quality, with the C2–C9 organic acids demonstrating the highest transport coefficients and airborne concentrations (D22). Using GC-FID (for

Table 2. Mobile Source Monitoring Methods. Gasoline and Diesel Engines

analytes	methods/comments	refs
VOCs and BTXE	activated charcoal sampling, carbon disulfide desorbing, internal standard GC/MS analysis; levels and types of VOC emissions from various internal combustion engines equipped with different catalytic converters were compared	E1
BTXE	on-line probe and/or automated Tenax sampler followed by membrane inlet MS analysis; analyzing 1 sample/s at the ppm detection limit	E2
PCDDs and PCDFs	on-road sampling of diesel engine emissions, high-resolution MS analysis; reported to have an average emission of 0.029 ng of I-TEQ/km at a 95% confidence level	E3
PAHs and hydrogenated PAHs (HPAHs)	real-time ion trap MS and MS/MS characterization of the organic composition of individual diesel engine smoke particles	E4
Pb, Ce, Pt, Mo, Ca, Na analyses	on-line aerosol TOF-MS characterization of particulate-associated, organic ($<1\ \mu\text{m}$) and inorganic ($>1\ \mu\text{m}$) compounds	E5

Table 3. Infrared Applications

analytes	methods/comments	refs
Method Development and QC and QA		
sulfur hexafluoride, Sarin, trichloroethylene, methyl isocyanate, mustard gas	theoretical detection limits of passive IR remote detection simulated and calculated using a 2-cm^{-1} resolution	E8
QC parameters	estimation of measurement uncertainty in open-path FT-IR (OP-FT-IR) remote sensing of fugitive emissions	E9
partial least-squares (PLS) OP-FT-IR analysis parameters	calibration models for the identification and quantitation of 23 gases in the presence of high background gases, e.g., H_2O , CO_2 , and CH_4 , established for indoor, ambient, and industrial monitoring purposes	E10
Field Applications		
fugitive VOC emissions from a gasoline station	extractive FT-IR monitoring of petroleum hydrocarbon at an active gasoline station	E11
methanol and DMMP	passive OP-FT-IR measurement and source monitoring of industrial chemicals at the low ppm·m detection limits	E12
CO and NO_x from air craft exhaust	passive OP-FT-IR emission measurement with an overall accuracy of $\pm 30\%$ and the ability to differentiate exhaust from an aged engine with a detection limit of 20–90 ppmv	E13
toxic and flammable air pollutants	a surveillance system using COMPAS feed-back algorithm that allowed the calculation of source concentration using the fence-line monitoring concentration obtained from OP-FT-IR	E14
<i>n</i> -hexane, <i>n</i> -heptane, <i>n</i> -octane, isooctane, 2,2,4-trimethylpentane	on-site, dynamic spiking using methanol, 1,3-butadiene, and <i>p</i> -xylene calibration allowing on-site monitoring of >100 VOC compounds and quantitative analysis of 20 VOCs using extractive FT-IR analysis of uncontrolled emissions at an asphalt coating process	E15
indoor tracer gas	OP-FT-IR combined with computed tomography for the analysis of gas concentrations in indoor environment demonstrated with a quantitative agreement within 50% of the true value	E16

hydrocarbons), GC-flame photometric detection (FPD, for reduced sulfur compounds), and a flux chamber, measurement of hydrocarbons and reduced sulfur compounds emitted from a wastewater treatment pond was done during the day (22 °C), the night (14 °C), and in warm and cold weather. It was demonstrated that the difference between day and night emissions during warm weather was greater than that during cold weather (D23). Measurement of landfill gas at seven U.K. waste disposal sites was done. More than 140 VOCs were identified, of which more than 90 were common to all seven sites. Correlations between the landfill-specific VOCs, the landfill component, and the stage of the decomposition were established (D24). Methyl mercury contamination and emission to the soil from solid amended with municipal sewage sludge (D25) and volatile transition metal compounds in landfill gas, especially the synthesis of molybdenum and tungsten carbonyls, were observed by GC/ICPMS and discussed (D26).

Mobile Sources, Optical Spectroscopy, and Chemometrics.

There were a total of 26 publications that were classified into three subsections: Mobile Sources, Optical Spectroscopy, and Chemometric. Emissions from vehicles with internal combustion gasoline engines (E1, E2, E5) and diesel engines (E3, E4) have been the center of studies for three decades. Optical spectroscopy monitoring of emissions and emission sources, either fixed or mobile, and/or ambient toxic air pollutants (E6, E7), followed by postdata processing assisted data interpretation, especially in the area of open-path Fourier transform infrared (FT-IR) spectrometry (E10–E16), has been a popular topic. In fact, extensive data processing has become an inherent nature of optical spectroscopy and chemometric applications (E8–E10, E14–E16). This is the reason

that the Optical Spectroscopy and Chemometrics subsection of Ambient Air was moved to this section. Similarly, modeling and source apportionment applications have been merged with the Chemometrics subsection.

(a) Mobile Sources. There were five publications under this subsection. Table 2 summarized these applications where method development and applications were done for the monitoring of emissions from motorized vehicles using internal combustion engines. Noteworthy is the projected U.S. annual emission of PCDD/PCDF from on-road diesel vehicles. Using the 1993 truck travel estimates, the total PCDD/PCDF emission is estimated to range from 4.4 to 16.1 g of I-TEQ/year, with a 95% confidence level (E4).

(b) Optical Spectroscopy. Applications in the UV region include the use of resonant UV laser pumping and fluorescence detection of ambient mercury at a detection limit of 2 ppb at 0.1 Torr air pressure. This technique is projected to have the ability at the sub-ppt level with further system optimization (E6). Formaldehyde detection at the 1–5 ppb level using differential absorption UV spectrometry was also demonstrated (E7). Infrared, especially remote FT-IR detection of hazardous vapors, has been popular. Table 3 summarizes publications related to this application in the area of method development, QC and QA, field applications, and postdata processing techniques.

(c) Chemometrics. This subsection includes chemometric, modeling, and source apportionment applications. Ambient air samples collected from Oporto, Portugal, were analyzed for 16 PAHs and 23 alkanes using GC/MS. Organic and elemental carbon contents of these samples were measured by thermal

Table 4. Sampling of Toxic Air Pollutants

analytes	methods/comments	refs
VOC		
highly volatile hydrocarbons	on-site comparison of canister and solid sorbent trap sampling cartridges such as CarboTrap B and C and CarboSieve S-III	F1
TO-14 target compounds	evaluation of multisorbent thermal desorption tubes for the use in TO-17 method	F2
BTX	evaluation of diffusive BTX sampling device and OP-UV measurement (OPSIS) against TO-17 equivalent method	F3
VOC	evaluation of TO-17 sampling protocol but with sampling cartridge cooled to -30°C followed by GC/MS analysis	F4
SVOC		
PAHs	evaluation of a low-volume ($24\text{ m}^3/24\text{ h}$), XAD-4 solid sorbent-based sampling method for PAH analysis	F5
two-ring PAHs, chlorophenols (CP), guaiacols, and benzene	evaluation of polyurethane foam and Tenax-GC sandwich cartridge using isotopically labeled compounds for surrogates	F6
Atmospheric Deposition		
organochlorine (OC) and PCB pollutants in cloudwater	evaluation of semipermeable membrane devices as time-integrated passive sampler	F7
mercury and trace element	development and evaluation of an automated cloudwater sampler for atmospheric monitoring network	F8
	development and evaluation of a new precipitation sampler for Hg and trace metal analysis	F9
Particulate, Inorganics, and Miscellaneous		
respirable particles	development of a high-throughput, liquid absorption air sampler for $2\text{--}10\text{-}\mu\text{m}$ particles and other applications	F10
lead	guidance for sitting air monitors around stationary Pb sources	F11
tritium	evaluation of silica gel adsorbent-based tritium sampling device	F12

Table 5. Monitoring Programs and Field Measurements

analytes	methods/comments	refs
photochemical initiators	description of a three-column, double-identification GC used in the Photochemical Assessment Monitoring Station (PAMS) and PAMS measurements in New York City	F13
ethanol and MTBE	monitoring of ambient level of ethanol and MTBE in Porto Alegre, Brazil, and its correlation to the local gasoline usages from March 1996 to April 1997	F14
formaldehyde and carbonyl compounds	DNPH cartridge sampling, liquid chromatography (LC)/UV analysis of oxygenated VOCs during the 1993 North Atlantic Regional Experiment in Nova Scotia, Canada	F15
HCFCs	calculated trends and atmospheric abundance of HCFC measured by automated in situ GC/MS at Mace Head, Ireland	F16
C2–C6 hydrocarbons	levels of biogenic and anthropogenic C2–C6 hydrocarbons measured at pristine sites and near oil and gas production facilities in Venezuela during the rainy season	F17
C2–C10 hydrocarbons	canister sampling followed by GC-FID analysis of C2–C10 hydrocarbons carried out at the summit (1400 m) of Whiteface Mountain, NY. QA of the analysis confirmed via the Nonmethane Hydrocarbon Intercomparison experiment and estimated to have an accuracy of $\pm 20\%$ for the method	F18
PAHs	profile analysis of PAHs in airborne particulates collected from Thessaloniki, Greece, done according to the EPA 16 PAHs and particle sizes of $\leq 2.5\text{ }\mu\text{m}$ and from $2.5\text{ to }10\text{ }\mu\text{m}$	F19
PCBs	temporal and spatial trends of PCBs in arctic air measured and reported for 1992–1994	F20
organophosphate pesticides (OP), chlorpyrifos	fate of chlorpyrifos, including the process of dilution, degradation, and sorption to plant foliage studied during the summer in an air corridor in the Southern Central Valley, CA, and moving into the nearby Sierra Nevada mountains	F21
OP, chlorpyrifos	chlorpyrifos in the air and surface water of Chesapeake Bay, MD, monitored and used to predict the atmospheric deposition fluxes in the area	F22
mercury	measurement results of atmospheric mercury in the Republic of Korea from 1987 to 1993	F23
lead isotope ratio	rates of zinc and lead fallout from atmospheric particulate $> 10\text{--}20\text{ }\mu\text{m}$, and Pb isotope ratio measured using passive sampling and ICPMS analysis at McArthur River Pb/Zn mine, Australia, from 1993 to 1997 using ICPMS; the Pb isotope ratio used to discriminate ore-derived Pb from local soil-derived Pb	F24
elemental analysis	PM-10 particulate samples collected from urban and rural sites in Belgium analyzed using PIXE and NAA and reported; there was minimum difference in the concentrations measured in samples collected from either the rural or urban sites	F25
elemental analysis	elemental concentrations in particulate samples collected from Zacatecas, Mexico, measured using NAA	F26

oxidation followed by the detection of CO_2 by infrared. Cluster analysis of these monitoring data and respective meteorological information revealed two PAH events that correlate well with the temperature and precipitation pattern. Principal component analysis also revealed two factors that can be attributed to nine PAHs characteristic of urban areas and four alkenes of biogenic origin (E17). Multivariate analysis of microbial air content from an air-conditioned building and a naturally ventilated building revealed that the air microbial content in the latter was much higher and much more variable. The interior fungal level in the air-conditioned building remained constant despite significant variations measured outside (E18).

In the application of source apportionment, Gelencser et al. showed that the toluene to benzene ratio is a more reliable tool characterizing the distance from vehicle emission sources than the conventional time-weighted average approach (E19). Adgate et al. used a chemical mass balance approach to apportion the major contributors of lead mass to house dust (HD_{Pb}). It was found that interior lead-based paints (pre-1960) accounted for about one-

third of the total HD_{Pb} while the other two-thirds was from outdoor contributors such as yard soils and air particulate (E20).

Baldasano applied receptor models, analyzed urban/suburban VOCs pattern in Martorell, Spain, and concluded that 62 and 23% of the VOC pollution was from road traffic and local industrials (E21). Air quality model evaluation data of C2–C36 nonaromatic hydrocarbons from a Los Angeles photochemical smog episode were analyzed for gas-phase VOC and SVOC and for particulate-phase organic compounds (E22). The possibility of applying a successive correction modeling technique to the monitoring of periodic excesses of the short-term threshold of several VOCs using a TAGA 6000E was demonstrated (E23). In the area of modeling pesticide drift and vaporization from agricultural fields, numerical model simulation and field measurement was used to derive qualitative and quantitative estimates of primary and secondary air drifts after different methods of pesticide application (E24). Yates conducted field experiments to measure the volatilization rate of soil fumigants. His work resulted in a procedure for the compilation and comparison of field experiment data to

