Field Analytical Chemistry

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Field analytical chemistry (FAC) is a rapidly growing area of chemical analysis in which the analytical measurement is completely accomplished at the site where the analyte is located. Traditionally, analysis is accomplished by collecting a sample from a remote site, transporting the sample to an analytical laboratory, which can and often is located several days of travel from the sample collection site, and finally storing the sample until the laboratory has completed the analysis of sample previously received.

The first and foremost problem associated with traditional sample collection, transportation, storage, and analysis is that it takes time. Days and often weeks are required before the information on the identity and quantity of chemicals in the sample is known. This delay in data availability delays decisions about the sampling site. Some decisions, such as those concerning human safety and product reliability, are desired in real time in order to prevent problems.

In addition to saving time, FAC can reduce the cost of analyses. Other real-time decisions, such as those concerning environmental remediation or site characterization, can significantly reduce the cost of an operation. Immediate analytical information on the level of success of a cleanup project can eliminate the need to return to a site to complete the cleanup after analytical data are finally returned from a traditional off-site laboratory. For site characterization, the site can be mapped with respect to analyte concentration in real time, decreasing the number of samples required to completely and thoroughly characterize a site. In general, if the costs associated with transportation and storage of samples are eliminated from the analytical protocol, the overall price per sample can be reduced.

Currently, laboratory-based instrumentation provides more precise and accurate analytical data than most field methods. Attracted by the promise of reduced cost and potential of real-time decisions, development of field analytical chemical methods has significantly increased over the past few years. This article reviews those developments made in the past two years. It is divided into eight sections: Chromatography, Mass Spectrometry, Ion Mobility Spectrometry, Chemical Sensors, Imunochemical Techniques, Optical Spectrometry, Radionuclear Methods, and Remote Sensing.

CHROMATOGRAPHY

High-Speed Gas Chromatography. The need for generation of analytical data in real time is pushing the field of analytical instrumentation toward portable, faster, and more cost-effective instrumentation for compound-specific analyses directly in the field at the point of sample collection (A1). To achieve portability, use of on-column resistive heating was reported (A2). This allows very rapid temperature programming and, thus, shorter analysis times. Phillips and Ledford (A3) reported on a thermal modulator that controls the temperature of a short length of the gas chromatographic column as a function of time and position. Thus, by simple rotation of the heater element over the column, analytes can be focused into sharp pulses and accelerated by the modulator second stage. Two chromatographic columns were used with the thermal modulator, which itself was a 0.3 m \times 100 μ m i.d. column coated with SE-30. One column was between the injection port and the modulator and the other column was between the modulator and detector.

Different inlet systems for fast gas chromatography have been reported (A4-A6). Li and Andrews (A4) investigated 10 different metal tubings that were used as trap tubes in a cryotrap/thermal desorption inlet, and Borgerding and Wilkerson (A5, A6) reported on two different cryofocusing injectors for fast GC. One is a ballistically heated metal injector; the other is a microloop injector that uses a PLOT column to trap volatile compounds such as ethane.

Microfabricated analytical instrumentation that relies on dedicated microprocessor control of all timing and temperature control functions was described by Overton et al. (*A1, A7, A8*). The small size of the instrument makes it very portable and the dual gas chromatographic column capability is a plus; however, the speed and selectivity advantages come at the cost of high detection limits (*A8*).

A crucial aspect of micromachined components that must be considered at the outset of any application is packaging (A9) because microsensors are particularly sensitive to stresses introduced during packaging.

Only a few applications using high-speed gas chromatography were reported during 1995-1996. Yun and Lee (A10) reported on the separation of light hydrocarbons and permanent gases using silica PLOT and carboxen PLOT columns, and Sacks et al. (A11) reported on the high-speed separation of gasoline to diesel range organic compounds. The gas chromatograph used by Sacks et al. was designed for use with a cone penetrometer system and employed a cryofocusing inlet to collect a soil gas sample.

Dagan and Amirav (A12) reported on the fast, very fast, and ultra-fast gas chromatography-mass spectrometry of the thermally labile carbamate pesticides.

Portable Gas Chromatography. Development of a fieldportable tandem GC (GC/GC) was reported by Arnold at FemtoScan Corp. and Meuzelaar and co-workers at the Center for MicroAnalysis Research Chemistry of the University of Utah (A13). The system uses two short columns; the GC peaks eluting from the first column are sampled repetitively at 1-5-s intervals into the second column. In combination with a suitable detector, this technology has potential for low power consumption and low weight and is relatively inexpensive.

Studies using hand-held instrumentation such as the Photovac Snapshot GC were reported by David and Pauls (A14), and Holland et al. (A15) reported on a low thermal mass miniaturized GC equipped with a miniature trap preconcentrator and miniature dual flame photometric detectors. The GC instrument developed by Holland et al. was designed specifically for Chemical Weapons Convention Treaty Verification Inspections; the system stores all data collected on PCMCIA memory cards that can be easily removed in the field and data transferred to a PC for archiving and further examination.

Recent developments in the detection of chemical warfare agents and of other volatile organic compounds in air were presented by Sides (A16). Among the detectors presented were a pulsed-flame photometric detector and a flameless halogenselective detector (does not require hydrogen). Identification of compounds relevant to the Chemical Weapons Convention using GC with element-selective detectors and GC/FT-IR was described by Soderstrom et al. (A17). Other detectors reported for portable GC include an argon ionization detector (A18) and a surface acoustic wave detector (SAW) (A19). More information on the SAW device will be given in the Chemical Sensors section. Other gas chromatographic detectors include the pulsed discharged detector in the electron capture and photoionization mode (A20), and the surface ionization detector (A21). The latter detector can be operated as a universal detector with an additional selectivity toward some species that have low ionization energy. New ionization sources for photoionization detectors were reported by Budovich et al. (A22).

MASS SPECTROMETRY

Developments and major applications of on-site/in situ environmental analysis using mass spectrometry as well as the performance characteristics have been reviewed by Kotiaho (B1). Sample introduction methods that have received considerable interest in the past two years are membrane introduction mass spectrometry (MIMS) and solid-phase microextraction (SPME). In MIMS, the analyte molecules are transported across a semipermeable membrane into the ion source of the mass spectrometer. A review with 28 references on the use of MIMS, including the membrane characteristics and instrumental configurations was published (B2). Various membranes made out of nonporous polymers such as silicones and less selective microporous Teflon membranes have been reported (B3). Virkki et al. (B4) reported that many environmentally significant compounds can be measured directly in water samples at sub-parts-per-billion levels in 1-2 min and that MIMS allows linear dynamic ranges of at least 4 orders of magnitude.

Direct determination of toluene and trans-1,2-dichloroethene in water at parts-per-quadrillion levels using MIMS and ion trap mass spectrometry was reported by Cooks and co-workers at Purdue University (B5). Use of MIMS in the determination of trihalomethanes and cyanogen chloride at parts-per-billion levels in treated drinking water was reported by Bauer et al. (B6); these authors injected water samples directly into the MIMS system without any sample preconcentration. The use of a two-stage hollow fiber membrane interface for ion trap mass spectrometry was reported by Hemberger and co-workers (B7); the instrument response was independent of the air flow rate from 0.2 to 3 L/min, and the response times were in the 60-90-s range.

Modification of a Hewlett-Packard 5971/5972 mass-selective detector for direct insertion of a capillary membrane probe was reported by Lauritsen and Gylling (B8), and affinity MIMS, which allows selective trapping of benzaldehydes by a chemically modified membrane, was described by Cooks and co-workers

SPME is a direct, solventless extraction technique, in which the analytes are adsorbed directly from an aqueous or gaseous matrix onto a fused-silica fiber to which a stationary phase has either been bonded or been applied as a coating. The adsorbed analytes are subsequently thermally desorbed into the injection port of a gas chromatograph/mass spectrometer (B10-B15). Analytes can be detected at parts-per-trillion to sub-parts-per-billion levels using ion trap mass spectrometry, and the precision of this technique was determined to be 1.5-6% relative standard deviation (B10).

Separation of as many as 28 compounds detected by SPME and ion trap mass spectrometry in less than 150 s was reported using internally heated fibers (B12). Heating was achieved by passing an electric current directly through a wire in place of the fiber or by using a hollow fiber equipped with an internal microheater (B12).

