

# Industrial Hygiene Chemistry: Keeping Pace with Rapid Change in the Workplace

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In the last two-year review, the authors posed a question as to whether industrial hygiene analytical chemistry was a “mature” field (*AI*). They concluded that this was not the case. Our survey of the recent literature leads us to the same conclusion. Analytical chemistry research remains highly innovative and productive in the industrial hygiene field. This makes perfect sense when one considers how rapidly the nature of work itself is changing in the industrialized world. New commercial processes and technologies in the workplace have lead to many technical challenges for the analytical chemistry laboratory. Even new industries have appeared in the past decade, and change promises to accelerate as we enter the next century.

Acknowledging trends in industrial activity and the technology revolution, we have added new emphasis to various sections of the review. Immunochemical methods themselves are a part of the technology revolution, for example, and their applications in industrial hygiene analytical chemistry are growing rapidly.

A view held by some on the margins of the field is that chemical exposures in the workplace either do not exist or are universally insignificant from a human health perspective. These attitudes continually reveal weaknesses as workplace injuries and illnesses are reported, occasionally in highly publicized and dramatic cases. Chemical exposures in the workplace (as in the home and ambient environment) are ubiquitous, a fact that increasingly sensitive and available analytical techniques reveal. The challenges in industrial hygiene and occupational medicine will be in the management and minimization of workplace exposures as we pass into the next century. The desire for regulators and the regulated community to deal with undetectably

low exposures is unrealistic—developments in analytical chemistry will further the needs for health risk assessment and risk reduction.

As new analytical techniques become available such as electrospray mass spectrometry or ICPMS, the biological markers routinely accessible grow tremendously. Applications of these new technologies in industrial hygiene monitoring have been hampered by a lack of research funding, as well as stagnation in regulatory incentives. The low cost and high sensitivity of immunologic methods is beginning to drive the development of conventional laboratory-based, instrumental methods that have wider technical acceptance and regulatory cognizance. Positives detected by immunology methods automatically lead to a demand for confirmation or corroboration because cross-reactivity must be considered.

Even advances in telecommunication tools such as the “Web” are having an impact and will stimulate interest and demand. While the information available on the Web is variable in quality and not subject to peer review, its fundamental strengths are universal access and availability. The deluge of new information available on workplace exposures and chemical toxicity data provides a powerful catalyst. This too will push the development of analytical chemistry techniques to address new or perceived health risks.

With these general thoughts and ideas as a backdrop, we turn to the task at hand. The objective of this review is to provide a critical overview of the published research in industrial hygiene chemistry for the years 1997 and 1998. During this period, a total of ~3000 papers and abstracts were examined—captioned here is a synopsis representing a fraction (~15%) that appeared to the authors to have the greatest significance and importance. In the past, *Analytical Chemistry* readers have indicated a strong preference for critical as opposed to encyclopedic or all-encompassing reviews. Thus, we have endeavored to include only the most relevant work although omissions are inevitable.

## GENERAL REVIEWS

Numerous reviews of progress in the areas of industrial hygiene sampling and analysis have been published since the last Analytical Chemistry Application Review in 1997 (*AI*). Some of the reviews are very large and broad in scope such as the review of Environmental Analysis by Clement, Yang, and Koester (*B1*) that contains 959 references. Prokhorova (*B2*) reviewed the global problems of safety and health in industrial environments while

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Table 1. General Reviews

topic	authors	refs
heterocyclic amines	Kataoka	B16
hydrogen sulfide	Hu and Du	B17
dust and ammonia in animal production buildings	Maghirang	B18
crystalline silica	Smith	B19
airborne particles	Injuk et al.	B20
aerosols	Baltensperger	B21
mutagens and carcinogens	Watanabe and Hirayama	B22
radiation	Matel	B23
nuclear techniques in particulate matter studies	Zeisler et al.	B24
polycyclic aromatic hydrocarbons	Zhu and Matsushita	B25
diesel exhaust aerosol	Cantrell and Watts	B26
airborne particulates	Harper	B27
organometallics	Battenberg et al.	B28
nitrogen oxides	Korenaga	B29
selenium	Magee and Tariq	B30
headspace sampling	Keil et al.	B31
organic acid anhydrides	Jonsson et al.	B32
oxygen sensors	Mills	B33

Wolkoff and colleagues (B3) asked "Are we measuring the relevant indoor pollutants?", as they looked at oxidizable volatile organic compounds (VOCs). Other broad scope reviews dealt with the bioanalytical perspectives of biomonitoring and specimen banking (B4), atmospheric pollution (B5), and occupational safety via workplace analysis (B6).

A large review by Zygmunt (B7) concentrated on sampling and sample preparation for chromatographic analysis. Sherwood (B8) reviewed the realization, development, and applications of personal air samplers. Olsen (B9) looked at the effect of sampling on measurement errors, Tamakawa et al. (B10) discussed the use of passive gas tube samplers, and Harper (B11) asked us to consider Anasorb 747, a universal sorbent with a very few exceptions (e.g., aldehydes). Finally, Griffiths (B12) offered sampling guidelines for bioaerosols.

The generation of known atmospheres was addressed by Kleinman and Phalen (B13), and Amagai (B14) and Fujimoto (B17) focused on sampling and analysis of indoor air for a variety of contaminants. Other reviewers were interested in single compounds, chemical families, or techniques. These are listed in Table 1.

A large increase was noted in reviews of environmental immunoassays and bioanalytical methods. Sherry (B34), Weller (B35), Gerlach and Van Emon (B36, B37), Wengatz et al. (B38), and Sadik and Van Emon (B39) all dealt with topics associated with immunoassays including fundamentals, cost-effectiveness, detection, and applicability. Diaz-Ferrero et al. (B40) discussed bioanalytical methods applied to endocrine disrupters while Lutz et al. (B41) focused on coupling liquid chromatography to a bioanalytical detector. The sampling and/or analysis of bioaerosols was covered by Boehm (B42), Jacobs (B43), and Eduard and Heederik (B44) while Tang et al. (B45) reviewed the monitoring of proteolytic enzymes. The following sections of this applications review contain numerous specific references in many areas of industrial hygiene sampling and analysis.

#### MONITORING INSTRUMENTS

Many papers have been published that deal with the measurement of chemicals of interest in workplace air, indoor air, ambient air, and emissions. Monitoring instruments for the analysis of air

samples are usually based on chromatographic and/or spectrometric techniques, although additional methods (e.g., microscopy, immunoassay) have made important contributions. Increasingly, portable instrumentation is being applied for industrial hygiene measurements, as reflected in pertinent publications.

**Spectrometry.** Infrared spectrometry continues to be used extensively for monitoring of airborne pollutants for environmental and occupational health applications. Makepeace et al. (C1) have employed "extractive" Fourier transform infrared spectrometry (FT-IR) for monitoring indoor air quality. This technique was used to monitor contaminant concentrations over time in a print shop and in a dry cleaning establishment, and it was possible to relate contaminant outputs to specific work activities. D'Arcy and Levine (C2) used FT-IR spectrometry for workplace air monitoring and employed an expert system to enable a nonspectroscopist to obtain meaningful analytical data. To date, the expert system coupled to FT-IR for air monitoring has only been applied in occupational environments with known atmospheric contaminants, but the presence of "unanticipated" compounds can be detected and made known to the user. In related work, Kanagawa et al. (C3) have used infrared cameras to monitor concentrations of flammable gases. Simulations were compared with experiments for the simultaneous detection of methane, butane, and propane. The expected application of this technique is leak detection. Toxic organic compounds have been monitored by open-path FT-IR in research by Russwurm (C4). The anticipated use of this device is FT-IR remote sensing for compliance with U.S. Environmental Protection Agency (EPA) air quality regulations. FT-IR and other methods have been employed by Siskos et al. (C5) to monitor pollution from environmental VOCs. Lindh et al. have used "transportable" FT-IR to measure an anhydride in workplace air (C6). The technique was useful for rapid mapping of exposure levels in the workplace. Pengelly et al. (C7) used infrared spectroscopy to monitor formaldehyde and related vapors which were generated from different source materials in the workplace. Comparison of the infrared technique was made with diffusive samplers, which may give erroneous results. Todd (C8) and Samanta and Todd (C9) have evaluated open-path FT-IR in an exposure chamber. In this research, concentrations of various gases within the air of the exposure chamber were varied in time and space. The within-chamber evaluations will prove to be useful for industrial hygiene applications for organic vapor detection. In related work, Sandsten et al. (C10) also used infrared spectrometry to image gases in workplace air. Last, Hearl (C11) used infrared ion mobility spectrometry (IMS) for industrial hygiene and related applications. Carnahan et al. (C12) described refinements to an IMS system that was developed for military and occupational hygiene applications. In this work, toxic gases such as those generated by explosives, chemical weapons, and OSHA-regulated gases could be successfully monitored by using a "novel" photo-ionizer. Graeme (C13) employed a hand-held IMS device to monitor volatile organic compounds in manufacturing industries. By use of different "ion acquisition" modes, it was possible to discriminate between a wide range of airborne volatile substances. In related research, Liess (C14) described the use of IMS for analysis of trace gases at the parts-per-billion level in occupational and environmental settings. Gordon et al. (C15) used IMS to quantitate chlorine dioxide in workplaces where chlorine was

present in the air. While previous IMS devices have not been able to discriminate between airborne chlorine and chlorine dioxide, refinements have been made to enable the new IMS instrument to differentiate between the two substances. Finally, Puskar and Plese (C16) evaluated IMS and other real time techniques for the measurement of hydrogen peroxide in workplace air. Experiments suggested that the IMS technique meets the National Institute for Occupational Safety and Health (NIOSH)  $\pm 25\%$  accuracy criterion for the determination of  $H_2O_2$ .

Not surprisingly, atomic absorption spectrometric methods continue to be used widely for occupational health monitoring. Often atomic spectrometric techniques are utilized as reference analytical methods for method evaluation purposes. For instance, Tsai et al. (C17) employed atomic absorption (AA) spectrometric analysis as a reference technique for the characterization of lead aerosols. Size-selective fractions of aerosols containing lead were collected from different work environments, and lead was determined by AA spectrometry. Schlecht et al. (C18) examined the performance of atomic spectrometric techniques for the analysis of lead in paints, soils, and dusts. In this work, interlaboratory evaluation of various atomic spectrometric methods (e.g., flame and graphite furnace AA) was found to give excellent laboratory-to-laboratory reproducibility and repeatability. The results offered support for a performance-based approach to proficiency testing for lead analyses. Bellido-Milla et al. (C19) used flame or electrothermal AA spectrometry to measure a series of metals (Fe, Mn, Zn, Cu, Cr, Ni, Cd) in samples of welding fume. In this application, the analytical techniques were used as reference methods for the determination of factors giving rise to variabilities in metals content. In related work, Roig-Navarro et al. (C20) used AA spectrometry to measure heavy metals and boron contamination in workplace air collected from ceramic factories.

Another atomic spectrometric technique, inductively coupled plasma (ICP) spectrometry, continues to be used extensively in the industrial hygiene field. For example, ICP spectrometry was used by Lemus et al. (C21) to determine concentrations of As, Cd, Cr, Cu, Fe, Pb, Hg, and Ni in household dusts. By using ICP as a reference analytical technique, exposures to toxic elements could be characterized as a basis for health risk assessment. In an interesting application, Hulanicki et al. (C22) employed inductively coupled plasma emission spectrometry for the determination of hafnium in workplace air filter samples. Millson and Ashley (C23) used ICP with atomic emission detection to measure lead in environmental reference materials following ultrasonic extraction. For this element, ultrasonication was found to give quantitative recoveries and, therefore, may be useful in lieu of hot plate or microwave digestion techniques. ICP with mass spectrometric detection (ICPMS) has been used by Pecheyren et al. (C24) for the simultaneous determination of a suite of elements (Pb, Hg, Sn, In, Ga, Se, P, As) in different atmospheres. While the high sensitivity of ICPMS may be unnecessary for many workplace monitoring applications, this technique could be required for ambient air measurements of metals and metalloids at very low concentrations.

Ultraviolet/visible (UV/visible) spectrophotometry and related techniques are frequently used in occupational and environmental health monitoring. UV/visible techniques offer certain advantages for industrial hygiene applications, such as low cost, ease of use,

and field portability. UV/visible methods also sometimes are relied upon as dependable reference methods (e.g., refs C5, and C20). Nevertheless, new spectrophotometric techniques continue to be developed for industrial hygiene applications. To wit, Turek et al. (C25) used spectrophotometry to determine dimethyl disulfide in complex gas mixtures emitted in the rubber industry. It was found that the method allowed for the simultaneous measurement of disulfides and mercaptans. Elsewhere, Verma and Gupta (C26) used a visible spectrophotometric method to determine hydrogen sulfide in the air of steel plants. The results were found to compare well with the popular and widely accepted methods for measuring  $H_2S$ . Visible spectrophotometry is widely employed for the measurement of environmental hexavalent chromium, as exemplified by the work of Kuo et al. (C27), where Cr(VI) in electroplating factories was monitored by the often-used diphenylcarbazide (DPC) method. Wang et al. (C28) developed and evaluated a flow injection analysis (FIA) method for Cr(VI) in workplace samples. Visible spectrophotometric detection was used following basic ultrasonic extraction, FIA, and derivatization with DPC. With the ultrasonic/FIA technique it was possible to determine Cr(VI) as well as total chromium. In other work, Patterson et al. (C29) developed a novel open-path, Fourier transform UV spectrometer to detect benzene and other gases. Wollaston prisms were used to form an interferogram in the spatial domain which was recorded by an array detector; the applicable wavelength range was 200–270 nm. Huang et al. (C30) used a photometric method to monitor the spatial variability of aerosols penetrating respirator filters. The technique can be used to test the performance of respirator filters and to detect leakage. Schiff et al. (C31) employed a near-IR tunable laser diode for air pollution monitoring. Examples of applications for near-IR monitoring include remote sensing of automobile emission of hydrocarbons and plant HF emissions. In related research, Stepanov et al. (C32) employed tunable diode lasers for monitoring gaseous CO, NO,  $NH_3$ ,  $CH_4$ ,  $SO_2$ , and so on. The device described is a multicomponent gas analyzer which is based on tunable diode lasers and is computer-controlled. Applications in human exposure monitoring were proposed.