Table 6. Organic Monitoring Methods

analytes	methods/comments	refs
VOCs		
method TO-15 targets	description of method TO-15 and its comparison with TO-14	F28
method TO-14 targets	performance evaluation of TOFMS and a field-portable GC, respectively, compared to that of method TO-14	F27, F29
method TO-14 targets	performance evaluation of ambient air VOC analysis using automated field GC and method TO-14	F30
C5–C10 and C2–C6 VOC	continuous solid sorbent VOC sampling and analysis using a programmed temperature vaporization injection system for field applications	F31
halogenated VOCs	high-humidity and stagnant VOC sampling using Tenax GR-based adsorbent, a dehumidified and ventilated diffusive sampler, followed by thermodesorption GC-FID analysis demonstrated good repeatability and linear range over a wide concentration range and up to 21-day storage time	F32
methylcyclopentadienylmanganese tricarbonyl (MMT)	high sensitivity and specificity developed for the analysis of airborne MMT using GC and plasma atomic emission detection (GC-AED), with absolute 0.5-fg detection limits of MMT as Mn	F33
VOC	applications of ion store/TOF-MS to the direct analysis of ambient VOCs described	F34
BTX	combining direct probe GC/MS analytical results and information from a global positioning system, the possibility of mapping VOC gradients and trends in space and time demonstrated at low-ppb BTX level at a time resolution of 15 s	F35
formaldehyde	denuder tube sampling of formaldehyde, in situ 2-hydroxymethylpiperidine derivatization, thermal desorption, and GC/MS analysis of the target demonstrated with limits of detection in the range 0.03–0.51 mg/m ³	F36
formaldehyde	solid-phase microextraction of formaldehyde in air followed by DNPH derivatization, GC-ECD analysis method with a detection limit of 0.17 mg/m ³ and a RSD of 9.6%	F37
formic and acetic acids	method for the analysis of formic and acetic acids measured the concentration of these two targets in Los Angeles	F38
organic nitrates and halocarbons	method using cryogenic sampling and GC-ECD analysis developed for the analysis of C1–C4 organic nitrates and halocarbons with a >95% recovery and sub-ppt detection limits using 50 mL of air sample	F39
Title III hazardous air pollutants (HAPs)	recoveries of EPA method 18 evaluated in field test for the analysis of HAPs	F40
VOCs	membrane inlet MS method developed for the analysis of VOCs at the low- $\mu\text{g}/\text{m}^3$ detection limits, over 4 orders of dynamic range, and fast response time (less than 5 s)	F41
C ₂ –C ₉ non-methane hydrocarbons (NMHC)	dual-column (porous-layer open tubular (PLOT) column and capillary columns) GC method for the simultaneous analysis of airborne NMHC developed with a high degree of flexibility and precision	F42
SVOCs		
polychlorinated naphthalene (PCN)	PCN sampling and analytical method using GC/negative chemical ionization MS and Halowax 1014 as a secondary standard; this method was validated to be within $\pm 20\%$ of the true value against five of the six pure PCN congeners used	F43
PCDD and PCDF	evaluation of pressurized liquid extraction and Soxhlet extraction for the fly ash, dry dust preparation for PCDD and PCDF analyses	F44
aliphatic isocyanates	method using midget impinger sampling, in situ derivatization of airborne aliphatic isocyanates (as dibutylamine derivatives), followed by LC/MS analysis with detection limits of 0.1–0.2 $\mu\text{g}/\text{m}^3$	F45
muramic acid	analytical for airborne muramic acid (a chemical marker for bacteria peptidoglycan) analysis developed using 96-h Teflon air filter sampling, acid digestion, and alditol acetate derivatization for GC/ion trap tandem MS analysis	F46
organic acid hydrides	method using Tenax and XAD-2 sampling followed by pentafluorobenzyl bromide derivatization, and GC/MS analysis, for the analysis of hexahydrophthalic anhydride, methyl hexahydrophthalic anhydride, tetrahydrophthalic anhydride, and octenylsuccinic anhydride	F47
carboxylic acids and phenols of biogenic nature	derivatization of acids and phenols using pentafluorobenzyl bromide followed by GC electron impact ion trap MS (GC/IT-MS) analysis or chemical ionization IT-MS analysis using pentafluorobenzyl alcohol as a reagent gas was developed; using this method, the formation of methacrylic acid in an isoprene/O ₃ demonstrated experimentally for the first time	F48
bis(chloromethyl) ether (BCME)	sampling and analytical method for BCME developed using Tenax sampling, solvent desorption and derivatization using <i>p</i> -fluorophenolate, followed by GC/MS analysis	F49
Particulate and/or Aerosol Associated Organics		
PAHs	TOF-secondary ion MS (TOF-SIMS) used to analyze PAHs on individual environmental particulates at <1-m lateral spatial resolution; method especially useful for the phase distribution studies of PAHs	F50
PAHs	air sampling method developed using various samplers to collect different air samples of different particulate size for the phase distribution determination of 21 PAHs; using this method, depending on the sampling location, PAHs can be found on particulate with submicrometer to 44 μm in size	F51, F52
oxygenated PAHs (OPAHs)	GC/MS analysis method of size-segregated atmospheric particulates for seven OPAHs; patterns of distribution of these seven OPAHs on different ranges of particulate sizes developed	F53
PAHs	evaluation using microwave-assisted solvent extraction of particulate-bound PAHs carried out using ambient air samples and CRMs; good recoveries of PAHs demonstrated using this sampling preparation method	F54
PAHs	sampling and GC/isotope dilution MS (GC/IDMS) analytical method for the analysis of ambient PAHs with the largest possible volatile range of PAHs including the abundant naphthalene	F55
nitro- and hydroxy-PAHs (NPAHs and HPAHs)	SFE method developed for the fractionation of NPAH and HPAH followed by GC/MS analysis	F56
azaarene	results of an extensive study of 47 azaarene in the aerosol of an urban environment; percentage of concentrations of azaarenes showed that these compounds tended to associate with larger particles in warm periods and enrich in fine particles in cold weather	F57
humic-like substances	capillary electrophoresis method developed for the characterization of humic-like substances extracted from dust samples via a microextraction procedure	F58
Pesticides		
OC, organonitrogen (ON), phenoxy acids (PA), and OP pesticides	analysis of OC, ON, OP, and PA pesticides in fogwater, rainwater, and ambient air samples; atmospheric concentrations were almost constant throughout the year in all atmospheric phases	F59
dichlorvos	sampling and analytical method developed and validated for the determination of airborne dichlorvos using XAD-4 resin and GC-NPD analysis	F60
azinphosmethyl and azinphosmethyl-oxon	sampling and analytical method developed and validated for the determination of airborne residues of azinphosmethyl and azinphosmethyl-oxon pesticides using XAD-4 resin sampling cool on-column injection and GC-FPD analysis with good recovery (93 ± 18 and $104 \pm 16\%$, respectively)	F61
chlordane and chlorpyrifos	extraction thimbles used in the supercritical extraction extractor modified to accommodate Florisil for the sampling and SFE sample preparation for GC-ECD analysis; method achieved a detection limit of 1 and 0.1 ng/m ³ using a 20-m ³ sampling volume for chlordane and chlorpyrifos, respectively	F62
pesticides	multiresidue method for the sampling and analysis of 13 pesticides in fogwater, rainwater, gas, and particulate matrices developed with 92–97% recovery and detection limits varying between 0.1 and 0.01 ppm depending on the sample matrices	F63

Table 7. Inorganic Monitoring Methods

analytes	methods/comments	refs
Hg ²⁺	selective sampling of ambient Hg ²⁺ developed using KCl-coated denuder with a recovery of >93%	F64
Hg	performance evaluation of gold trap sampling followed by atomic fluorescence at a detection limit of 4 pg	F65
Hg	method developed for the analysis of reactive gaseous mercury in flue gas and applied to the observation of Hg in wet and dry deposition	F66
metals	design and evaluation of an impaction-graphite furnace system for direct and near-real-time airborne metal analysis at the ng/m ³ level	F67
metal aerosols	modification of an ICP-OES-based continuous emission monitoring equipment applied to the monitoring of metal aerosols in air	F68
Al, As, Ba, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, S, Sb, V, Zn	evaluation of particulates collected by a β -gauge monitoring system followed by ICP-OES and ICPMS analysis	F69
Pb, Hg, Sn, In, Ga, Se, P, As	cryofocusing sampling followed by ICPMS analysis of volatile metals and nonmetal species in air	F70
Ge, As, Se, Cd, Sn, Sb, Te, I, Hg, Pb, Bi	GC/ICPMS calibration for the analysis of airborne volatile metals developed	F71
Na, Al, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Ag, Cd, Sb, Ce, W, Pb	evaluation of direct airborne particulate analysis using LA-ICPMS	F72
Cr(III), Cr(VI)	extractive separation and thermal ionization isotope MS analysis of Cr(III) and Cr(VI) in airborne particulate	F73
Fe, Ti, Ca, K, S, Si, Al	control and correction method for the direct XRF analysis of atmospheric aerosol samples	F74
Al, Ca, Cl, Cu, K, Mn, Ti, V	XRF analysis of airborne particulate samples collected on Teflon-coated filter paper	F75
Mn, Fe, Ni, Cu, Zn, Se, Rb, Pb	evaluation of BLPI sampling followed by XRF analysis	F76
Al, Si, P, S, Cl, K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Br, Pb	evaluation of different types of filters for atmospheric trace element analysis using XRF, PIXE, and SEM	F77
Si, S, Cl, Ca, K, Ti, Va, Cr, Mn, Fe, Ni, Cu, Zn, Br, Pb	PIXE studies of size distribution and solubility of elements in atmospheric aerosols	F78
Al, As, Br, Ca, Ce, Cl, Co, Cr, Cs, Cu, Eu, Fe, I, In, K, La, Mn, Na, S, Sb, Sc, Se, Ti, V, Zn	NAA monitoring of size distribution of atmospheric pollutants collected by Berner low-pressure impactor (BLPI) in urban and rural sites	F79
Ni, Va	HPLC/UV analysis of ambient air samples at the ng/m ³ level	F80
Pb, Cd, Cr	distribution of elements as a function of particle size in aerosol samples by sequential leaching	F81
Ni, V, Pb, Pt, Pd	evaluation of microwave-assisted vapor-phase acid digestion of cellulose nitrate filters for airborne dust monitoring	F82

that obtained from the modeling (E25). Using an atmospheric transport model, Rogge et al. explored the relationship between source emissions and ambient air quality for particulate-associated organic compounds that existed in the source emissions (E26).

Ambient Air. The subsection Optical Spectroscopy and Chemometrics was merged with the Mobile Source section to reflect analytical trends observed in the last two years.

(a) Sampling. The collection of representative air samples for laboratory analysis remained as the most difficult part in environmental air analysis. Discussed in this subsection are 12 publications related to the procedures, sampling apparatus, and/or evaluation for the collection of airborne VOC samples (F1–F4), SVOC samples (F5, F6), atmospheric deposition samples (F7–F9), and particulate and inorganic samples (F10–F12). Table 4 summarizes these 12 different applications according to the four categories listed above.

(b) Monitoring Programs and Field Measurements. There were 14 publications where airborne organics were monitored in urban centers, rural areas, and the arctic. Analytes ranged from photochemical initiators, VOCs, HCFCs, biogenic VOCs, PAHs, PCBs, pesticides, and inorganic elements. Table 5 summarizes these activities according to the analytes measured in the programs.

(c) Methods. This subsection is devoted to publications where sample preparation and/or instrumentation techniques were improved to resolve existing analytical problems and were demonstrated by the measurement of real world samples. Publications where novel analytical techniques were used to enhance sensitivity, operating efficiency and, above all, the data quality, though without real world applications, could be included in this section because of their potential contributions in environmental analysis. Like prior reviews, methods are divided into organic and inorganic subsections where Tables 6 and 7 are used to summarize organic and inorganic applications, respectively. Organic applications are listed in the order of the type of analytes while inorganic

applications are listed in the order of the elements and then followed by the type of analytical technologies.

It is worth noting that, with the requirement of monitoring of PM_{2.5}, much effort has been devoted to the method development and studies correlating the particle size and pollutants of airborne particulate and aerosols. Noteworthy is that more than 35 and 60% of the organic and inorganic methods, respectively, are analytical applications and/or studies on the airborne particulate. Table 5 summarizes organic methods in five categories according to the type of analytes: VOCs and oxygenated VOCs, SVOCs, particulate-/aerosol-associated organics, and pesticides. In addition, efforts were made in the organization of the table such that subgroups were created under each category according to analytes and specific methods used in each publication. Noteworthy is the development of many new analytical methods for the analysis of oxygenated VOCs (F36–F38) and SVOCs (F46–F49). All of these new methods used a new derivatization agent that allowed the application of GC instead of LC-based separation, followed by MS analysis of this class of compounds. Also worth noting are the increased applications and method development on various polycyclic aromatic compounds such as PAHs, hydroxy-PAHs, nitro-PAHs, oxygenated PAHs, and azaarenes in air particulates and aerosols, and their affinity to different particle sizes (F50–F57) and the relatively few publications on the PCDD/PCDF analysis.

Table 7 summarizes inorganic methods according to analytes and analytical technologies. There were mercury analysis (F64–F66), metals by graphite furnace and ICP-OES (F67–F69), metals and nonmetals by ICPMS and MS (F69–F73), XRF (F74–F77), PIXE (F77, F78), NAA (F79), and HPLC/UV (F80). Application and evaluation of sample preparation methods were also reported (F81, F82). Noteworthy is the increased use of XRF because of its portability, operation efficiency, and ability to produce data that can meet different data quality objectives.

(d) Personal Exposure. Indoor and Microenvironment Air. This subsection covers publications in the area of sampling,

Table 8. Monitoring of Indoor and Microenvironment Pollutants

analytes	methods/comments	refs
VOC		
benzene and 1,3-butadiene	exposure to emissions in the cabins of moving vehicles and buses	F83
benzene	evaluation of personal exposure to benzene with results correlated to an activity diary	F84
trimethylbenzenes	blood and urine samples taken from human volunteers subject to trimethylbenzene exposure analyzed	F85
carbonyl compounds	DNPH cartridge sampling followed by HPLC analysis of samples collected from urban roadside and indoor sites analyzed for formaldehyde, acetaldehyde, hexanal, and crotonaldehyde	F86
nitrosamines	worker exposures to nitrosodimethylamine and nitrosodiethylamine measured in a rubber vehicle sealing plant	F87
VOC	more than 150 VOC compounds identified in a test chamber where different furniture coatings were monitored	F88
SVOC		
phenols, PAH, and OPAH	source profiles for smoke from burning pine, oak, and synthetic log studied	F89
polychlorinated terphenyls (PCT)	monitoring of PCBs led to the discovery of PCTs, contaminants ubiquitously distributed in the environment, in indoor air; indoor PCTs were particulate phase associated	F90
1-hydroxypyrene and monohydroxylated metabolites of phenanthrene	occupational exposure to PAHs for workers in a graphite-electrode producing plant done using personal air sampler and human biological samples such as urine	F91
SVOC	evaluation of Tenax/filter sorbent sandwich sampling followed by thermal desorption GC analysis method	F92
organophosphate ester flame retardant	sampling and analytical method developed for the analysis of indoor concentrations of organophosphate ester flame retardant with tris(2-chloroethyl)phosphate identified as one of the main components	F93
Pesticides		
chlorpyrifos	indoor level of chlorpyrifos monitored throughout the year in Louisiana	F94
wood preservatives	Tenax sampling followed by GC analysis identified diazinon, phenthoate, phoxim, propoxur, dichlofluanid, endosulfan, permethrin, and tributyltin at various concentrations in homes that were treated by wood preservatives	F95
lindane, α - and β -endosulfan, endosulfan sulfate in greenhouse	evaluation of sampling media (Chromosorb, Porapak, Supelpak, XAD-2, XAD-4) and analysis of greenhouse air by GC	F96
Inorganics		
lead	lead particle content of indoor dust analyzed before and after lead paint abatement	F97
Pb, Cd, As, Hg, Fe, Mn, Ni	evaluation and analysis of surface and airborne element in the workplace	F98
Cr	assessment of biological chromium among stainless and mild steel welders in relation to welding processes	F99

method development, and studies of organic and inorganic pollutants in indoor and microenvironment air. The purpose of these studies was for personal exposure monitoring and they included samples collected from home (indoor) and other microenvironments such as automobile, public transportation, workplace, restaurant, and shopping centers. Table 8 summarizes these applications in four categories according to the type of analytes: VOCs and oxygenated VOCs, SVOCs, pesticides, and inorganics. Noteworthy is the characterization of pesticides used for wood preservation and their abundance in the indoor environment (F95).

Accidents and Emergencies. Six publications discussed topics related to this section and were summarized in two categories: monitoring of fallout from the Chernobyl nuclear facility (G1–G5) and an environmental survey on the possible identification of neurobehavioral consequences in the Gulf region following the Gulf War (G6).

The consequences of the Chernobyl fallout on some Italian underbrush produce (G1) because of ^{134}Cs and ^{137}Cs were reported. Using a multifractal approach, cumulative ^{137}Cs contamination measured from the soil samples was used to aid in describing the spatial distribution of radioactivity in Europe and northern Italy (G2) and in Austria (G3). Determination of the isotopic composition of plutonium in hot particles in the Chernobyl area was carried out to gain more insight about the origin of these manmade trans-uranium elements (G4). Straume et al. investigated the feasibility of using ^{129}I to reconstruct ^{131}I deposition from the Chernobyl reactor accident (G5).