Fibers coated with poly(dimethylsiloxane) of 15-um film thickness were found to work well for chlorinated solvents (B12), whereas polyacrylate worked better for heteroaromatic compounds such as pyrroles, substituted pyridines, quinolines, benzofurans, dibenzothiophenes, carbazole, acridine (B13), and nitrogen-containing herbicides (e.g., triazines, nitroanilines, thiocarbamates) (B15).

Soil samples can also be analyzed by SPME/GC/MS, but the target analytes have to be preconcentrated first by a headspace technique. To improve the sensitivity of the technique, the headspace volume above the soil layer should be kept as low as possible and the soil volume should be as large as possible (*B14*).

Portable Instrumentation. The trend in the development of portable GC/MS systems is toward lighter, more compact, modular instruments, with modest power consumption and low maintenance requirements. Baykut (B16, B17) reviewed the capabilities of the Bruker-Franzen portable GC/MS system. The samplers and air inlets on this instrument allow analysis of volatile organics in water samples by spray-and-trap, purge-and-trap, or direct injection methods. A polysiloxane inlet membrane seals the vacuum chamber while the various GC modules are being exchanged. Other GC/MS systems that have been modified to make them suitable for field use are the Viking SpectraTrak GC/MS system (*B18*) and the Fisions MD800 system (*B4*).

A newly redesigned SpectraTrak system was described in refs B19 and B20; this system weighs \sim 20 kg without the laptop computer and is contained in a 33-L box; this represents a reduction in size of \sim 50% and a reduction in weight of \sim 25% over the first-generation system. Its mass range is 1.6–700 amu with a unit resolution at 700 amu and it can detect picogram levels of hexachlorobenzene when scanned from 50 to 300 amu (at a signal-to-noise ratio of 10).

Inficon, Inc. (B21) recently patented a portable GC/MS system, which is battery-powered, lightweight, and weatherproof. A sorption pump, connected to the ion chamber, is capable of maintaining the ion chamber at high vacuum (10^{-6} Torr) (B21). The pump consists of a diffusion barrier that allows smooth pulseless pumping.

A portable quadrupole GC/MS system designed to be contained in a standard size suitcase, which weighs \sim 35 kg and requires \sim 600 W of electric power, was recently patented (B22).

A simple, compact, and inexpensive mass spectrometer for plasma discharge ion analysis was reported by Tuszewski (B23). The mass separation is achieved by combining a variable extraction voltage with a fixed magnetic field $(0.28\ T)$. The ions are first accelerated from the plasma potential $(10-20\ V)$ to the floating potential $(0-4\ V)$ and then accelerated or repelled by the varying voltage $(\pm 100\ V)$.

Kogan et al. (B24) described a portable static mass spectrometer with a static magnetic mass analyzer designed specifically for ecological studies. In situ quantification of gas mixtures by a novel technique identified as matrix interval algebra-assisted quadrupole mass spectrometry was reported by Kaiser et al. (B25). This technique has applicability in space missions to identify chemical compounds in planetary atmospheres. Another technique that may have applicability in establishing life on the planet Mars is the secondary ion mass spectrometry (SIMS); SIMS can measure isotopic abundances in mineral or organic phases without any sample processing (B26).

A compact hydrogen/helium isotope mass spectrometer, which combines low mass resolution mass spectrometry and beam-foil interaction technology to achieve complete separation of molecular species of equivalent atomic mass, was described (*B27*).

Combined ultraviolet detection/gas chromatography/mass spectrometry using a heated remote flow cell was reported for analysis of Aroclors and unleaded gasoline (*B28*). Calibration curves for aromatic hydrocarbons indicate linearity of detector response over a range of 100, and single-wavelength detection limits of 90 pg for naphthalene

In situ mass spectrometry for real-time analysis of reactant and product species in single-wafer rapid thermal chemical vapor deposition was reported by Tedder et al. (*B29*) and Thomas and Blanquet (*B30*).

Field Methods and Applications. A performance-based field method for analysis of various substituted phenols using thermal desorption and GC/MS was reported by Jiao and Robbat (*B31*); MS with selective ion monitoring mode and a compound-specific data analysis algorithm provides data with a precision better than 40% and an analysis time of almost 5 min/sample.

Use of portable GC/MS for on-site verification of compliance with the terms of the Chemical Weapons Convention was reported by Slivon et al. (*B32*), who evaluated several ion trap mass spectrometers available commercially. The use of ion trap MS/MS to detect chemical warfare agents in the presence of a large chemical background was investigated by Lammert and Palausky (*B33*), who reported that use of MS/MS allows use of shorter columns and consequently can increase sample thoroughput considerably.

Globetrotting with a modular GC/MS system was reported by Heyl (*B34*); this system is part of a modular laboratory designed to support chemical weapons inspections and compliance to such inspections. A roving GC/MS system consisting of a Hewlett-Packard mass-selective detector and a FemptoScan Enviroprobe repetitive vapor sampling inlet with a short capillary column and a notebook computer was used to measure aromatic solvents along a 6-km urban route at 15-s intervals while moving at an average speed of 35 km/h (*B35*).

A brief review of the development of mobile GC/MS procedures and applications at the Technical University of Hamburg—Harburg over the last decade was reported by Matz and Schröder (*B36*).

Use of a field GC/MS procedure developed by Sandia National Laboratory to characterize sites contaminated with volatile organic compounds was described by Ardito et al. (*B37*).

On-site analysis of landfill gas and landfill gas-fired boiler exhaust using a Viking Spectra Trak 620 field-mobile GC/MS system was reported by Williamson (*B38*).

The field evaluation of a man-portable GC/MS system consisting of a Hewlett-Packard 5971 mass-selective detector and a unique automated vapor sampling transfer line GC system was reported by Arnold et al. (*B39*).

The Instrumentation Group and the Environmental Monitoring Group from the Oak Ridge National Laboratory reported on the direct sampling ion trap mass spectrometry (DSITMS) technique for field screening and compliance monitoring of volatile organic compounds in groundwater (*B40*). DSITMS is the technique in which sample components are introduced into the mass spectrometer via a short capillary restrictor or a polymeric membrane, resulting in a fast-response quick analysis time and thus high sample throughput (typically, 15 samples can be analyzed per hour). Several validation studies have been conducted in which the DSITMS technique has been compared to the EPA standard method involving purge-and-trap GC/MS.

A field-deployable ion trap mass spectrometer with a direct air sampling interface and an air sampling flow discharge ionization source was reported by Gordon et al. (*B41*). This allows organic compounds in air samples to be ionized externally before they are injected into the ion trap where they are stored and dissociated using the filtered noise fields (FNF) technique.

The same technique was used by McLuckey et al. (*B42*) to detect traces of high explosives. Atmospheric sampling glow discharge allows anion formation while the quadrupole ion trap provides for efficient tandem mass spectrometry (*B42*).

Direct sampling interfaces, a sorbent trap/thermal desorption interface, and a continuous air monitor for direct sampling ion trap mass spectrometry have been described and use of these interfaces for dual gas monitoring is described (*B43*).

An in situ volatile organic compound sampling system to detect parts-per-billion levels of BTEX and TCE using the cone pen-

etrometer system was described by Myers et al. (B44). The CPT probe is pushed to a desired depth into the subsurface, and an interior rod is retracted to form a sampling chamber in which a measured volume of soil is purged in place. The volatile compounds that are purged from the soil matrix are transferred to the surface where they are collected on a Tenax trap and subsequently desorbed and analyzed by GC/MS. Stainless steel tubing heated at 100 °C and a carrier gas flow rate of 40 mL/min resulted in recoveries of > 90% in a 30-min sampling period (*B38*).

ION MOBILITY SPECTROMETRY

Applications of ion mobility spectrometry (IMS) to field analytical chemistry are rapidly increasing due to the development of hand-portable ion mobility instruments. Unlike mass spectrometry, ion mobility spectrometers do not require high-vacuum pumping devices that limit portability. IMS instruments operate at ambient pressure and can use pumped, purified room air for the drift and carrier gas. During the past two years, improvements and advancements in hand-held gas chromatography/ion mobility spectrometry were reported (C1) along with a discussion of engineering approaches and compromises required for the construction of small ion mobility spectrometers (C2). The performance characteristics of a new miniature IMS instrument was compared to those of a standard hand-held ion mobility spectrometer (C3).