Mass spectral techniques have been the subject of several reports having to do with air monitoring, e.g., VOCs (C5). Time-of-flight mass spectrometry (MS) was employed by Noble and Prather (C33) to characterize aerosol particles. In this work, a technique was devised that allowed for the measurement of both the size and composition of individual aerosol particles. This research may prove to be useful for epidemiological studies. White et al. (C34) described a transportable time-of-flight MS device for analysis of gases. The instrument demonstrates detection limits approaching those of laboratory-based time-of-flight MS and offers potential for environmental monitoring. In an interesting application, Nusko and Heumann (C35) used thermal ionization MS following extractive separation for speciation of trivalent and hexavalent chromium in collected aerosol particles. The extraction method described is tedious but effective, and isotope dilution MS offers extremely low detection limits, enabling chromium speciation to be conducted on very small samples. The technique was tested successfully in an interlaboratory comparison of a new European welding fume reference material containing certified concentrations of total and hexavalent chromium.

X-ray fluorescence (XRF) was used for multielement analysis of airborne particulate matter in several noteworthy papers. Schmeling and Klockow (C36) described a total reflection XRF spectrometric technique for the multielement analysis of air particulate samples. Analytical figures of merit were calculated for V, Mn, Fe, Ni, Cu, Zn, Se, Rb, and Pb. Dost (C37) described the use of a field-portable XRF device for monitoring inorganic contaminants in surface dust and airborne particulates collected from workplace environments. Efforts were made to compare in situ surface dust portable XRF measurements with results from wipe sampling. For workplace air filter samples, detection limits were sufficiently low to allow for the analysis of 18 elements at or below the required sensitivity (excepting Pb and Cd). In another study, XRF was used by Thomson et al. (C38) for the measurement of heavy metals in workplace aerosols collected on filter paper tape. A prototype XRF device was found to give acceptable analytical performance on the elements U, Cr, Pb, Hg, W, Br, Mo, Cu, Zn, Fe, and As. XRF and neutron activation analysis were employed by Martinez et al. (C39) to measure metallic pollutants in household dust. In some cases, it was found that concentrations of Pb and Hg in dust samples posed unacceptable health risks. Portable XRF and other on-site techniques were used by Ashley et al. (C40) to measure lead in paint during a field investigation. This study showed that the use of portable XRF is questionable for the in situ determination of lead in paint but may be applicable for screening purposes. XRF analysis of lead in collected and ground paint samples (ex situ) gave results that were quantitative, with acceptable detection limits.

Other spectrometric techniques have been used in novel ways for industrial hygiene or related applications. In an interesting investigation, Andreson (C41) used a microwave resonator to analyze a gaseous mixture of polar substances. The technique entails qualitative and quantitative analysis by high-resolution rotational emission spectrometry and is applicable to gas mixtures of rather high concentrations. Sallsten and Nolkranz (C42) used atomic fluorescence to determine trace mercury in humid air. Analytical figures of merit for the atomic fluorescence technique following sample collection and preconcentration by amalgamation demonstrated excellent performance of the method. Last, Bard et al. (C43) employed Raman microspectroscopy for the identification of asbestos fibers. For samples collected on cellulose acetate filters, this Raman technique was found to be useful for the identification and characterization of single micrometer-sized asbestos particles, both fibrous and nonfibrous. The technique could be used to distinguish between species such as amosite, anthophyllite, chrysotile, and crocokolite.

**Chromatography.** By far the most widely used chromatographic technique for industrial hygiene analysis is gas chromatography (GC). This has been reflected in a great many publications on applications of GC to occupational air monitoring and related subjects in the past two years. In one investigation of agricultural importance, Dimitriou and Tsoukali (C44) used capillary GC with flame ionization detection (FID) to monitor methyl bromide in greenhouse air. Personal and environmental measurements revealed high airborne concentrations of methyl bromide, which is a widely used soil fumigant. In related work, Martinez Vidal et al. (C45) used GC to monitor greenhouse air, in this case for several pesticides. Atmospheres containing known

concentrations of airborne pesticides of interest were generated, and the pesticides were sampled using several common sorbents. The samples were then analyzed by GC with electron capture detection (ECD) and MS detection. Huether et al. (C46) used a GC method for the simultaneous detection of methane, nitrous oxide, and carbon dioxide in the presence of high levels of oxygen and nitrogen. The monitoring system described has been employed for several years for the determination of these gases in trace concentrations in agricultural settings.

Methanol, phenol, *o*-, *m*-, and *p*-cresols, and formaldehyde were determined by GC in work by Posniak and Politowicz (C47). Conditions were optimized to sample and analyze for the tested compounds emitted during the hardening of phenol-formaldehyde resins. In related research by Martos and Pawliszyn (C48), formaldehyde was determined by GC-FID following solid-phase microextraction (SPME). A very good detection limit and broad linear dynamic range were achieved. In other work, David and Pauls (C49) measured concentrations of aromatic hydrocarbons in petrochemical plants using three different GC techniques. One of the GC devices tested was a portable unit, and the performance of the instrument for the measurement of benzene, toluene, ethylbenzene, and *o*-, *m*-, and *p*-xylene was investigated. Khalitov et al. (C50) also used GC to determine xylene isomers and other hydrocarbons in ambient air. Bassford et al. (C51) described an automated GC system for monitoring trace halocarbons in the atmosphere. Excellent precision was achieved, and extremely low concentrations could be detected. Hallama et al. (C52, C53) compared GC methods for the sampling and analysis of VOCs in workplace air. GC was also used by Qin et al. (C54, C55) to monitor trace methanol, ethanol, acetone, and pentane in air following preconcentration on solid sorbents. The procedure described is purportedly simple and is applicable to low-molecular-weight VOCs. Shumilov and Ustinova (C56) employed GC-FID to determine tetrahydrofuran and carbon tetrachloride simultaneously in workplace air. The method is stated to be fast and simple and requires no sample pretreatment, a decided advantage for potential industrial hygiene applications.

Aromatic hydrocarbons in the air of gasoline service stations were measured by Periago et al. (C57) with GC. It was found that personal time-weighted average benzene exposures exceeded the threshold limit value in 20% of the sampled population. Temporal variations in ambient concentrations of other VOCs (i.e., toluene and *m*-, *o*-, and *p*-xylene) were found to correlate with gasoline sales. Begerow et al. (C58) employed capillary GC with tandem FID and electron capture detection (ECD) for the simultaneous detection of 28 VOCs in air. The procedure, which is a screening method, was successfully applied to field studies of variations in VOC concentration with changes in traffic density. In a noteworthy study, airborne hydrocarbons were analyzed by Martos et al. (C59) using a temperature-programmed capillary GC method. The technique proposes to quantify hydrocarbons in air on the basis of retention indexes, thereby precluding the need for standard calibration. Clausen and Wolkoff (C60, C61) used a capillary GC technique to test a new sampling device for monitoring semivolatile organic compounds in indoor air. Evaluations were conducted on different sorbent materials, and figures of merit were reported. Jonsson et al. (C62) employed GC-FID to measure cyclic organic acid anhydrides in air. The overall

sampling and analytical method was validated using GC/MS and atmospheric standards. Headspace GC has been used by Balyatinskaya et al. (C63) for the analysis of toxic organics in air. Preconcentration in 1-butanol or 1-pentanol (for the analysis of alcohols or ketones, respectively) was used for sample preparation prior to GC analysis. Brixie et al. (C64) also used a headspace GC technique to analyze gases captured by a new sampling technique.

In other work, GC with atomic emission detection (AED) was used by Becker et al. (C65) for the speciation of organotin compounds released from poly(vinyl chloride) (PVC). It was found that significant amounts of dibutyltin were emitted into the air from PVC samples stabilized with dibutyltin maleate at high temperatures. Elsewhere, Chen et al. (C66) employed GC-ECD for the measurement of toluene-2,4-diisocyanate (TDI) vapor. TDI was measured as a pentafluorobenzyl derivative after collection using an impinger, with excellent sampling efficiency demonstrated. Nuclear magnetic resonance (NMR) spectroscopy was used to unambiguously identify the derivative. In additional research, Clausen et al. (C67) used GC to analyze free fatty acids and fatty acid salts extracted from house dust. By examining results from different extraction conditions, efforts were made to avoid false positive results at low concentrations. Continuous monitoring of phosgene, cyanogen, and cyanogen chloride in ambient air was the object of a GC method developed by Lattin and Paul (C68). The method is intended for on-site use during field operations. Aldehydes in indoor air were measured by GC following solvent desorption in research by Mori et al. (C69). Good recoveries for several aldehyde species were obtained.

Thermal desorption (TD) is often used for GC sample introduction of sorbed gases and vapors. Prado et al. (C70) evaluated a method for the determination of styrene in air by using TD-GC. The method was evaluated in the laboratory as well as in the field and was found to give no significant differences in performance from existing methods. In an interesting application of TD-GC techniques, Rowell et al. (C71) tested the recovery of various solvents from adhesive skin adsorption pads. A desire of this work was to monitor solvent exposure to workers' hands, with a goal of detecting solvent breakthrough through protective gloves. In another workplace application, Bianchi and Joyner (C72) used TD-GC-FID to measure low concentrations of TDI in air; a similar study employing a different detection technique was already mentioned previously (C66). A low detection limit for TDI was determined, and the analysis duration was less than 30 min. TD-GC was used by Yang and Lo (C73) to evaluate Tenax TA adsorption for measuring a series of toxic organic compounds in workplace air. The method was found to perform well for the sampling and analysis of toluene, styrene, isobutyl acetate, and *o*-, *m*-, and *p*-xylene. In related work, Rothberg et al. (C74) employed TD-GC to measure nicotine and other VOCs in indoor air. The detection of nicotine in air is an indication of exposure to tobacco smoke spreading from smoking areas. McKenzie et al. (C75) used TD-GC-ECD to monitor 2,3-dimethyl-2,3-dinitrobutane (DMDNB) vapor in workplace air. This compound has been proposed as a tag molecule to aid in the detection of plastic explosives, which cannot be easily detected by conventional methods. DMDNB sublimates at room temperature, and the TD-GC technique was useful for its detection. Scobbie and Groves

(C76) used a similar TD-GC approach to measure dimethyl- and diethyl sulfate in workplace air, following sampling onto Tenax TA sorbent tubes. In a study with important implications, Hung et al. (C77) investigated problems with interference from artifacts in the analysis of vinyl chloride by TD-GC. An artifact from water vapor collected by the sorbent gives rise to an interference in the analysis of vinyl chloride. Procedures for removing the artifact arising from water vapor were investigated. Finally, Yao et al. (C78) used passive dosimeters and TD-GC for the collection and determination of 1,3-butadiene in ambient air. The sampling and analytical procedure was tested against NIOSH statistical protocols, and it was found that the results obtained met NIOSH accuracy criteria for occupational exposure monitoring.

Gas chromatography with ion mobility spectrometry (GC/IMS) has been used by some researchers in investigations of interest to the military. For example, in studies with potential applications in detecting exposures to soldiers, Arnold et al. (C79, C80) employed GC/IMS for personal monitoring of chemical weapons. Efforts to improve the performance of hand-held GC/IMS devices centered on an automated vapor sampling-transfer line GC approach. Other refinements to the GC/IMS methodology for the detection of airborne chemical weapons agents were also described.

Gas chromatography with mass spectral (GC/MS) detection was used by a number of researchers in studies pertinent to the field of industrial hygiene. In one study, Smith et al. (C81) used GC/MS for the detection of resin acid compounds emitted from rosin used in soldering flux. Resin acids can cause dermal and pulmonary sensitization in workers, hence the interest in their analysis. A method was described for the detection of resin acids, such as abietic and dehydroabietic acid, found in aerosols from heated rosin flux. In another study, Mangani et al. (C82) used solid-phase extraction and GC/MS for the determination of semivolatile organic compounds in workplace atmospheres. Elsewhere, Hammond et al. (C83) employed GC/MS to monitor exposures to glycol ethers in the semiconductor industry. It was found in this investigation that measured concentrations of several glycol ethers were low, although there were differences in the magnitude of exposure for workers performing different tasks. Benzene and other VOCs were detected in cigarette smoke in a study by Darrall et al. (C84), where GC/MS was the method of choice. Concentrations of these compounds were measured in smoke emanating from numerous cigarette brands, and implications for indoor air monitoring were suggested. Radolovich et al. (C85) developed a dual-column GC device that uses both mass spectral and flame photometric detection and used this instrument to measure airborne chemical agents. Several chemical agents could be detected simultaneously with this approach. Scobbie et al. (C86) described a GC/MS method and a liquid chromatographic (LC) method for the determination of coal tar pitch volatiles. It was suggested that the GC/MS or LC approach for the analysis of polycyclic aromatic hydrocarbons (PAHs) would be superior to the analysis of coal tar pitch fume as a whole, the latter of which can give variable responses for different source materials.

Several efforts focused on the development of portable or "transportable" GC/MS devices. Piltingsrud (C87) described a field-deployable GC/MS instrument for industrial hygiene ap-

plications and conducted field tests involving the monitoring of complex mixtures of gases and vapors. In related work, Matz et al. (C88) developed an on-site GC/MS approach for the analysis of toxic substances produced by fires and chemical accidents. Efforts were made to ruggedize the mobile GC/MS device. Elsewhere, Simon and Farant (C89) described a portable GC/MS instrument for long-term personal monitoring of VOCs and other air pollutants. Prototype devices were made using small evacuated canisters, which in turn were connected to the GC/MS device by a "novel" flow controller. Last, McClennan et al. (C90) used a mobile GC/MS system to map VOC gradients in time and space in ambient environments. It was stated that the device could be used for more rapid profiling of VOC concentrations than previous GC/MS instruments.

