

# Industrial Hygiene

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## Review Contents

Introduction	133R
General Reviews	133R
Dynamic Generation of Mixtures and Standards	134R
Gas Monitoring Instruments	135R
Gas Monitoring Sensors	137R
Sorbents and Filter Media	138R
Derivatizations	138R
Detector Tubes	138R
Specific Chemical Analyses	138R
Polynuclear Aromatic Hydrocarbons	139R
Biological Monitoring	144R
Passive Dosimeters	144R
Specific Chemical Dosimeters	145R
Sampling Strategy and Quality Assurance / Quality Control	145R
Indoor Air Quality	147R
Future Needs and Trends	148R
Conclusions	148R

## INTRODUCTION

Since the last review article on industrial hygiene in *Analytical Chemistry* in 1989 (A1), the realm of industrial hygiene monitoring in the workplace environment has expanded to include both the community and global environments. This expansion, due primarily to environmental concerns, regulations, and increased public awareness, has led to the development of improved analytical monitoring techniques and methods for detecting trace airborne contaminants and emissions. Included in this expansion is the indoor air environment, where sick building syndrome and other illnesses have been linked to various air contaminants. Monitoring for these unknown confined contaminants can be a challenge because of the multitude of building materials, supplies, and products that contribute to the complex matrix of potential contaminants.

This review covers the four-year period, 1989-1992, which includes well over 2000 articles in the field of industrial hygiene chemistry. It was impossible to reference and comment on all of the articles; however, the authors believe they have covered the field as comprehensively as possible.

Basically, industrial hygiene monitoring involves sampling and analysis. Both of these processes go hand in hand and are dependent on each other for monitoring personal exposures in the workplace to assess human health risk. Selection of suitable sorbents, filter media, impinger solutions, and sufficient air volumes are necessary in order to assure compatibility with analytical methods having specific detectors and sensitivities for various contaminants. Typically, these air contaminants are gases, vapors, mists, aerosols, fumes, fibers, dusts, and other particulates that can be trapped, adsorbed, and desorbed in an appropriate analytical medium.

## GENERAL REVIEWS

In the past twenty years, there has been a great improvement in analytical instruments in the laboratory and for field applications, especially in the environmental area. The emphasis remains on determining the concentration of toxic

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chemicals in the workplace, but also in the community at large. In community and ambient air analyses, the concentration range has been lowered significantly, sometimes even to the part-per-trillion range. Spengler and Wallace (B1) have written a rather comprehensive review concerning ways to estimate potential exposures to chemicals in the environment. They state that, "The emphasis has been towards the criteria pollutants of  $O_3$ ,  $NO_2$ ,  $SO_2$ , and  $CO$ . However, recently more attention has been given to an array of organic materials: formaldehyde from resins; alkanes and aldehydes from woodburning and vehicle exhaust; halogenated hydrocarbons from solvents, adhesives, and consumer products; and polynuclear aromatics and nitrosamines from combustion." Discussions of air pollutant exposures relate to measuring the concentration of the polluting chemicals in microenvironments involving people or to measurements made while people are carrying or wearing the monitoring device. This review describes monitors for personal exposure

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monitoring, sampling equipment for CO, NO<sub>2</sub>, volatile organic compounds in indoor air, formaldehyde, pesticides, and polychlorinated biphenyls (PCBs), particles, and polycyclic aromatic hydrocarbons. The review ends with recommendations for (1) the development of test protocols for evaluating equipment to be used as indoor/personal monitors, especially for NO<sub>2</sub>, CO, and formaldehyde, taking into account interferences from tobacco smoke, organic and nitrogenated compounds, ammonia, and other volatile compounds found in cleaning agents, solvents, and aerosol spray propellants; (2) portable, continuous NO<sub>2</sub> monitors; (3) portable, continuous CO monitors; (4) a passive CO survey sampler; (5) a particulate sampler, size selective for collecting inhalable particles ( $d < 10 \mu\text{m}$ ); (6) a monitor for tobacco smoke or a stable indicator thereof, such as certain nitrosamines or nicotine; (7) organic compound determination of volatile and nonvolatile materials in indoor air; and (8) a passive formaldehyde sampler.

Principles and methods for air sampling and evaluation of threshold concentrations are presented in a Polish article describing methods of stationary measurements for the assessment of occupational exposure to toxic substances in the working environment (B2). Recent trends in instrumentation for environmental monitoring have been described in a report by Bhatnagar which includes design requirements of instruments for air and water monitoring for an integrated environmental monitoring program for a countywide monitoring system which is satellite based. Development of instruments based on piezoelectric microbalance systems for personal monitoring as well as for ambient and occupational monitoring are described (B3).

Atmospheric sampling methods in the workplace are described by Carton, et al., in a French article which summarizes those methods and the apparatus used to assess workplace environment toxicity (B4). Miller has published an overview of methods available for the measurement of toxic substances in workplace air. This review contains references for methods and instruments for the determination of toxic substances in workplace air, including short-term or spot tests, continuous (portable) instruments, personnel monitors, leak detectors, fixed installations, and monitoring of emissions in waste gas stacks or in waste gas plumes for the prevention of ground-level pollution threatening the work force (B5). Another review, in Russian, covers methods available for the determination of toxic chemicals in workplace air and the chemical reagents used with the methods (B6).