One major problem with portable ion mobility spectrometers has been the use of radioactive ionization sources. Both photoionization and corona discharge ionization were investigated as alternative ionization sources for IMS (C4, C5). A second problem is the lack of response specificity. Increased selectivity of response has been reported by several methods. A more selective response can be obtained by using a high-resolution instrument with an IMS resolution up to 200 (C6). Selectivity was also obtained by using selective chemical ionization methods (C7) and by derivatization of the sample before analysis (C8). Discrimination between classes of compounds was reported using multivariate statistical analysis of the ion mobility spectra (C9).

In addition to advancements in application of IMS for monitoring traditional targets such as drugs of abuse (C10-C14), chemical warfare agents (C15, C16), and explosives (C17), a number of nascent applications were reported. The more creative of these were the use of IMS in space exploration (C18-C20), biological warfare agents (C21), and the determination of the freshness of fish (C22). Other promising applications reported in the past two years were the determination of aqueous ammonia (C23, C24), the on-site monitoring of nicotine vapors in air during the manufacture of transdermal systems (C25), and the characterization of SF6 decomposition products caused by discharges in switchgears (C26). Environmental applications were also investigated including the detection of polychlorinated aromatics (C27), organic priority pollutants (C28), and vinyl chloride (C29).

CHEMICAL SENSORS

Portable monitors based on recent developments in chemical sensors (e.g., quartz crystal microbalance, surface acoustic wave devices, electrochemical sensors, and fiber-optic sensors), biosensors, gas sensors, and disposable test-strip sensors have been reviewed by Alexander (D1). Despite impressive advances in the field of chemical sensors and biosensors, most sensors have been designed for specific applications and show practical constraints

(D2, D3). A typical example is a solid state chemical sensor that exhibits a high degree of selectivity in detecting chlorinated solvent vapors at levels of \sim 0.2 ppm in \sim 90 s was reported by Buttner et al. (D3). Current strategies that deal with certain shortcomings of the sensors based on thick-film technology have been summarized by Galan-Vidal et al. (D2). The silicon-sensor field, which is now more than 35 years old, has been reviewed by Middelhoek (D4) and a review on microfabrication and micromachining techniques used in the manufacture of these sensors has been published by Liu (D5).

Quartz Crystal Microbalances (QCMs). QCM devices are finding their way into the world of chemical sensors and new applications of these type of mass sensors became possible with the development of QCMs for use in liquids (D6). A QCM device consists of a piezoelectric crystal (i.e., a small quartz disk with electrodes plated on it). When an electric field is applied across the device, an acoustic wave is induced, whose frequency is a function of the mass of the entire device (crystal and electrodes). Several QCMs that can measure mass changes of up to 100 μ g with a minimum mass change of 1 ng/cm² (D6) are commercially available; these devices are fairly inexpensive but their main disadvantage is lack of sensitivity. Developments in the selection of coatings that react specifically with target analytes are a major technology challenge (D7-D13).

The selection of the particular sensor technology and the appropriate coating must consider the application for which the sensor is intended. A very careful selection of coatings is required since noise level, thermal effects, and the limit of detection of the sensor were shown to depend on the type of polymer and its film thickness (D14). Sensors that combine the sensitivity of the QCM device and the specificity of immunochemical reactions, immunosensors, have been reported by Welsch et al. (D15) and Rapp et al. (D16). To convert the QCM device into an immunosensor, several steps, including precoating with a polyimide layer to prevent corrosion of the aluminum transducers, activation of the polyimide, and covalent precipitation of the antibody, are required

Combinations of sensors including metal oxide, conducting polymer, and surface acoustic wave, packaged with application software and automation features are available commercially and have been described in detail elsewhere (D17-D19). Applications of this emerging technology called electronic nose in the food industry (e.g., aroma detection in the brewery) and environmental (e.g., monitoring of air in buildings, cars, and planes), biotechnology, and medical diagnostics have been reported (D18, D20,

Applications of SAW sensors coated with various polymeric films to analysis of organic vapor mixtures was reported by Zellers and his co-workers at the University of Michigan (D22, D23), Frye et al. (D24, D25), Grate et al. (D26-D28), Ellis et al. (D29), Rapp et al. (D30), and Zhang and Li (D31). Zhang and Zellers (D22) used a 60-MHz SAW oscillator coated with Pt-olefin coordination compound with the formula trans-PtCl₂(olefin) (pyridine) in which the olefin was either 1-hexene or ethylene. This prototype field instrument gave linear response isotherms from 0.05 to 3 and 0.6 to 50 ppm, for 1,3-butadiene and styrene, respectively, based on a 10-s measurement time. Zeller and his co-workers (D23), who have had many significant contributions in this field, reported on a database consisting of calibrated responses of 10 polymer-coated SAW sensors to each of six organic vapors (chloroform, hexane,

benzene, trichloroethylene, isooctane, xylene) and used pattern recognition and Monte Carlo simulation to rank the various possible coatings and to determine the ability of the sensor array to identify the set of analytes. The coating materials employed covered a wide range of polarities and functionalities; among the polymers used were polyisobutylene, polychloroprene, polysiloxane, acrylonitrile/butadiene copolymer, poly(methyl methacrylate), and others (*D23*).

Frye et al. (*D24*, *D25*) described portable SAW devices for on-line monitoring of soil gas and in situ monitoring of volatile organics brought to surface via a cone penetrometer probe. These systems use a thin polymer film (e.g., polyisobutylene) that can reversibly absorb the analytes within seconds. The detection limits of these devices is 1–10 ppm and can be lowered to 10–100 ppb by use of a preconcentrator device.

Selectivity patterns for the sorption of 11 organic vapors by α -cyclodextrin, polyepichlorohydrin, and 75% phenyl 25% methyl polysiloxane were reported by Grate et al. (D26), who found that sorption into cavitand monolayers on SAW is similar to that by amorphous polymers. Dewetting of the polymeric film can lead to isolated droplets of material and to degradation in sensor performance (D27); to prevent this, cleaning and surface preparation of the sensor prior to coating is important.

Grate et al. (D28) also reported on a technique for estimating the responses of polymer-coated SAWs to organic vapors; the authors determined the polymer/vapor (gas) partition coefficients by gas chromatography and used these partition coefficients to construct linear solvation energy relationships that correlate partition coefficients on a given polymer with various solute solvation parameters (D28).

Ellis et al. (*D29*) reported on an electrically conductive polymer, poly(3-hexylthiophene) as a thin-film sensor for detection of parts-per-billion levels of hydrazine and monomethylhydrazine in the presence of ammonia, amines, and ambient water, that has a shelf-life of several years when stored in dry, cold conditions.

The typical SAW device for detection of organic vapors uses two SAW oscillators; one device is coated with a selective polymer and represents the chemical sensor element; the noncoated device serves as the reference element. Rapp et al. (D30) reported on a compact automatic system consisting of nine SAW devices (eight sensor oscillators and one reference oscillator) mounted in a radial geometry with a diameter of 10 cm that uses horizontally polarized surface transverse waves; the device performs a measurement in about 5-10 s, depending on the type of polymer and its film thickness.

The effect of temperature and humidity on the performance of polymer-coated SAW sensor arrays was discussed by Zellers and Han (*D32*), who reported that small changes in temperature and humidity can have a large effect on baseline stability rather than on sensor response to vapors.

Pattern recognition and neural networking techniques were reported to assist in classifying the responses of a sensor arrany (*D21*, *D33*, *D34*).

Electrochemical Sensors. Chemical sensors and the development of potentiometic methods for analysis of liquid media have been reviewed by Vlasov et al. (*D35*).

The electrochemical sensor technology, based on stripping potentiometry at a gold fiber electrode, has been reported by Wang et al. (D36, D37). The gold microelectrode was used as a remote sensor for detection of Cu, Hg, Pb, and Se at 3-4 min

intervals and instrument/sample distances of up to 32 m. The low detection limits achieved with this sensor are attributed to its "built-in" preconcentration step. Furthermore, the sensor eliminates need for mercury electrodes and offers potential for miniaturization to allow adaptation to cone penetrometer technology.

A novel microfabricated electrochemical sensor was reported by Kovacs and co-workers at Stanford University and Kounaves and co-workers at Tufts University (*D38–D40*). The sensor consists of a silicon substrate on which is placed an array of thinfilm iridium microelectrodes; a thin film of mercury is electrodeposited on these electrically interconnected microelectrodes.

Polymers such as poly(4-vinylpyridine) as a coating for the gold film electrode showed increased sensitivity and better mechanical stability of the gold film when used with the square wave voltametric method (D41).