Numerous papers have appeared that describe applications of LC methods to workplace air analysis. For instance, Kennedy et al. (C91) developed a sampling and analytical method for the simultaneous determination of 14 organonitrogen pesticides in air. Sampling was conducted with an XAD-2 sorbent, and high-performance liquid chromatography (HPLC) with UV detection was used for analysis. It was found that, for 10 of the 14 pesticides investigated, NIOSH accuracy criteria were met. In another study, Zhu et al. (C92) used HPLC with spectrofluorometric detection to analyze PAHs in air. Very good recoveries and high sensitivities were found for 12 PAHs in air. Benassi (C93) employed a similar analytical protocol to determine trace phenolic compounds in air and reported low detection limits, good specificity, and overall simplicity. In another significant investigation, Dunzik et al. (C94) evaluated HPLC methods for the determination of benzene-soluble species in asphalt fume samples. In this work, the analytical performances of NIOSH and OSHA methods were compared, and it was found that a modified NIOSH procedure provided better accuracy and precision in the concentration range studied. Elsewhere, Sathra et al. (C95) used HPLC methods to isolate and identify contact allergens in rosins. Other analytical techniques were employed for confirmation purposes. Liquid chromatography with mass spectral detection (LC/MS) was used by White et al. (C96) for the analysis of hazardous agricultural and industrial chemicals. The LC/MS method compared favorably with GC/MS in the analysis of several compound classes not easily analyzed by GC.

The monitoring of isocyanates in the occupational environment has been a subject of much interest, and methods to measure these species in workplace air have most often been based on HPLC. Several studies have reported HPLC analysis of derivatized isocyanates, with a variety of derivatizing agents having been evaluated. For example, Streicher et al. (C97) investigated a novel derivatizing agent, 1-(9-anthracenylmethyl)piperazine (MAP), for its reactivity with mono- and difunctional isocyanates. The reactivity of MAP was compared with reactivities of other derivatizing agents that have been employed in the analysis of isocyanates, and the performance of MAP was found to be favorable. In related work, Rudzinski et al. (C98) evaluated the use of MAP and HPLC for the analysis of isocyanates in spray-painting operations. The performance of MAP was compared with that of another derivatizing agent, 1-(2-methoxyphenyl)piperazine (MOP), and the results were comparable for both mono- and polyisocyanate analysis. Elsewhere, Schulz and Salthammer (C99) used 1-(2-

pyridyl)piperazine as a derivatizing agent for monomeric 4,4'-diphenylmethane diisocyanate (MDI) prior to HPLC with fluorescence detection. The procedure employed an impregnated filter for sampling of isocyanates; this technique was described in OSHA method 47 over 10 years ago. Karlsson (C100) determined low-molecular-weight aliphatic isocyanates by means of their dibutylamine derivatives; LC/MS or GC/MS was used for analysis of the derivatives. Tinnerberg et al. (C101) utilized the same derivatizing agent for the determination of MDI and related compounds formed during the thermal decomposition of polyurethane. Analysis was performed by using reversed-phase LC with mass spectral detection. This LC/MS method was found to be effective in resolving a variety of structural isomers and analogues in polymeric MDI and also allowed for the detection of amines and other isocyanates. In related work, Tinnerberg et al. (C102) used dibutylamine and 9-*N*-methyl(aminomethyl)-anthracene as derivatizing agents for TDI in workplace air. Analysis of derivatized TDI was conducted by an LC/UV/MS procedure. While LC/MS techniques offer good analytical performance and provide much information, the cost of LC/MS instrumentation is probably prohibitive for routine use in industrial hygiene monitoring.

Ion chromatographic methods have been used in several studies of interest in occupational and environmental health. In a field investigation, Hethmon and Ludlow (C103) used ion chromatography (IC) to monitor sulfuric acid mist in copper electro-winning tank-house environments. A new procedure was developed which was stated to offer better analytical performance than existing OSHA and NIOSH methods. IC has also been used to monitor acid aerosols in the semiconductor industry, where Lue et al. (C104) used this technique to measure trace acidic airborne contaminants in cleanrooms. The method was capable of analyses in class 1 cleanrooms. In another novel study, Ito et al. (C105) used a continuous monitoring system based on IC for the measurement of strong acidity levels in workplace air. Interferences from weakly acidic or basic gases were found to be insignificant. In related work, Guglya (C106) determined orthophosphoric and orthophosphorous acids in workplace atmospheres by an IC technique. Finally, environmental hexavalent chromium can be measured using IC and other methods, as demonstrated by Vitale et al. (C107). Several standard methods for measuring Cr(VI) are based on IC, but refinements continue in efforts to stabilize chromium species during sampling, sample treatment, and analysis.

**Other Monitoring Techniques.** A number of other contributions that did not entail spectrometric or chromatographic monitoring means (per se) for industrial hygiene purposes were published in the last two years. These include techniques such as microscopy, X-ray spectroscopy, electroanalysis, immunoassay, and surface analysis, to name a few. Pertinent papers are summarized in Table 2 for ease of review and economy of space.

## SENSORS

Sensors for vapors and gases are widely used in the industrial hygiene field, and research on new sensors and improved sensor designs continued in the past two years. The majority of sensors available commercially are based on electrochemical or optical transduction, and improvements in the materials, design, and

Table 2. Other Monitoring Methods

topic/analytical technique	authors	refs
SEM and X-ray analysis of surface defects on protective gloves	Canning et al.	C108
particle-induced X-ray emission (PIXE) of metal particulates collected from welding	Formenti et al.	C109
Ni speciation in oil ash by X-ray absorption fine structure (XAFS) and anodic stripping voltammetry (ASV)	Galbreath et al.	C110
ultrasonic extraction and portable ASV of lead in particulate collected from workplace air	Ashley, Ashley et al.	C111, C112
adsorption stripping voltammetric determination of formaldehyde	Chan and Xie	C113
laser light scattering for detection of hazardous fibers	Kaye et al.	C114
portable vapor analyzer based on piezoelectric sorption	Fung and Wong	C115
isotachophoretic determination of ethanolamines	Sollenberg	C116
field-portable immunoassay of pentachlorophenol	Gerlach and Van Emon	C117
enzyme-linked immunoassay (ELISA) determination of Hg(II)	Marx et al.	C118
ELISA for detection of 1-nitropyrene in air particulate	Froeschl et al.	C119
electron spin resonance (ESR) for quantification of coal dust–diesel particulate	Dalal et al.	C120
SEM of diesel exhaust particulate for size distribution	Figler et al.	C121
interlaboratory analysis of carbonaceous aerosols	Birch	C122
monitoring diesel emissions in workplace air	Dahmann et al.	C123
portable volatile chlorinated solvent monitor	Singh	C124
portable instrument for real time analysis of chlorinated organic compounds	Buttner et al.	C125

construction of these types of sensors have been reported in the last two years. Other types of sensors, e.g., biosensors and mass-sensitive sensors, are also being investigated and are seeing use in the occupational and environmental health arena.

**Electrochemical Sensors.** Electrochemical sensors are used extensively in industrial hygiene for the detection of gases and vapors such as carbon dioxide, carbon monoxide, sulfur dioxide, and nitric and nitrous oxides. New materials continue to be developed and tested in efforts to improve the performance of electrochemical sensors. For instance, zirconia-based materials have been investigated by several workers striving to improve the sensitivity and stability of solid-state electrochemical CO sensors. In recent studies, Miura et al. (*D1*, *D2*) designed a solid-state electrochemical device, based on stabilized zirconia, for the selective detection of CO at high temperatures. CdO and SnO<sub>2</sub> electrodes were found to be best suited for selective response to CO (20–4000 ppm) at high temperatures, and interferences from hydrogen, NO<sub>x</sub>, carbon dioxide, oxygen, and water were minimal. In related work, Schwebel et al. (*D3*) designed a CO resistive sensor based on high-temperature stable Ga<sub>2</sub>O<sub>3</sub> thin films. Responses to organic solvents were virtually eliminated.

Stabilized zirconia has been used by Aono et al. (*D4*) in the fabrication of electrochemical sensors for carbon dioxide. These workers investigated the influence of humidity on a potentiometric CO<sub>2</sub> sensor using a solid electrolyte composed of stabilized zirconia and Na<sup>+</sup>-conducting glass. Excellent results were obtained in dry air, but significantly lower responses were obtained in humid environments. In other research by Zhang et al. (*D5*, *D6*), a solid-state electrochemical sensor for carbon dioxide was fabricated using a lithium ion conductor. The device was found to perform well for the detection of CO<sub>2</sub> at high temperature, and the response was not affected by water vapor. Essalik et al. (*D7*) developed a carbon dioxide microsensor based on a solid electrolyte thin film composed of Na<sub>2</sub>CO<sub>3</sub>/BaCO<sub>3</sub>/Ag<sub>2</sub>SO<sub>4</sub>. Enhanced performance characteristics over “bulky” CO<sub>2</sub> sensors were claimed, although stability problems were identified. In related work, Kale et al. (*D8*) employed Nasicon (an Na<sup>+</sup> conductor) in the fabrication of a potentiometric CO<sub>2</sub> sensor. It was mentioned that the emf response of the sensor was unaffected by water vapor. Nasicon was also used by Steudel et al. (*D9*) in the design of a tin oxide electrode modified carbon dioxide sensor. The response of the device was based on the cell reaction Na<sup>+</sup> +

OH<sup>-</sup> + CO<sub>2</sub> = NaHCO<sub>3</sub>. Kaneyasu et al. (*D10*) also used Nasicon in the fabrication of a carbon dioxide sensor and reported good emf response for CO<sub>2</sub> even in high humidity atmospheres.

Several studies having to do with electrochemical NO<sub>x</sub> sensors have been reported. Shimizu et al. (*D11*, *D12*) fabricated a solid-state sensor for NO and NO<sub>2</sub> using Nasicon and polychlor-type oxide electrodes. The potentiometric response was especially sensitive to NO, even in the presence of other gases. Lu et al. (*D13*) described a high-temperature NO<sub>x</sub> sensor based on stabilized zirconia and a spinel-type oxide, CdCr<sub>2</sub>O<sub>4</sub>. The emf was almost linear with log[NO<sub>x</sub>], and good selectivity was obtained in the presence of H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, and H<sub>2</sub>O gases. Asami et al. (*D14*) also investigated zirconia-based sensors for NO and NO<sub>2</sub>, where stabilized zirconia was coupled with indium oxide-based electrodes modified with rare earth metal oxides. Elsewhere, Hibino et al. (*D15*) detected NO in the presence of excess oxygen, water, and carbon dioxide by using a four-cell electrochemical system. Potentiometric and amperometric means were used to detect NO.

A few papers describing SO<sub>x</sub> sensors were published recently. In one study, MgO-stabilized zirconia (MSZ) was used by Muto et al. (*D16*) in the design of a solid-state electrochemical sensor for sulfur dioxide. Li<sub>2</sub>SO<sub>4</sub>/CaSO<sub>4</sub>/SiO<sub>2</sub> was modified with MSZ and then covered with a Pt net to produce a resistive electrode for the detection of SO<sub>2</sub>. In other work, a galvanic SO<sub>2</sub>/SO<sub>3</sub> solid-state sensor utilizing Ag-β-alumina as solid electrolyte was fabricated by Yang et al. (*D17*). The sensor response was tested in high-temperature environments, and there was no observed interference from CO<sub>2</sub> or NO<sub>2</sub>.

Electrochemical sensors for hydrogen were investigated by several workers in the past two years. For example, Fukui and Katsuki (*D18*) developed a semiconductor H<sub>2</sub> gas sensor based on tin oxide and vapor-deposited hexamethyldisiloxane. The device was selective for H<sub>2</sub>, demonstrated “minor” humidity dependence, and exhibited long-term stability. Elsewhere, Lu et al. (*D19*) fabricated a high-temperature hydrogen sensor from stabilized zirconia and a zinc oxide electrode. Excellent selectivity for H<sub>2</sub> in the presence of NO<sub>x</sub>, CO<sub>2</sub>, CH<sub>4</sub>, and H<sub>2</sub>O was claimed, and the device was reportedly stable for long time periods.

A variety of electrochemical sensors for other gases and vapors have been investigated. For instance, Nagasawa et al. (*D20*) reported the development of a highly sensitive and selective sensor

for methane, based on tin oxide thin films. By using an optimized microscopic structure of SnO<sub>2</sub> thin films, along with improved catalytic activity, the electrical response for methane far exceeded that for hydrogen, a common interference. In other research, Pisarkiewicz et al. (D21) designed a semiconductor ozone sensor based on indium oxide thin films doped with Fe or Ce. Elsewhere, Rao et al. (D22) described a rather simple electrochemical sensor for hydrogen cyanide gas. The sensor utilizes a porous membrane gold-poly(tetrafluoroethylene) (PTFE) working electrode for measurement of gaseous HCN at concentrations near the threshold limit value. In other work, Miura et al. (D23) investigated a hydrogen sulfide electrochemical sensor based on a stabilized zirconia oxide sensing electrode. An yttrium oxide-stabilized zirconia substrate was combined with a sensing layer of tungsten oxide to prepare a novel sensor for H<sub>2</sub>S. In another study, Ahmed et al. (D24) fabricated a solid-state sensor for gaseous HCl by using a combination of two solid electrolytes. The device was tested for its potential application in the monitoring of flue gases. Other research by Niizeki and Shibata (D25) described a galvanic chlorine gas sensor that operates at room temperature. A lead chloride/potassium sulfate solid electrolyte (with aluminum oxide for mechanical strength) was employed in combination with a ruthenium oxide sensing electrode. A rapid response time (<10 s) for Cl<sub>2</sub> gas was illustrated, and the stability of the sensor was reportedly in excess of six months. Chlorine dioxide gas in the presence of chlorine was detected by means of a voltammetric membrane electrode in work by Imaizumi et al. (D26). Water and dimethyl sulfoxide (DMSO) were used to collect ClO<sub>2</sub>, and the analyte was determined voltammetrically. While tedious, it offers a means of eliminating Cl<sub>2</sub> interference in the detection of ClO<sub>2</sub>. Huang and Dasgupta (D27) prepared electrochemical sensors for gaseous hydroperoxides based on renewable Nafion thin films. The novel sensor is reportedly inexpensive to fabricate and simple to operate. In other work, Kroll (D28) described gas sensors for toxic and explosive gases based on proton-conductive solid-state electrolytes. Macrocyclic Co(III) complexes with different ligands comprising the counter electrode material allowed for the development of sensors with reportedly excellent performance. Finally, Brailsford et al. (D29) described a "first-principles" model for the measurement of combustibles in air. It was proposed that the design and fabrication of electrochemical sensors for combustible gases could be improved by predicting the behavior of different sensor materials based on published data.