A wealth of information is contained in Vol. 46 of the British publication *SCOPE*, in an issue entitled *Methods for Assessing Exposure of Human and Non-Human Biota*. One of the articles contains a review giving state-of-the-art analytical methods and instruments for the determination of pollutants in air, including personal exposure monitors for gases and particles and the filter media used in them, plus analytical methods for atmospheric aerosols, with special attention to mass analysis, elemental analysis, energy-dispersive X-ray fluorescence, PIXE (particle-induced X-ray emission), neutron activation analysis, atomic absorption spectrometry, and carbon analysis (B7). Rappaport has written a review on the assessment of long-term exposures to toxic substances in air, which integrates biological concepts, statistical methods, and the philosophical bases for exposure limits and shows that a sampling strategy consistent with the current understanding of exposure-response relationships can be adopted (B8).

A Czech review by Pitter and Capkova supports the preference for amount concentrations (e.g., micromoles per cubic meter) vs mass or equivalent concentrations to characterize quantitative relationships of the chemical and biological effects of compounds and make possible a comparison of results. The claim is supported by applied chemical and biological examples from analytical chemistry, biochemistry, industrial hygiene, hydrochemistry, and physical chemistry (B9). Mass spectrometry in environmental analysis is reviewed by Medved, showing how the coupling of gas chromatography and mass spectrometry provides the most useful technique for structure elucidation and quantitative analysis of organic compounds in environmental samples in all media, air, water, and soil, particularly in connection with reliable sampling and sample preparation (B10). A German review by Siekmann deals with the standardization of measurement technology for hazardous materials in the

workplace (B11). The use of nuclear analytical techniques for environmental studies in South America is selectively reviewed to study air pollution problems in large cities, such as Sao Paulo and Rio de Janeiro, focusing briefly on natural radioactivity and fallout from nuclear weapons in South America (B12). A review with the intriguing title "Hairs, criminals, moonrocks, metals, diseases, polluters! Where next for nuclear analytical chemistry?" is presented by Jervis, who shares his reflections on nuclear activation methods and their trends and cites some unique and interesting applications and mileposts in the development of this field of analysis (B13).

A rather extensive review by Barcelo discusses the occurrence, handling, and chromatographic determination of pesticides in the aquatic environment, i.e., in soils, water, and biota. Sample handling and the choice of analytical techniques to be used are discussed—e.g., whether to use gas chromatography or liquid chromatography/mass spectrometry for the characterization of pesticides and their breakdown products in environmental matrices (B14). A Russian review by Gorban describes the current status of monitoring the concentration of toxic substances in workplace air in welding operations. Analysis of gaseous and solid components of the welding aerosol is discussed (B15).

Odor threshold values for 223 odorants are discussed in a Japanese review by Nagata and Takeuchi (B16), using the triangle odor bag method. A very wide range of concentration values,  $2 \times 10^9$ , was observed. Isoamyl mercaptan had the lowest odor threshold, at 0.77 ppt, whereas propane was not perceived at <1500 ppm. They found that, in a homologous series, the odor threshold concentration gets lower as the molecular weight increases.

## DYNAMIC GENERATION OF MIXTURES AND STANDARDS

Several devices have been developed for the generation of mixtures which have been used in animal inhalation studies, aerosol generation for adsorption tube sampling and analytical method development, and a diffusion tube system for generating a standard composition gas mixture.

An improved spinning top aerosol generator has been developed in Germany to produce spherical magnetic iron oxide particles (Fe<sub>3</sub>O<sub>4</sub>) which are highly monodisperse and have a high particle number concentration above  $10\,000/\text{cm}^3$  (C1). An inhalation system has been developed for generating fumes from high melting point metals, e.g., Cr, Ni, Mn, and Fe, consisting of a plasma flame metal sprayer as a fume generator, a granular bed-type fume collector, a fluidized-bed aerosol generator, an exposure and a control chamber of a horizontal-flow type, and inhalant monitoring and controlling units (C2).

Two mercury vapor generators have been described. The first is a system for generating low-dosage mercury vapor for animal experiments, consisting of four acrylic plastic chambers: one chamber containing the mercury source, one for mixing the mercury vapor with air, one exposure chamber, and one chamber containing activated coal filters and mechanisms for regulating the airflow. The system is easy to set up and requires minimal attendance during use (C3). The second mercury vapor generating system is suitable for long-term inhalation experiments and is based upon the reduction of HgCl<sub>2</sub> by SnCl<sub>2</sub> in the input airstream of the inhalation chamber. An experimental example with mice is given (C4).

A programmable gas-dispensing system for exposing plants to dynamic concentrations of air pollutants has been described, along with a protocol for assessing the precision and accuracy of the system, which is used for ozone generation (C5). A laboratory apparatus used for the generation of phosgene mixtures in air has been described, in which the phosgene is produced by the pyrolysis of tetrachloromethane on heated platinum wires. Part of the phosgene-air mixture is absorbed in a glass absorber and is hydrolyzed in a NaOH solution. The chloride produced is measured photometrically (C6).

A dynamic vapor generator is described for the generation of atmospheres containing stable concentrations of moderate to low volatility organic chemicals. The generator is a glass

tube packed with an inert support matrix coated with an appropriate test compound to be added to the test atmosphere. Test atmospheres containing chlorpyrifos were produced for the testing of adsorbent tubes used in air monitoring for industrial hygiene purposes (C7). Nitrous acid is generated from the reaction of HCl, produced by a low-pressure permeation device, with a  $\text{NaNO}_2$  fluidized bed. The gas mixture is analyzed by using a  $\text{Na}_2\text{CO}_3$ -glycerol diffusion denuder and an instrumental method which uses a chemiluminescence detector, allowing the continuous monitoring of  $\text{NO}$ ,  $\text{NO}_2$ , and  $\text{HNO}_2$ . The system shows very good reproducibility and stability, with a 2% standard deviation over 3 months (C8).