To increase the selectivity of the electrochemical sensor, the polymer membranes have been doped with a palladium organophosphine complex (D42) which may function as a neutral or charged carrier-type ionophone within the polymer membrane phase.

Fouling problems are a main limitation of direct voltametric measurements in industrial wastewaters. To minimize it, a novel antifouling membrane-covered voltametric microsensor was reported by Tercier and Buffle (D43). This agarose membrane-covered, Hg-plated, irridium microelectrode was used to detect Pb and Cd in water in the presence of high concentrations of both humic and fulvic acids.

Electrochemical techniques can also be applied for the determination of neutral compounds in air or water (D44-D46). The challenge of extending stripping voltametry to organic compounds is in the fact that the analyte needs to be electroactive in the sorbed state, and electrochemical activity of organic compounds often requires a catalyst. A polymeric ruthenium oxide film retained its electrocatalytic activity in the presence of a Nafion overlayer and was used in the anodic stripping voltametry of various N-nitroso compounds (D44). A remote electrochemical sensor for monitoring hydrazine compounds using an electropolymerized film of 3,4-dihydroxybenzaldehyde was reported by Wang et al. (D45).

Determination of As(III) in aqueous solutions by flow injection coupled with potentiometric stripping analysis (PSA) was reported by Alstadt et al. (*D47*, *D48*). This technique can be applied to the determination of dichloro(2-chlorovinyl)arsine or Lewisite in air samples at levels ranging from 0.5 to $50 \,\mu\text{g/m}^3$ using a 15 min total cycle time (*D47*).

A portable, battery-operated flow injection system containing an eight-sensor flow cell was developed by Alexander and co-workers (D49); electrode responses to silver and iodide solutions were used to evaluate the performance of the flow cell.

Fiber-Optic Sensors. Fiber-optic technology has grown significantly since the early 1970s when the first demonstrations of fiber-optic gyros and acoustic sensors were performed. The number of fiber-optic sensors is expected to grow as rapid progress is being made in the optoelectronic and communication fields. An overview of fiber-optic sensors was reported by Udd (*D50*). The use of fibers as light pipes to transmit spectroscopic information to a detector is the easiest way of remote sensing. The main advantages of fiber-optic chemical sensors (FOCS) and examples of sensors used in field measurements were described

by Camara et al. (D51). Other examples of FOCS published during 1995-1996 include sensors for the determination of methanol (D52); organic acids (D53); polycyclic aromatic hydrocarbons (D54); petroleum products (D55-D59); trichlorethylene and other chlorinated solvents (D60-D63); benzene, toluene, ethylbenzene, and xylenes (D63); other volatile solvents such as acetone, chloroform, toluene, and cyclohexane (D64); and nitric oxide (D65). A multianalyte imaging fiber-optic sensor that allows simultaneous pH, carbon dioxide, and oxygen sensing was reported by Pantano and Walt (D66). These authors also reported on the fabrication and performance of a biosensor that measures simultaneously penicillin and pH.

Tapered silver halide fibers were used to enhance the sensitivity of chemical sensors for chlorinated hydrocarbons in water (D67); a minimum detectable level of 50 μ g/L was reported for a polyisobutylene-coated fiber with a 1:4 tapering ratio. The latest developments in this field include the capillary waveguide sensors that represent not only optical waveguides but also act as sample cavities, sampling devices, flow-through cells, and wavelength discriminators (D68), and a fiber-optic spectrometer, manufactured by Ocean Optics, that uses a bundle of six 200-um-diameter multimode silica fibers to illuminate the sample being tested (D69).

Biosensors. The biosensor is an analytical tool in which the sensing element is an enzyme, antibody, DNA, or microorganism and the transducer is an electrochemical (measures change in voltage or current), acoustic (measures change in frequency as a result of a change in mass bound to the surface of the device), or optical device (measures change in fluorescence, absorbance, or reflectance). Several review articles on the status of the environmental biosensors have been published during 1995–1996 (D70– D73), and a whole issue of Trends in Analytical Chemistry was devoted to this subject (D70, D74-D82). Another review article on biosensors for chemical and biological agents of defense interest was prepared by Paddle (D83). The reader should consult refs D84-D88 for additional reviews on this subject.

Potentiometric and amperometric biosensors for the determination of organophosphorus pesticides in environmental samples were described by Trojanowicz and Hitchman (D89), and Besombes et al. (D90) reported on an amperometric biosensor for the determination of cyanide, chlorophenols, atrazine, dithiocarbamates, and carbamate pesticides that was constructed by the electropolymerization of a pyrrole amphiphilic monomer-tyrosinase coating. A remote electrochemical biosensor for field monitoring of phenolic compounds that consists of a specially designed tyrosinase enzyme electrode coupled to a 50-ft shielded cable was reported by Wang and Chen (D91). In another publication, Wang and Chen (D92) report on the phenol biosensor strips microfabricated by screen printing of tyrosinase-containing carbon inks. The ability of the enzyme to tolerate the 2-h firing process was attributed to its organic-phase activity.

Catalytic materials, membranes, and fabrication technology for the construction of amperometric biosensors have been reviewed by Newman et al. (D93). Other examples of electrochemical biosensors published during the review period include the following: a nitrite sensor based on a highly sensitive nitrite reductase mediator-coupled amperometric detector (D94); a phenol sensor based on the enzyme polyphenol oxidase immobilized in a glycerol-based gel (D95); an aldehyde biosensor based on the determination of NADH generated by the enzymatic activity of immobilized aldehyde dehydrogenase (D96); a phenol sensor consisting of either a solid graphite or a carbon paste electrode incorporating the enzyme tyrosinase (D97); and a cyanide sensor using cytochrome oxidase immobilized in a matrix of lipid-cytochrome *c*-modified carbon paste electrode (*D98*).

The use of QCM as a biosensor was reviewed by Minunni (D99) and Tom-Moy et al. (D100) reported on a biosensor using the surface transverse wave device. By immobilizing atrazine to the surface of the sensor, the authors demonstrated that atrazine can be detected at levels ranging from 0.06 parts-per-billion to 10 parts-per-million in less than 3-min analysis time. Investigation of liposome-based immunomigration sensors for the detection of polychlorinated biphenyls was reported by Roberts and Durst (D101).

A flow immunosensor, developed by researchers at the Naval Research Laboratory, for the detection of TNT in water using the evanescent wave fiber-optic technology was reported by Liegler and co-workers (D102). This device is inexpensive and was demonstrated to be comparable in performance to standard methodologies currently in use for the determination of TNT in environmental samples.

IMMUNOCHEMICAL TECHNIQUES

Immunoassays have continued to gain popularity in the regulatory and user community as rapid field-screening techniques, and there are currently 13 immunoassay methods listed in the U.S. EPA Office of Solid Waste Manual (E1-E12). Field evaluation of two of the immunoassay methods approved by EPA was reported (E13, E14). Applications are widespread, covering many fields including human and ecological exposure, foods and pharmaceuticals, groundwater and soil monitoring, agricultural runoff, etc. Pros and cons, applicability of this technique in water quality control studies with emphasis on validation/standardization of immunoassays, and results of field tests were discussed by Muelenberg et al. (E15). Enzyme immunoassay test kits available commercially are reviewed with respect to the requirements for analysis of pesticides in drinking water (E16).

Use of immunoassays in the area of human exposure monitoring was reported by Knopp (E17) and by Biagini and co-workers at the National Institute of Occupational Health (NiOSH) (E18). The NiOSH group evaluated the body burdens of alachlor and alachlor metabolites in urine samples collected from pesticide applicators and also reported use of circulating antibodies to morphine to monitor workers' exposure (*E18*). The NiOSH group also compared ELISA and HPLC methods in the determination of alachlor and alachlor mercapturate in urine samples.

Monitoring of parathion-methyl and chlorpyrifos in saliva by ELISA was reported by Ferguson and Nigg (E19), who fortified six saliva samples with chlorpyrifos at 1 ppb and achieved an average recovery of 91%. The precision and reproducibility of the enzyme immunoassays were correlated with both gas chromatography and high-performance liquid chromatography data (E19). Manclus and Montoya (E20) reported on ELISAs for chlorpyrifos and its degradation product (3,5,6-trichloro-2-pyridinol, TCP) and its suitability for the determination of the two analytes in environmental waters. The detection limits of the two ELISAs were 6 ppb for chlorpyrifos and 0.034 ppb for TCP.