**Optical Sensors.** A wide variety of optical sensors are commonly used in the industrial hygiene field, and research continues in efforts to improve the technology and expand applications. Reports describing the development and evaluation of new or improved optical sensors based on fluorescence, infrared, UV/visible, chemiluminescence, etc., have been published recently. Also (not surprisingly) fiber optics are seeing increased use in the design and fabrication of optical sensors.

In a recent study, Peck et al. (D30) reported a new design for a fiber-optic fluorescence sensor for the detection of fugitive emissions in industrial plants. The device, which incorporates a fluorescent dye, is meant to be used as a chemical dosimeter for various regulated compounds, including VOCs. In other research, King and Webb (D31) described a novel infrared waveguide sensing device for the detection of airborne pollutants, e.g.,

amines. The sensor incorporates a functionalized self-assembled monolayer on the metallized interior surface of a tubular IR waveguide. In this manner, the self-assembled monolayer functions as a medium for the adsorption of target analytes, the IR spectra of which may then be recorded. Elsewhere, Drescher et al. (D32) used an open-path FT-IR system to measure indoor time-dependent tracer gas concentration profiles. A computed tomography technique was employed to maximize sensing abilities of the system. In other work, Murray et al. (D33) used IR sensors to detect leaks of fugitive gases such as methane. A highly sensitive, portable device was developed for proposed applications in a vehicle-mounted optical leak detection systems. In a related investigation, Franks et al. (D34) reported the development of a remote optical sensor for methane for applications in mining. The operation of the device is based on IR radiation and reportedly is an improvement over earlier designs. A miniature solid-state optical waveguide sensor for airborne pollutants was described by Klainer et al. (D35). In this work, fiber optics were employed in concert with different sensing mechanisms (such as absorbance, refractive index, and fluorescence) for the detection of hydrocarbons, CO, O<sub>2</sub>, and pesticides in air. Potential applications for portable, on-site field monitoring were readily apparent.

A novel chemiluminescence sensor for carbon dioxide was developed by Lan and Mottola (D36). These workers described the continuous monitoring of CO<sub>2</sub> by means of an enhanced luminol-Co(II) phthalocyanine chemiluminescence. In another study, Chang et al. (D37) described a chemiluminescence alarm sensor for nickel carbonyl. A very low detection limit (0.5 ppb) and reasonably rapid response time (2 min) were reported. In an interesting application, Dickert and Thierer (D38) used a "molecularly imprinted" polymer as a basis for the fabrication of optochemical sensors for solvent vapors. By intercalation of a dye within a polyurethane substrate, it was possible to measure changes in absorbance due to the adsorption of solvent molecules. Elsewhere, Drummond (D39) described a technique for correcting for variable responses of photoionization detectors (PIDs) to airborne contaminants. PID responses due to partially evaporated hydrocarbon fuels and solvents can be calibrated using a method that is simple to implement. In other work, Karanassios and Sharples (D40) fabricated a microsensor for gaseous "micro-samples" by using {100} crystalline silicon wafers as "micro-channels" for optical signals. The device was applied successfully to the detection of mercury vapor by atomic fluorescence. A novel colorimetric sensor for formaldehyde was developed by Feldbruegge et al. (D41). These workers described an enzyme-based "diffusion badge" that was used in concert with colorimetry for the selective detection of formaldehyde in air. A paper by Miller (D42) detailed the application of fiber-optic detection systems in potentially explosive atmospheres. The focus of this work was on design and installation guidelines for the use of fiber-optic sensors in hazardous locations. In a separate investigation, Tanaka et al. (D43) described a method for sensing carbon disulfide vapor by using a stain tube following diffusive sampling and thermal desorption. In this respect, the procedure functions as a colorimetric optical sensor for CS<sub>2</sub>, and may have applications in worker protection. Elsewhere, Danielsson and Mecklenburg (D44) used an optrode biosensor for detecting airborne organic solvents and heavy metals. The sensing mechanism, which is based on the

Table 3. Other Sensors

topic	authors	refs
methanol biosensor	Neassens and Tran-Minh	D52
formaldehyde/ethanol biosensors	Dennison et al.	D53
immunosensor determination of 2,4-dichlorophenoxyacetic acid	Bier et al.	D54
immunoassay biosensors for PCB	Del Carlo et al.	D55
fluorometric enzyme sensor for ammonia	Subramanian et al.	D56
waveguide interferometric atrazine sensor	Schipper et al.	D57
surface acoustic wave (SAW) array sensors and chemometrics for solvent vapor detection	Boess	D58
SAW organophosphorus dosimetry sensor	Nieuwenhuizen and Harteveld	D59
quartz microbalance and SAW sensors for detection of diesel and solvent vapors	Dickert et al.	D60
chemical composite coupling vapor sensor	Chen et al.	D61
quartz crystal sensor for dust monitoring	Wilson et al.	D62
pyridine sensing using porphyrin probe molecules in polystyrene films	Leray et al.	D63
isocyanate sensing using phthalocyanines	Agbabiaka et al.	D64
quartz microbalance and SAW sensors for nitric acid	Meadows et al.	D65
passive monitoring of methylene chloride	Charron et al.	D66
passive sampling technique for nitrogen dioxide and sulfur dioxide	De Santis et al.	D67

use of nucleic acids and a long-wavelength fluorophore, offers a variety of potential applications. Last, Geyh et al. (D45) described the development and evaluation of an "active" ozone sensor which employs an etched glass denuder as the collection substrate. The collection device can be used in a variety of optical sensing schemes, and its performance compares well with that of commercial ozone sensors.

**Sensor Arrays.** Arrays of chemical sensors, including electrochemical, optical, and other designs, have been applied to problems pertinent to occupational and environmental health. For example, Oyabu et al. (D46, D47) used a multisensor system for the detection of gaseous indoor air pollutants. A computerized multisensor system was described for the detection of combustible gas, NO<sub>x</sub>, ammonia, CO, carbon dioxide, humidity (absolute and relative), and temperature. In other work, Baumgart et al. (D48) described a vehicle-mounted multisensor array system for characterizing hazardous waste sites. Elsewhere, Hoefler et al. (D49) employed tin oxide thin-film sensor arrays for monitoring gas mixtures. By using principal component regression analysis and by varying the contact potential of different sensors in the arrays, it was possible to simultaneously monitor NO<sub>2</sub>, CO, CH<sub>4</sub>, and H<sub>2</sub>O mixtures in the range of the threshold limit values for target analytes. In another investigation, Doleman et al. (D50) used a statistical procedure to evaluate the performance of sensor arrays for classification of responses. The approach was applied to the case of a 14-element array of carbon black-insulating polymer composite chemiresistor sensors and was used to distinguish between 19 solvent vapors. Finally, Onodera et al. (D51) examined the use of four types of tin oxide gas sensors for the detection of various gases in indoor air. The utility of a multisensor system for indoor air monitoring purposes was evaluated.

**Other Chemical Sensors.** Other sensor devices for airborne gases and vapors (neither optical nor electrochemical, per se), such as biosensors, piezoelectric sensors, and immunosensors, have been applied to problems of interest in the industrial hygiene field. Recent papers that fit in this "other" category are summarized in Table 3.

## DUSTS, AEROSOLS, AND FIBERS

Inhalable airborne particulates, whether solid or liquid, are of interest to the industrial hygienist. This section covers publications that deal with the sampling of particulates from the air and the determination of their size distribution and composition. A brief summary of airborne particles was prepared by Harper including a history of regulations, measurement, and monitoring technology (B27). For particles, size distribution is particularly important because size or aerodynamic diameter largely determines whether a particle is inhalable and if it is likely to reach the nasal passages, the airways in the lung, or the lung's alveolar gas-exchange region. Because many air sampling methods ultimately deposit particulates on filters, there is some overlap with the section on Sorbents and Filters. Interested readers should refer to that section and note materials that were collected on filters.

**Real Time Monitoring.** Instruments for real time monitoring of airborne particles have been available for many years. Lilienfeld reviewed the development of instruments based on nephelometry over the last few decades (E1). Karpisek et al. compared the performance of two personal dust monitors (E2). Papers were also published that described a new device for personal sampling and on-line monitoring of inhalable, thoracic, and respirable particles (E3) and a new personal dust monitor based on a piezoelectric quartz crystal sensor (E4). Kaye et al. reported on a real time monitor for respirable particles based on laser light scattering that was said to be capable of detecting 0.1 fiber/cm<sup>3</sup> after a sampling time of only a few seconds (C114). The instrument uses a diode array to sense scattered light and then assigns a particle to a particular class (crocidolite, chrysotile, etc.) based on its scattering characteristics. Lehocky and Williams compared a direct-reading real time aerosol monitor to respirable samplers for monitoring coal dust (E5). Ito et al. reported on a continuous monitoring instrument for strong acid aerosols (E6), and Noble and Prather characterized aerosol particles using an aerosol time-of-flight mass spectrometer (C33).

**Sample Collection.** Techniques and equipment used to collect particles from the air receive special attention because of the degree of difficulty in obtaining samples that are truly representative of ambient air. Two reviews of aerosol samplers were published (E7, E8). Several papers were concerned with the comparison of different aerosol samplers and the comparability of data obtained when sampling an atmosphere with identical samplers (E9–E13). Techniques for assessing the performance of aerosol samplers were the subject of three papers: a simplified method for testing personal inhalable aerosol samplers (E14), a study of how air flow near samplers is effected by the orientation of the inlet orifice and its position near other bodies (E15), and a quantitative study of the decrease in efficiency of glass impingers due to evaporation of the trapping liquid (E16). New designs for personal samplers were presented for cotton dust (E17), for particulates based on diffusive sampling (E18), for respirable and nonrespirable welding fumes (E19, E20), and for total aerosols (E21). The latter was said to correlate well with data obtained from a standard 37-mm cassette but was subject to less variability. Hoke reported on a continuous denuder for sampling acid aerosols and gases (E22). Sioutas et al. described a high-volume rectangular geometry impactor for sampling particles between 0.1 and

2.5  $\mu\text{m}$  (E23). Glass fibers on surfaces were sampled with tape and microvacuum techniques (E24).

**Bioaerosols.** Currently there are many different approaches for the determination of bioaerosols. Reviews were published on methods for sampling (B12, B42, E25) and for measuring (B44, E26) bioaerosols. Pipke reported on German efforts to standardize methods for determining airborne biological materials including yeast and fungi (E27). Reviews also covered sampling and analysis methods for proteolytic enzymes (B45) and for endotoxins (B43). One paper described a field evaluation of different endotoxin sampling and assay methods (E28). Several bioaerosol papers were concerned primarily with the sampling aspect: a comparison of four bioaerosol samplers (E29), an aqueous spinning vortex collector (E30), and a low-energy wetted surface denuder for viable and nonviable aerosols (E31). One paper addressed the difficulty in sampling airborne microorganisms and concluded that assays based on colony-forming units tended to be low due to mechanical and dehydration stress caused by sample collection (E32). The authors advocated assays based on lipid and hydroxy fatty acids as indicators of microorganism mass. Other authors reported general assays based on bioluminescence (E33) and ELISA (E34). Several papers described assays for specific materials: genetically modified organisms by PCR (E35), rat allergens by a sensitive monoclonal ELISA method (E36), and rat urinary proteins (E37).

**Particle Analysis and Characterization.** Four reviews were published on analytical approaches to characterizing particles (B20, B21, B24, E38). Two papers dealt with techniques applied to the characterization of intact particles, one using Auger electron spectroscopy and scanning electron microscopy (SEM) (E39) and the other using ion beam analysis for airborne particles collected as thin films on filters (E40). Elemental analysis of particles was achieved by sequential leaching (E41) and by acid digestion and plasma ashing followed by X-ray fluorescence (C36).

Fiber identification and counting continues to be an area of some interest. Baron discussed the characterization and counting criteria for thoracic fibers (E42). Raman spectroscopy was presented as a useful tool for differentiating different types of asbestos but was applicable only to single fibers (C43). Maddalon et al. compared the use of optical and electron microscopy for asbestos fibers and concluded that optical is more cost-effective and is the basis for most exposure limit regulations worldwide (E43). Zamataro et al. reviewed the development of the Brazilian method for determining airborne fibers, concluding that it differed from other accepted methods only in the counting criteria (E44). Silicate fibers from rock quarries were quantified by electron microscopy (E45). Hengstberger compared the use of phase contrast microscopy and SEM for counting organic fibers (E46). Bahners et al. studied the effect of textile foreign substances on the analysis of fiber dust by both SEM and energy-dispersive X-ray spectroscopy (E47).