Aerosols of hexamethylene diisocyanate (HDI) are generated dynamically in the 0.01–0.25 ppm range, based upon precise control of the evaporation of pure liquid HDI followed by dilution with air, which is passed through a nebulizer coupled with a separation stage to exclude nonrespirable droplets  $>10\ \mu\text{m}$ . The diisocyanate concentration was determined from samples collected in impingers by a high-pressure liquid chromatographic method. The atmospheres were used in provocation tests in studying clinical asthma (C9). Capillary tubes have been used to prepare standard gas mixtures. Diffusion rates were quantified by gravimetric methods. General procedural suggestions concerning the development of gas mixture systems are proposed (C10).

## GAS MONITORING INSTRUMENTS

Several reviews have been published concerning the determination of hazardous chemicals in the workplace. A general review on measurement methodology needs from the occupational hygienist's point of view has been published by Hafkenscheid (D1) and includes objectives of air measurements and measurement methods. Cottica has presented another review on the determination of hazardous pollutants in workplace air and discusses the various instruments available (D2). A review concerning direct-reading instruments for measuring pollutant gases and vapors in workplace air, by Siekmann and Kleine (D3), includes applications, requirements, choice of suitable instrumentation, types of apparatus, and future needs. Squirrell has published a review on measurement technology to control occupational exposure to hazardous gases and vapors. This review includes fixed plant monitoring systems, portable apparatus for leak detection, miniaturized instrumentation, and personal monitoring, calibration, and special requirements (D4). A French review by Vincent describes sampling apparatuses and analytical procedures used to monitor occupational exposure to airborne pollutants (D5). Basic principles and guidelines for monitoring air pollution are given in a German review by Hobelsberger (D6).

Another review describes the principles of detection and measurement of gases by direct-reading instruments (D7). The techniques described by Nader and Lauderdale range from traditional methods involving the principles of conductivity, coulometry, and colorimetry to advanced methods using Raman scattering and chemiluminescence, and from the traditional point-sampling methods to the long-path, electrooptical schemes.

Sampling with test tubes and hand pumps and principles and design of mobile gas chromatographic systems with photoionization (PID) and electron capture detectors (ECD) for environmental analysis are described in a German review by Teske and Karl (D8). They have devised a floating stripper system for the determination of volatile compounds in water. The system can be coupled with GC-PID or GC-ECD instruments.

The use of plantwide computer networks to obtain and analyze data from continuous air monitors will allow databases to be developed on a plantwide basis so that the data can be utilized more completely by the industrial hygienist. Smith, Rafales, Feldman, and Hilbert at NIOSH have developed a local area network (LAN) which has been used to collect data locally from continuous air monitors and transmit them to a central computer for further display and processing (D9).

Several specific techniques have been used in monitoring the workplace air. Ion chromatography has been used in a research laboratory for the analysis of anions in the steel

industry. Several applications are described in an article by Goddard (D10): the analysis of acid mists and sulfur dioxide emissions; the determination of anions in rainwater, sand, and soil; and the determination of anions on the surface of steel, which may contribute to corrosion.

Experiences after 3 years of practical application of a mobile laboratory for testing the safety of transport of hazardous materials has been described in a German article by Kirch-nawy (D11). The trailer is located at border-crossing points at Austrian customs offices to check the loads of trucks carrying hazardous materials. Using energy-dispersive X-ray fluorescence analysis, chemical elements with atomic numbers  $>10$  may be determined quickly without sample preparation. Volatile organic substances are identified using gas chromatography/mass spectrometry for headspace analysis.

The use of photoionization detectors in gas chromatography has been reviewed in a Chinese article by Tu et al. (D12), giving the advantages and applications of PIDs in the determination of toxic substances in industrial and environmental samples and in the determination of biological substances and pharmaceuticals. Modification of a commercial portable gas chromatograph equipped with a PID, to make possible the determination of hazardous compounds in ambient air without preconcentration at the ppb level, has been described by Berkley (D13).

A German paper written by Manns, Nitz, and Striefler (D14) includes a description of measurement systems for selectively determining volatile organic compounds with health risk in ambient air. Samples may be taken for 30 min in glass sampling vessels and then determined by multidimensional chromatography, using a two-column system of CP Sil 5 CB and CP Wax 57 CB, for continuous measurements of benzene, toluene, and xylene or C3 and C4 olefins.

A German review by Scharfenberger (D15) contains information on short-term air sampling using headspace chromatography, long-term sampling for the determination of occupational exposure by absorption, the determination of inhomogeneities in air by a flow-detecting device, and the reconstruction of a laboratory gas chromatograph for on-line air analysis as a part of an automated process control system.

The monitoring of air in coal mines, using a gas chromatographic system is described by Braithwaite, Cooper, and Bovell (D16). The system identifies and determines gases which indicate spontaneous combustion in the mines:  $\text{CO}$ ,  $\text{CO}_2$ , acetic acid, acetone,  $\text{COS}$ , ethylene, and propene. A micro thermal conductivity detector and a photoionization detector are used together to monitor methane, propene, alkenes, and oxygenates. The use of optical fiber sensors and transmission of data using optical fibers for environmental monitoring in underground coal mines, monitoring methane,  $\text{CO}$ , temperature, and air velocity is described by Srivastava, Kumar, and Prasad (D17).

Mouradian has described two fast gas chromatographic systems developed for use in industrial hygiene and environmental applications. The systems feature a gas-cooled and resistively heated cold trap inlet that produces injection bands with widths of 15–25 ms. Separation is achieved with 2–5 m  $\times$  0.25 mm i.d. capillary columns in a system built around flame ionization detectors coupled with a fast-responding electrometer and a personal computer based-data system. One system was designed for the analysis of organic vapors and the other for the analysis of liquids. Liquid mixtures of aromatics and aliphatics were often separated in less than 10 s, with relative standard deviations (RSD) for peak areas of replicate samples between 1 and 10%. RSDs for retention times were  $<1\%$ . Dilute solutions with concentrations  $<50\ \mu\text{g/mL}$  could not be analyzed due to the poor signal-to-noise ratios associated with fast-responding electrometers (D18).