Yu and Stopa (E21) reported use of an immunomagnetic assay (IMAS) with electrochemiluminescence detection for rapid and sensitive bacterial detection of Bacillus anthrax spores, Escherichia coli, and Salmonella typhimurium in biological samples. The IMAS procedure takes less than 1 h and has a detection limit of about 100 cells/mL in phosphate buffer and 1000 cells/mL in most biological samples.

Determination of PCBs in fish extracts and dialysates of semipermeable membrane devices using ELISA was reported by Zajick et al. (*E22*). Lopez-Avila et al. (*E23*) reported on the determination of PCBs in soils/sediments by microwave-assisted extraction (MAE) and ELISA and compared the results of MAE-ELISA with those obtained by Soxhlet-GC/ECD; these authors recommended that a percentage of the ELISA results be verified by GC or other means whenever dealing with an unknown source of PCB contamination.

On-site analysis of PCBs on surfaces (concrete floor, concrete walls, bricks, motor housing, pipes, metal pipes, etc.) using ELISA was reported by Harrison (*E24*). A cellulose wiper saturated with methanol was used to wipe surfaces, and the wiper was extracted with methanol in a propylene jar. The performance of the ELISA kits was established by comparison to the GC/ECD data from 64 pairs of field samples collected over a period of 3 months.

A portable 96-well plate format chemiluminescence immunoassay with a 5-min analysis time and a working range of 0.1-50 ppm was reported by Gui and co-workers (E25). In this enzyme assay, PCBs in the sample to be analyzed are displacing an alkaline phosphatase-bromobiphenyl conjugate from the binding site of an anti-PCB antibody and the residual alkaline phosphatase catalyzes the chemiluminescent reaction of an adamantyl dioxetane phosphate. The same authors (E25) report on the synthesis of various haptens to produce various anti-PCB antibodies (polyclonal and monoclonal). A monoclonal immunoassay for coplanar PCBs was reported by Karu and co-workers (*E26*); this assay is highly specific for non-ortho-substituted congeners and does not recognize the noncoplanar PCB congeners or the 2,3,7,8-tetrachlorodibenzo-p-dioxin, 2,3,7,8-tetrachlorodibenzofuran, or dichlorobenzenes. The limit of detection for PCBs 77 and 126 were 0.2 and 1.0 ppb, respectively, depending on the format.

Monoclonal antibodies specific for complexes of indium and cadmium and ethylenediaminetetraacetic acid (EDTA) were reported by Blake and co-workers (*E27*). The heavy metals were extracted from soil samples with a 230 mM solution of EDTA and the immunoassay was able to differentiate between a heavily contaminated and a minimally contaminated soil sample, but the values obtained for cadmium using the immunoassay were approximately twice as high as those reported by NIST.

Determination of mercury in environmental samples using ELISA was reported by Schweitzer et al. (*E28*). The assay, which is commercialized by BioNebraska, Inc., is available in both plate and tube format and measures mercuric ions in environmental samples (the lower limit of quantitation is 0.25 ppb and the linear range of the tube assay is 0.25–25 ppb). Three of 15 metals tested affected mercury detection; these included gold, silver, and chromium.

Detection of mercuric ions at low-ppb levels using dithiocarbamate chelators in place of antibodies was reported by Szurdoki et al. (*E29*).

An immunoassay test kit for trichloroethylene (TCE) with a detection range of 1.5-75 ppm in water and 5-250 ppm in soil was reported by Hudak et al. (*E30*). The kit contains a TCE-specific antibody covalently linked to small latex particles and the

TCE conjugate linked to alkaline phosphatase. The cross-reactivity data for various chlorinated compounds indicate that only perchloroethylene (PCE) shows high cross-reactivity and none of the dichloroethylenes or vinyl chloride responds in the assay.

The performance of two commercially available test kits for detecting petroleum hydrocarbons in soil was evaluated by Waters et al. (*E31*). Experimental data for the semiquantitative test kit indicate that 44 of the 50 samples tested were correctly determined (the other six samples were false positives).

Experimental data for the quantitative test with benzene, toluene, ethylbenzene, and xylene (BTEX) exhibited correlation coefficients of 0.976–0.983 (slopes of 0.94–0.97), and the sensitivity of either kit for BTEX is in the 1–2 ppm range.

Immunoassays for the organophosphorus pesticides chlorpyrifos-methyl and chlorpyrifos-ethyl have been reported ($\it E32, E33$). Chlorpyrifos-ethyl can be quantified in soils at levels varying from 50 to 500 mg/kg, and no false negatives were reported at these concentrations.

Screening of cyclodiene insecticides (aldrin, chlordane, dieldrin, endosulfan, endrin, heptachlor) in groundwater by ELISA was also reported (*E34*). The least detectable concentrations reported by the manufacturer of this ELISA kit ranged from 0.15 mg/L for endrin to 17 mg/L for aldrin. The ELISA showed the greatest sensitivity for endrin and endosulfan and displayed a positive bias when correlated with gas chromatography. Field screening for aldrin in soil by immunoassay was reported by McCain and Myers (*E35*). Although the test was qualitative and did not distinguish between various cyclodienes, the field immunoassay helped establish the extent of contamination and provided the quick turnaround needed by the field crew. A quantitative ELISA for detection of cyclodiene insecticides in soil or water using paramagnetic particles was reported by Selisker et al. (*E36*).

Hapten synthesis and development of two ELISAs (a laboratory assay based on microtiter plates and a field assay using polystyrene tubes) for the determination of endosulfan in water and soil was reported (E37). One of the haptens was obtained by derivatization of endosulfan diol and the others used derivatives of the five-membered ring adjacent to the cyclodiene ring. The assay had a detection limit of 0.2 ppb and a linear range of 0.2–20 ppb. The assay can detect endosulfan sulfate with a sensitivity similar to endosulfan and endosulfan diol (in this case, sensitivity is 4-10 times lower than for endosulfan).

The determination of chlorothalonil was reported by Matsunaga et al., who used a magnetic particle-based enzyme immunoassay with a detection limit of 0.07 ng/mL (*E38*).

An evaluation of a microtiter plate ELISA for the determination of triazines and chloroacetanilide herbicides in slightly over 1700 storm runoff samples was reported by Pomes et al. (E39). The least-squares analysis showed a relatively good agreement between ELISA and GC/MS (median slope of 1.01 for 363 samples) for atrazine but not for chloroacetanilide because of the presence of a degradation product of alachlor. Increased sample throughput for these two ELISA methods was reported (E40). As many as 86 water samples could be analyzed in \sim 2.5 h (including sample and instrument setup time). The precision of the automated assay varied from 28% for 10 ppt fortification levels to 3.4% at 500 ppt fortification levels.

Comparative trials for triazine ELISA kits distributed by Millipore, Riedel-de Haen, Transia, and CIFEC were conducted

by Mouvet et al. (41) for a set of 41 water samples, who reported that Riedel-de Haen tests gave the best overall results.

Determination of atrazine and alachlor in various types of waters including distilled water, groundwater, estuarine water, and seawater using the magnetic particle ELISA and the microtiter plate assay were reported by Barcelo and co-workers (E42–E44). These authors reported high cross-reactivity values for deethylatrazine that varied from 65 to 81% depending on the water type, and much lower cross-reactivity values for deisopropylatrazine and simazine (E42). In the case of alachlor, the presence of metolachlor was not significant because of the very low crossreactivity of metolachlor (1.1%). The ELISA results were compared with either gas chromatographic data (E43) or the data obtained by solid-phase extraction followed by liquid chromatography with diode-array detection (SPE-LC/DAD). The ELISA was found to detect lower levels of chlorotriazines than the gas chromatographic technique (nitrogen phosphorus detection). The correlation between ELISA and SPE-LC/DAD for seawater samples was good in the 0.01-5 mg/L range, at salt concentrations varying from 0 to 35 g/L and pH values from 2 to 10 (E44).

Determination of carbaryl and naphthol by ELISA was compared to the EPA method 531.1, which involves HPLC postcolumn derivatization and fluorescence detection; the data reported by Marco et al. (E45) indicate both techniques are comparable in terms of precision, reliability, and reproducibility.