Smith reviewed the determination of respirable crystalline silica by X-ray powder diffraction (B19). Peng discussed the performance of a respirable dust sampler for  $\text{SiO}_2$  and compared three methods for  $\text{SiO}_2$  recommended by WHO (E48). Ojima estimated the size distribution of quartz dust by IR (E49). Hearl concluded that reference materials for ambient silica particles smaller

than 10  $\mu\text{m}$  should be developed because modern analysis methods (X-ray, IR) are size-dependent, and current reference materials are based on respirable size particles (C11). Harrison et al. analyzed respirable silica particles for an aluminosilicate clay coating by SEM energy-dispersive X-ray analysis because the coating may reduce the bioavailability of the particles (E50).

Heavy metals are a common constituent of airborne particles and are of significant interest because of their toxicity. Reviews were published on the analysis of heavy metals in environmental particles by ultrasonic extraction (E51) and on ASTM methods for sampling and analysis of lead-containing particles (E52). Millson and Ashley concluded that ultrasonic extraction with dilute nitric acid and analysis by ICP gave acceptable results for lead in a number of environmental reference materials (C23). Ashley reported favorable results using a field-portable analyzer for lead based on ultrasonic extraction and anodic stripping voltammetry (C111). Others also addressed the collection (E53) and characterization (C17) of lead-containing aerosols. Chromium, especially Cr(VI), in aerosols was the subject of two papers (C35, C27). ICP was used to measure platinum (E54) and hafnium (C22) collected on sampling filters.

Other inorganic species that received specific mention were ammonium perchlorate (E55) and sulfuric acid mist (C103). The work on sulfuric acid eliminated interference from other sulfate-containing species by extraction with anhydrous 2-propanol, in which sulfuric acid is highly soluble, leaving behind the insoluble sulfate salts.

Determination of organic materials on particles was the subject of a number of papers. Four dealt with the characterization of diesel exhaust particles (C120, C121, E56, E57). A related paper described an interlaboratory comparison of the determination of elemental and organic carbon in carbonaceous aerosols (C122). Studies of machine shop oil mists exemplified the difficulty in obtaining reproducible, representative samples. Wilsey et al. found higher concentrations of inhalable oil mists on collection filters when using the European IOM sampler than when using the standard 37-mm cassette specified by U.S. authorities (E58). Leith et al. found higher oil mist concentrations using an electrostatic precipitator than when using a filter as a sample collector (E59). They hypothesized sample loss from the filter due to evaporation or migration through the filter pores. The possibility of sample loss was also investigated by Ding and co-workers in the analysis of airborne particles for *N*-nitroso and nitrite-containing organic compounds (E60). They noted that some of the target compounds are volatile and may be stripped from the collected particles during protracted sample collection. Specific compounds measured on airborne particles included pyrethrins by high-resolution GC-ECD (E61), PAHs by ultrasonic extraction and HPLC (E62), and 1-nitropyrene by ELISA and HPLC (C119). A pilot study of children exposed to pesticides via contaminated house dust was published by Bradman and co-workers (E63). This paper reported practical techniques for sampling house dust as well as novel approaches to extraction, analysis, and confirmation of pesticide residues in this complex matrix. House dust has long been recognized as a source of exposure to heavy metals, particularly lead.

Table 4. Methods for Determination of Radon and Radon Progeny

subject	authors	refs
activated charcoal adsorption coefficients for $^{222}\text{Rn}$	Zikovsky	F1
bronchial dosimeter for studying deposition of Rn/Tn progeny in nasal and tracheobronchial regions	Yu et al.	F2
cascade impactor for particle sampling in niobium mines	Da Cunha et al.	F3
coal slag abrasive analysis	Spitz and Rajaretnam, Cox et al.	F4, F5
exhalation rates from solids using solid-state nuclear track detectors (SSNTD)	Singh et al.	F6
filter sampler with improved sample handling and performance characteristics	Parulian et al.	F7
flat phosphate glass pulsed luminescence dosimeter (PLD) and LiF thermoluminescent dosimeter (TLD) comparison	Burghardt et al.	F8
ground exhalation rate measurement with large-scale ZnS(Ag) scintillator	Saegusa et al.	F9
hypersensitive TLD for determination of low doses	Saez-Vergara and Romero	F10
$^{210}\text{Pb}$ in indoor air by total $\alpha$ -measurement	Fisenne et al.	F11
passive local and personal dosimeters in waterworks	Schneider	F12
long- and short-lived Rn progeny in mines based on dust analysis	Holub et al.	F13
portable instruments based on combined $\beta/\alpha$ and Si detectors	Rolle and Lettner	F14
$^{222}\text{Rn}$ progeny by $\alpha$ - and $\beta$ -spectroscopy	Ruzer and Sextro	F15
rotating filter device for radon progeny	Pressyanov et al.	F16
soils $^{222}\text{Rn}$ estimation by combined SSNTD and TLD	Pressyanov et al.	F17
SSNTD: properties, calibration and use	Jonsson	F18
SSNTD and active electronic devices compared	Jonsson et al.	F19
uncertainty in charcoal and $\alpha$ -track Rn monitors	Mushrush et al.	F20
USEPA charcoal sampler modification for active sampling	Yu et al.	F21

### RADIATION DOSIMETRY

The majority of publications in radiation dosimetry are concerned with the determination of naturally occurring radioactivity and, in particular, radon and radon progeny. Advancements in Rn dosimetry are summarized in Table 4. Radon research not noted in the table focused on Rn exposures in various work environments including underground mines (F22–F25), waterworks (F12, F26), show caves (F27, F28), radon spas (F29, F30), and niobium metal works (F3, F31). Studies by Annanmaki et al. in Finland establish that integrating measurements usually overestimate exposures during working hours, especially if mechanical ventilation is used (F32). Mushrush and co-workers reported that uncertainty in any single radon determination can be very high, e.g.,  $\pm 50$ –90%, depending on the technique (F20). The relationship between routine Rn measurements and dose has been discussed by Porstendorfer (F33).

Radon exhalation rates from building materials including phosphogypsum plaster board (F34) and marble (F35) have been determined. Certain granite samples contain high levels of  $^{232}\text{Th}$  and  $^{226}\text{Ra}$ , but the limited use of decorative granite in buildings does not represent a significant radiation hazard according to Chen et al. (F36). Exposures from building materials relate to Rn emanation as well as  $\beta$ - and  $\gamma$ -radiation. Hee and Zerbib reported that welders using tungsten electrodes are exposed to thorium-containing fumes (F37).

Pashchenko and Barboza-Flores have developed a method to correct fluctuations in thermoluminescent dosimeter (TLD) response related to climatic conditions (F38). Alvarez and Navarone recommended sequential determination of actinides ( $^{239}\text{Pu}$  and  $^{241}\text{Am}$ ) and  $^{90}\text{Sr}$  in urine for monitoring workers decommissioning or decontaminating nuclear facilities (F39). A collimated NaI (Tl) scintillation spectroscopy meter provides a low-cost alternative to whole body counters for monitoring  $^{131}\text{I}$  in the human thyroid gland (F40). Whicker et al. evaluated air monitor placement in plutonium facilities using a polydisperse oil aerosol tracer and a laser particle counter detector—the optimal location of emergency release detectors is not at exhaust registers (F41). Ibach and Scheuer described analysis of uranium and transuranium elements in indoor air based on the  $\alpha, \beta$ -pseudocoincidence

difference technique that compensates for interference from Rn daughters (F42). Radioactive Sr in environmental samples has been determined by Abbadi et al. using ion chromatography with an HPLC  $\beta$ -detector (F43).

O'meara and co-workers have investigated in vivo XRF for determination of bone uranium (F44, F45). XRF has adequate sensitivity for monitoring workers with known significant exposures, but it is not sensitive enough for routine occupational monitoring. Exposures of health care workers performing positron emission tomography imaging have been evaluated—the highest doses were associated with positioning patients and administering radioactive drugs in a study by Mcelroy (F46). Cardarelli and co-workers determined external radiation doses in buildings with  $^{60}\text{Co}$ -contaminated structural steel using survey instruments and TLD (F47).

### SORBENTS AND FILTER MEDIA

To measure low concentrations of materials in air or to obtain time-averaged air concentrations, a known volume of air is sampled by a collection device, usually containing a sorbent material or filter, retaining the materials of interest. If air is mechanically pumped through or over the device, it is commonly referred to as an "active" sampler. If the sampler functions by means of convection and diffusion, it is commonly referred to as a "diffusive" or "passive" sampler. After sample collection, the device is then analyzed by an appropriate technique to identify and to quantify the target materials. Papers reviewed in this section deal primarily with new collection devices, new applications of existing devices, or performance evaluations and field studies.

Several papers described general applications of sorbent tubes for sampling VOCs (C73, G1, G2). Tubes filled with multiple sorbents or with different sorbents connected in series were used to collect compounds with a wide range of volatility and functionality (G3–G5). Two papers described sorbent tubes applicable for VOCs in human breath as well as air which were subsequently analyzed by thermal desorption directly into a GC (C54, C55). One paper compared the performance of Carbosieve, activated charcoal, and the porous polymer Tenax for collecting VOCs (G6). Another compared four different solid sorbents for sampling polar

VOCs followed by thermal desorption and analysis GC-FID (C53). McClenny and Colon evaluated EPA method TO-17 for 41 VOCs using synthetic mixtures in purified air and residential indoor air (G7). One author described a combination quartz fiber filter/Tenax tube for sampling semi-VOCs which was said to overcome problems associated with memory effects and poor recoveries at low-nanogram levels (G8). Hallama et al. touted the benefits of thermal over solvent desorption especially when using diffusive samplers (G9). One paper discussed artifacts that could arise due to reactive species and sorbent degradation when Tenax and thermal desorption was used for measuring low concentrations of VOCs in indoor air (C61). In one novel application, a charcoal tube was attached to the outlet of a real time air monitor based on a nominally nondestructive photoionization detector (D39). After obtaining instantaneous composite VOC concentrations in the field, which can be useful for identifying emission sources and exposure areas, the tube can be analyzed in the laboratory to identify compounds and to calculate average calibration factors.

Although the vast majority of industrial hygiene air-sampling methods developed by OSHA and NIOSH rely on active sampling devices, diffusive samplers are generally accepted alternatives and are often preferred due to their small size and ease of use. Harper and Guild discussed the use of the NIOSH diffusive sampler protocol to validate sampler performance (G10). Others described the effect of face velocity (G11), subzero temperatures (G12), and humidity (G13) on sampler performance. Pengelly et al. highlighted the dependence of a diffusive sampler's effective sampling rate on diffusion coefficients (G14). They demonstrated that active and diffusive samplers can produce different results for formaldehyde depending on whether pure formaldehyde monomer or a mixture of monomer and other species (arising from monomer equilibration with water vapor and methanol stabilizer) is sampled.

Several papers dealt with VOC surveys using diffusive samplers (B11, C58, G15–G17). Several authors advocated the use of diffusive samplers for long-term monitoring and assessing indoor air quality (G18–G20). Kawamoto investigated the performance of a diffusive sampler for chlorinated VOCs at low-ppb concentrations and found an inverse relationship between sampling rate and relative humidity (G21). Because they require no power source, diffusive samplers will continue to find applications for long-term monitoring at low concentrations. However, rigorous validation of these devices for extended monitoring periods (>24 h) and at sub-ppb concentrations still remains an opportunity for future research.

A new high sampling rate diffusive sampler amenable to both solvent and thermal desorption was described (G22). Another new design incorporated provisions for automated injection into a GC following in situ solvent desorption (G23). Still another new design allowed for microwave desorption directly into a GC (G24). Other papers described diffusive samplers designed to be worn underneath personal protective equipment to assess personal exposure and thereby assess the effectiveness of the equipment (G25, G26).

A number of authors cited alternatives and noted their advantages to conventional practice in certain situations. Diethyl ether was suggested as an effective and a more "friendly" solvent than the more universal CS<sub>2</sub> when a mass spectrometer was used as a detector (G27). A mixture of DMSO and CS<sub>2</sub> was recommended as an effective general purpose desorption solvent when

a mixture of polar and nonpolar compounds (G28) was being analyzed. Anasorb 747, a beaded activated carbon, was proposed as a more universal sorbent than the commonly used charcoal (B12). Advantages were cited to using hot 2-hydroxypyridine as a desorption solvent when for certain volatile organic solvents were analyzed (G29).

Two papers described miniature Summa canisters (evacuated canisters with passivated surfaces) for use as personal sampling devices (C89, G30). These devices, almost a hybrid between an active and a diffusive monitor, are engineered with small low-flow controllers and are said to be capable of sampling at constant flow rates for periods exceeding 40 h.

Applications of sorbents and filters for specific materials are arranged alphabetically by analyte in Table 5 for organic compounds and in Table 6 for inorganic compounds. Active samplers were used unless noted for the organic compounds, and diffusive or passive samplers were used in all cases for the inorganic compounds unless noted.

## BIOLOGICAL MONITORING

Biological monitoring is an increasingly important approach used to ascertain exposure levels in workers. Biological samples such as exhaled breath, urine, or blood are obtained at an appropriate time after exposure. Chemical residues known as biological monitoring "determinants" are compared to the biological exposure index (BEI), the expected concentration under acceptable exposure levels.

Biological markers provide information on the internal chemical dose and integrate exposures from all routes including inhalation, dermal, and oral pathways. Air monitoring, even when samples are taken in the worker's breathing zone, provides an estimate of the exposure potential from only one pathway. There are numerous examples where cutaneous absorption of chemicals in the workplace predominates, and the recent literature provides much additional evidence of this. Biological monitoring is particularly useful for evaluating the efficacy of exposure prevention strategies, differences in exposures associated with particular tasks or processes, and even differences in hygienic practices among workers.