Volatile organic compound (VOC) emissions from consumer products were determined by Bayer and Black (D19), using an environmental chamber coupled with a capillary gas chromatographic system, which included an introduction system for gaseous samples and a cryogenically cooled chromatographic column. Indoor air pollution could be determined with this system. A portable spirometer has been used by Raymer, Thomas, Cooper, Whitaker, and Pellizzari to collect primarily alveolar breath in 1.8-L canisters for subsequent gas chromatographic/mass spectrometric analysis. Based on  $\text{CO}_2$  measurements,  $>97\%$  of the breath collected

was alveolar in origin. The device can be used to monitor human breath for organic compounds with volatilities greater than that of *p*-dichlorobenzene (*D20*).

A portable ambient air monitor based on infrared photoacoustic spectroscopy has been developed and used for measuring propane, tetrachloroethylene, sulfur hexafluoride, paint booth solvents (e.g., toluene, acetone, and 2-propanol), and ethylene oxide from a hospital sterilizer and has been described by Jalenak (*D21*). A hand-held electronic sensor for the detection of hydrogen sulfide has been described by Luke (*D22*). The sensor provides visual and audible signals when the  $\text{H}_2\text{S}$  concentration is  $>10$  ppm.

Diesel exhaust in tailpipe emissions in underground mines has been determined using an apparatus described by Carlson, Taubert, and Johnson (*D23*). The apparatus dilutes the exhaust and measures the concentration of diesel particulate matter,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{NO}$ , and  $\text{NO}_2$ . Diesel soot extract has been collected from diesel exhaust particles using glass filters and toluene extraction. The extracts were separated into alkanes, polycyclic aromatic hydrocarbons (PAHs), mononitro-PAHs, oxidized PAHs, dinitro-PAHs, and other polar compounds. Gas chromatographic and high-performance liquid chromatographic analyses were used to determine the composition of the fractions extracted, as discussed in a German article by Levens (*D24*).

Ozone is commonly analyzed by chemiluminescence. The performance characteristics of an analyzer which uses the chemiluminescent reaction of ozone with a stoichiometric excess of 2-methyl-2-butene has been described by Ben-Jebria, Hu, and Ultman (*D25*). The goal was to be able to measure the distribution of ozone in a single human breath. The minimum detection limit was reduced to 0.01 ppm. Humidity and temperature did not have an effect on the analyzer output. When correction for  $\text{CO}_2$  is made, the instrument can be used to monitor inhaled and exhaled ozone concentrations over a broad range of values. Ultman and Ben-Jebria (*D26*) continued their work on developing a chemiluminescent ozone analyzer and constructed an ozone bolus generator to use a bolus-response method to measure noninvasively the longitudinal distribution of ozone absorption in human lungs. The analyzer has a rapid 90% step-response time (110 ms), a linear calibration from 0.03 to 10 ppm, and a minimum detection limit of 0.017 ppm ozone.

A newly developed computer-aided ozone measuring system can be used to evaluate gas-shielded welding, taking measurements simultaneously at several points, e.g., near the arc and in the welder's breathing zone. Ozone abatement measures are proposed by Brehme, Farwer, and Sroka in this German study (*D27*). A review has been prepared by Hughes, describing the development of a direct-reading method for the determination of methylenediphenylene isocyanate (MDI) in air (*D28*).

Several compounds have been determined by the relatively new technique of ion mobility spectrometry (IMS). Karpas, Pollevoy, and Melloul have described their study of the negative ion chemistry of bromine, in which they monitored its concentration in the ambient air of a chemical plant (*D29*). Three bromine ions were produced,  $\text{Br}^-$ ,  $\text{Br}_2^-$ , and  $\text{Br}_3^-$ , which were not adversely affected by the presence of chlorine, other bromides, and chlorides. The maximum bromine levels measured in the plant were  $<30$  ppb, well below the 8-h time-weighted-average threshold limit value of 100 ppb. Eleven hazardous gases can be determined in less than 10 s using IMS with a simple finite impulse response (FIR) digital filter, as described by Davis and Kroutil (*D30*). A hand-held IMS vapor monitor, used to monitor mixtures (2:1–1:2.5) of bis(2-chloroethyl) sulfide and phenol, detected and identified the sulfide peak. Linear discriminant functions were shown to be useful for spectral identification. Toluene diisocyanate (TDI) vapors in the 1–50 ppb range were monitored in the laboratory and in a polyurethane plant by Brokenshire, Dharmarajan, Coyne and Keller (*D31*) using IMS, even in the presence of gross excesses of amine and tin catalysts, blowing agents, and surfactants.

A field evaluation of a direct-reading continuous ethylene oxide monitor has been described by Hossain and Carpenter (*D32*).  $\text{Et}_2\text{O}$  is used as a sterilizing agent and can cause adverse health effects. Two gas monitors were evaluated vs the OSHA charcoal tube sampling method and the 3M passive monitor for  $\text{Et}_2\text{O}$ . Both instruments gave higher readings than the

charcoal tube or passive monitor methods. Further study is needed here. Methyl mercaptan in pulp mill air has been monitored by Bhatia, with a portable analyzer having a detection limit of 0.25 ppm (*D33*). A Chinese study by Kong, Xu, Chen, and Guan (*D34*) describes the determination of sulfur dioxide in air by flow injection analysis colorimetry, based upon the pararosaniline method. The method has been modified to increase its speed to 90 samples/h. Average recovery was 99.0–102.3%, relative standard deviation was 0.9, and detection limit was  $1 \mu\text{g/mL}$ . Results compared favorably with those from the standard method.