Pentachlorophenol (PCP) ELISA test kits available commercially have been evaluated (E46, E47). When using the Ensys ELISA kit, and comparing the ELISA results with the GC/FID or GC/MS data, the authors reported a statistically significant higher extraction efficiency for the GC/FID data. However, the procedure for the ELISA kit involved extraction with 20 mL of methanol/water whereas the extraction procedure for GC/FID used extraction with acetone/methylene chloride (1:4) (E46). Good correlations were found between one of the ELISA test kits data and liquid/solid extraction followed by liquid chromatography with UV detection when PCP concentrations were greater than 2,4,6-trichlorophenol concentrations (*E47*).

Recent developments in the field include the microformat imaging ELISA (E48), electrochemical immunosensors (ref E49; also see below review of biosensors), use of near-infrared fluorescence detection (E50), and use of chelators instead of antibodies to detect heavy metals (E29). The flat-well microformat enzyme-linked immunosorbent chemiluminiscent assay for the detection of 2,4-D was reported by Dzgoev et al. (E48). The detection limit of the microformat assay was 6 pg of 2,4-D and the linear range was in the 10-pg to 10-ng range. In addition to its superior sensitivity, the microformat assay has a number of advantages including reduction in the absolute amounts of reagents required in the assay, reduction in the optimal concentration of the coating antigen and antibody, and the complementary scalable nature of the patterning technology and charge-coupled device camera-based detection system (E48).

Near-infrared fluorescence (near-IR) allows detection of near-IR-tagged small molecules, antigens, and antibodies with high sensitivity and is faster than ELISA because the near-IR assay does not require as a step the substrate turnover by the enzyme as in ELISA. The near-IR dyes allow use of inexpensive laser diodes and thus allow design of systems with small footprint (E50).

Rapid detection of Fumonisin B1 in corn-based food by a membrane-based enzyme immunoassay in dipstick and immunofiltration format was reported (E51). A nylon membrane was coated with anti-fumonisin B1 (FB1) antibodies and with antihorseradish peroxidase (HRP) antibodies. A FB1-HRP conjugate was used as the labeled antigen for the competitive assay. The assay time for the dipstick assay was 60 min and for the immunofiltration assay was 10 min. Both assays have detection limits in the 40-60 ng/g range.

Trucksess used a commercial ELISA test kit for Fumonisin B1 and an immunoaffinity column in which the anti-Fumonisin B1 antibodies are attached to an agarose bead support and compared the results of the two techniques with the results obtained by HPLC (E52). For naturally contaminated corn samples, she reported that ELISA results were 2-100% higher than the HPLC results but with the immunoaffinity column the results were \sim 71% of the levels found by HPLC.

An enzyme immunoassay for the detection of pyrethroid insecticides using either polyclonal (E53) or monoclonal antibodies (E54) was reported by Pullen and Hock. Antisera were raised in rabbits using 1R-trans-permethric acid coupled to bovine serum albumin (BSA) as the immunogen.

Pichon et al. (E55) reported on the development of immunosorbents for preconcentration of phenyl urea and triazine herbicides from environmental waters.

Coupling of enzyme immunoassays with supercritical fluid extraction (SFE) was reported by King and Nam (E56), Lopez-Avila et al. (E57), and Stearman et al. (E58). Coupling of ELISA with SFE is attractive because it allows extraction with nonhazardous solvents and the extraction and analysis time are significantly reduced over the conventional techniques.

Multianalyte immunoassays in which as many as nine different antibodies can be assayed on one microtiter plate have been reported by Strahan (E59). This ELISA format was used to screen 1500 samples of Benlate fungicide for nine sulfonyl urea herbicides at a limit of detection of 5 ppb in the formulated product. A similar concept but using a combination of enzyme immunoassays for triazine herbicides with antibodies of different cross-reactivities for the triazine herbicides was reported by Wortberg et al. (E60).

Quality assurance indicators for the immunoassay test kits (E61) and how to maximize the information from the field immunoassay evaluation studies (E62) were reported. Methods for minimizing false negatives and false positive rates were suggested, and recommendations for calculating confidence intervals of different sample sizes are given (E62).

Dill reported on a sensitive detection system available commercially that employs a unique detection system based on microchip technology (E63). This technology known as the threshold immunoassay system is at the forefront of sensors currently available for the detection of small molecules.

Johns et al. (E64) described the execution of ELISA in immunosorbent columns with antibodies immobilized on porous particles and in which samples and reagents are metered by valves and syringe pumps and are transported into or through the system by high-pressure liquid chromatography pumps.

OPTICAL SPECTROMETRY

Analysis of diesel fuel in soils is commonly accomplished by methods based EPA methods requiring Soxhlet or ultrasonic extraction. Unfortunately, these methods are not suitable for field analysis. Field analytical methods involve Freon extraction followed by infrared methods to measure the aliphatic C–H stretch or a Fridel–Crafts alkylation method to measure the aromatic component of the fuel mixtures. Schabron et al. have developed a simple extraction-UV absorption method which can measure the aromatic components directly at a level as low as 75 mg/kg (F1). This is below the cleanup level normally required of 100 mg/kg. A portable absorption fluorometer for the detection of organic substances in fluids has been reported by Mittenzwey et al. in which they monitored the presence of p-nitrophenol and 2-nitroaniline in drinking water (F2).

In a general review, Steinberg and co-workers have discussed the advantages and disadvantages of fluorescence, phosphorescence, bioluminescence, and chemiluminescence for the analytical determination of environmental pollutants, the measurement of biomass, and the elucidation of toxic and mutagenic effects (F3). Earley and Rapp compared laser-induced fluorescence (LIF) with cone penetrometer testing to more conventional screening methods (F4). A rapid optical screen tool (RPST) (F5) and a site characterization analysis penetrometer system (SCAPS) (F6) for site characterization were reported using fluorescence-based technology. LIF methods have proved particularly useful for the in situ, real-time measurements of polynuclear aromatic compounds in soils (F7, F8). Gerlach has described a novel fieldportable scanning spectrofluorometer (FPSS) for this purpose (F9). The application for LIF for the determination of phytoplankton and polycyclic aromatic hydrocarbons in water was investigated by Uebel et al. (F10). Using a frequency-doubled dye laser as an excitation light source, substances dissolved in water were excited with light in the range 265-400 nm and fluorescence was monitored in the range 310-750 nm.

Surface-enhanced Raman scattering (SERS) has been investigated over the past two years as a potential field method because of its adequate sensitivity for trace organics and its potential selectivity due to the inherent characteristics of Raman spectroscopy (F11). SERS can be used for field and in situ monitoring by coating different sensors onto fiber-optic probes. Vo-Dinh reviewed recent advances in the development of SERS-active substrates for in situ analysis in liquid and vapor samples of environmental and biological origin (F12). In further studies Vo-Dinh and co-workers investigated the potential of poly(vinylpyrrolidone) as a coated substrate for the detection of benzoic acid, isonicotinic acid, and 2,4-dinitrophenol (F13). Carron and coworkers modified SERS surfaces with a 4-(2-pyridylazo)resorcinal disulfide derivative for the detection of copper, lead, and cadmium (F14) and investigated other hybrid substrates for the development of real-time SERS-based chemical sensors (F15). Detection of aromatics in aqueous solutions was demonstrated using substrates chemically modified with p-tert-butylcalix[4] are netetrathiol (F16). The determination of BTEX compounds in water was demonstrated by Wittkamp and Tilotta (F17).

Field-portable Fourier transform infrared spectrometers have also been reported during the past two years (*F18*). The most common use of FT-IR is for monitoring air. Griffiths and Richardson described open-path atmospheric monitoring with a low-resolution FT-IR spectrometer (*F19*). Chaffin and co-workers discussed passive FT-IR data collection and reduction strategies (*F20*). Demirgian and co-workers looked at various emission sources using passive-remote FT-IR (*F21*). A related method for

air monitoring called coherent infrared differential absorption LIDAR was described by Richter and co-workers (*F22*, *F23*). For more information on FT-IR and lidar methods, see the section on remote sensing.