Chemicals are initially absorbed into the circulatory system; thus chemical analysis of blood is of fundamental importance in pharmacokinetic studies. Blood analysis is advantageous since the parent chemical can be measured directly, sometimes using very straightforward techniques—for example, analysis of volatile chemicals in blood by headspace gas chromatography. While important in biological research, blood sampling in the workplace is invasive, requires a trained phlebotomist, and has other drawbacks. Most determinants are urinary metabolites present in end-of-shift or first void urine samples. Variations in the urine output must be controlled, usually by relating the urinary determinant concentration to creatinine or specific gravity. Excretion in the urine is often preceded by either phase I (oxidation, hydrolysis) or phase I and phase II (conjugation) metabolism which increases a chemical's polarity and water solubility. In phase II metabolism, functional groups are conjugated with glucuronic, sulfuric, or amino acids. The enhanced polarity, which facilitates excretion, is generally unattractive from the viewpoint of the analytical chemist who cleaves the conjugate moiety by enzymatic

Table 5. Sorbent Media for Organic Compounds

compound or analyte	sorbent (sampler)	analysis/comments	refs
acrylamide	charcoal	acetone desorption, GC-FID	G31
alcalase	filter	rapid ELISA	G32
alcohols	1-butanol impinger		C63
aldehydes	silica gel coated with pentafluorobenzyl hydroxylamine	GC-ECD	C69
aldehydes	various	European interlab study	G33
amines	cation-exchange resin	GC/MS, FT/IR	G34
amines	impingers with ethyl chloroformate	HPLC/MS	C101
anhydrides (cyclic)	various	GC-FID, GC/MS	C62, G35
antibiotics	filter	direct on-filter ELISA	G36
aromatic hydrocarbons	diffusion denuder		G37
asphalt fumes		comparison of NIOSH 5023 and OSHA 58	C94
bromomethane	Tenax	GC-PID	G38
2-bromopropane	carbon cloth (diffusive sampler)		G39
1,3-butadiene	(diffusive sampler)	thermal desorption	C78
carbon disulfide	porous polymer (diffusive sampler)	thermal desorption, length of stain detection	D43
ceftazidime	filter	rapid ELISA	G32
chemical warfare agents (GB, HD, VX)		dual-column GC, heart cut, MS and FP detection	G40
chlorothalonil	various	GC-ECD or MS	G41
coal tar pitch volatiles	filter and graphitized carbon	HPLC or GC/MS	C86
cyanogen chloride		GC in mobile laboratory	C68
cyclosiloxanes		purge and trap GC/MS	G42
dibenzothiazole disulfide	perchloroethylene filter	spectrophotometric analysis	G43
dichloflumid	various	GC-ECD or MS	G41
diisocyanates	glass fiber filter coated with pyridylpiperazine		G44
4,4'-diphenylmethane- diisocyanate (MDI)	glass fiber filter coated with pyridylpiperazine	HPLC	C99
toluene diisocyanates (TDIs)	glass fiber filter coated with pyridylpiperazine	HPLC	G45
TDIs	impinger with dibutylamine or methylamino- methylanthracene	HPLC	C102
TDIs	impinger with triethanolamine and pentafluoro- benzyl alcohol	GC-ECD	C66
TDIs	Tenax TA	thermal desorption, GC-FID or ion trap GC/MS	C72
2,3-dimethyl-2,3-dinitrobutane	Tenax	ethyl acetate desorption, GC-ECD	C75
dimethyl and diethyl sulfate	Tenax TA	thermal desorption, GC/MS	C76
dimethyl sulfide	porous polymer	capillary GC	G46
endosulfan and endosulfan sulfate	various	GC-ECD or GC/MS	C45
3-ethenylpyridine	various	capillary GC and HPLC	G47
formaldehyde	glass fiber filter coated with Girard's reagent T	detection limit of 1 $\mu\text{g}/\text{m}^3$ for 30-L sample	C113
formaldehyde	enzyme (diffusive sampler)	colorimetry	G48
formaldehyde	impinger with acetylacetone or DNPH	spectrophotometry or HPLC	G49
formaldehyde	water	derivatized with DNPH, GC-ECD	G50
formaldehyde	(active/diffusive samplers)	DNPH and AHMT compared	G51
formaldehyde	SPME fiber coated with pentafluorobenzylamine hydrochloride	GC-FID	C48
glutaraldehyde	(active/diffusive samples)	comparison of four methods	G52
glycols and glycol ethers	XAD-7	methylene chloride desorption, GC/MS	G53
imidachloprid	Tenax		G54
indole and methyl indole	alkaline porous silica gel	GC-FID	G55
isocyanates	impinger with anthracenylmethylpiperazine	comparison to NIOSH 5521	C98, C97
isocyanates	dibutylamine impingers	LC/MS or GC/MS	C100, C101
isocyanates	impinger with butyl acetate and isooctane	advantages over NIOSH 5522 cited	G56
isoprene	porous polymer	capillary GC	G46
ketones	1-pentanol impinger		C63
lindane	various	GC-ECD or GC/MS	C45
methylene chloride	(diffusive sampler)	field validation of different samplers	D66
nicotine	treated filter	capillary GC-FID	G57
nicotine	various	capillary GC or HPLC	G47
N-nitrosoethylphenylamine and active methylphenylamine	denuder with triethanolamine and tetraethylene glycol	GC-thermal energy analyzer	G58
ondansetron	filter	rapid ELISA	G59
pentachlorophenol	(diffusive sampler)	enzyme immunoassay	G60
N-containing pesticides	filter/XAD-2	HPLC-UV	C91
phenols	C <sub>18</sub> SepPak	HPLC-FLD	C93
phosphate esters	glass fiber filters/PUF	ultrasonic extraction	G61
phosgene		GC in mobile lab	C68
PAH		monoclonal antibody immunoassay	G62
PAH		review	G63
procymidone	PUF	GC-ECD or GC/MS	G64
pyridine	activated charcoal	methylene chloride desorption, GC-FID	G65
styrene and substituted styrenes	(diffusive samplers)	evaluation of 2 samplers	G66
organotin compounds		GC-AED	C65
trihalomethanes	XAD	desorbed with pentane, GC-ECD	G67
vinclozolin	PUF	GC-ECD or GC/MS	G64

Table 6. Sorbent Media for Inorganic Compounds

compound/analyte	sorbent	analysis/comment	refs
ammonia	phosphoric acid-impregnated filter	spectrophotometric analysis	G68
ammonia		fluorometric enzyme assay	D56
ammonia	various	evaluation of different samplers	G69
arsenic oxides	filter (active sampler)	colorimetric analysis	G70
hydrogen cyanide	(active sampler)	GC in mobile lab	C68
mercury vapor	charcoal		G71
metals and metalloids	glass wool at -175 °C (active sampler)	GC-ICPMS	G72
NO <sub>2</sub>			G73
NO <sub>2</sub>	triethanolamine solution		G74
NO <sub>2</sub>		5 ppb detection limit for 24-h sample	D67
NO <sub>2</sub>		evaluation of two samplers	G76
NO <sub>2</sub> and NO <sub>x</sub>		review	G75
NO <sub>x</sub>		improved stability with ionic surfactant	G77
ozone	KI-treated paper	colorimetric analysis	G78
ozone	sodium nitrite and others		G79
ozone	nitrite-coated denuder (active sampler)		G80
SO <sub>2</sub>			G73
SO <sub>2</sub>	triethanolamine solution		G74
SO <sub>2</sub>		10 ppb detection limit for 24-h sample	D67

or chemical hydrolysis and forms derivatives with desirable separation and detection characteristics.

Industrial hygiene monitoring often involves the use of portable or hand-held monitoring devices. The determination of biomarkers, particularly metabolites, requires a well-equipped laboratory with capabilities for sample preparation, chemical manipulation and cleanup, and quantitative analysis using spectroscopic and chromatographic techniques. Immunoassays are being developed that offer accessibility—the fundamental techniques required are well within the capability of minimally equipped laboratories. Immunochemical techniques can be accomplished with the same procedures used for over-the-counter clinical test kits such as those available for blood glucose, pregnancy, and HIV status.

Aside from exposure measurement, biomonitoring can provide additional information such as susceptibility or genetic predisposition to disease and evidence of disease processes at a very early or preclinical stage. The goals of a health and safety program first and foremost, however, are prevention of disease and promotion of safe work practices.

Over the past several years, research in all aspects of biological monitoring has been very active. We focus here only on developments in analytical chemistry of exposure markers. Because effect markers serve as convenient surrogates for exposure, some of the advances in this area of research are noted. Routine techniques used widely in exposure and epidemiologic research and monitoring occupational and ecological exposures (e.g., determination of blood Pb) have been omitted for the most part. Because of the large number of published papers, key work has been tabulated according to organic compounds (Table 7), pesticides and pharmaceuticals (Table 8), miscellaneous biological markers (Table 9), and metals (Table 10).

A comprehensive volume dealing with biological monitoring in occupational hygiene appeared (*H130*)—this volume, edited by Mendelsohn, Pelters, and Normandy, includes chapters covering ethical and legal aspects and study design as well as technical developments. Biological monitoring in occupational and environmental health also was the subject of a meeting held in Finland in 1996. Papers from this meeting were published in a single journal volume (*H131*). A review of quality assurance issues in biological monitoring was published by Menditto (*H132*). The

fundamentals of pharmacokinetics and their relationship to the timing of biological monitoring sampling was considered in a useful report by Fiserova-Bergerova and Vlach (*H133*).

Research into new biological monitoring techniques usually addresses specificity and dietary confounding. Metabolites may arise from more than one chemical, the chemical of interest may occur in the diet, or the chemical specificity of the technique may be limited. Improved specificity for measurement of low-level benzene exposure has been demonstrated with *S*-phenylmercapturic acid (*H11*, *H14*, *H15*, *H134*, *H135*) and *trans,trans*-muconic acid (*H12*, *H14*, *H134*–*H136*). *o*-Cresol is reported to be a specific marker of toluene exposure (*H48*, *H49*, *H134*, *H137*–*H140*). Hippuric acid is not a recommended exposure marker due to dietary interferences or those from pharmaceuticals. In the case of low-level or “ecological” benzene biomonitoring (e.g., in the general population), debate continues as to whether phenylmercapturic (*H135*) or muconic acid (*H14*) is more specific.

Cigarette smoke is a tremendously complex mixture containing many toxic substances and chemicals regulated in the occupational environment. Accordingly, the impact of active and passive smoking on biomarkers must be ascertained in their development. In the past several years we have learned that cigarette smoke contains acrylamide (*H1*)—in addition to acrylonitrile and ethylene oxide known previously—and styrene oxide (*H44*). A study by Karita et al. correlated blood lead with lead in facial skin wipes and fingernails (*H141*). Using electron microscopic techniques, the authors identified lead-containing particles on the skin which were associated with exposure via the oral route.

Urinary cobalt is a useful determinant for occupational cobalt exposures including those incurred by workers wet sharpening hard metal and stellite blades (*H113*). Linnainmaa and Kilunen observed that smoking habits, beer drinking, and vitamin B<sub>12</sub> dietary supplements are confounders. Moreover, as Co is absorbed through the skin, personal hygiene also affects urinary Co concentrations. Similarly, Valkonen and Aitio found that antacid drugs affect aluminum concentrations in the urine and serum (*H104*).

As natural product chemists can attest, secondary plant metabolites are numerous and varied in chemical structure. De Rooij and co-workers reported that garlic consumption and occupational exposure to 3-chloroprene both yield allylmercapturic