A commercially available differential optical absorption spectroscopy instrument was used by Neftel, Blatter, and Staffelbach (*D35*) for continuous determination of ammonia concentration over a path length of 80–250 m in the 200–230-nm UV region, for agricultural monitoring (*D35*). An integration time of 1 min yielded detection limits of  $5 \mu\text{g/m}^3$  for a 75-m path and  $2 \mu\text{g/m}^3$  for a 250-m path. A two-channel gas stripping scrubber system combined with flow injection analysis was also developed to test the results. Ammonia was fluorometrically measured by the reaction of *o*-phthalaldehyde in the presence of  $\text{NaHSO}_3$  buffered at pH 11. One channel, equipped with a glass spiral scrubber, measures ammonia and ammonium ion, whereas the second channel, equipped with a diffusion scrubber, measures ammonia only. Continuous measurements can be made with a time resolution of 2 min with a detection limit of  $1 \mu\text{g/m}^3$ .

Leonhardt et al. have increased the sensitivity of ionization analysis of chemicals in air, workplace air, and waste gases by ionizing a portion of the gas mixture in an ionization chamber using a radioactive source under a pulsed positive, negative, or alternating potential and analyzing the resulting current as a measure of the concentration of the chemicals in the gas (*D36*). The method is suitable for the determination of organic phosphorus-containing compounds.

Several articles have described monitors for radon in indoor air. A system for monitoring and controlling the level of  $^{222}\text{Rn}$  in indoor air, described by Hurst (*D37*), includes a collector of  $^{222}\text{Rn}$  and its daughter atoms, a detector for  $\alpha$  particles, and a means to calculate  $^{222}\text{Rn}$ ,  $^{218}\text{Po}$ , and  $^{214}\text{Po}$  levels in the air. A vent in the heating, ventilating, and air conditioning (HVAC) system can be used to reduce the  $^{222}\text{Rn}$  levels. The system can be used to monitor the efficiency of a dust filter in a HVAC system. An automated radon monitoring system for continuous measurement of Rn concentration in air was developed by Paoletti and Shirripa Spagnolo (*D38*). The system electrostatically collects the daughter products of  $^{222}\text{Rn}$  (or  $^{220}\text{Rn}$ ) and observes the  $\alpha$ -decay events from  $^{218}\text{Po}$  and  $^{214}\text{Po}$  (or  $^{212}\text{Bi}$  and  $^{220}\text{Rn}$ ). The detector is well-suited for routine measurements in enclosed environments such as residences. A system for the detection and measurement of indoor Rn progeny activity-weighted size distributions (particle size  $>0.5$  nm) and concentration levels has been developed by Ramamurthi and Hopke (*D39*). The system is microcomputer-controlled and involves a combination of multiple wire screen (graded screen array) sampler-detector units operated in parallel. System development, including prototype design, experimental characterization in a Rn aerosol chamber, and numerical studies for optimization of the design and operating parameters are described.

An oxygen deficiency monitoring system which measures the oxygen content of ambient air in the lower levels of a laboratory, where air circulation may be insufficient, allowing the creation of an oxygen-deficient environment, has been described by Devlin (*D40*). Three methods for the determination of volatile chlorocarbons in the atmosphere using chemical ionization mass spectrometry were described in a Japanese article by Daishima, Iida, Koshikawa, and Kawabayashi (*D41*). Detection limits were 0.5–1 ppb by discharge chemical ionization mass spectrometry and 0.01–0.1 ppb by gas chromatography/negative chemical ionization mass spectrometry with or without preconcentration using a collection tube packed with Tenax GC and activated charcoal. Relative standard deviations were 3–6% for the analysis of 1,1,1-trichloroethane, tetrachloroethylene, 1,2-dichloroethylene, chloroform, and carbon tetrachloride.

A gas analyzer with an electrochemical cell for the measurement of oxygen and an IR spectrometer for the measurement of  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{NO}$ , and Freon in indoor air and



industrial processes has been described in a German article by Ascherfeld and Fabinski (D42).

The infrared determination of silica is hindered by an almost complete spectral overlap in the region around 800  $\text{cm}^{-1}$ , where the various forms of silica have their main absorption band. To overcome this problem, Bjoersvik and Bye have developed a multivariate calibration technique using partial least-squares regression techniques applied to the analysis of mixtures of  $\alpha$ -quartz and fumed amorphous silica, by calibrating the wavelength region from 600 to 900  $\text{cm}^{-1}$  at 10- $\text{cm}^{-1}$  intervals. The two silica components can be determined simultaneously at concentrations of interest in industrial hygiene work. Standard deviation is  $\pm 1.8\%$ , and relative prediction error is better than  $\pm 5.2\%$  (D43).

A device has been described by Hahn, Ruettinger, Matschiner, and Lenk (D44) for the rapid coulometric determination of adsorbable organic halogen (AOX) compounds. After incineration, the halide ions are titrated against electrolytic silver. Computer-controlled square-wave polarization of the indicator electrodes allows very low noise titration curves in this automated analysis. The determination of the AOX fraction in liquid samples, elementary analysis for halogen in organic compounds, and the determination of AOX in air can be done with this device.