Field-portable X-ray fluorescence (XRF) has been an important area of development for monitoring metal compounds. A general description of the technique was given by Fordham (F24). Bernick et al. (F25-F27) have reported the used of XRF for the determination of metals in soils. Instrument calibration methods, precision, and detection limits were presented along with data collected from a battery breakage and scrap metal site. Detection limits for arsenic, barium, cadmium, chromium, cobalt, copper, lead, mercury, and nickel in soil were reported to be in the 10-100 mg/kg range (*F28*). Elam et al. have reported methods for improving detection limits down to those of the regulatory limits for water (F29). Driscoll et al. (F30) have reported the use of a high-resolution semiconductor detector for portable XRF units, and Potts et al. (F31) used a Hg(II) iodide detector for highresolution measurements. Swift and co-workers reported that XRF methods correlated well with data obtained from standard laboratory methods for lead and arsenic (F32, F33). They have compared on-site analysis using XRF with flame atomic absorption spectrometry (F33). Hewitt, however, found that nickel, cobalt, and chromium did not correlate well with expected values in soil samples (F34). Parametric effects such as measurement time, specimen size, etc., have been reported by Wang (F35). Goldstein et al. (F36) found a large positive bias between XRF and the standard EPA nitric acid digestion methods. Their explanation for this discrepancy was that XRF methods measured the total amounts of analyte while the EPA method measured only the "components labile in nitric acid and not the matrix". Applications other than for soil analysis have also been reported with XRF. The body burden of metals by the analysis of bones have been reported in which Bloch and Shapiro (F37) monitored the depletion of uranium in bones and Ryde et al. (F38) measured the bone lead concentration. Brubaker et al. have reported the potential of XRF in assessing the contamination of building demolition rubble (F39). Airborne particulates collected on filters have been monitored for the presence of metals with XRF methods (F40, F41).

Laser-induced breakdown spectroscopy (LIBS) is the newest of the analytical techniques used for field analysis. As with XRF, LIBS is used for screening of soils and surfaces for metal contamination. Cremers and co-workers have developed a portable instrument which weighs 14.6 kg and fits completely into a small suitcase (F42). The hand-held probe contains a small laser that generates light emission from elements on surfaces at which it is aimed. The breakdown emissions are collected with fiberoptic cable and spectrally resolved to identify the elemental emission source. The detection of metals in soils (F43-F47), Pb in paint (F48), Pb in concrete (F49), Fe in water (F50), and Be and Pb in particulate matter (F42) has been reported. The concentration range in which LIBS is most effective is from 0.01 to 1% (F50). Remote sensing with LIBS has been investigated up to 100 m from the laser source and spectrometry via fiberoptic cable (F51). As a remote sensing tool, analysis can be accomplished in conjunction with a cone penetrometer (F52) and in a bore hole for subsurface rock analysis (F53). Reviews of the LIBS techniques can be found in refs F54 and F55.

RADIONUCLEAR METHODS

Monitoring radionuclear species outside of the laboratory environment is somewhat less difficult than monitoring nonradioactive species. The principal advantage of radioactive materials is that an energy source need not be transported to the field. Radionuclear species have sufficient energies to promote measurable physicochemical events. The most common method for the field detection of radionuclear species is the detection of α , β , and γ particles produced from the naturally occurring nuclear reactions. No field methods in which an excitation source, such as neutron activation analysis, was required to be brought into the field was reported. In this review, field methods for radionuclides are categorized by the emission particle measured.

Alpha-contaminated soils were screened using a novel approach called the Victoreen Alpha activity monitor (G1). With this method 100 Pu-contaminated soils were successfully screen 70% of the time for levels greater than 5 pCi/g and 90% of the time for levels greater than 30 pCi/g. A direct large-area α spectrometer for the analysis of airborne dust particles was reported by Sill (G2). Two type of passive detectors were reported for the measurement of indoor radon (G3). They were electret ionization chambers (EICs) and α track detectors (ATDs). When applied to soils, it was found that the α emission rates varied with soil composition. Thus, it was necessary to develop site-specific calibrations for each type of detector. Nuclear track detectors (NTDs) in general require two to seven days for analysis. Recently, however, Buckner et al. have developed a prototype analysis kit which can be carried into the field (G4). In addition, the presence of hot particles was determined by image analysis of the characteristic starbrust patterns and the energy of the α particle was determined by the size and shape of the track. Passive α detectors have also been used to screen uranium contamination in fields (G5).

Methods for the field detection of β particles were also reported. Noakes et al. have developed a prototype field-deployable tritium analysis system (FDTAS) which was capable of measuring tritium in water samples at environmental levels (G6). In situ measurements of weak β -emitting radionuclides were described using thermally stimulated exoelectron emission (TSEE) and EIC methods (G7). Rego and Smith have developed robust sample handling and liquid scintillation counting procedures to routinely monitor tritium in the field (G8).

Portable γ -ray detectors are needed for the search and verification of nuclear materials for arms control, nuclear safeguards, environmental management, emergency response, and treaty verification. Although most γ -ray detectors require nitrogencooled photomultiplier tubes, Lavietes et al. recently reported on a field-portable instrument base on a new micronomad multichannel analyzer (G9). Xu and Williams developed a 12 in. \times 7 in. portable y-ray spectrometer that does not require cryogenic cooling (G10). Durability, low power consumption, low noise operation, the ability to record data anywhere, autonomous and continuous operation, and a self-monitoring algorithm are reported as integral parts of this CsI-based detector. A third portable detector for γ -ray detection as been reported by Meisner et al. which consisted of a CsI(Tl) scintillation detector with two sidemounted photodiodes (G11). It is cylindrical in shape and operates inside steel pipes pushed into the ground to depths up to 20 m by a cone penetrometer system. Ballard et al. have also reported on a cone penetrometer γ probe for radiation waste detection (G12). A performance test for the photon analysis spectrometer system (PASS) was reported by Gehrke et al. (G13). The use of underwater γ detectors was reported at the Savannah river site (G14). Troyer and co-workers have described the combination of portable γ -ray detectors with an artificial neural network to automatically identify radioactive isotopes in real time (G15).

Field monitoring of radioactivity has been applied to a number of sites over the past two years. Tykva has surveyed analytical methods for environmental monitoring (G16); Renken and Coursin have described a contemporary method for remote monitoring of radon (G17); Cornelius et al. assessed radiation contamination at an abandoned 10-smelter facility (G18); and Rego et al. described a field strategy for monitoring radioactivity associated with wastes derived from deep drilling sites (G19). Coal mining wastes (G20), γ radioactivity in the Kara Sea (G21), and the γ spectrometry of a CANDU heat transport system (G22) have also been reported. Methods for monitoring the dose rate from the gaseous radionuclides were reported by Chung and Tsai (G23, G24). Finally, Perkins et al. have reported a field device that can discriminate between types of radiation and energies providing the measurement of concentrations of ¹³⁷Cs, ⁹⁰Sr, and ²³⁸Ur in soil and 85 Kr in air (G25).

REMOTE SENSING

Field analysis is often accomplished by remote sensing methods. The ability to acquire chemical measurements in physically, chemically, or politically hostile environments depends on the development of analytical methods that can acquire and transport chemical information without the presence of an analyst at the sampling site. In practice, only two approaches are possible: on-site and off-site methods. On-site methods are those in which the analytical measurement device is positioned at the sampling site and left unattended in continuous, intermittent, or radiocontrolled operation. Off-site methods are those in which a probe signal, usually optical, is produced from a remote location, reflected from or passed through the sample, and then detected at a remote location. Remote sensing methods have found a variety of applications such as making chemical measurements in stack and exhaust gases, waste streams, process streams, the atmosphere, oceans, and space.

Optical remote sensing (ORS) of the atmosphere has been the most thoroughly investigated application. Inorganic atmospheric gases such as CO₂, CO, N₂O, NO₂, H₂O, ClO, OH, and O₃ as well as organic vapor-phase components such as CH₄, C₂H₆, C₂H₄, C₂H₂, CFCs, etc., have been measured by ORS techniques.

Passive ORS methods of measurement include those which do not require an source of artificial light. Most of these methods rely on solar radiation as the initial source of energy. Mixing ratios of oxygen, water vapor, ozone, and chlorine monoxide have been measured from the space shuttle by a method known as millimeter-wave atmospheric sounder (MAS) in which the thermal microwave radiation from Earth was monitored at 60, 183, and 204 GHz (H1). Kampfer has reviewed the concepts of microwave remote sensing and its applications to atmospheric analysis (*H2*). Space-borne measurements of NO and NO2 using atmospheric trace molecule spectroscopy (ATMOS) solar occultation were reported by Newchurch et al. (H3). Traces of SO₂ and NO₂ have been measured by an improved UV/visible method called DI-COSPEC (DIgital COrrelation SPECtrometer) (H4). An intercomparison of UV/visible spectrometers for measurements of stratospheric NO_2 has been conducted with $\pm 10\%$ agreement (*H5*). The design of a spectral radiometer for the frequency band of SO_2 was reported by Markina et al. (*H6*).