Deposition, Atmospheric Transport, and Atmospheric Chemistry. (a) **Wet Deposition, Including Rain, Snow, and Fog.** Using concentrations of dissolved ions as an indicator, Ranalli et al. evaluated the use of bulk collectors in place of the wet/dry collectors for the monitoring of wet deposition at high-altitude sites in winter (H1). Concentrations of PCB in precipitation samples were measured by Offenberger and Baker and proved that

elevated levels of atmospheric contaminants in urban areas would enhance atmospheric deposition to adjacent surface waters (H2). A new method for the speciation of precipitation-borne trimethyllead was developed using sodium tetraethylborate (ethylation) followed by GC/MS analysis. The result was evaluated against other alkylation method with comparable results (H3). Atrazine and alachlor were detected in rain samples collected throughout the midwest and northeast United States during the spring and summer seasons. Evidence of atmospheric degradation of atrazine was confirmed by the existence of deethylatrazine (H4). Evaluation of an enzyme immunoassay method and a GC/MS method was done for the analysis of terbutylazine and deethylterbutylazine in surface water with comparable results (H5). An enzyme-linked immunosorbent assay (ELISA) for the analysis of triazine and chloroacetanilide in precipitation samples using samples collected from 23 states and/or regions was done. The possibility of false-positive identification was investigated for both compounds (H6).

Reimann et al. analyzed precipitation samples collected from eight Arctic catchments in northern Europe and found that rainwater was enriched in Ni, Co, Cu, A, Mo, Al, V, Cd, Sb, Pb, Zn, Fe, Sr, Na, S/SO₄, Cl, Cr, Se, and Ag when compared to a Finnish background site (H7). Using HPLC/ICP-isotope dilution MS analysis, organic lead (trimethyl- and triethyllead chloride) compounds were identified in rainwater (H8). A new approach was developed using the calibration-less determination of Cu and Pb by anodic stripping voltammetry at thin mercury film micro-electrodes for the analysis of well water and rainwater (H9). Seasonal variations in heavy metal (Pb, Cd, Zn, Cu, Al) concentrations in present-day Greenland snow (H10) and the application of ICP-double focusing MS for the analysis of Cu, Zn, Mo, Pd, Ag, Cd, Sb, Pt, Pb, Bi, and U at the low- and sub-pg/g range in Greenland and Antarctic snow samples (H11) were reported.

(b) **Dry Deposition.** Dry deposition of atmospheric pollutants to the LOIS area, United Kingdom, was used to estimate a range of other pollutants to the river catchments in eastern Britain (H12).

Dry deposition velocities were determined for PAHs in the ambient air of traffic intersections collected using dry deposition plates and PS-1 samplers in the fall and winter seasons. Analytical results revealed that high-molecular-weight (HMW) PAHs, because of their association with particulates, had a higher deposition velocity and more than 94% of the HMW PAH mass was contributed by the particle phase (H13). PAHs in dry deposition in the ambient air in central Taiwan were monitored by using 21 PAH target compounds (H14). Ambient air concentrations, deposition fluxes, and grass concentrations/offtakes of PCDD/PCDF were measured concurrently for the spatial and temporal differences investigation and the possible mechanism of deposition (H15).

Different concentration gradients of Pb, Sb, and Cd versus distance to heavily traveled roads were observed by monitoring grass and deposition samples (H16). Atmospheric emission data were entered into the TRace toxic Air Concentrations in Europe (TRACE) model to predict depositions of Cd, Pb, and Zn from 1955 to 1987 in Europe (H17). Recent trends of trace elements in deposition, including V, Zn, As, Mo, Ag, Cd, Sn, Sb, Hg, Tl, Pb, and Bi, in Norway were reported using a 1995 moss survey (H18). The association of antimony with traffic pattern and its occurrence in airborne dust, deposition, and accumulation in standardized grass cultures was carried out using a dichotomous sampler (fine and coarse mode). It was demonstrated that the recovery of antimony in grass samples could be increased by a second digestion. The second digestion would have minimum effect on the recovery of the target in particulate samples (H19).

(c) Atmospheric Transport. Eight pesticides (atrazine, terbutylazine, malathion, alachlor, fenithrothion, pendimethalin, diuron, trifluralin) were used to demonstrate that particulate-associated pesticides can be used as geochemical tracers of domestic or agricultural activities. It was noted that atrazine, terbutylazine, and alachlor, due to their relatively low vapor pressure, were the best tracers (H20). Deposition patterns of long-range atmospheric transport of Ag, As, Cd, Hg, Mo, Sb, and Ti were studied by using terrestrial moss samples collected in a 188 000 km² area in the European Arctic (H21). Using energy-dispersive X-ray fluorescence spectrometry, airborne particulates collected using virtual impactors at four sites were analyzed for S, Cl, K, Ca, V, Cr, Fe, Ni, Cu, Zn, Br, and Pb. Intersite concentration ratios of these element were demonstrated to be a useful tool in the verification of air mass trajectories in long distance transport studies (H22). The ²⁰⁶Pb/²⁰⁷Pb isotopic ratio in lake sediment was also demonstrated to be a useful tracer in the assessment of atmospheric transport of Pb in eastern Canada (H23). The enantiomer ratios (ERs) of the persistent chiral pesticide α -hexachlorocyclohexane (α -HCH), was analyzed as (+)- α -HCH and (–)- α -HCH and calculated as (+)- α -HCH/(–)- α -HCH, by chiral phase capillary gas chromatography. The ER was demonstrated to be a useful tracer of air–water gas exchange in Lake Ontario (H24).

(d) Atmospheric Chemistry. ER of chlordane in the air near Sturgeon Point, NY, Eagle Harbor, MI, and Sleeping Bear, MI, were analyzed and compared for α -chlordane, γ -chlordane, and heptachlor epoxide B and were used to substantiate their persistent nature in the environment (H25). UV and IR absorption cross sections, vapor pressure, and rate coefficients of reaction

of α -pinene and D³-carene with OH and NO₃ radicals were determined to gain an in-depth understanding of the atmospheric fate of their carbonyl oxidation products (H26). Airborne carboxylic acids, phenols, and methacrylic acids were demonstrated to be the major oxidation products (with O₃) of airborne isoprene and toluene and were analyzed as their pentafluorobenzyl derivatives using either electron impact or chemical ionization mass spectrometry (H27). Based on either single or multiple soil measurements and a dynamic modeling framework, estimated deposition of air emissions in soil was reported. These dynamic estimates, made in the context of the uncertainty and variability associated with environmental systems, differ significantly from those based on a steady-state single-valued assumption. A case study on the global mass balance for PCDD and PCDF, for example showed a discrepancy of 6–20-fold between deposition and source emissions (H28). The applicability of the gas-phase chemistry of the EURAD model was determined and compared to the experimental data on the hydroxyl radical, a key photochemical substance (H29).

Biomonitoring. Air pollution control made by means of living organisms has been a popular approach for environment monitoring. Lichens, tree leaves, tree barks, grass, and vegetation are suitable tools for monitoring the relative levels of atmospheric pollution because of their ability to accumulate substances present in the environment. This section reviews publications where biological materials or living organisms were used as an integral part of environmental analysis, i.e., biomonitoring.

Concentrations of alkylated dibenzothiophenes were assessed using pine needles obtained from a recipient area of a pulp and paper mill environment (I1). Spatial distribution of PAHs in the U.K. atmospheric environment was monitored in 1994 using pine needles collected across the country (I2). Using electrothermal atomic absorption spectrometry, a method for the determination of As, Cd, and Pb in epicuticular waxes of pine and spruce needles was developed (I3). Santamaria et. el. demonstrated that tree bark could be used as a bioindicator of air pollution as good correlation could be obtained from the pH of bark extracts and defoliation levels (I4). Al, As, Ca, Cd, Cl, Cr, Cu, Fe, K, Mg, Mn, Ni, P, Pb, S, Si, and Zn were measured by XRF in Scots pine needles collected from transects across a subarctic area (Finnish Lapland and the Kola Peninsula in Russia). Analytical results indicated that Ni, Cu, Fe, P, and S were from a local smelter plant while concentrations of Mn and Zn remained constant (I5). Adsorption rates of atmospheric formaldehyde by deciduous broad-leaved, evergreen broad-leaved, and coniferous tree species were studied and demonstrated that trees could act as an important sink for atmospheric aldehydes (I6). Comparison of the bulk deposition of PCDD/PCDF in a spruce forest and its adjacent clearing was made and, due to the atmospheric deposition process, there were more elevated levels of PCDD/PCDF in forest soils than in agricultural soils (I7). Pb and PCB transport from local sources in the Canadian Arctic was demonstrated by using vascular plants (I8). Concentrations of Al, B, Ca, Cu, Fe, K, Mg, Mn, and Zn from treated experimental forest watershed and heavy metal depositions (Cu, Cd, Pb, Zn, Fe, Mn) were monitored using mosses (I9) and moss bags (I10), respectively. A comparison of levels of atmospheric metal pollution using low-volume aerosol samplers and moss *Sphagnum auriculatum* bioindicator for Cr, Cu, Fe, Mn, Ni,

Pb and Zn analysis was carried out. The correlation between the rate of metal uptake of moss correlates very well with that of atmospheric deposition (I11). Changes in atmospheric trace element (Pb, Cd, Cr, Ni, Mg) deposition were monitored by *Isoetecium stoloniferum* moss in Fraser Valley, BC, Canada (I12).

Hg was monitored by epiphyte plants in Brazil (I13) and by transplanted *Hypogymnia physodes* lichens downwind of Wisconsin (I14). Correlation of concentrations of trace elements (As, B, Cd, Cu, Fe, Hg, Mn, Pb, Zn) released by geothermal power plants in Italy suggested the possible transfer of atmospheric depositions from tree leaves to Epiphytic lichens (I15). Epilithic Antarctic lichens was used to determine the deposition patterns of heavy metals in the Shetland Island, Antarctica (I16). ICPMS and NAA methods were developed for the analysis of lichens and tomato leaves for the multielement characterization (I17) and metals such as Al, As, Br, Ca, Cd, Cl, Co, Cr, Cs, Fe, Hf, K, La, Ce, Nd, Sm, Eu, Tb, Yb, Lu, Mg, Mn, Na, Rb, Sb, Sc, Se, Th, Ti, U, V, and Zn (I18).

WATER ANALYSIS APPLICATIONS

This year, as with previous reviews, we emphasize the analysis of trace pollutants in environmental waters. We have selected articles containing methods that will lead to an understanding of the environmental concentrations, transport, and fate of pollutants. Issues pertaining to the general quality of the water supply, such as the analysis of dissolved gases, analytical methods to monitor drinking water quality, and detection of bacteria will be covered in the Water Analysis review in this issue.

Reviews and Articles of Broad Interest. Pharmaceutical compounds, which contaminate sewage, surface waters, and groundwaters, are the newest class of contaminants to generate interest (J1). Monitoring, sampling, and automated analysis of pollutants were reviewed with 63 references (J2). One review (190 references) discussed methods to detect both inorganic and organic compounds (J3). Methods for sampling, storage, and determinations of polar pesticides were reviewed with 155 references (J4). Matrix removal and sample concentration techniques for ultratrace metal analyses were reviewed with 81 references (J5). Several reviews described the determination of specific inorganic compounds, such as antimony (J6), thallium (J7), and arsenic (J8). Reviews of specific techniques, such as the use of neutron activation analysis in the marine environment (J9) and the use of remote electrochemical sensors for monitoring inorganic and organic contaminants (J10), were presented.

Sampling and Storage Issues. A review of sampling techniques for trace metals in sea- and freshwaters emphasized contamination prevention (K1). The sorptions of organic solutes by six polymeric materials used in groundwater well casings were studied. Analytes sorbed least to PVC and fiberglass-reinforced epoxy; acrylonitrile butadiene styrene sorbed analytes most rapidly and also leached contaminants into samples (K2). Hollow-fiber modules provided better filtration of lake water, with minimum artifact formation, than conventional membrane filters (K3). Mechanical energy, used to force contact between a sample and solvent, improved the efficiency of continuous extraction of dissolved and colloidal compounds; the extraction of colloidal triglycerides was enhanced 9 times with respect to classical flow extraction (K4). Tangential flow filtration, with 0.003- μ m pore size,

allowed more accurate determinations of dissolved Al than dead-end filtration with 0.45- μ m filters (K5).

Semipermeable membrane device (SPMD) uptake was not affected by suspended solids loads of <100 mg/L; however, reduced SPMD uptake occurred at high solids loads (K6). Water concentrations for PCDD/F and PCB obtained by SPMD and by Infiltrax resin samplers were comparable (K7). PAHs in urban streams were monitored by SPMD; reproducibilities ranged from ~15 to ~55%, with a detection limit of ~10 ppt (K8). SPMDs were also used to measure residence times of PAHs in an irrigation canal (K9). Uptakes of PCB by triolein-filled SPMD and trout were correlated (K10). Polyethylene bags filled with deionized water served as inexpensive diffusion samplers for VOCs in groundwater and yielded results comparable to those of traditional methods (K11). A new sampler was designed to collect seawater, at depths of 800 m, for ppt mercury analyses (K12).

Acidification was inadequate for preservation of dissolved Cu; oxidative pretreatment prior to acidification resulted in less analyte loss (K13). Approximately 30% of Se(IV) oxidized to Se(VI) in less than one month in acidic, oxygenated water in the presence of Cl^- (K14). Phenmedipham, Ethofumesate, and fenamiphos were extracted from groundwater with styrene divinylbenzene cartridges, and their stabilities under a variety of cartridge storage conditions were studied; 20–80% of these pesticides degraded after 20 days at room temperature (K15). Methylmercury was formed as an artifact during aqueous distillation; an extraction method incorporating leaching with $\text{KBr}/\text{H}_2\text{SO}_4/\text{CuSO}_4$, extraction into CH_2Cl_2 , back extraction into water, and subsequent ethylation minimized artifact formation (K16).

Groundwater. Analyses of groundwater were performed with goals of understanding groundwater flow (via tracer experiments) and of assessing environmental pollution. Indicators such as pH, temperature, and conductivity have been used to determine the time for optimum groundwater sampling—now, ^{222}Rn concentration has been proposed as another indicator (L1). The advantages and disadvantages of various samplers, including the Hydropunch and Geoprobe, were discussed (L2). A passive, discrete-level, multilayer groundwater sampler provided accurate vertical contaminant distributions (L3). The use of a PVC bailer adversely affected dimethoate recoveries, but not those of other pesticides studied (L4).

(a) Gases. Different methods for measuring hydrogen concentrations were evaluated; the gas-stripping method yielded results comparable to those of the diffusion sampler and was adapted for field use (L5). A quadrupole mass spectrometer was used to continually measure dissolved gases and $^{40}\text{Ar}/^{36}\text{Ar}$ isotope ratios (L6). Headspace GC/MS was used to measure methane, ethane, and ethylene to access intrinsic bioremediation (L7). ^{222}Rn was measured with a detection limit of ~0.05 Bq/L by liquid scintillation spectrometry (L8), ~0.1 Bq/L using a low-background liquid scintillation counter with a pulse shape analyzer (L9), and ~1 Bq/L by direct γ -spectrometry (L10).