A compact differential optical absorption spectrometer (DOAS) with a 2.5-m base path was constructed for indoor air measurements by Biermann, Green, and Seiber. With proper mirror setting, the system has a total path length up to 220 m. With 2-min averaging, the cooled photodiode array detector has a detection limit equivalent to an absorbance of  $<0.0001$ . The instrument was tested initially with xylene isomers (D45).

Polar solvent vapors, such as ethylene oxide, tetrahydrofuran, and ethyl acetate in air are continuously and reversibly determined by contacting the gas sample with the color-forming agent, *O*-carboxytriphenylmethane, in a German patent by Wolfbeis and Koller (D46). The device is a thin optically transparent test strip coated with the color-forming agent to give a color reaction with the test gas, a light source, a light detector, and an optoelectronic measurement unit.

Sulfur hexafluoride has been used by Ritter (D47) to determine leaks from asbestos removal containment enclosures. Measuring the presence of  $\text{SF}_6$  outside the enclosures indicates leaks through which asbestos fibers may escape into the surrounding area. Air pollutants released during the production of integrated circuits have been monitored by Ponsold and Kath in a German article (D48). They monitored for  $\text{HCl}$ ,  $\text{NO}_x$ ,  $\text{NH}_3$ , silanes, phosphines,  $\text{As}$ ,  $\text{O}_3$ , and  $\text{HF}$  and found that  $\text{NO}_x$ , silane, and phosphine concentrations exceeded the limit values.

A new photopyroelectric device has been described by Christofides and Mandelis (D49) for the detection of hydrogen. It uses poly(vinylidene fluoride) coated with a Pd layer on one side, as the active electrode, and a reference electrode of Ni-Al on the other side. It can detect H at concentrations of  $\sim 40$  ppm and is sensitive to hydrogen at  $-63^\circ\text{C}$ . An iridium laser irradiating the polymer produces alternating temperature gradients, resulting in an alternating voltage. Exposure to H produces an increased differential signal between the electrodes. The detector is sensitive, has a fast response, high selectivity, durability, and reversibility at room temperature.

The use of IR spectrometry for on-line measurement of organic solvents in the waste air of a pharmaceuticals plant is described in a German article by Dueblin and Thoene (D50). Fifteen components were analyzed simultaneously every 6 min. The emission sources within a building, mass flux evaluation, and the control efficiency of waste air cleaning measures can be determined.

Four commercially available high-sensitivity  $\text{NO}/\text{NO}_x$  analyzers were tested indoors and outdoors by Jassim, Stedman, Steenson, Kita, and Solomon at the University of Denver (Denver, CO) to determine their suitability as low-level  $\text{NO}_x$  monitors (D51). Interferent effects from nitric acid, ammonia, and peracetonitrile (PAN) were determined. The most sensitive instrument was the TECAN parts-per-trillion (ppt) analyzer, equipped with a photolytic converter. This instrument showed negligible interferences from the interferences, whereas the other three instruments did respond to them. The TECAN ppt instrument was the best for the sub-

ppb concentrations. The other three, TECO Model 42, TECAN ppb, and Monitor Labs Model 8841 analyzer were not as sensitive. At the 100–200 ppb levels, all instruments were comparable.

The use of IR methods for remote automatic monitoring of the concentrations of gaseous components ( $\text{CH}_4$ ,  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{SO}_2$ , etc.) in mining atmospheres has been described in a Russian article by Onishchenko, Krichko, and Ivashov (D52). The instrument can be used also for studying the condition of the rock massif and for prediction of abrupt releases of  $\text{CH}_4$ . Remote control of the mining equipment is discussed. A European patent by Lee and Van Ewyk (D53) contains a description of a gas detector for determining toxic or combustible gases in air, using infrared techniques in coal mines, on oil platforms, or in chemical plants.

The design and application of a very sensitive ultraviolet flame photometric detector (UV-FPD) analyzer has been described by Yuen, Gavin, and Brand (D54). It has been used for the rapid determination of low-ppb concentrations in air of certain organophosphorus esters and organophosphorus fluorides used as intermediates in pesticide and insecticide manufacture, which are serious health hazards according to OSHA. The detector measures the concentration of H-P-O species produced by chemiluminescence in a hydrogen-enriched flame. This is a continuous monitoring system with alarms when concentrations reach alarm levels.

Sampling and analysis of soil gas along a 5-mile section of a New York State highway in Manhattan, NY, showed moderate-to-high levels of  $\text{CH}_4$  and nonmethane volatile organic compounds in various areas, according to a report by Rizvi and Fleischacker (D55). Sampling and analytical methods are described in detail. A review of personal monitoring systems for occupational exposure to chemicals has been presented by Manning (D56). It describes first-, second-, and third-generation personal monitors. Fourier transform infrared (FTIR) remote sensing was used by Brandon and Trautwein (D57) to monitor ambient air in an electronics assembly area and in an aluminum smelter. Methanol and Freon-12 were determined at the electronics facility, and HF was determined at the smelter.

Fast response monitors coupled with video filming of tasks were used by Unwin, Walsh, and Worsell (D58), with real-time exposure level being dubbed onto the video picture of the work activity. The videotape can also be used to demonstrate good working practices and improve awareness of the hazard.

A Fourier transform infrared spectrometer has been interfaced to a 2.5-m base path, open multiple-reflection cell to monitor indoor air, as described by Green, Biermann, and Seiber (D59). Methyl bromide, a fumigant, and chloropicrin, its odor additive, can be detected to the ppb level. This method gave higher results at low pollutant concentrations than did the conventional adsorption-desorption GC technique.