Other passive ORS methods have included those sponsored by the Passive Infra-Red Atmospheric Measurements of HYDroxyl (PIRAMHYD) program of the European Space Agency for the measurement of OH. Under this program a high-Tc superconductor bolometer was developed for the detection of OH emissions (H7). Ozone measurements have been made by Puckrin and Evans by thermal emission spectroscopy (H8) and calculated from direct solar radiation extinction measurements by Butov and Lognov (H9). Jiyao and Yingjian have developed a method for increasing the spectral resolution of passive ORS instruments (H10). Reine et al. have discussed the advantages of photovoltaic over photoconductive HgCdTe detectors for advanced remote sensing instruments (H11). Johnson et al. reported on the use of a far-infrared spectrometer to measure the mid- to far-infrared thermal emission spectra of the stratosphere during balloon and aircraft flights (H12, H13). Camy-Peyret reported another balloonborne FT-IR technique for monitoring trace levels of molecular species in the earth's atmosphere (H14). A passive IR remote sensing system was also described by Xiaolin et al. for monitoring pollution clouds, demonstrating the difference between laboratory and field spectra (H15). In one application, the infrared emission from a lava fountain was used to measure the SO2/HCl ratio in fumarolic plumes (H16). Space-based thermal IR detection based on neural network techniques was reported for CO (H17). A comprehensive review of spaceborne infrared Fourier transform spectrometers for remote sensing has been written by Persky (H18). Fischer and Oelhaf provide a review of both balloon-borne and space-borne Michelson interferometer for passive atmospheric sounding (MIPAS) techniques (H19). The results of IR remote sensing from satellites is discussed with respect to global atmospheric chemistry (H20). Spatial distribution of gaseous pollutant concentrations were improved by smooth basis function minimization reported by Drescher et al. (H21). Wang et al. used ground-based and airborne interferometer observations to validate FASCOD3 and MODTRAN3 (H22).

Active ORS methods of measurement have been reviewed by Vazquez (H23). Svanberg has also reviewed general chemical sensing methods using laser spectroscopy (H24). The most common type of lidar investigated during the past two years was differential absorption lidar (DIAL). Gelbwachs developed a DIAL technique for monitoring stratospheric ozone depletion in the plume of a launch vehicle (H25). Goers described the optimization of the wavelength pair for the measurement of tropospheric ozone (H26). Langford examined the impact of digitizer nonlinearity on DIAL (H27). Related to DIAL. Warren described a frequency-agile lidar (FAL) technique which scans as many as 60 wavelengths to provide multiple vapor detection (H28). Detection ranges for differential absorption measurements were conducted by Staicu et al. and found to be 6.4 ppm for SO₂ and 18.4 ppm for NO₂ (*H29*). A pulsed xenon arc lamp was used to provide a broad-band radiation source which allowed the simultaneous measurement of the vertical profiles of such atmospheric constituents as O₃, H₂O, and NO₂ (H30). Spatial-resolvable remote sensing was also reported in which CW low-power lasers were used (H31). Nelson et al. used lead salt, liquid nitrogen-cooled diode lasers to improve atmospheric trace gas monitoring (K32).

Remote sensing of organic air pollution was accomplished by a variety of methods during the past two years. FT-IR methods were used for the detection of CFCs (H33), a variety of air pollutants above a petrochemical industrial park (H34), and in agriculture and urban areas (H35). These methods were also used to monitor smoke stack and test flare emissions (H36), accidental releases of toxic and flammable substances (H37), and natural gas leaks (H38). Freon-12 in alcohol/air flames was studied by remote sensing FT-IR (H39). Part-per-million quantities of volatile organic compounds were measured by reflectometric interference spectrometry (H40). Flanigan reported the development of a novel laboratory cell to be used for testing IR remote sensing vapor detectors (H41). Other methods for organic pollution monitoring were cross dispersion infrared spectrometry (CDIRS) (H42), multispectral ultraviolet fluorescence lidar (H43), differential absorption lidar by topographic reflection (H44, Raman scattering (H45-H48), and tunable diode laser heterodyne spectrometry (TDLHS) (H49). One unique method, called surface plasmon resonance spectroscopy (SPRS), used a fiber-optic sensor for remote gas monitoring (H50). Combinations of remote optic sensors were reported for the characterization of air pollutants over an industrial zone (H51) and during biomass burning in Africa (H52, H53).

Combustion products were monitored extensively as exhausts from automobiles and airplanes. FT-IR was used to monitor the impact of air traffic on the upper and lower troposphere (*H54*). Stedman and co-workers have developed a new detector for the remote sensing of on-road carbon monoxide, carbon dioxide, and hydrocarbon from vehicles passing the sensor (*H55*, *H56*). As a result of this work, he received the ACS Award for Creative Advances in Environmental Science & Technology (*H57*).

ORS methods were also of considerable interest for the detection and monitoring of chemical warfare agents (H58). Chemical plumes from organophosphate liquids deposited on the ground were detected with CO₂-based DIAL (H59). Development of a coherent transceiver for the CO₂ DIAL was reported in which a wavelength-agile CW CO2 local oscillator's wavelength was synchronized with that of the transmitter. (H60). IR imaging of remote targets was coupled with remote spectral sensing to provide both spatial and spectral information simultaneously (H61). Althouse et al. reported the use of a Michelson interferometer as a lightweight standoff chemical agent detector (LSCAD) designed to operate from a moving vehicle and provide 360° coverage (H62). Another mobile sensor for chemical/biological warfare agents was described by Kurmer and Leonelli (H63). In their design, both active, multiwavelength, laser remote sensing technologies and passive multispectral imaging systems were utilized. Costello et al. reported improvements in the M21 remote sensing chemical agent alarm (RSCAAL) and tested it for the detection of hydrazine (H64). A review of remote sensing technology for chemical warfare agents has been written by Quinn et al. (*H65*).

Remote sensing of non-vapor-phase samples is also possible. Remote sensing of industrial processes, waste streams, freshwater, seawater, oil contamination, mixed wastes, soils, and planetary surfaces have all been reported during the past two years.

One of the most promising methods developed for industrial process monitoring has been those in which fiber-optics technology is employed. TeX glasses were investigated for use with remote IR spectroscopy (H66). A two-wavelength photometer

with fiber optics was described by Danigel in which the photometer could be located more than 100 m away from the analysis site (H67). A fluorometer with a fiber-optic probe and a directly modulated light source was developed for a wide range of remote sensing applications (H68, H69). Other methods suitable for process monitoring included an acousto-optic tunable filter nearinfrared spectrometer (H70) and an amperometric electrochemical device (H71).

Pollution due to urban effluents was monitored with molecular absorption and turbidimetry (H72). Laser-excited synchronous fluorescence was reported for the detection and monitoring of polynuclear aromatic compounds in aquatic systems (H73), and chlorinated hydrocarbons in water have been investigated by fiber evanescent field analysis (FEFA) (H74). Wang and Chen reported the use of a tyrosinase enzyme electrode coupled to a 50-ft-long shielded cable as a submersible probe for monitoring phenolic compounds in untreated river water and groundwater (H75).

Airborne monitoring of rivers, lakes, and oceans has been investigated for bulk information such as water temperature, oil pollution, phytoplankton behavior, etc. Vorosmary et al. demonstrated the use of passive microwave satellite sensor data to infer the discharge dynamics of large river systems (H76). Dekker et al. used remote sensing to provide additional information on chlorophyll-a in shallow lakes (H77). Dor and Ben-Yosef used multispectral satellite data to evaluate effluent quality of hypertrophic waste water (H78). Bostater et al. evaluated reflectance data from Delaware Bay estuarine waters and determined that the most important parameters are the absorption coefficient, the internal diffuse reflectance coefficient, and the back-scatter coefficient (H79). Fluorescence lidar (FLIDAR) was described by Pantani et al. for monitoring marine environments (H80). Other methods reported for oceanic remote sensing included mediumresolution imaging spectrometry (MERIS) (H81) and satellite synthetic aperture radar (SAR) (H82). The coupling of geographic information systems (GIS) with remote sensing data was discussed by Sweeney (H83).

Remote sensing methods have also been used for oil spill identification (H84, H85), nuclear materials (H86), and the identification of plastics among non-plastics in mixed waste (H87, H88). Optical polarimetry for probing planetary surfaces was reported by Koshiishi et al. (H89).

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