(b) Inorganics. Arsenic contamination in Bangladesh is a problem; 38% of water samples analyzed in 27 districts contained more than 50 ppb As (L11). Ultrafiltration through membrane filters with 30- and 1-nm pores was used to distinguish dissolved As and Se species (L12). With the addition of HCl and excess Fe(II), As(III) was stable in solution for 1 week; treated samples

could be analyzed by hydride generation atomic absorption spectrometry (L13). An electrochemical-based probe was developed for in situ measurements of metal species; this probe used square wave anodic stripping voltammetry to measure ppb Cu, Pb, Cd, and Zn at a landfill (L14). X-ray absorption spectroscopy was used to determine Pb and Zn speciation in an aquifer (L15). A proportional correction factor was used to reduce errors associated with matrix effects in ICP AES (L16). $^{234}\text{U}/^{238}\text{U}$ α activity ratio was used to determine contamination from a uranium mill (L17).

(c) Organics. The organic compounds of most interest in groundwater were herbicides and pesticides. Herbicide contamination beneath claypan soils was studied; variability in hydrology was more important than differences in herbicide application rates (L18). In Iowa, the occurrence of herbicides and degradation products, including alachlor ethanesulfonic acid, atrazine, and deethylatrazine, changed depending on aquifer type, most likely because of different groundwater ages (L19). In the top 15 m of the saturated zone, 1,2-dichloropropane (fumigant component) concentrations decreased with depth (L20). In Arkansas, pesticide use and pesticide occurrence was correlated (L21). Large-volume extraction allowed detection of ppt concentrations of pesticides—atrazine, picloram, and 2,4-D were most frequently detected in Saskatchewan (L22). Liquid/liquid microextraction followed by GC/MS was used to detect 3–35 ppt concentrations of endosulfan and pyrethroids (L23). Sulfonic acid degradation products of acetochlor, alachlor, and metolachlor were detected at 0.1 ppb by electrospray LC/MS/MS (L24). ELISA overestimated atrazine, alachlor, and metolachlor concentrations because of cross-reactivity of the antibodies (L25); however, ELISA yielded results that correlated well with those of GC/MS for cyclodiene insecticides and atrazine herbicides (L26).

Gasoline oxygenates, including 100 ppt methyl *tert*-butyl ether (MTBE), and degradation products, were determined by direct aqueous injection and GC/MS (L27). Solid-phase extraction (SPE) and GC/MS were used to characterize the biogeochemical processes affecting the transport of semivolatile hydrocarbons, including PAHs (L28). LC, preceded by SPE, was used to determine substituted benzoic acids (0.1–2 ppm) resulting from degradation of aromatic compounds (L29). The Hydrosparge VOC sensor, consisting of a direct push well for groundwater sampling, an in situ sparge module, and a direct-sampling ion trap MS, was described (L30). SPME was used to concentrate heteroaromatic compounds, at 20–40 ppt, prior to detection with an ion trap MS (L31) and to concentrate aromatic amines at ~50 ppt (L32). LC and GC/MS were used to identify alkylphenol from septic systems (L33). Particle beam and thermospray LC/MS were used to detect ionic, organic components near Superfund sites (L34). Groundwater from a former ammunition plant was analyzed for nitrophenols by ^1H NMR, GC/MS, and LC (L35). Thermospray MS was used to identify 31 compounds associated with explosives, including TNT and RDX; thermospray was not well suited to quantification (L36). TNT was detected at 5 ppb using a fiber-optic biosensor (L37). A continuous-flow immunosensor was also developed to measure TNT (L38). Drugs and drug metabolites were detected at ppb concentration by GC/MS (L39).

(d) Tracers. Chlorofluorocarbons, $^3\text{H}/^3\text{He}$, and ^{85}Kr were used to date groundwaters (L40). ^3H and chlorofluorocarbons were

used to assess water protection zones (L41). SF_6 was used to measure residence times in streams (L42). A fluorescent dye was injected into groundwater in order to understand groundwater migration between two sites; ppt concentrations were measured by capillary electrophoresis/laser-induced fluorescence detection (L43). Rhodamine WT was also used as a tracer to monitor groundwater movement (L44). Chemical and isotopic analyses of $^{18}\text{O}/^{16}\text{O}$, $^2\text{H}/^1\text{H}$, $^{13}\text{C}/^{12}\text{C}$, and $^{87}\text{Sr}/^{86}\text{Sr}$ were performed to study the interaction of surface waters and groundwaters (L45). $^{11}\text{B}/^{10}\text{B}$ isotope ratios were measured to determine the sources of groundwater contamination (L46, L47).

Surface Waters. The number of publications dealing with surface waters has increased dramatically. We have cited twice as many publications as in our last review. Of the organic compounds analyzed, herbicides are the most studied. In addition, the analyses of degradation products of herbicides are becoming of interest. GC/MS was used to identify degradation products of alachlor (M1). Hydroxylated atrazine degradation products were measured at ~40 ppt in streamwater (M2). Desethylatrazine was found at concentrations similar to atrazine in Swiss lakes (M3). Atmospheric pressure chemical ionization (APCI) MS was used to identify degradation products of organophosphorus pesticides (M4). HPLC was used to measure the degradation products of isoproturon (M5). Derivatization GC/MS was used to determine degradation products of the fumigant 1,3-dichloropropene at detection limits of ~10 ppt (M6). Metabolites of dimethyl tetrachloroterphthalate were confirmed by derivatization and GC/MS (M7). Transformation rates of aldicarb, simazine, MCPA, and mecoprop were determined in surface waters and found to be variable (M8). Immunochemical techniques continue to be used and improved; a neural network was used to identify terbutylazine in samples (M9). Pesticides and other organic compounds, which were determined in surface waters, are listed in Tables 9 and 10. Both electrochemical (M71) and nonelectrochemical (M72) methods for studying trace metal speciation were reviewed with 213 and 135 references, respectively. The analyses of trace metals are summarized in Table 11.

Seawaters and Coastal Waters. The analysis of seawater, including particles, colloids, and trace elements and their speciation, was reviewed with 66 references (N1). Most studies focused on the analysis of inorganic compounds. An automated system was designed to provide on-line addition of isotopic spikes to aqueous samples for ICPMS analysis; the system was applied to the detection of Mo in saltwater (N2). A method using $\text{Mg}(\text{OH})_2$ coprecipitation and isotope dilution ICPMS was developed to measure Pb, Cu, and Cd in 1-mL seawater samples; detection limits for Pb, Cu, and Cd were 1, 40, and 5 pM, respectively (N3). ICPMS was used to determine heavy metals after extraction with dithiocarbamate and diisobutyl ketone; detection limits were element dependent and ranged from 0.2 to 20 ppt (N4). ICPMS was also used to determine trace metals after complexation with 2-(5-bromo-2-pyridylazo)-5-(*N*-propyl-*N*-sulfopropylamino)phenol; detection limits were 1–20 ppt (N5). A total of 10 ppt of trace metals were obtained using an automated flow injection system; iminodiacetate resin sequestered the trace metals, which were detected by ICPMS (N6). Ion chromatography was interfaced to ICPMS to determine sub-ppb concentrations of trace metals (N7). Pneumatic nebulization was compared to electrothermal vaporiza-

Table 9. Pesticides and Herbicides in Surface Water^a

analyte (no. of compounds)	comment/analysis technique (detection limits)	refs
atrazine, diuron, simazine, terbutylazine	LC/APCI-MS/MS (~100 ppt)	M10
atrazine	ESI-ion mobility spectrometry	M11
atrazine, simazine, terbutylazine, terbumeton, metribuzin, terbutryn	liquid/liquid microextraction and SPE, GC-NPD (10–40 ppt)	M12
atrazine	immunoassay screening compared to GC	M13
atrazine, simazine, ametryn, cyanazine, isoproturon, linuron, diuron, chlortoluron	mid-ppt	M14
triazines, acetamides, phenoxy acids	SPE with graphitized carbon black, derivatization with diazomethane, GC/MS (~10 ppt)	M15
bipyridyliums (diquat, paraquat, difenzoquat)	SPE with graphitized carbon black, LC (100 ppt)	M16
bromocyclen	chiral GC, ~300 ppq measured in waters	M17
carbendazim	conc with protein G immunoaffinity column and LC/MS/MS (25 ppt)	M18
dinitroanilines	SPME and GC-ECD (~40 ppt)	M19
fenthion, temephos, and degradation products	LC/APCI-MS	M20
lindane	~10 ppt	M21
organonitrogen (10)	SPE with graphitized carbon black or liquid/liquid with Gouliden extractor and GC-NPD (0.4–4 ppt) or LC/APCI-MS (0.6–3 ppt)	M22
organophosphorus	SPE with on-line LC (30–100 ppt), unattended, automated operation	M23
phenoxyalkanoic acids	<100 ppt	M24
pyrethroids	GC/negative ion MS (0.01–5 pg)	M25
sulfonylureas (12)	CE/UV and LC/ESI-MS (200 ppt)	M26
sulfonylurea, imidazolinone, sulfonamide (16)	SPE with RP-102, LC/ESI-MS (100 ppt)	M27
pesticides (17)	SPE with GC/MS or LC/UV detection	M28
pesticides	automated, on-line SPE, LC/APCI-MS (~10 ppt)	M29
pesticides (28)	SPE with styrene divinylbenze and GC/MS (~20 ppt)	M30
pesticides, including organochlorines	SPE with LiChrolut EN, GC-ECD or GC/MS (low ppt)	M31
pesticides, including chlorotriazine metabolites	SPE with C18, GC/MS (50 ppt)	M32
pesticides	SPE with styrene divinylbenze and GC (50–100 ppt)	M33
pesticides (alachlor, triazines, diazinon, cyclodienes, paraquat, 2,4-D)	immunoassay compared to GC/MS and LC methods	M34

^a Other abbreviations given in text. CE, capillary electrophoresis; ESI, electrospray ionization; NPD, nitrogen-phosphorus detector.

Table 10. Organic Compounds in Surface Water^a

analyte (no. of analytes)	comment/analysis technique (detection limits)	ref
acidic compounds	Gouliden extractor evaluated for acidic compounds, recoveries 20–90%	M35
amines, aliphatic	derivatization in water with 2,4-dinitrofluorobenzene or benzenesulfonyl chloride, extraction with CH ₂ Cl ₂ , GC/MS (sub-ppb)	M36
azo dyes, sulfonated	ion chromatography (low ppb)	M37
bisphenol A	GC/MS (600 ppt)	M38
CFC-11, CFC-12, N ₂ , Ar	GC-ECD (0.007 and 0.02 pmol/kg for CFC-11 and CFC-12, respectively, and 0.9 mmol/kg for N ₂ and Ar)	M39
chloro-bispropyl ethers	enantioselective GC/MS	M40
chloro ethers	SPME GC/MS (~100 ppt)	M41
chlorolignosulfonic acids	pyrolysis GC/MS/MS (500 ppt)	M42
clofibrate and clofibrac acid	brief discussion of U.S. research efforts	M44
dimethyl disulfide, dimethyl trisulfide, 2-isobutyl-3-methoxypyrazine	closed-loop stripping and GC/MS used to identify compounds associated with decaying vegetation	M45
1,3-dioxanes, 1,3-dioxolanes	closed-loop stripping and chemical ionization/MS or electron ionization/MS/MS	M46
ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid	esterification and GC-NPD	M47
flucufuron, sulcofuron (mothproofing agents)	LC/ESI-MS/MS (100 ppt)	M48
clofibrac acid	derivatization and GC/MS or GC/MS/MS (~1 ppt)	M43
haloacetic acids	derivatization with 2,4-difluoroaniline using dicyclohexylcarbodiimide as a catalyst, GC/MS	M49
haloacetic acids	negative ion ESI-MS (3–70 ppt)	M50
haloacetic acids	ion pair agent as modifier for in situ methylation, headspace sampling, GC/MS (~1 ppb)	M51
methylisoborneol, geosmin	microextraction with hexane, GC/MS (1–25 ppt)	M52
linear alkylbenzene sulfonates	no environmental accumulation observed	M53
linear alkylbenzene sulfonates	~5 ppb observed in rivers	M54
naphthalene sulfonates	CE-laser-induced fluorescence (1–35 ppt)	M55
organolead	GC-MIP-AED (~1 ppt)	M56
organotin	aqueous ethylation followed by liquid/liquid extraction or SPE and GC/MS (~1 ppt)	M57
PCB	extraction with XAD-2 (low ppt)	M58
PCB	seasonal variations in PCB observed	M59
PCB	130 ppq measured in Antarctic waters	M60
phosphonic acids	BSTFA derivatization and GC/ion trap MS	M61
phthalate esters	GC/MS	M62
misc compounds	identified by GC/MS and FAB/MS	M63
misc compounds	screened by GC-MIP-AED (pg of compounds containing C, H, Cl, Br, S, N)	M64
misc compounds	programmed temperature vaporizer inlet used to directly inject 1 mL of water into GC/MS; 20 compounds identified in river water (10 ppt)	M65
misc polar compounds	on-line (coupled) SPE and LC/particle beam MS (100 ppt)	M66
misc polar compounds (40)	LC/MS/MS (50–100 ppt)	M67
VOC	use of “presolvent” allowed SPE to include compounds traditionally considered too volatile for SPE (20–50 ppt)	M68
VOC	occurrence and distribution in British estuary	M69
VOC (55)	occurrence in Japanese rivers	M70

^a FAB, fast atom bombardment; MIP, microwave-induced plasma.