Table 7. Biological Monitoring Methods. Organic Compounds

compound	analysis/matrix	authors	refs
acrylamide, acrylonitrile, ethylene oxide	Hb adducts determined by modified Edman degradation procedure	Bergmark	H1
acrylonitrile	Hb adduct by GC/MS and ELISA	Wong et al.	H2
acrylonitrile, ethylene oxide	Hb adducts by SIM-GC/MS, adducts characterized by NMR, FAB, and ESP-MS	Lawrence et al.	H3
acrylonitrile, ethylene oxide, methylating agents	Hb adducts by SIM-GC/MS	Van Sittert	H4
aflatoxin	aflatoxicol in urine by HPLC-FLD after immunoaffinity column cleanup	Kussak et al.	H5
aflatoxin B1	aflatoxin M1 in urine by coupled SPE-HPLC	Simon et al.	H6
aflatoxin B1	serum albumin adducts by radioimmune assay	Kensler et al.	H7
alcohols, ketones	headspace GC-FID (blood, urine)	Angerer and Gundel	H8
allyl chloride	allylmercapturic acid by GC/MS	de Rooij et al.	H9
aromatic amines	headspace SPME GC/MS (urine, blood, milk)	De Bruin et al.	H10
benzene	mercapturic acid by GC/MS	Muller and Jeske	H11
benzene	urinary <i>trans,trans</i> -muconic acid by HPLC	Angerer et al.	H12
benzene	phenylmercapturic acid by HPLC after isolation by immunoaffinity chromatography	Ball et al.	H13
benzene	phenylmercapturic and <i>trans,trans</i> -muconic acids (urine)	Boogaard and Van Sittert	H14
benzene, toluene	mercapturic acids by GC-ECD	Einig et al.	H15
benzene, VOC	cumulative dose estimate with PBPK	Roy and Georgopoulos	H16
butadiene	Hb adducts by NCI-CI-MS	Richardson et al.	H17
chlorinated solvents	bile acids in serum by HPLC	Neghab et al.	H18
chloroform	real time breath monitor with ion trap MS <sup>2</sup>	Gordon et al.	H19
dichlorobenzidine	Hb adducts by NCI-GC/MS	Joppich-Kuhn et al.	H20
dimethylacetamide	monomethylacetamide in urine	Spies et al.	H21
4,4'-diphenylmethane diisocyanate (MDI)	serum-specific IgG antibodies to an MDI-albumin conjugate by immunoassay	Lushniak et al.	H22
epichlorohydrin	mercapturic acid by GC/MS	de Rooij et al.	H23
ethoxypropanol isomers	ethoxypropanol or propanoic acid metabolite in urine	Bader et al.	H24
ethylene glycol monomethyl ether or its acetate	urinary alkoxyacetic acids	Vincent et al.	H25
hexahydrophthalic anhydride (HHPA)	Hb adducts by NCI-GC/MS	Lindh and Jonsson	H26
HHPA	urinary hexahydrophthalic acid and Hb-HHPA adducts by GC/MS	Jonsson et al.	H27
hexamethylene diisocyanate	urinary hexanediamine from acid hydrolysis by GC/MS	Maitre et al.	H28
2-methoxyethanol, 2-ethoxyethanol	alkoxyacetic acid metabolites in urine	Kezic et al.	H29
1-methoxy-2-propanol	urinary 1,2-propanediol by capillary GC	Laitinen et al.	H30
methyl <i>tert</i> -butyl ether	MTBE and <i>tert</i> -butyl alcohol metabolite in blood and breath	Buckleley et al.	H31
monoterpenes	verbenols ( $\alpha$ -pinene metabolites) in urine	Eriksson et al.	H32
<i>n</i> -heptane	urinary 2-heptanone, valerolactone, and 2,5-heptanedione by GC/MS	Sturaro et al.	H33
<i>N</i> -methyl-2-pyrrolidone	<i>N</i> -methylsuccinimide and <i>N</i> -methyl-2-hydroxysuccinimide in urine by NCI-GC/MS	Jonsson and Akesson	H34
PAH	1-hydroxypyrene in urine by HPLC	Bentsen et al.	H35
PAH	plasma benzo[ <i>a</i> ]pyrene-albumin adducts by ELISA	Kure et al.	H36
PAH	hydroxypyrenes and phenanthrenes in urine by coupled SPE-HPLC	Mannschreck et al.	H37
PAH	urinary 1-hydroxypyrene	Jongeneelen	H38
PCB	plasma PCB by GC-ECD and ELISA	Griffin et al.	H39
PCB and PCDD/F	hair analysis demonstrates potential	Schramm	H40
PCDD/F, coplanar PCB	isotope dilution GC/high-resolution MS serum analysis	Gilroy et al.	H41
styrene	mercapturic acids (four diastereoisomers) by HPLC-FLD	Maestri et al.	H42
styrene	mercapturic acids by HPLC-FLD	Maestri et al.	H43
styrene oxide (SO), styrene	DNA and albumin adducts in blood are employed as SO-specific biomarkers	Rappaport et al.	H44
TCDD	Analysis of blood samples from Seveso incident reveals serum half-life of 7 yr	Needham et al.	H45
TCE	trichloroacetic acid in urine	Goh et al.	H46
TDI- and MDI-based polyurethane thermal decay products	corresponding amines from hydrolysis of urine and plasma by NCI-GC/MS	Dalene et al.	H47
toluene	urinary <i>o</i> -cresol and alveolar toluene	Tardif et al.	H48
toluene	toluylmercapturic acid	Angerer et al.	H49
toluene	headspace isotope dilution GC/MS (breath, blood)	Pierce et al.	H50
toluene diisocyanate (TDI)	2,4- and 2,6-toluenediamines from urine hydrolysis by isotope dilution GC/MS	Tinnerberg et al.	H51
toluene diisocyanates	plasma albumin adducts by NCI-GC/MS	Lind et al.	H52
1,2,4-trimethylbenzene (TMB)	TMB in blood and urinary 3,4-dimethylhippuric acid by HPLC	Jarnberg et al.	H53
trinitrotoluene (TNT)	TNT's major urinary metabolite, 4-amino-2,6-dinitrotoluene	Robert et al.	H54
VOC	static headspace GC-PID/ECD/FID (blood)	Schroers and Jermann	H55
VOC	SAW sensor array with breath preconcentration	Groves et al., Groves and Zellers	H56, H57
VOC	single-breath canister sampling with GC/MS determination	Pleil and Lindstrom	H58
xylene	dimethylphenol, methylbenzyl alcohol, toluic acid, and methylhippuric acid in urine and blood by ID-GC/MS	Pyon et al.	H59

Table 8. Biological Monitoring Methods. Pesticides and Pharmaceuticals

compound	analysis	authors	ref
atrazine	mercaptopuric acid by a commercial immunoassay kit	Brady et al.	H60
atrazine	atrazine and dealkylated chlorotriazine metabolites in urine by GC/MS-SIM	Bhodalbhai et al.	H61
atrazine	atrazine determined by ELISA in whole saliva and arterial plasma correlate in the rat	Lu et al.	H62
benomyl	5-hydroxy-2-benzimidazole carbamate metabolite in urine	Hoekstra et al.	H63
chlorophenols	SPME-NCI-GC/MS of urine	Lee et al.	H64
chlorpyrifos	plasma mean cholinesterase activity	Burns et al.	H65
creosote	urinary 1-naphthol from naphthalene, the most abundant compound in creosote vapor	Heikkila et al.	H66
cyclophosphamide	probe for occupational exposure to antineoplastic drugs (urine)	Sessink et al.	H67
DDT	workers applying pesticide in malaria mosquito control exhibit levels between 10 and 300 ppm in adipose	Rivero-Rodriguez et al.	H68
dichlorvos	urinary dimethyl phosphate levels correlate with drop in serum cholinesterase activity	Okuno et al.	H69
fluorouracil	$\alpha$ -F- $\beta$ -alanine in urine by GC/MS/MS	Bos et al.	H70
HCH and DDT	surface extracted skin lipids or blood	Dua et al.	H71
hexachlorobenzene, octachlorostyrene	parent compounds in plasma by GC/MS	Selden et al.	H72
lindane	blood lindane	Jung et al.	H73
methyl bromide	compound in blood or urine	Acuna et al.	H74
OP insecticides	urinary alkyl phosphate metabolites by GC-FPD after SPE and derivatization	Apra et al.	H75
paraquat/diquat	capillary electrophoresis of urine	Wu and Tsai	H76
parathions	<i>p</i> -nitrophenol in urine	Yeung et al.	H77
pesticide screen	biomarkers from 20 or more pesticides are determined by isotope dilution GC/MS/MS after enzyme digestion, extraction and derivatization (urine)	Shealy et al.	H78
phenoxy herbicides	HPLC-DAD or GC-ECD of pentafluorobenzyl derivatives from urine	Apra et al.	H79
phenoxy herbicides	urinary residues isolated by SPE	Thompson and Treble	H80
phenylmercury chloride	mercury in urine—increased elimination after administration of complexation agent	Gombos et al.	H81
Pt-based antineoplastic drugs	plasma or leukocyte Pt by ICPMS	Webster et al.	H82
propanil	3,4-dichloroaniline—Hb adducts by GC/NCI-MS—detected 4 months postexposure	Pastorelli et al.	H83
propoxur	2-isopropoxyphenol in urine	Mueling et al.	H84
pyrethroid screen	carboxylic acid metabolites are determined at parts-per-trillion concentrations by GC/high-resolution NCI-MS	Leng et al.	H85
pyrethroid screen	determination of carboxylic acid metabolites by stereoselective capillary GC/MS following acid hydrolysis, extraction, and esterification	Kuehn et al.	H86
pyrethroids	pyrethroid in blood by GC-ECD, urinary carboxylic acids metabolites by GC/MS	Leng et al.	H87
pyrethroids	3-phenoxybenzoic acid metabolite in urine	Choi et al., Apra et al., Tuomainen et al.	H88, H89, H90
ribavirin	ribavirin in urine	Shults et al.	H91
zinc phosphide	serum Zn and Ca elevated on exposure	Amr et al.	H92

Table 9. Biological Monitoring Methods. Miscellaneous

compound	analysis	authors	refs
ammonia	breath by tunable diode laser spectroscopy	Moskalendo and Nadezhdinskii	H93
carbon disulfide	urinary 2-thiothiazolidine-4-carboxylic acid (TTCA)	Cox et al.	H94
carbon monoxide	breath by portable electrochemical cell or "length of stain" tubes	Kuster and Sherwood	H95
cigarette smoke	urinary cotinine by HPLC-APCI MS/MS	Bernert et al.	H96
cigarette smoke (smoking status)	colorimetric assay of nictotinic metabolites in urine	Smith et al.	H97
musk xylene	GC-ECD and GC/MS—detected in blood from 92% of subjects from unknown origin	Angerer and Kafferlein	H98
oleandrin	apparent digitoxin in serum by immunoassay	Dasgupta and Hart	H99
poppy seeds	urinary thebaine by ion trap GC/MS	Casella et al.	H100
sarin	methylphosphonates by GC-FPD urine analysis	Minami et al.	H101
VX	ethyl methylphosphonic acid in serum by GC/MS	Katagi et al.	H102
xenoestrogens	progress toward determination of "estradiol equivalents" based on bioassay of serum	Soto et al.	H103

acid in urine (H9). Thus, excessive garlic intake is a potential confounder of allyl chloride exposure based on this exposure marker. Bastos De Sigueira and co-workers studied reference values for urinary and plasma thiocyanate (a biological marker for cyanide exposure)—age, sex, and alcohol were not critical, but intake of cooked cassava caused a sharp increase in plasma thiocyanate (H142).

As the feasibility of biological markers is investigated, particularly in controlled exposure studies with human subjects, there

has been an increasing understanding that intraindividual differences may be substantial. In some cases, differences are conferred by a single genetic trait or polymorph. Kawamoto and Kodama (H143) studied the affect of aldehyde dehydrogenase polymorphs on urinary hippuric acid (HA), an exposure marker for toluene. When toluene inhalation exposures were matched, the urinary HA levels are significantly lower in two of the three possible genotypes. A second Japanese study (H144) examined the influence of acetylation phenotypes on the disposition of the weak

Table 10. Biological Monitoring Methods. Metals

metal	analysis/matrix	authors	refs
Al	platform GFAA with Zeeman background correction after dilution with nitric acid (urine or serum)	Valkonen and Aitio	H104
As	arsenic metabolites in urine (inorganic, monomethylarsonate, dimethylarsinate) by hydride cold trapping method	Ng et al.	H105
AsH <sub>3</sub>	MMA, DMA, As <sup>3+</sup> , As <sup>5+</sup> , and arsenobetaine in urine by LC/ICPMS	Apostoli et al.	H106
Cd	intraindividual and analytical variation studied (urine)	Mason et al.	H107
Cd	current urinary Cd predicts kidney and liver burden determined by in vivo XRF	Boerjesson et al.	H108
Cd	metallothionein in lymphocytes by flow cytometry	Yurkow and Makhijani	H109
Cd	metallothionein mRNA correlates with blood Cd levels	Taioli et al.	H110
Cd	in vivo neutron activation analysis of kidney and liver	Gerhardsson et al., Perrin et al.	H111, H112
Co	urinary Co confounders include smoking and vitamin B <sub>12</sub> from dietary supplements and beer—cobalt absorbed through the skin	Linnainmaa and Kiilunen	H113
Hg	atomic fluorescence following breath preconcentration by amalgamation	Sallsten and Nolkrautz	H114
Mo	adsorption voltammetry after oxidative digestion of urine	Seiler and Seiler	H115
Cr	erythrocytes and lymphocytes provide a Cr <sup>6+</sup> exposure marker—urine and whole blood or plasma reflect total Cr exposure	Mikshe and Lewalter, Lukanova et al.	H116, H117
Pb	ICPMS after sample dilution without clean room facility (plasma or whole blood)	Schutz et al.	H118
Pb	ICP- high-resolution MS, bias attributed to subtle details of methodology (plasma, serum)	Smith et al.	H119
Pb	in vivo XRF—historical blood lead can be estimated from bone Pb and information on exposure duration	Bergdahl et al., Hanninen et al., Boerjesson et al., Boerjesson et al.	H120, H121, H122, H123
Pb, Cd, Zn, Cu	differential pulse anodic stripping voltammetry after microwave digestion of hair samples	Wasiak et al.	H124
Pb, Zn	hair analyzed in cross section by synchrotron radiation XRF imaging microprobe	Shimojo et al.	H125
Pt	adsorptive voltammetry following UV photolysis of urine	Schierl et al.	H126
Si	GFAA with nickel chloride matrix modifier after dilution of urine or blood	Kobayashi et al.	H127
silica and silicate dusts	BAL samples by electron microscopic microanalysis	Monso et al.	H128
multielement	teeth analysis by ICP-AES with MSA	Capota et al.	H129

carcinogen hydrazine. Using polymerase chain reaction technology, Koizumi and co-workers established the phenotypic frequency in 297 male workers—the phenotype frequency, as well as the biological half-life of hydrazine, was distinctly different among Asian and Caucasian groups.

Temporal studies are critical in the development of each biological monitoring determinant for use in monitoring workers. The relationship between exposure concentration, the concentration in the workplace air regulated by the TLV, and the determinant concentration in the biological matrix is of central importance. Wu and co-workers investigated coke oven emissions and urinary 1-hydroxypyrene (1-HP) (H145). A 10-fold increase in occupational exposure to coke oven emissions (measured as the breathing zone, benzene-soluble fraction) resulted in a 2-fold increase in daily postshift urinary 1-HP. During subsequent work periods the urine concentrations increased, stressing the need to obtain biological samples before and after the work shift.

The first report of a mercapturic acid from toluene in humans was noted in the literature by Angerer et al. (H49). Mercapturates are exposure biomarkers for many other chemicals in the workplace including acrylonitrile (H146), allyl chloride (H9), atrazine (H60), butadiene (H147), epichlorohydrin (H23, H148), and styrene (H42, H43, H149, H150). Low detection limits for mercapturic acids are afforded by use of fluorescent *o*-phthalaldehyde derivatives determined by HPLC-FLD (H42, H146, H149).