A prototype automated formaldehyde monitor was developed and evaluated for EPA and was used in testing during a 10-day field study involving ambient air and indoor air measurements, as part of the Atlanta Ozone Precursor Study, as described by Fortune (D60). Analytical and sampling protocols are discussed and data are reported. An analyzer for the determination of formaldehyde in mine air has been reported by Pochenkova, Zemskii, Gavrilenko, and Tureeva, in a Russian article, and is based on colorimetric measurement of a rose-colored quinoid compound formed by condensation of  $\text{HCHO}$  with aromatic hydrocarbons in the presence of concentrated  $\text{H}_2\text{SO}_4$ , followed by oxidation of the intermediate (D61).

## GAS MONITORING SENSORS

Gas monitoring sensors provide the industrial hygienist with a wide range of air monitoring capabilities, from simple leak detection to fixed and portable area monitoring and personal monitoring. A variety of air monitoring instruments equipped with specific sensors are available at low cost for direct reading and continuous unattended operation. The function of sensors is to identify a specific chemical in air and produce a signal related to the concentration of that chemical. The sensors should be specific, sensitive, have an applicable

concentration range, be unaffected by humidity, temperature and coexisting species, and be stable and responsive to recovery after exposure. They can be as simple as a color reaction on paper tape or as complex as the reactions that occur on coated piezoelectric crystals. The most common sensor used is infrared (IR), because of its versatility for monitoring different gases and vapors at selected wavelengths. In the last review article, sensors were classified as electrochemical, thermochemical, biochemical, optochemical, and solid-state semiconductors (*A1*). A review of sensors for analytical purposes, including vapors, was also referenced. A more recent update on the criteria for selection and placement of selective sensors for toxic and hazardous gases in the chemical industry has been reported (*E1*). Additional reviews on chemical sensors for gases and solvent vapors, including a new type of gas sensor based on a film coated with substituted phthalide layers, have been reported (*E2, E3*). Some of the latest sensors include fiber optics, coated films, surface acoustical waves (*E4*), lasers (*E5*), and biosensors (*E6, E7*) as sensing techniques. As the need for fast, responsive, sensitive sensors increases, so will the intensity for developing other innovative techniques that provide accurate gas monitoring data.

## SORBENTS AND FILTER MEDIA

A number of different sorbent media have been utilized for collecting various gases and vapors. Charcoal is still the most predominant choice for organic vapors, while silica gel is used for polar inorganic and organic vapors. Tenax, Ambersorb, and XAD resins also have proved to be useful for specific vapors. These sorbents can be used for both active and passive sampling and for personal and area monitoring. For each of these sorbents, adsorption capacity, stability, and desorption efficiency must be evaluated to substantiate collection ability. In some cases desorption is accomplished by solvent elution, e.g., CS<sub>2</sub>, while with others, thermal desorption is used if the analytes are not heat-labile and there is no need for subsequent analysis. Improvements in these sampling techniques have been made by impregnating some sorbents with reagents capable of reacting with, stabilizing, and derivatizing such analytes as amines, isocyanates, and aldehydes (*F1-F3*). Various presorbent tubes have also been utilized to eliminate interfering contaminants and the effects of relative humidity. Another area where improvements have been made is in sampling for mixtures, where preferred adsorption and desorption on sorbents has been investigated and suitable solvents found to separate and improve desorption efficiency, e.g., the addition of 5% 2-butoxyethoxyethanol to CS<sub>2</sub> for desorption of oxygenated solvents (*F4*).

For most sampling needs, solid sorbents still continue to remain the most popular technique for sampling gases and vapors because they can be readily packaged and contained in sampling devices which are easily transported, worn, and processed for subsequent analyses. Liquid sorbents are still used with impingers for various gases, mists, and aerosols. This form of active sampling has its drawbacks because of the potential for spills and leaks of reagents and because of the inconvenience of wearing a glass container and sample pump. Inert plastic passive liquid dosimeters have been introduced for area and personal monitoring and are made to contain liquid sorbents that can be sealed after sampling and sent to a laboratory for subsequent analyses. Of course, the key to this technique is analyte solubility in the liquid sorbent. In some cases it is also feasible to dilute, concentrate, or extract these liquid sorbents to allow for concentrated or diluted samples to be analyzed. This has resulted in improved analytical sensitivity and eliminated matrix interferences.

Specific filter media continue to be used for monitoring particulates, dusts, mists, fibers, fumes, and metals. Poly(vinyl chloride) (PVC) filters are used for gravimetric determination of total dusts, while cellulose ester membrane filters are used for metals. Fibers such as asbestos are still collected on membrane filters in nonconductive cassettes. These filters are then cleared in acetone and mounted on a microscope slide for counting by phase-contrast light microscopy (PCM). Qualitative bulk fiber identification is done by polarized light microscopy (PLM). For confirmatory analysis, transmission electron microscopy (TEM) is used.

When TEM is inconclusive, scanning electron microscopy (SEM) is used to confirm the elemental composition of the minerals present.

## DERIVATIZATIONS

Derivatization of airborne contaminants is another technique employed in air sampling and analysis, since some contaminants are unstable and require a derivatizing reagent to stabilize them prior to analysis. Aldehydes, ketones, amines, and isocyanates were examples of some of the most frequently monitored unstable air contaminants cited in reference articles for this review (*G1*). By precoating solid adsorbent tubes with derivatizing agents, these reactive chemicals were trapped, stabilized, and tagged with chromophores or other chemical groups that increased detection sensitivity and selectivity. For aldehydes and ketones, samples were collected and derivatized on solid sorbent tubes treated with 2,4-dinitrophenylhydrazine and the corresponding hydrazone derivatives were determined by HPLC with UV detection (*G2*). Two new derivatizing agents were reported for monitoring airborne amines. A polymeric anhydride containing an *O*-acetylsalicyl group as the labeling moiety was used for the collection and simultaneous derivatization of primary aliphatic amines, with *n*-butyl amine as the model. The reaction product, *n*-butyl-*O*-acetylsalicylamide, was determined quantitatively by HPLC and UV detection (*G3*). In another method, naphthyl isothiocyanate coated on Amberlite XAD-2 or glass fiber filters was used to determine gaseous and particulate polyamines. The poly-(thiourea) derivatives were then determined by HPLC with UV detection (*G4*). For isocyanates, two papers reported the use of tryptamine as a derivatizing agent for the determination of airborne isocyanates. Phenyl isocyanate was evaluated using tryptamine-coated solid sorbent tubes and subsequent analysis by HPLC with UV detection (*G5, G6*).