Table 11. Inorganic Compounds in Surface Water

analyte	comment/analysis technique (detection limits)	ref
As	electrothermal vaporization ICPMS (~10 ppt); NaCl interferences reduced	M73
As, Se, Sb	electrothermal vaporization ICPMS (~10–100 ppt)	M74
As	derivatization with thioglycolic acid methyl ester and GC/MS (100–3000 ppt)	M75
As(III)	differential pulse anodic stripping voltammetry using disk Au electrode (150 ppt)	M76
As	collected on activated carbon as molybdoarsenate, AAS (20 ppt)	M77
As	13–160 nmol/kg measured in Sierra Nevada rivers	M78
B	negative thermal ionization MS (1–10 ng) compared with ICP-OES	M79
Ca, Mg	LC on graphitic stationary phase with <i>o</i> -cresolphthalein in mobile phase (100 ppb Ca, 50 ppb Mg)	M80
Cd	continuous hydride generation, in situ preconcentration (5 ppt)	M81
Cd	cold vapor AAS (50–150 ppt)	M82
Cd	battery-powered, tungsten coil AAS (3 ppb)	M83
Cd, Pb	sequential metal vapor elution analysis and AAS used to separate Cd and Pb from interferences	M84
CN ⁻	biosensor based on immobilized bacteria and O electrode (50 ppb)	M85
Co	ligand exchange with dimethylglyoxime and differential pulse cathodic stripping voltammetry (total Co in Swiss lakes ~1 nM)	M86
Co	preconcentration on XAD-16 as Co–(2-thiazoylazo) resorcinol complex	M87
Co	flow cell with on-line UV digestion, voltammetric detection, 60 measurements/h	M88
Cr	graphite furnace AAS (10 pg)	M89
Cu (II)	preconcentration Cu(II) <i>N,N</i> -diethyl dithiocarbamate on thin film, spectrophotometry (sub-ppb)	M90
Cu(II)	comparison of ligand exchange and cathodic stripping voltammetry and ion-selective electrode; at low Cu concentrations, ion-selective electrode gave erroneous results	M91
Cu	ozone used to destroy dissolved organic matter prior to voltammetric determinations	M92
Cu, Fe	liquid/liquid extraction as picrate ion pairs, second-derivative spectrometry (3000 ppt)	M93
Cu, Pb, speciation	metal speciation based ion-exchange procedure	M94
Cu, speciation	Cu(I)–bathocuproine complex concentrated on C18, eluted, determined by spectrophotometry (4 ppb)	M95
Fe(II), Fe(III)	preconcentration on melamine–formaldehyde resin and spectrophotometric detection (400 ppt)	M96
³ H	impact of power plant quantitated and discussed	M97
³ H	0.1–0.9 Bq/L observed in surface waters	M98
Hg	adsorbed on dithione immobilized on sodium dodecyl sulfate-coated alumina, cold vapor AAS (sub-ppb)	M99
Hg, methyl	extraction with dithiocarbamate resin, derivatization, GC-MIP-AES (0.008 ppt)	M100
Hg, methyl	steam distillation, GC/atomic fluorescence spectrometry (~0.02 ppt)	M101
Hg, methyl Hg	Hg and methyl Hg measured in Anacostia River; total Hg concentrations <10 ppt–800 ppm	M102
Ir	coprecipitation with ferric hydroxide, anion exchange chromatography, UV irradiation to decompose organics, isotope dilution negative thermal ionization MS, concentrations in rivers ~20 × 10 ⁸ atoms/kg	M103
Mo(VI)	absorptive stripping voltammetry of benzoinoxime complex (10 ppt)	M104
Pb	electrothermal AAS (140 ppt)	M105
Pb	preconcentration based on formation of thenoyltrifluoroacetone complex and AAS; low ppb concentrations measured in real water	M106
Pb, Cd	rotating disk electrode voltammetry/anodic stripping voltammetry; Nafion-coated thin Hg film electrodes block transport of metal–fulvic acid complexes to the electrode surface	M107
Pb, Cr	differential pulse polarography, 15 ppb Pb measured in water	M108
Pb(II), Cu(II)	passive monitor based on polyacrylamidoxime cloth, energy and wavelength-dispersive XRF	M109
Ra	precipitation with acetic acid, α -spectrometry	M110
rare earth elements	hydrated Fe(III) hydroxide coprecipitation, ICPMS detected low ppt of rare earth elements in Antarctic lakes and streams	M111
Rh	Mg-W cell electrodeposition, AAS (13 ppb)	M112
Sb	hydride generation, ICP-AES, Chelex 100 used to remove transition metal interferences (~200 ppb)	M113
Se	Se ⁴⁺ and tetrahydroborate ³⁺ retained on anion-exchange resin, AAS (100 ppt)	M114
Se speciation	Se ⁴⁺ , Se ⁴⁺ + Se ⁶⁺ , and Se ⁴⁺ + Se ²⁻ determined, in turn, by differential pulse cathodic stripping voltammetry, UV light destroyed organic interferences (0.2 ppt)	M115
Sr	extraction with Empore strontium rad disks	M116
⁹⁰ Sr	accelerator MS (1 pCi/L)	M117
thiosulfate	indirect spectrophotometry using oxidation of thiosulfate by iodine (~10 ppb)	M118
U	waters and sediments sampled to determine contamination	M119
V	adsorption on Sephadex G-25, catalytic photometric detection (20 ppt)	M120
V	preconcentration with Sephadex DEAE A-25 with Eriochrome Cyanine R, speciation of V(IV) and V(V), spectrophotometry (1 ppb)	M121
Y	preconcentration with Chelex 100, ultrafiltration, ICPMS	M122
trace elements (V, Co, Cd, Sb, Pb)	flow injection manifold developed for preconcentration followed by ICPMS	M123
trace elements (Mn ²⁺)	voltammetric, in situ profiling system monitored to 500 m depth, useful for seawater (5 ppt)	M124
trace elements (Pd, Cd)	voltammetric, in situ sensor consisting of array of Hg-plated, Ir microdisk electrodes with agarose gel membrane	M125
trace elements (U, Ni)	integrated membrane sampling/absorptive stripping sensor, Pr gallate and dimethylglyoxime chelating agents used to monitor U and Ni	M126
trace elements	preconcentration with disodium piperazino-1,4-bis(dithiocarbamate), wavelength-dispersive XRF, compared to ICPMS and AAS	M127
trace elements (Zn, Cd, Pb, Cu, Ni, Sn)	anodic and adsorptive stripping voltammetry in the presence of complexing/chelating agents used in automated measuring system (1–10 ppb)	M128
trace elements	NAA used to monitor metals in various waters in India	M129
trace elements	solid-state electrode developed for square wave anodic stripping voltammetry, Ag/AgCl wire coated with electrolyte immobilized with Nafion or polyurethane	M130
trace elements	preconcentration on CeO ₂ , AAS	M131

tion as sample introduction for ICPMS; electrothermal vaporization removed some polyatomic interferences caused by Na and Cl

(N8). A combination of ligands were used so that different trace elements could be simultaneously determined, at ~0.1 nM

Table 12. Inorganic Compounds in Seawaters and Coastal Waters

analyte	comment/analysis technique (detection limits)	ref
Al	complex formation with salicylaldehyde picolinoylhydrazone and fluorescence detection (10 pM)	N13
Br ⁻	CE (0.5 ppm)	N14
Cd, Pb	anodic stripping voltammetry; ~0.7 nM Cd and ~0.1 nM Pd measured in Antarctic waters	N15
Cd	C18-bonded silica gel coated with methyltricaprylammonium chloride used to concentrate Cd, GF/AAS (0.2 ppt)	N16
Cr(VI)	ion pair extraction with tricaprylmethylammonium chloride, GF/AAS (0.1 nM); Cr(VI) in frozen seawater stable for 8 months	N17
Cu, Mn	Mg(NO ₃) ₂ and Pd(NO ₃) ₂ used as modifiers, GF/AAS (~100 ppt)	N18
Cu, Cd, Pb, Bi, Se	analytes complexed with <i>O,O</i> -diethyl dithiophosphoric acid on C18 silica column coupled to electrothermal vaporization ICPMS	N19
Cu, Zn, Cd, Pb	chemical speciation of dissolved elements determined by differential pulse anodic stripping voltammetry; Narragansett Bay estuary, Rhode Island, studied	N20
Fe	shipboard measurement, preconcentration on 8-hydroxyquinoline resin, chemiluminescence detection (40 pM with 3-min analysis)	N21
Fe	Mg(OH) ₂ coprecipitation, isotope dilution ICPMS (~0.1 nM)	N22
Hg	liquid chromatography ICPMS (30–100 ppt)	N23
I ⁻	poly(styrene–divinylbenzene) anion-exchange resin preconcentration, ion chromatography (200 ppt)	N24
I (total reducible)	cathodic stripping voltammetry (~0.2 nM)	N25
Mn(II)	cathodic stripping voltammetry with rotating glassy carbon disk electrode (6 nM)	N26
Na ⁺ , K ⁺ , Mg ²⁺ , Ca ²⁺	CE with indirect UV detection (~100 ppt)	N27
²³⁷ Np, ¹²⁹ I	monitoring studies indicate ²³⁷ Np from Sellafield can be measured in Arctic Ocean	N28
³² P, ³³ P	ultra-low-level liquid scintillation counter developed; energy-specific β -spectra obtained and used to identify other species	N29
Pb	differential pulse anodic stripping voltammetry used to measure dissolved and particulate Pb in South San Francisco Bay	N30
²³⁹ Pu, ²⁴⁰ Pu, ¹³⁷ Cs	monitored in Japan Sea; total Pu in surface water ~7 mBq/m ³ ; total Pu at 500 m ~40 mBq/m ³	N31
Pu	basic alumina microcolumn used with flow injection ICPMS (ppq)	N32
Se(IV), Sb, As, Ge	continuous-flow hydride generation ICPMS; methanol matrix modifier (~1 ppt)	N33
Se(IV), Se(VI)	continuous-flow hydride generation atomic fluorescence spectrometry (1 ppt)	N34
thiosulfate	linear sweep voltammetry	N35
Tc	concentration with anion-exchange resin; ultrasonic nebulization with membrane desolvation and ICPMS (0.03 ppt)	N36
V	V-catalyzed oxidation of Bindschedler's green leuco base, spectrophotometry (0.2 nM)	N37
V	liquid chromatography/ICPMS (30–40 ppt)	N38
Zr, Hf	concentration with Chelex 100, isotope dilution ICPMS (pM–fM)	N39
rare earth elements	preconcentration with ion-exchange chelating fiber with aminophosphonic and dithiocarbamate groups, ICPMS (ppt)	N40

Table 13. Organic Compounds in Sea and Coastal Waters

analyte	comment/analysis technique (detection limits)	ref
atrazine	immunoassay compared with SPE and GC/MS; 20–120 ppt atrazine detected in coastal waters	N41
butyltin	in situ derivatization with sodium borohydride and simultaneous extraction into dichloromethane, GC flame photometric detection (5 ppt)	N42
butyltin	GC-negative chemical ionization MS (25 ppq)	N43
CH ₄ , N ₂ O	automated GC with ECD (N ₂ O) and with FID (CH ₄) (~50 pM/L)	N44
chlorinated hydrocarbons (volatile)	13 compounds studied in North Sea and Scheldt Estuary; average water concentrations of individual compounds 2–70 ppt	N45
dimethyl sulfide, dimethyl sulfoniopropionate, dimethyl sulfoxide	dimethyl sulfoxide dominant compound in Mediterranean Sea; average concentrations of dimethyl sulfide, dimethyl sulfoniopropionate, and dimethyl sulfoxide of 3, 3, and 20 nM, respectively	N46
Irgarol 1051 (antifouling agent)	ELISA (20 ppt) compared with on-line SPE-LC with diode array detection (1 ppt)	N47
organoiodine, organobromide	purge-and-trap GC-ECD (0.3–15 ppt); presence of CH ₂ ClI, CH ₂ I ₂ , CH ₃ CH ₂ CH ₂ I documented in Atlantic Ocean	N48
PAH, alkylated	63 compounds (at 5–100 ppt) identified by GC/MS	N49
PCB	dissolved and particulate fractions measured; 300–1600 ppt total PCB in San Francisco Bay	N50
pesticides	SPE combined with large-volume injection using a programmable temperature vaporizer and GC/MS/MS (ppt with 200-mL sample)	N51
sulfonates, linear alkylbenzene (LAS)	fate of LAS in Bay of Cadiz (Spain) studied; nonconservative behavior due to degradation observed	N52
sulfonates, LAS	LC/ion spray MS used to identify degradation products; biodegradation of LAS slow in an oxygen-deficient marine environment	N53

concentrations, by cathodic stripping voltammetry (N9). X-ray fluorescence spectrometry was used to monitor trace metals in Barbados Bay, Colombia (N10). Neutron activation analysis, preceded by Mg(OH)₂ coprecipitation, was used to determine trace metals; Hg²⁺ and Ni²⁺, which form stable complexes with Cl⁻, were inefficiently adsorbed (N11). On-line precipitation electrothermal atomic adsorption spectrometry was developed to measure ppt concentrations of trace elements (N12). Other methods for specific inorganic analytes are summarized in Table 12. Methods for the analyses of organic compounds are listed in Table 13.

Wastewaters. (a) Organics. Techniques used to analyze organic compounds in leachates of industrial waste landfills and results of analyses were discussed (O1). A study of 168 samples of New York City municipal wastewaters, collected from 1989 to

1993, identified 37 different compounds; the occurrence of priority pollutants was infrequent (O2). A chapter reviewing organotins in wastewater, their speciation, and analytical methods was written; ~400 ppt total butyltins were observed in a landfill leachate (O3).

GC/MS continues to be a standard technique for the analysis of organic compounds in wastewaters. Volatile organic compounds, including chloroform, saturated carboxylic acids, phenols, and alkylbenzenes, were removed from wastewaters by SPME and analyzed, from 10 to 170 ppb, by GC/MS (O4). SPME with a polyacrylate-coated fiber was used in the analysis of phenols; results were compared to liquid/liquid extraction (O5). Wastewater from a pharmaceutical factory was preconcentrated and introduced into a GC/MS by direct aqueous injection—acetone, acetonitrile, and other compounds were detected at 0.1 ppb (O6). Screening of heteroatom-containing compounds at 20–1000 ppt

was accomplished by GC-atomic emission detection (AED) (O7). GC/MS, GC-AED, pyrolysis GC/MS, and pyrolysis GC-AED were used to characterize coal wastewaters. Phenols, polycyclic aromatic hydrocarbons, indenols, and heterocyclic compounds were detected; polymers of natural and anthropogenic origins could be distinguished (O8). Hydrothiol 191 (amine salt of endothall) was determined to 50 ppb with solid-phase extraction, derivatization with heptafluoro-*p*-tolylhydrazine, and detection with GC-ECD (O9). Phenolic compounds were extracted with poly(styrene-divinylbenzene) resin (more effective than graphitized carbon black) and derivatized on-column with 3,5-bis(trifluoromethyl)-benzylidimethylphenylammonium fluoride; ppt concentrations were detected by negative ion chemical ionization GC/MS (O10). Polar pollutants from textile industries were monitored by LC/MS and LC/MS/MS (O11). Solid-phase extraction followed by APCI LC/MS was used to determine 0.2–50 ppb of various phthalates, phenols, and tributyl phosphates (O12) and 1 ppb thiourea (O13).

Short-chain aliphatic acids were determined by ion-exclusion chromatography; two systems differing in retention characteristics were used; amino acids were determined by ion chromatography (O14). HPLC with diode array and fluorescence detection was used to determine aminobenzoic acids in wastewaters from a former ammunition plant (O15). Nonylphenol polyethoxy carboxylate metabolites were extracted with strong anion-exchange disks, derivatized, and analyzed by GC/MS, operated in positive chemical ionization mode; detection limits were 0.2–2 ppb (O16). A method for the determination of phenolic lignin degradation products in Kraft black liquors used liquid extraction and capillary electrophoresis with UV detection (O17). The 17- β -estradiol present in runoff from poultry litter applied to pastures was determined by ELISA; amending the poultry litter with alum reduced 17- β -estradiol in runoff (O18). ELISA and LC/APCI MS were used to determine pollutants such as pentachlorophenol, phthalates, and nonylphenol; the advantages and limitations of three ELISA kits used to characterize industrial effluents were reported (O19).