Adducts to macromolecules in the blood are used increasingly as biomarkers. Hemoglobin (Hb) adducts have been used in determination of exposure to acrylamide (H1), acrylonitrile (H1,

H3, H4), 1,3-butadiene (H17), 3,3'-dichlorobenzidine (H20), *N,N*-dimethylformamide (H151), ethylene oxide (H1, H3), ethene (H152), and propanil (H83). Albumin adducts are useful in biological monitoring of toluene diisocyanate (H52) and styrene (H44).

Some of the biomonitoring techniques are not readily accessible due to requirements for highly trained technicians or sophisticated instrumentation. As an example, Hb adducts from hexahydrophthalic anhydride exposure are determined by lysing red blood cells, dialysis, acid hydrolysis, LLE/SPE, and derivatization prior to instrumental analysis by isotope dilution ammonia NCI-GC/MS (H26). Immunochemical methods improve accessibility. For example, immunoassays have been reported for aflatoxin—(H7) and benzo[*a*]pyrene—(H36) albumin adducts and acrylonitrile—Hb adducts (H2). Brady and colleagues reported that a commercially available test kit was useful for determination of atrazine's urinary mercapturic acid metabolite (H60).

While immunological methods are potentially inexpensive and sensitive, cross-reactivity is occasionally observed. Drug screens for opiates can give false positives for poppy seed consumption (H100)—Cassella et al. confirmed poppy seeds by determination of urinary thebaine by ion trap GC/MS. Rifampicin also has been found to give false positives in screening (H153). Cross-reactivity has been exploited by Dasgupta and Hart in diagnosing oleander poisoning (serum oleandrin) using an immunoassay developed for another cardiac glycoside, digitoxin (H99). Immunological methods have been reported for anabolic steroids in bovine hair (H154), domoic acid (H155), dexamethazone in equine urine

(H156), ochratoxin (H157), and polychlorinated biphenyls in blood plasma (H39). Immobilized antibodies have been used in the enrichment of *S*-phenylmercapturic acid (H13) and aflatoxicol (H5) from urine.

Developments in analytical instrumentation have influenced biological monitoring. Static headspace sample introduction is advantageous for determination of volatiles in blood and urine. There is minimal sample preparation, and many sample matrix problems are avoided such as foaming in purging liquid samples. The headspace technique has been used in the determination of alcohols and ketones (H8, H158, H159) and volatile aromatics (H50) and in screening VOC exposures in the background or "normal" concentration range (H55). An increasingly popular technique, solid-phase microextraction (SPME), has been used in conjunction with headspace sampling in determination of urinary toluene and xylene (H160) and aromatic amines in urine, blood, and milk (H10). To improve the partitioning of polar compounds into the headspace, Debruin et al. modified the matrix by adjustment of pH or ionic strength or by heating the liquid—thus, sub-parts-per-billion detection limits have been achieved for substituted amines in biological samples (H10). SPME also has been used in sampling breath for ethanol, acetone, and isoprene (H161) as well as chlorophenols in urine (H64).

GC/MS is among the most useful analytical techniques in biological monitoring. In many cases though, biological samples require extensive preparation to obtain the final extract. In such cases, isotope dilution is the preferred quantitation technique. Shealy and colleagues reported ~100% accuracy in a multiresidue screen for metabolites of 20 pesticides in human urine (H78). Obtaining isotope-labeled compounds, particularly metabolites, is a constraint. Deuterated xylene metabolites (dimethylphenol, methylbenzyl alcohol, methylhippuric acid) have been prepared by dosing rats with xylene- $d_{10}$  (H59). Isotope dilution has been used in GC/MS determination of hexahydrophthalic anhydride-Hb adducts (H26). Isotope dilution substantially improves quantitation accuracy in the HPLC/MS determination of styrene metabolites in urine (H162). The improved accuracy in particle beam LC/MS is attributed to increased linear dynamic response according to Draper and co-workers. Headspace GC/MS determination of toluene in blood also has benefited from use of deuterated internal standards (H50). In this novel toxicokinetic study published by Pierce et al., subjects inhaled the labeled solvent allowing determination of background exposures using a physiologically based pharmacokinetic (PBPK) model.

Important developments in instrumentation have been applied to biological monitoring of metals. ICPMS is a tremendously versatile and sensitive technique capable of sequential (single element) or simultaneous (multielement) determination. ICP-atomic emission spectroscopy (ICP-AES) has similar versatility but relies on an optical system for detection and differentiation of elements. Computerized data acquisition and processing are integral to these techniques and are required for background correction (e.g., elimination of spectral interferences in AES and chloride interferences in ICPMS).

ICPMS has proven useful in simultaneous determination of multiple elements in urine at ecological levels with low parts-per-trillion detection limits (H163, H164). Scalp hair (H165) and seminal plasma (H166) also have been analyzed by ICPMS.

Sequential or simultaneous measurements in urine have been accomplished by ICP-AES (H167) as well as determination of trace elements in teeth (H129).

Graphite furnace atomic absorption (GFAA) is finding many new uses, especially for the L'vov platform or stabilized temperature (STGFAA) modification. In platform GFAA, the atomization of elements is delayed, greatly limiting matrix effects and often eliminating the need for correction by the method of standard additions (MSA). Platform GFAA methods have been described for thallium (H168) and beryllium (H169) in urine where no sample preparation is required. Additional GFAA methods have been reported for Al in plasma and urine (H170), Cr and Cu in urine (H171), Si in urine and blood (H127), and Pb, Mn, Fe, and Cr in hair (H172).

The optimal determination of some other elements requires gaseous hydride generation atomic absorption (e.g., As, Se, and Sb). Cold vapor atomic adsorption continues to be the preferred method for determination of mercury. Cold vapor AA methods for mercury in urine (H173, H174) and blood, milk, and tissues (H175) have been reported.

Adsorption voltammetry has been applied to the determination of urinary Pt (H126) and Mo after wet oxidative digestion (H115). Differential pulse anodic stripping voltammetry has been used in the determination of urinary Ni (H176) and Zn, Cd, Pb, and Cu (H177). The same elements have been determined by voltammetry in hair after microwave sample digestion (H124).

Inhaled particulate materials can be examined using bronchoalveolar lavage (BAL) sampling. BAL samples have been analyzed by electron microscopic microanalysis to determine silica and silicates (H128, H178) and aluminum oxide/Al fibers (H179). Electron microscopic microanalysis was used in a unique Pb exposure study—lead in blood, skin wipes, and fingernails was associated with smoking and Pb-containing smoke particles on the skin (H141). Capellaro and co-workers report that determination of asbestos bodies in BAL and sputum provide complementary information for monitoring asbestos-exposed workers (H180).

Instrumental neutron activation analysis (INAA) has been used widely in the determination of trace elements in hair (H181–H185) with sufficiently low detection limits for ecological studies. Trace elements in toenails also have been quantitated by INAA (H183). Trace element composition in hair may be influenced by hair dyeing and natural hair color according to one study utilizing atomic absorption analysis (H186), and this obviously could be a critical underlying factor in any biological monitoring program. Reference ranges for elements in hair have been reported in a publication by Druyan et al. (H187).

Speciation of certain metals is critical in risk assessment. The coupling of ICPMS with liquid chromatography offers tremendous potential for problem solving in this arena. Almost all lead in blood is bound in erythrocytes. Using LC/ICPMS, Bergdahl and others established that most erythrocyte lead is bound to a single protein,  $\delta$ -aminolevulinic acid dehydratase (ALAD) (H188, H189). ALAD production is a polymorphic trait under the control of a single gene, suggesting a simple hereditary factor controlling disposition of lead and possibly predisposing some individuals to lead toxicity. Isotopic composition analysis and the finding of ultralow Pb contamination in marine mammals by Owen and Flegall suggests

Table 11. Proficiency Evaluation Programs and Interlaboratory Comparisons

topic	authors	refs
PAT program, background and current status	Esche and Groff	112
PAT program update	Esche and Groff	113, 114
ELPAT program	Esche and Groff	115
lead measurements	Schlecht et al.	116
asbestos	Webber et al.	117
European Union residues	Caroli et al.	118
pentachlorophenol and lindane	Angerer et al.	119, 120
aromatic and chlorinated hydrocarbons	Goelen et al.	121
aldehydes	Goelen et al.	122
lacquer aerosols and solvent vapors	Lichtenstein et al.	123
biological agents "molds"	Averdick et al.	124
formaldehyde via NIOSH method 3500	Akbar-Khanzadeh and Park	125
lead sampling methods	Reynolds et al.	126
carbon in diesel exhaust particulates	Guillemin et al.	127, 128
aromatic and chlorinated hydrocarbons	Sato et al.	129
nitrogen dioxide passive monitors	Van Reeuwijk et al.	130
glutaraldehyde	Wellons et al.	131
PM10 and black smoke	Hoek et al.	132
ASTM sampling and analytical methods for lead	Ashley et al.	133
between laboratory differences in method detection limits examined	Draper et al.	134

that current human baseline levels may be 2–3 orders of magnitude greater than preindustrial levels (*H190*). Levels of Pb in perspiration are related to blood lead levels (*H191*), suggesting a possible noninvasive technique for Pb biomonitoring. Application of this approach, however, will need to address the pervasive sample contamination problems in the workplace environment. Additional examples of biological monitoring techniques for metals are summarized in Table 10.

With this arsenal of established or improved analytical techniques, the available exposure markers in biological samples is extensive and growing. Moreover, there is often more than one technique for reliable analysis of the determinant in the biological matrix. Arsenic, for example, is determined by platform GFAA, ICPMS, and ICP-AES. For organic determinants a GC-based procedure (with appropriate derivatization as necessary) is often available as an alternative to an HPLC method, and the availability of multiple separation and detection systems provides effective confirmation.

The utilization of biological markers in chemical health and safety programs varies from country to country. And the majority of published research originates from outside the United States, as is readily apparent from reviewing the literature cited. There is increasing interest and acceptance of biological monitoring in this country, however, and this trend can be expected to continue with the effort to harmonize environmental and occupational regulations in the world economy.

#### QUALITY ASSURANCE

Trends/changes in quality control and quality assurance continue to occur as the effects of additional factors on data quality are better understood. The AIHA laboratory accreditation programs have expanded, more laboratories have satisfied the requirements of ISO 9000, and the National Environmental Laboratory Accreditation Program (NELAC) and the National Cooperation for Laboratory Accreditation (NACLA) are no longer dreams. Additionally, the expansion of ISO Guide 25 into ISO Standard 17025 will make the establishment and implementation of a quality system in the industrial hygiene laboratory a requirement within a few years, especially for those laboratories doing

business internationally. Many of the publications reviewed for this article report on components of a quality system such as proficiency samples and reference materials or interlaboratory round robins. It is expected that future articles will include the description of laboratory quality systems that encompass all aspects of a project from the development of the sampling strategy through report generation to provide the client with *data of known quality consistent with its intended use*.

Publications of a general nature whose concepts might apply to a range of subjects include a technique for evaluating a mathematical model used to calculate the consequences of major hazards (*I1*). Ratti et al. (*I2*) provided risk assessment and analysis protocols, Kobayashi (*I3*) compared one- and two sided tests for evaluating toxicological data, and Stewart et al. (*I4*) used statistical techniques to evaluate the error of historical data. Finally, Menditto (*I5*) provided a review covering many of the factors associated with biological monitoring.

Method evaluation guidelines used at NIOSH were the subject of a paper, and computer program, by Abell and Kennedy (*I6*). Method performance criteria are based, in part, on the use of certified reference materials. Maier et al. (*I7*) reviewed the importance of European Union certified reference material programs, while Egmore and colleagues (*I8*) considered the preparation of reference materials for air analysis, and Morabito and Quevauviller (*I9*) dealt with reference materials for marine ecosystems. Finally, Inn (*I10*) discussed the NIST program for environmental radiation measurements, and an international group lead by Broadway (*I11*) outlined a prototype international quality assurance program. Several papers discussed domestic and international proficiency analytical testing programs and interlaboratory comparison studies. These are shown in Table 11.

Other authors dealt with quality assurance procedures in sampling (*I35–I37*) and analysis (*I38*).

Although the initial cost of quality assurance and quality control may be significant, there is long-term financial benefit in doing it right the first time. It has been said, "The bitterness of poor quality remains long after the sweetness of low price is forgotten."

## CONCLUSIONS

Research is an iterative process. Thus, the tremendous progress evident from this review leads us to a point where substantial advancements can be anticipated. Emerging and highly fertile research areas will certainly include new biological monitoring tools, especially those relying on immunochemical techniques and new mass spectrometry instrumentation. LC/MS, for example, is capable of direct analysis of polar metabolites and conjugates and there is a new generation of these instruments "in the field". Refinements in instrumentation and computerization will be incremental, for the most part, leading to gradual improvements in sensitivity, lower costs of analysis/higher throughput, greater operator friendliness, and chemical speciation not known today. There will be greater emphasis on field-portable techniques and screening analysis (vs "definitive"). There may be increased reliance on surface wipe samples, which may supplement information on airborne residues and provide needed information on cutaneous exposures. Evolutionary advancements will occasionally be upset by revolutionary developments in instrumental analysis.

As we observed in the introductory comments, there is little doubt that there will be many new industrial processes and exposure scenarios that are not even imagined at this point, and they will arrive on the scene abruptly. Accordingly, there will be many challenges and research opportunities for the analytical chemists interested in the industrial hygiene field.

In the traditional fields of work, we will know more and more about the trace chemical composition of the work environment. And greater detail will be available on internal doses and biological responses to these chemicals. Faced with new information from the analytical chemistry laboratory, the ability to recognize and characterize occupational health hazards and to prioritize and manage risks will continue to be important.

**Disclaimer.** Mention of company names or products does not constitute endorsement by the Centers for Disease Control and Prevention.

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#### MONITORING INSTRUMENTS

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