(b) Inorganics. Ultrafiltration and neutron activation analysis or atomic adsorption spectroscopy (AAS) were used to determine the speciation of trace metals in discharges from the pulp industry; almost all the alkali metals, Cs, Rb, Sr, and Ni were present in the low-molecular-weight fraction (O20). As(III) was determined indirectly by oxidation with a known excess of Cr(VI); 2-(α -pyridyl)thioquinaldinamide was added and fluorescence was measured; detection limit was 0.3 ppb As (III) (O21). Competitive ligand equilibration cathodic stripping voltammetry and column partitioning-graphite furnace AAS were used to determine speciation of Cu and Ni in South San Francisco Bay; the existence of strong metal-complexing ligands in wastewater effluent must be considered in remediation strategies (O22). LC/ICPMS was used to study an industrial Te removal process (O23). Electrothermal AAS was used to determine sub-ppm concentrations of Os in wastewaters; thiourea was used as a matrix modifier to minimize interferences (O24). Hg in pulp effluents, at ppb concentrations, was measured, by spectrophotometry, as a Hg iodide–brilliant green complex (O25). Fluorescence quenching of Rhodamine B with Hg²⁺ was used to measure total Hg, at 10 ppb, in wastewaters (O26). Flow-through stripping chronopotentiometry was used to determine ppb Hg in wastewaters (O27). LC coupled with UV

detection, AAS, and ICPMS was used to determine Cr (ppb) speciation (O28). In another study, ICPMS was used to determine Cr (0.5 ppb) speciation; separation of interfering ions was discussed (O29). Beryllium in mining wastes was determined to 200 ppt by complexation with Chrom Azurol S, preconcentration with an anion-exchange resin, and spectrophotometry (O30). Membranes, impregnated with MnO₂, were used to collect ionic actinides (U, Pu, Am, Cm) in coolant waters of nuclear power plants; α -spectrometry was used to detect 0.04 Bq/L (O31). ⁸⁹Sr and ⁹⁰Sr, at 800 Bq/L, in nuclear reactor effluent was measured by ion chromatography and liquid scintillation (β) spectroscopy (O32). γ -Spectrometry was used to determine ²²⁶Ra in uranium mine process waters (O33). Sulfide in wastewater from a dairy farm was determined, indirectly, with a reactor containing PbCrO₄ suspended on alumina; spectrophotometry was used to obtain 3 ppm detection limits (O34). A total of 80 ppb sulfide was measured using an inexpensive, compact, solid-state detector and the standard “Methylene Blue method” (O35). Sparged H₂S was precipitated in an acetate-supporting electrolyte by silver ions; 2 ppb sulfide was detected by coulometry (O36). On-line dialysis, performed in a flow injection analysis system, was interfaced to capillary electrophoresis for the determination of small anions in various matrixes, including pulp effluents (O37). A quartz crystal balance was used to monitor 1–800 ppm CN[−] in electroplating wastewaters; the results produced by this rapid technique were comparable to those of the ASTM method (O38). An anion-exchange column was developed to allow isocratic separation of weakly retained organic and inorganic anions at low-ppb concentrations (O39). An ion chromatography method, which overcomes problems of low recovery of chlorine dioxide and poor resolution of chlorite from organic anions, was developed to measure inorganic chlorine species in kraft mill bleach effluents; the use of ethylenediamine as a preservative for chlorite was assessed (O40).

Instruments and Methods. (a) Inorganics. Included in this section are instruments and methods which, although not routinely applied to the analysis of environmental waters, are worthy of note. A sensor array of ion-selective electrodes was used to simultaneously measure a variety of chemical species; chemometrics and neural networks were used to optimize information obtained (P1).

An integrated membrane sampling/adsorptive stripping sensor was designed to allow detection of metals that are not readily accumulated by amalgamation. This sensor detected 0.01 μ M U and Ni, using praseodymium gallate and dimethylglyoxime chelating agents, and has the potential to be used as a remote sensor (P2). Capillary electrophoresis continues to be developed for the analysis of charged metals; Fe(II), as a phenanthroline complex, was detected, at 5 nM, by its absorbance at 270 nM (P3). Laser-induced breakdown spectroscopy was developed as a fast, sensitive technique for the measurement of trace elements in water (P4). There is a trend toward connecting ICPMS with sample preconcentration and/or chromatographic separation methods. A preconcentration system, using two parallel columns of AG50W-X8 resin, was joined with ICPMS and provided sample enrichment factors of 10–95 in less than 5 min (P5). Silica gel functionalized with 1,5-bis(di-2-pyridyl)methylene thiocarbohydrazide served as an on-line preconcentration system for ICP atomic emission

spectrometry; 1 ppb Hg was detected (P6). LC/ICPMS was applied to the determination of organotin compounds; mono-, di-, and trisubstituted tins were separated and detected at ppt concentrations (P7). Gas chromatography/chemical reaction interface mass spectrometry was used to detect 60 pg of Se (P8). Mass spectrometry has also been used to detect analytes traditionally determined by ion chromatography. Negative thermal ionization isotope dilution mass spectrometry and ICPMS were used to determine bromide and bromate; detection limits of 30–90 ppt, over 1 order of magnitude better than those obtained with ion chromatography, were reported (P9). Chlorite, chlorate, bromate, and iodate were determined with ion spray MS/MS; this method provided high specificity and sensitivity (P10). SPME, typically thought of as a technique for the extraction of organic compounds, was applied to the analysis of metal ions. SPME fibers doped with crown ether were used to extract Hg from water; the resulting complex was desorbed from the fiber and analyzed by LC with UV detection (P11). A spectroscopic method for the detection of both inorganic and organic compounds in water using in situ corona reactions was described; initial data were presented on the measurement of Cl and BTEX in waters (P12).

(b) Organics. A heated extraction cell was developed to remove volatile and semivolatile compounds, at 50–200 ppt, from 1–2-mL aqueous samples. Analytes were removed from the sample with elevated temperatures (~90 °C) and a 30 mL/min gas flow; a reflux condenser was used for water vapor management (P13). SPME was used to extract hydrocarbons from water; a linear temperature-programmed retention index was developed to aid quantification (P14). SPME, using three different fiber types, was evaluated for the extraction of 49 organophosphorus pesticides; XAD and polyacrylate fibers performed best (P15). SPME coupled to GC/isotope ratioing MS was used to measure ^{12}C to ^{13}C ratios for toluene, methylcyclohexane, hexanol, and acetic, propionic, and valeric acids (P16). Isotope ratios can potentially be used to identify contamination sources. GC/isotope ratioing MS was also used to analyze benzene, toluene, xylene, and ethylbenzene (BTXE) (P17) and aroclors, clophens, kaneclors, and phenoclor (P18). LC/electrospray MS, preceded by SPE, has gained wide acceptance for the detection of organic compounds including ~1 ppt of 45 pesticides (P19), ~30 ppt glyphosate and aminomethylphosphonic acid (P20), and ~10 ppt poly(ethylene glycol)s (P21). Novel techniques developed for the detection of organic compounds in water included an evanescent fiber-optic chemical sensor (P22), fiber-optic-based biomonitoring of benzene compounds by recombinant *Escherichia coli* bearing a luciferase gene fused to TOL plasmid (P23), a purge and membrane mass spectrometer, which detected sub-ppb concentrations of volatile organic compounds (P24), and a mass spectrometer with a rotating ball inlet for continuous monitoring of aqueous solutions (P25).

Quality Control and Quality Assurance. An inventory of available CRMs for inorganic analytes in various aqueous matrixes was prepared (Q1). General considerations in the preparation of CRMs were presented (Q2). New concentration values for trace elements in more than 100 U.S. Geological Survey standard reference water samples were reported (Q3). Results of an interlaboratory study to test the feasibility of preparing groundwater reference materials and to eliminate problems with trace

element determinations were reported (Q4). Studies describing the certification of aqueous matrixes for several trace elements, including mercury (Q5), cadmium (Q6), rubidium (Q7), copper (Q8), chromium (Q9), and selenium (Q10) were reported. Results of an interlaboratory study to measure specific activity associated with tritium were given (Q11). The European Union project on Quality Assurance of Information in Marine Environmental Monitoring in Europe was reviewed with 15 references (Q12). A multilaboratory round robin showed that only three out of eight organic compounds spiked into effluents were isolated and positively identified by a proposed GC/MS library search method (Q13). Twenty laboratories participated in a validation study of SPME; the reproducibility and accuracy of SPME was good and headspace sampling, instead of direct sampling of the liquid, provided the best precision (Q14). Forward censored regression was introduced for selecting an appropriate model to treat data sets with values that are below the detection limit; this procedure was applied to concentrations of atrazine, deethylatrazine, and deisopropylatrazine in U.S. groundwaters (Q15).

SOIL AND SEDIMENT ANALYSIS APPLICATIONS

Sampling. The whole field of the sampling of biological, environmental, and technical materials was reviewed by Stoeppler (R1). What is good practice in sampling was discussed by Epps, including the effect of sampling variables on test results and the need for standardization of sampling methods (R2). For individual investigations of contaminated soil, sampling errors were much greater than analytical errors; therefore, the theory and practice of sampling contaminated bulk material must be developed (R3). Methods for evaluating the performance of sampling, sample preparation, and subsampling have also been reviewed (R4).

Several new methods and apparatus for sampling environmental matrixes have been described in the past two years (R5–R12). A new sampling model was developed that is especially adapted to the specific conditions of sampling contaminated bulk soil masses (R5). The use of dynamic (window) sampling in site investigations of soil and groundwater was discussed by Eccles (R6). Reduced costs and improved statistical performance can be obtained by application of composite sampling methods (R7). New sampling devices include a gravity-driven, hydraulically damped multipiston corer for fine-grained sediments (R8) and a time-series sediment trap that can collect 21 samples at programmed intervals (R9). Problems and solutions with respect to sampling riverine sediments impacted by acid mine drainage were discussed by Herr (R10). Evaluation of sampling and analytical errors for the determination of manganese in soils (R11) and protocols for a sampling campaign for explosives-contaminated sites (R12) were also presented.

Sample Preparation. A number of studies on sequential extraction of trace metals were reported (R13–R20). Metal distributions were significantly different among three sequential extraction procedures compared (R14). Silty soils may have a relatively high heavy metal retention capacity due to the presence of carbonate, and this retention capacity can be comparable in magnitude to that of certain clayey soils (R18). Soil amended with sewage sludge exhibited a difference in the distribution of metals in soil (R19). Bodog extended the sequential extraction procedure of Ure, and observed good agreement with a total acid extraction

procedure (R20). Vacuum microwave drying of marine sediments can be completed in 10 min compared to 8 h or more by oven-drying (R21). Compared to fresh sediment, not one of three drying methods studied (freeze-drying, air-drying, oven-drying) completely preserved the distribution of selected metals in the various geochemical fractions of the sediment (R22).

Various digestion methods were compared for metals in sediments (R23–R25). The comparability of the preparation procedures (drying, grinding, sieving, etc.) of sediments is just as important as the extraction and instrumental analysis steps (R26). In one comparison of various digestion methods for metals in sediments, sonication with reverse aqua regia followed by hot plate treatment was found superior to other methods examined, which included use of a microwave oven (R27). Notwithstanding this specific study, the trend is clearly toward increased use of microwave-assisted extractions as shown in several studies (R29–R31) and a review (R32). The technique of ultrasonic extraction for heavy metals should not be overlooked because of its simplicity, speed, and ease of use, as was discussed by Ashley in a review (R28).

Inorganic Analytes. (a) Mercury. The determination of mercury (S1) and mercury and organomercury compounds (S2) in environmental samples has been reviewed. In a comparison of various acid dissolution methods, only the use of a HNO_3/HF mixture in a closed microwave container gave values for both As and Hg that matched certified values of a reference material (S3). Total Hg concentrations determined after a pyrolysis treatment showed lower values and up to 3-fold higher RSD than those obtained after wet digestion (S4). Distillation as a method of sediment sample preparation was combined with on-line solid-phase preconcentration to give excellent recoveries for methylmercury (S5, S6). A simple and selective method for leaching methylmercury from sediments was based on ultrasonic extraction with 0.5% (m/v) thiourea aqueous solution (S7). Lyophilized sediment samples exhibited better homogeneity and analytical reproducibility for methylmercury compared to wet samples (S8). For measuring mercury soil emissions, a Teflon dynamic flux chamber was developed by Carpi (S9). For the instrumental determination of mercury and methylmercury, AAS remains a popular choice (S10–S14). In one report, a method was described for the direct AAS determination of Hg in sediments with no sample treatment (S10). One analysis could be performed in 2 min, including sample weighing. The use of sequential injection (S11), slurry sampling (S12), and cryofocusing techniques (S13) with AAS have all been reported. Energy-dispersive X-ray fluorescence spectroscopy gave results for total Hg in various environmental samples after digestion that were comparable to results from AAS analyses (S14). Other analytical methods reported for Hg determinations included an isotope dilution ICPMS procedure (S15), and a GC-ECD method for speciation of methylmercury (S16).

(b) Chromium. Marques reviewed the analytical literature concerning Cr speciation in solid sample types (S17). In another review, the reagents for spectrophotometric determination of Cr were covered (S18). The effects of soil sample preparation procedures for Cr in soil samples were reported (S19). The optimum conditions determined included use of a homogeneous sample with mass of >4000 g, grain diameter of <0.25 mm,

digestion with a solution of $\text{HNO}_3 + \text{HClO}_4$ (3:2), and HCl after dry ashing, with addition of 1% La or 1% NH_4Cl to eliminate interferences. Cr was determined in soil and sediment samples by modifier-free slurry sampling with overall analytical repeatability better than 10% (S20). Determination of the Cr(VI)/Cr(III) ratio was accomplished without any sample pretreatment by using X-ray absorption fine structure spectroscopy (XAFS) (S21), and after extraction from contaminated soils by using X-ray absorption near-edge structure spectroscopy (XANES) (S22).

(c) Arsenic and Selenium. A method for As in sediment was reported that combined slurry sampling, microwave-assisted extraction, and hydride generation AAS (S23). Lopez-Garcia determined both As and Sb in soil and sediment by slurry sampling and graphite furnace AAS (S24). For the high-pressure microwave digestion AAS determination of total As and Se in sediments, a study of five different acid mixtures showed that methods using aqua regia were best (S25). Organoarsenicals in soils and sediments were determined after dithiol derivatization with solid-phase microextraction and GC/MS (S26). Demesmay showed that the use of microwave extraction procedures work well for mineral As, but may affect the relative amounts of various As species (S27). To determine the various forms of As present in soil and sediment samples, the techniques of coupled HPLC/ICPMS (S28), high-performance ion chromatography with plasma source MS (S29), and capillary electrophoresis with ICPMS (S30) have all been applied. The determination of various Se species in soil and sediment samples has also been studied (S31–S34). Martens employed sequential extractions and hydride generation AAS and achieved excellent precision (S31). Speciation of SE(IV) in sediments was accomplished using neutron activation analysis after coprecipitation with dibenzylthiocarbamate with phenolphthalein (S32). Selenium species were also determined by using HPLC (S33) and GC/MS (S34).

(d) Organotins. After an evaluation of extraction variables affecting the stability of butyl- and phenyltin compounds, a toluene/HOAc mixture (10:4) was found to give the best extraction efficiency and to minimize the degradation of trialkyl- and triaryltins during the extraction (S35). Another procedure for extracting organotin species was based on soaking sediments in a mixture of water/hydrogen bromide (1:1), followed by extraction with 0.04% (w/v) tropolone solution in dichloromethane for 2 h (S36). Cai performed an extensive study of complexing agents and acid modifiers on the supercritical fluid extraction of phenyltins and butyltins from sediment and obtained the highest efficiency at 30 MPa and 50 °C by using CO_2 modified with acetic acid (200 μL in the cell) (S37). Other extraction procedures for recovery of organotins include the following: use of accelerated solvent extraction (S38), microwave-assisted extraction and Na-BEt_4 derivatization (S39), direct derivatization on sediment slurries (S40), and ethylation followed by GC-FPD (S41) or GC/ICPMS (S42). For the rapid speciation of trace concentrations of organomercury, organolead, and organotin compounds, isothermal multicapillary GC/plasma emission spectrometry was employed (S43).

(e) Other Metals. Reviews on the determination of metals in soils and sediments have been published (S44, S45). Reddy determined Cd in soil and leafy samples by differential pulse anodic stripping voltammetry after extraction as xanthate into

chloroform (S46). One study showed that complementary procedures for Al speciation provided more comprehensive information when used together (S47). The three methods used in this study were cation-exchange fast protein LC/ICP-atomic emission spectrometry, microcolumn chelating ion-exchange chromatography-AAS, and the 8-hydroxyquinoline spectrophotometric method. Al speciation was also reported by using cation-exchange and size exclusion HPLC methods with postcolumn fluorescence detection using 5-sulfo-8-quinolinol (S48). A rapid method for Cu, Cd, and Pb was reported, based on on-line preconcentration on a microcolumn packed with C18 material (S49). Sample throughput was 30/h, with a loading time of 1 min. Johnston reported that microscope spectroscopy (scanning electron microscope energy-dispersive X-ray spectroscopy, laser Raman microscopy) can provide detailed information on the location, elemental composition, and chemical speciation of soil contamination (S50).

Organic Analytes. (a) Volatile Organic Compounds. Three vapor partitioning headspace and three solvent extraction methods for the preparation of soil samples for VOCs determination were compared (T1). Methanol extraction was the most efficient method of spiked VOCs recovery, which depended on soil organic carbon content, octanol–water partition coefficients of analytes, and extraction time. Ball employed hot (70 °C) methanol extractions for quantitative recovery of VOCs from subsurface core samples (T2). Solid-phase microextraction was found to be an effective technique for VOCs in landfill site samples by sampling the headspace layer above a soil sample (T3). Pervaporation as an alternative to headspace has been proposed (T4). Good precision and recoveries were observed using this technique for VOCs in soil samples, with detection limits on the order of 1 ng/g. Stuart addressed the analysis of VOCs from various soil samples by using an automated, static headspace method (T5). VOC recoveries were found to decrease in the order water, pure sand, sandy soil, clay, and top soil. A full evaporation technique that uses little or no aqueous phase and higher equilibration temperature was found to give the most reproducible analyte recoveries. Kawata described the effects of headspace conditions on VOC recoveries from sediments (T6).

(b) Pesticides. The application of supercritical fluid extraction (SFE) for extracting pesticides from soil samples was investigated in a number of studies (T7–T10). The best efficiency for SFE extraction of fenpropimorph, pirimicarb, parathion-ethyl, triallate, and fenvalerate from soil was achieved at 60 °C extraction temperature, CO₂ pressure of 3.8×10^7 Pa, 5% methanol modifier, diol-modified silica gel trap, and ethyl acetate as eluent (T7). More rapid analysis times and minimum sample preparation times were achieved by the on-line coupling of SFE extraction and HPLC (T8). SFE and subcritical water were compared for the extraction of a metabolite of chlorpyrifos from soil (T9). Complete extraction by using subcritical water was achieved within 15 min without additives. Chlordane was extracted from soil by using SFE, Soxhlet, and accelerated solvent extraction by Brumley, who developed improved approaches to Superfund sample analyses (T10). Low-ppb detection limits were achieved for the determination of organochlorine insecticides from soil and sediment samples by using accelerated solvent extraction followed by GC/ion trap tandem mass spectrometry (T11). Other extraction methods tested for pesticides in soils and sediments included solid-

phase extraction (T12), solid-phase microextraction (T13), and microwave-assisted extraction (T14–T16). The selectivity of microwave-assisted extractions was found to be highly dependent on soil composition (T15).

A variety of techniques were investigated for the detection and quantitation of pesticides in soils and sediments (T17–T32). The determination of chlorpyrifos in soil by GC analysis was compared to p-ELISA and t-ELISA methods, and the p-ELISA was found to be unreliable for this application (T17). Static secondary ion mass spectrometry for the direct surface examination of soil, leaves, grass, and stainless steel was found to be an effective screening method for 16 of 20 pesticides examined (T18). Polar pesticides were the most easily detected. Amperometric biosensors based on acetyl- or butyrylcholinesterase were used for the kinetic determination of organophosphate and carbamate pesticides (T19), and the ¹³C-labeled fungicide cyprodinil in soil was investigated by using NMR spectroscopy (T20).

GC-, GC/MS-, and LC/MS-based methods for pesticides are still preferred for many applications (T21–T32). Sanchez-Rasero developed a rapid, precise, and sensitive HPLC method for mecoprop and dichlorprop in soil, supplemented by GC/MS with a chiral stationary phase to determine the *R* and *S* enantiomeric forms of these herbicides (T21). The use of LC-MS was reported for polar pesticides (T22) and herbicides (T23) in soil. For soil fumigants, which have high vapor pressures and low boiling points, static headspace GC was found to be an effective analysis technique (T24). GC-based methods that were reported included the use of nitrogen–phosphorus detection of herbicides (T25, T26, T30), the determination of pendimethalin and chlor/bromfenvinphos by flame ionization or ECD detection (T27), GC/MS determination of dimethylhydrazine after derivatization (T28, T29), GC-ECD determination of aged residues of diflufenican (T31), and a simple GC method for tolclofos-methyl in a variety of soil types (T32).

(c) Organochlorines. A number of papers reported on various extraction techniques for organochlorines in soils and sediments. Trapping efficiencies for selected polychlorinated benzenes, polybrominated biphenyls, and polybrominated diphenyl ethers were determined (T33). SFE extraction produced recoveries comparable to Soxhlet PCBs in standard reference materials (T34). A rapid method for PCBs in soils and sediments was developed by combining static subcritical water extractions with solid-phase microextraction (T35). Accelerated solvent extraction gave essentially equivalent recoveries of chlorinated dibenzo-*p*-dioxins and dibenzofurans from solid sample types, compared to Soxhlet extraction, but in less time and by using much less solvent (T36). Microwave-assisted extraction (T37), headspace solid-phase microextraction (T38), and solid-phase microextraction (T39) were all effective means of recovering organochlorine and other organic compounds from solid sample types. Sample preparation can be greatly reduced for some applications by the application of immunochemical test kits. Pullen showed that results of a PCB immunochemical test kit for soil analysis corresponded well with those obtained from GC-ECD determinations (T40).

(d) Polycyclic Aromatic Hydrocarbons. A number of studies on the extraction of PAHs from soils and sediments were reported (T41–T49). SFE was investigated by several researchers (T41–T45). In general, PAH extraction efficiency increased with reduced

polarity of the SFE modifier used and at higher concentrations of cosolvent (T41). By using BF₃ in methanol as modifier, PAH recoveries from a soil containing high humic acids content were significantly improved (T42). One study validated an SFE extraction and analysis procedure from filtered suspended solids, including an initial sample drying step, and obtained overall accuracy and precision of 91 and 8% RSD, respectively (T43). SFE was combined on-line with cold-trap GC/MS to enable multistep extractions based on the addition of a CO₂ modifier directly into the extraction cell (T44). SFE using CO₂ modified with 10% methylene chloride was shown to be more efficient than Soxhlet extraction for all PAHs (T45). Other extraction methods evaluated include coupled subcritical water with solid-phase microextraction (T46), focused microwave-assisted extraction (T47), extraction with *N*-methyl-2-pyrrolidinone in a microwave oven (T48), and ultrasonic extraction combined with solid-phase extraction cleanup (T49).

Methodologies for compound-specific analysis of 63 parent, alkylated, and heterocyclic PAHs using GC/MS have been reported by Means (T50). He found that analysis of sediments for parent PAH alone vastly underestimated the total PAH concentrations. Novel methods for the determination of PAH in solid sample types include on-line polarization synchronous fluorescence with HPLC (T51) and an optical fiber fluorescence method for in situ detection of PAH in porous media (T52). By employing GC/MS for structural information, isotope ratio MS for ¹³C/¹²C ratio information, and accelerator mass spectrometry for ¹⁴C age determination, Lichtfouse demonstrated that soil PAHs were mainly of pyrolytic origin (T53).

(e) Petroleum Hydrocarbons. The determination of petroleum hydrocarbons, and specifically attempts to determine the sources of contamination, continues to be an important area of investigation. Various extraction methods were applied to recover hydrocarbons from soils and sediments including microwave-assisted extraction (T54) and supercritical fluid extraction coupled with on-line infrared spectroscopy detection (T55, T56). The on-line SFE-IR method produced results that were comparable to those obtained by using a standard Soxhlet extraction-IR method (T55).

The determination of petroleum hydrocarbons in soil can be complicated by the presence of natural organic matter. White inferred the presence of biogenic interferences in Alaska soil samples by using pyrolysis GC/MS and looking for a suite of biogenic indicator compounds (T57). Conrad described a method to monitor bioremediation of petroleum hydrocarbons in situ by employing stable C isotope ratio measurements of microbial metabolic end products (T58). A practical method to distinguish between liquid- and dissolved-phase hydrocarbons and to assess the extent of nonaqueous-phase liquids in gasoline-polluted soils was described by Peuron (T59). The method is based on the ratio of soil pollutant concentrations to the soil pore water capacity to retain dissolved-phase pollutants. To study the origin and evolution of an oil spill, high-resolution GC/MS and GC/MS/MS metastable reaction monitoring were used (T60). Interim progress was reported of a study to use multivariate analysis of mid-IR FT-IR spectra of hydrocarbon-contaminated wet soil to develop an analytical tool suitable for real-time in situ underground measurements where a suitable reference spectrum is not available (T61).

Simultaneous quantitation of moisture content and identification of soil composition may be achieved by using this approach.

(f) Other Organics. Novel analytical applications were reported for a wide range of organic compound types in soils and sediments, including phenolics (T62–T65), surfactants (T66, T67, T70), nitroaromatics (T68), pyrethroid mothproofing agents (T69), phthalates and carboxylic acids (T70), and polyaromatic quinones (T71). Priority phenolic compounds can be recovered effectively by employing microwave-assisted extraction except for nitrophenols, which suffer degradation (T62). By using microwave extraction methods, it was reported that phenol and methylphenol isomers could be determined directly by GC/MS without any preliminary cleanup or concentration steps (T63). Phenolic priority compounds were separated by using capillary electrophoresis with theoretical plate numbers of >100 000 (T65).

BIOLOGICAL SAMPLE ANALYSIS APPLICATIONS

Overall, the number of environmental analysis references published over the past two years has increased substantially over the number of references found when the previous review in this series was prepared (A1). However, the number of references concerned with the determination of metals and organics in biological environmental samples has decreased. It is not yet clear whether this is a real trend or not.

(a) Mercury and Organomercury. How to optimize sample preparation for total Hg in biota was investigated by Saraci (U1). Aqueous-phase derivatization of methylmercury followed by solid-phase microextraction and GC-atomic fluorescence detection has been applied to fish analysis (U2). Ethylation of fish extracts by sodium tetraethylborate was a central step in the GC speciation of methylmercury (U3, U4). In one study, absolute detection limits in the low-picogram range were obtained (U3). A method for the fully automated determination of both organomercury and inorganic mercury species in fish has been reported (U5). This method is based on solid-phase extraction, flow injection, HPLC separation, reduction combined with thermolysis, and detection by cold vapor AAS. Fast Hg determination in biological samples was accomplished by ICPMS using minitube furnace catalytic combustion (U6).

(b) Other Metals and Organometallics. A study on various grinding methods for plant material showed some differences in results, although the authors were not sure the differences were due to grinding or sample contamination (U7). A simple procedure for metals in biological and botanical materials was based on an extraction with a water-soluble, tertiary amine solution (U8). Preashing of biological materials was used as a concentration technique to achieve low detection limits for selenium (U9). Lucker determined the distribution of Pb, Cd, and Hg in avian kidneys by direct solid sampling electrothermal AAS (U10). A method for Se in fish was reported, based on a slurry technique without sample pretreatment (U11). The determination of six arsenic species in marine organisms was accomplished by HPLC using ICP-optical emission spectrometry and hydride generation-quartz furnace AAS (U12). Other methods reported for metals in biota include the determination of As and Bi by total reflection X-ray fluorescence after separation and collection of their hydrides (U13), determination of Co, Ni, and Cu in plants using ultrasonic slurry sampling electrothermal AAS (U14), and determination of

Cu, Mn, and Ni in biological samples by flow injection on-line sorption preconcentration in a knotted reactor coupled with electrothermal AAS (U15).

(c) Organics. For the extraction of both PCBs and total lipids from fish, the Bligh and Dyer method recovered a greater amount of total lipid, but a lesser quantity of total PCBs than Soxhlet extraction (U16). Organochlorine pesticides and PCBs were determined by simultaneous extraction and cleanup followed by GC-ECD and GC/MS confirmation (U17). The method was based on supercritical fluid extraction with Florisil sorbent in the extraction cell. An automated HPLC method for the fractionation of PCBs and chlorinated dibenzo-*p*-dioxins/dibenzofurans in fish tissue was based on use of a porous graphitic carbon column (U18). Chiral organochlorines were separated by using modified cyclodextrin phases (U19). Congener-specific methods were also reported for toxaphene in Antarctic seals (U20) and fish (U21). Other reported methods for organics in biota included spectrofluorometry estimation of petroleum hydrocarbon levels in benthic organisms (U22), heterocyclic aromatic amines in fish (U23), bacteriophanepolyols in bacterial extracts (U24), and synthetic pyrethroids in fish (U25).

RADIONUCLIDES

(a) General Papers and Radon. The importance of separation chemistry for the determination of radionuclides in the environment has been reviewed (V1). Hilton found that concentrations of several major ions in pore waters, extracted by centrifugation, were lower than those obtained by employing similar procedures under a nitrogen atmosphere (V2). This can affect estimates of radionuclide distribution coefficients in sediments. A new bone ash SRM has been developed for environmental radioactivity measurements (V3). Bujdoso has presented an extensive bibliography concerning Ra in the environment (V4). Ra was determined in soil by using a new arrangement of two sheets of parallel track-etch detectors (V5) and in soil gas by a combination of thermoluminescent and solid-state nuclear track detectors (V6). A rapid and precise method for Ra adsorbed on sediments is based on measuring the daughter ^{220}Ra in a closed-loop counting system (V7). Bode has demonstrated that the $^{226}\text{Ra}/^{226}\text{Ba}$ ratio can be used as an indicator of phosphogypsum contributions to ^{226}Ra in sediments (V8). Direct measurements could not be used because of particle size effects.

(b) Strontium and Plutonium. The radiochemical analysis of environmental samples for Sr radionuclides and transuranium elements has been reviewed (V9). Satisfactory precision and accuracy for Sr in soils were obtained by using microwave-assisted digestion (V10). ^{90}Sr , Pu, and ^{241}Am were all determined in soils and sediments after application of a sequential separation procedure (V11). After isolating Sr from sediment samples previously dated by using the standard ^{210}Pb dating method, a possible Sr transport pathway through the outflowing waters of a lake could be deduced (V12). In a study of the $^{87}\text{Sr}/^{86}\text{Sr}$ composition in marine sediments, excellent correlation was observed between results of ICPMS and thermal ionization MS analyses (V13). A fast nonradiometric technique for untrace analysis of ^{89}Sr and ^{90}Sr in environmental samples combined mass separation with collinear laser resonance ionization spectrometry (V14). Tinker reported a method for the determination of ^{90}Sr at low activities

in the presence of Th by separating and measuring the Ra progeny ^{90}Y (V15). ^{90}Sr and ^{147}Pm determination in Chernobyl hot particles was accomplished using a β absorption method (V16). Other methods reported for Pu in environmental samples included the use of liquid scintillation counting (V17), resonance ionization MS (V18), isotope ratios by using ICPMS and α -spectrometry (V19), and luminescence detection (V20). A simple method for simultaneous determination of Pu and Am in lichen and moss samples was described by Jia (V21). After HCl leaching, he isolated Pu by using a Microthene-TNOA column, and Am by using a KL-HDEHP column followed by purification by a PMBP-TOPO extraction (V21).

(c) Uranium and Thorium. The application of UV-visible and time-resolved laser-induced fluorescence spectroscopies to the direct speciation of U(VI) in environmental samples was reviewed by Meinrath (V22). Good results for the extraction of U from aqueous solution were obtained by using polyurethane foam imbedded with phosphonic acid (V23). A new sample preparation method was reported that allowed the determination of isotopes of U, Th, and Ra in soil samples without employing a tracer for determining chemical yields (V24). Methods for U and/or U/Th were also reported using thermal ionization MS (V25), neutron activation analysis (V26), neutron-induced fission activation analysis (V27), and double γ -irradiation (V28).

(d) Miscellaneous Radiochemical Applications. The determination of thallium by electroanalytical techniques was reviewed by Esteban (V29). New Th methods were based on laser-induced fluorescence (V30) and AAS (V31). Technetium was determined in the environment by using ICPMS (V32) and by employing a Tc-selective extraction chromatographic resin (V33). Chesnokov measured a ^{137}Cs deposit in soil under a clean protected layer by using a collimated detector technique (V34). The method permits predictions to be made of the work needed for land decontamination. α -Activity of soil samples can be measured using liquid scintillation counting combined with time interval analysis and pulse shape discrimination (V35) and by the use of dielectric track detectors (V36). An in situ γ -spectroscopy method for radionuclides in desert soil was found to give results comparable to laboratory measurements (V37). Neutron activation analysis was found to be a superior technique for the quantitation of background radionuclide levels in soils, compared to acid digestion-based spectrometry methods (V38).

MISCELLANEOUS APPLICATIONS

Endocrine Disruptors. Endocrine-disrupting chemicals encompass a large range of organic, metallic, and organometallic substances. Many of the citations in this review are for the determination of chemicals that may be classified as endocrine disruptors. However, significant regulatory attention is starting to be directed to this very broad class of chemicals, and environmental analysis methods are being developed for this application. A handbook of property data for endocrine disruptor chemicals has been published (W1). Challenges to the study of such a diverse group of chemicals were discussed by Ashby (W2), and public health implications of environmental exposures of these chemicals were presented by Derosa (W3). Novel monitoring strategies for these chemicals have also been reviewed (W4). A study of U.K. river systems showed that high levels of estrogenic

potential exist in the effluent and upstream water from any sewage treatment plant (W5). A list of 56 validated reference standards was proposed (unlabeled and isotope labeled) to facilitate the development of reliable measurement systems for the endocrine disruptor class of compounds (W6). A biomarker approach for endocrine disruptors in the aquatic environment was studied by Bjerregaard (W7). In vitro assays represent one approach to endocrine disruptor testing (W8–W10). Assessing human exposure was discussed by Rivas (W11), and markers for the detection of estrogenic effects in fish were discussed by Hylland (W12). A GC/MS method for the determination of trace endocrine active phenolic compounds was also presented (W13).

Quality Control and Reference Materials. Several reviews on reference materials (RMs) have been published (X1–X7). A framework for the role of RMs in the future was presented by Raspberry (X1). The International Atomic Agency in cooperation with the United Nations Environment Program has published a free compendium on internationally available RMs (X2). Many modern techniques use small solid samples, which necessitates the development of a new genre of highly homogeneous natural matrix materials (X3). The use of natural matrix RMs is increasing at a faster rate than existing producers can meet (X4). Other reviews discussed metal speciation and CRMs used in support of European Union legislation (X5), the use of neutron-capture prompt γ -ray activation analysis for RM certification (X6), and the importance of CRM programs for the European Union (X7).

Quality system requirements for the production of reference materials are described in ISO Guide 34 (X8). Some problems related to the use of RMs by environmental laboratories and their linking to accreditation were discussed by Quevauviller (X9). Various issues regarding RMs were discussed by others, including a synopsis of different approaches to the certification of RMs (X10), homogeneity testing of RMs (X11), and the determination of the expected shelf life of CRMs (X12). RMs for long-term environmental programs were described, in context of the practices followed at the German Environmental Specimen Bank (X13). In many cases, CRMs can be used for noncertified analytes by pooling results for these other species as reported by several laboratories, to develop “consensus value” certifications (X14). The development and certification of many new RMs has been reported, including the following: trace organics in frozen mussel tissue (X15), As and Hg in tuna fish (X16), trace elements in sewage-amended soils (X17), acid-extractable metals in sludge (X18), heavy metals in single-cell green algae (X19), trace elements in Virginia tobacco leaves (X20), multielements in coal ash (X21), and solid calibration standards for trace elemental analysis of tree rings (X22).

Other general quality control-related publications included a review of some of the main aspects of quality control for environmental analysis (X23). Critical evaluation of analytical methods based on their figures of merit has been discussed (X24, X25). If enough measurement data are available and the distribution of these data can be established, then look-up tables can be used to estimate nondetects using statistically based procedures, without introducing bias in a calculated mean (X26). A computer program has been developed for ranking analytical methods according to their fitness for purpose (X27). A number of papers were published concerning measurement uncertainty (X28–X32). Ellison reviewed the interpretation of the ISO *Guide to the*

Expression of Uncertainty in Measurement (X28), and Horwitz presented views on uncertainty with regard to its estimation, error structure, and practical statistical problems (X29). Quantitation of uncertainty in qualitative analysis has also been discussed and the reporting of “identification uncertainty” figures described (X30). A graphical approach to identifying and structuring input effects on a measurement result has been presented (X31). The use of arithmetic rather than geometric means for concentration data is recommended, with some exceptions (X32).

Toxicity, Biomonitoring, and Bioindicators. The increasing importance of biomonitoring and related topics can be seen by the large number of reviews in this area (Y1–Y17). In Herkimer's review are discussed regulations, models, methods, inorganics and organics, NH₃, water and wastewater, test organisms, and soil and sediments (Y1). Emerging technologies for environmental biomonitoring were reviewed by Beattie (Y2). Biomarker strategies to evaluate the environmental effects of chemicals present an alternative approach to destructive animal testing (X3). Contemporary biogenic, fossil, and anthropogenic markers, which provide information about sources of organic pollutants in soil, have been discussed (X4). Recent advances related to environmental biomonitoring and specimen banking have been presented (X5). Burlage reviewed the use of bioreporters and biosensors for environmental analysis (X6), and Albertini discussed the use and interpretation of biomarkers of environmental genotoxicity in humans (X7). Reviews of specific organisms for toxicity and biomonitoring included the toxicity analysis of meio- and macrobenthic organisms (Y8), sponge cells and tissue as monitors of aquatic pollution (Y9), vascular plants as field biomonitoring (Y10), and biomarkers of marine pollution by mutagen content and metabolic activation of promutagens by molluscs (Y11). The use of caged bivalves in aquatic monitoring programs was reviewed by Salazar (Y30). Incorporation of appropriate surrogate species and biomarkers of chronic toxicity into standard toxicity characterizations was proposed as a way to significantly refine the ecological hazard assessment process (Y12). Specific groups of chemicals reviewed with respect to toxicity and biomonitoring include PAHs in soil (Y13), organotin compounds in marine molluscs (Y14), and the sources and environmental fate of the polychlorinated diphenyl ethers (Y15). Other reviews covered electrochemical biosensors for monitoring environmental pollutants (Y16) and biosensors based on bilayer lipid membranes for continuous monitoring or rapid screening of environmental pollutants (Y17).

Specific biomonitoring have been proposed for environmental effects' monitoring. Moss and lichen (Y18) and bee pollen (Y19) were found to be useful biomonitoring of heavy metal pollution. Tissue-level biomarkers were found to be cost-effective tools to assess metal pollution in soils (Y20). Mutagenic activation of arylamines by mollusk S9 could be a reliable biomarker for marine pollution (Y21). The use of algal assays to detect toxic organometallics in wastewater was studied by Wong (Y22). Duckweed bioassays were found to be useful in detecting heavy metal availability in elutriate and leachate samples from smelter soils (Y23), while a new on-line biomonitoring was studied for use in monitoring downstream of a copper effluent (Y24). Tree rings were not found to provide a reliable record of the pollution history of an area, because single trees reflected individual uptake of heavy metals rather than the history of the region (Y25).

Table 14. Technology Cross-Reference

technology/method	citations in review with reference to technology/method
sampling	A6, B1, B2, A63, F1–F12, F92, F96, H13, J2, J4, K1, K2–K16, L1–L4, L12, R1–R3, R4, R5–R12
SFE	A18, S37, T7–T10, T41–T45, T55, T56, U17, Z5
SPE	A19, L28, L29, M12, M15, M16, M22, M23, M28–M33, M58, M66, M87, N19, N41, N47, N51, O9, O12, O13, P19, S5, S6, T12, T34, T49, U5
SPME	A9–A11, A20–A22, C15, L31, M19, M20, O4, O5, P11, P14–P16, Q14, S26, T3, T13, T35, T39, T46, U2
microwave-assisted extraction	R27, R29–R31, R32, S3, S23, S25, S27, S39, T14–T16, T37, T47, T48, T54, T62, T63, V10, Z4
LC, HPLC	M5, N23, O15, S33, S48, T51, U5, U12, U18
IR spectroscopy, FT-IR	E8–E16, T55, T56, T61
AAS	A54, C14, F67–F69, L13, M89, M99, M105, M106, O20, O22, O24, O28, S10–S14, S24, S31, U5, U10, U12, U14, U15, V31, Z2
ICP, ICPMS	A29, A31, A53, C7, D26, F70–F72, I17, I18, M73, M74, N19, N23, N2–N8, O28, O29, P5, P6, S15, S28, S42, U6, V13, V19, V32
MS methods	D11, D12, E1, E2, F45–F56, F73, H27, L7, L24, L26, L30, L34, L36, M4, M6, M10, M18, M25–M27, M63, M66, M67, N51, N53, O6, O8, O10–O13, O16, O19, O23, P7–P10, P16, P17, P19, P24, P25, S26, S29, S30, T18, T21–T32, T50, T53, T57, T60, V25, Z6
immunochemical methods	L25, L38, M13, M34, N41, N47, O18, O19, T17, T40
NAA, XRF, PIXE	A15, A32, A33, A37–A39, C10–C13, F25, F26, F76–F79, H22, I5, J9, M129, N10, N11, S14, S32, U13, V26, V38

Table 15. Analyte Cross-Reference

analyte	citations in review with reference to analyte
Hg, organomercury	A59, B12, D25, F9, F23, F64–F66, K12, K16, M99–M102, O25–O27, Q5, S1–S16, U1–U6, U10, X16
Pb, organolead	B25, E5, E20, F11, F24, F70, F78, F81, F82, F97, F98, H3, H8–H11, H16, H17, H21, L15, M56, M84, M105–M109, N3, N15, N30, S49, U10
As, organoarsenic	D7, F69–F72, F77, F79, F98, H17, H21, J8, L11, M73–M78, O21, S23–S30, U12, U13, X16
chlorinated organic compounds (dioxins, PCBs, toxaphene, other chlorinated organic compounds)	A46, A68, B7, B24, C16, D8, D13, E3, F7, F20, F43, F44, F59, F90, H2, H15, H28, I7, M58–M60, N50, P18, T33–T40, U16–U18, U20, U21
PAHs	B5, B6, D2, D12, E3, E4, E17, F5, F6, F19, F50–F57, F89, F91, H13, H14, I2, L28, N49, T41–T53, Z9
VOCs	B14, B15, B17, B20–B22, B26, C6, D4, D9, D10, D15–D24, E1, E2, E21–E23, F1–F4, F14–F18, F28–F42, F83–F88, H20, L30, M68–M70, N45, O4, P13, P17, P24, T1–T6
pesticides	B18, C16, E24, E25, F21, F22, F59–F63, F94–F96, H4, H5, H24, H25, K15, L18–L26, M1–M34, N41, P15, P19, P21, Q15, T7–T32
radionuclides	D6, L1, L8–L10, L17, M110, M116, M117, M119, N28, O32, O33, Q11, V1, V2, V3, V4, V5–V8, V9, V10–V21, V22, V23–V28, V29, V30–V38
organotin compounds	A60–A62, J7, M57, N42, N43, O3, P7, S35–S43

Approaches to toxicity testing of chemicals included use of a bacterial indicator for rapid, inexpensive assays (Y26) and a microalgal solid-phase test to assess the toxic potential of freshwater sediments (Y27). The selection of an optimum toxicity assay test battery for screening environmental samples was described (Y28). Wall developed a continuous-flow renewal system for sediment toxicity testing (Y29).

Miscellaneous Applications. Considerable attention recently has been given to the determination of various pollutants in sewage sludges (Z1–Z6). Rapid strategies for the rapid determination of metals in sewage sludge were described by de la Guardia (Z1). In the determination of Se in sewage sludge by electrothermal AAS, the use of microwave-assisted extraction to shorten digestion times helped to minimize the potential of Fe interference (Z2). The analytical assessment of two sequential extraction schemes for metals in sewage sludge has been reported (Z3). In a study to optimize digestion methods for metals in sewage sludge, no significant difference was found in the three methods evaluated, which included microwave oven digestion (Z4). Organic methods reported included the determination of nonylphenol polyethoxylates and their carboxylic acid metabolites in sewage sludge by supercritical fluid extraction (Z5) and the application of TLC and GC/MS to the identification of amino-PAH in sewage sludge (Z6).

Carpenter reviewed methods for the extraction and detection of tracer organosilicon materials in environmental samples (Z7). The review focused primarily on the poly(dimethylsiloxane)s, which are the largest contributors to organosilicon materials in wastewater. Semipermeable membranes can be used as passive samplers to determine organochlorine pollutants in compost (Z8). Although the membranes are simple and efficient sampling devices, they may be best suited to applications that need only semiquantitative results. O'Malley described a unique way of

tracing PAHs in the environment, by using $^{13}\text{C}/^{12}\text{C}$ ratios (Z9). Other applications included the determination of organic components in leachates from waste disposal sites by GC/MS (Z10) and the use of accelerated solvent extraction for various toxic organics in soil waste samples (Z11, Z12).

TECHNOLOGY AND ANALYTE CROSS-REFERENCE

Tables 14 and 15 list key technologies and analytes, respectively, and the associated citations in this review where these were central to the reported study. These tables are included to allow readers to quickly access information concerning these technologies and analytes, regardless of the environmental application. Some topics such as gas chromatography and environmental endocrine disruptors were not included in the tables, because there are so many applications in this review that would have to be included. Citations that appear in *italic* font are review articles.

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