## DETECTOR TUBES

Length-of-stain detector tubes continue to find varied uses in industrial hygiene monitoring. Three commercial carbon dioxide detector tubes were evaluated by Ancker, Goethe, and Bjurstroem for their accuracy by comparison with an infrared spectrophotometer. They were not suitable for measuring CO<sub>2</sub> less than or equal to 500 µL/L (ppmv) for estimating the extent of air recirculation in ventilation systems (*H1*).

Several mercaptan gas detector tubes were evaluated by Bhatia (*H2*), using synthetic gas mixtures containing methyl mercaptan, hydrogen sulfide, dimethyl sulfide, dimethyl disulfide, and sulfur dioxide in air. Only the Draeger 0.5/a tube was sensitive enough for measuring methyl mercaptan at the threshold limit value concentration of 0.5 ppm, using 20 strokes on the Draeger bellows pump.

The use of detector tubes to monitor chemical exposure of personnel in the construction industry was evaluated by Ruehl and Knoll (*H3*). Results compared with mobile laboratory gas chromatography showed that the detector tubes could be used in many cases. A simple method for measuring cleaning solvent in air, using an ethyl acetate detector tube, was described in a Japanese article by Yoshikawa, Arashidani, and Kodama (*H4*). Color developed whenever toluene and ethyl acetate coexisted. Acetone, methanol, and trichloroethylene did not interfere with color formation.

## SPECIFIC CHEMICAL ANALYSES

References to the analysis of specific chemicals over the past four years are too numerous to include brief remarks on each. Therefore, a tabular alphabetical listing has been employed in this section.

As with all "shortcuts", however, there are exceptions. An exceptional book has been published, *Metals and Their Compounds in the Environment*, edited by Ernest Merian. It contains numerous reviews on 36 metals and groups of elements in the environment—from aluminum to zirconium—including physical and chemical properties; ana-

lytical methods; sources; methods of manufacture; important compounds; uses; waste products; recycling; distribution in the environment, foods, and living organisms; uptake, absorption, transport, distribution, metabolism, and elimination in plants, animals, and humans; effects on plants, animals, and humans; and hazard evaluation and limiting concentrations (150-185). Another unique method, in Chinese, by Zhang, Xu, and Zhang, describes a qualitative analysis method of fingerprint UV spectra for 70 common organic toxicants, using a method of fuzzy clustering analysis to classify and file the spectra, forming a UV spectrum data bank. Unknown toxicants are identified through diet spectrum pattern recognition (1118).

Two other articles, which list several chemicals whose desorption efficiency has been improved by the addition of other chemicals to carbon disulfide to improve the recovery of polar compounds from charcoal, are referenced in section L of this review (118, 119), and their specific chemicals are listed in the Table I.

## POLYNUCLEAR AROMATIC HYDROCARBONS

Nilsson and Colmsjoe (11) studied retention characteristics in normal-phase HPLC of highly biologically active chloro-added polycyclic aromatic hydrocarbons. Silica and (cyanopropyl)dimethylsilyl- and (aminopropyl)silyl-modified stationary phases were investigated. A fast cleanup procedure for chloro-added PAHs in complex samples is outlined. The effect of solvent polarity on the determination of oxo- and nitro-PAHs was described by Galceran and Moyano (12). They used capillary gas chromatography with splitless injection for analysis. The solvents which worked best were methylene chloride and acetone vs methanol and acetonitrile. PAHs studied were 2-methyl-1-nitronaphthalene and 9,10-phenanthroquinone.

Sampling, analysis and data validation of indoor concentrations of PAHs was reported by Offermann, Daisey, Loiselle, and Gundel (13). They developed methods for sampling and analyzing PAHs in particulate and vapor phases. Highest indoor PAH levels were found where sources such as wood smoke and tobacco smoke were present. Airborne particulate matter collected from a coal field in India was analyzed by Mukherjee and Chakraborty for benzo[a]anthracene, benzo[a]pyrene, and perylene, by a combined thin-layer chromatography/gas chromatography method (14).

Gas chromatography/matrix isolation infrared spectrometry (GC/MI-IR) has been developed by Childers (15) for environmental air sample extract analysis. The samples analyzed included extracts from woodsmoke-impacted air, XAD-2 blanks, indoor air, and carpet samples. The analytical technique was used to identify semivolatile organic compounds in the extracts. This technique discriminates between isomeric compounds that are difficult to separate chromatographically and to distinguish by GC/electron impact ionization MS methods.

An improved dilution sampler was developed by Wall (16) for source sampling for PAHs and other semivolatile organic species. The new sampler reduced the artifacts formed during the analysis of flue gas by the EPA modified method 5 sampling procedure. The method provides an accurate measure of the particulate-phase PAHs. PAHs in work areas in a rubber processing plant were measured by Kisselov and Hill (17). Oil plasticizers may cause PAH pollution at such workplaces. Fifteen PAH parent compounds were determined, using benzo[a]pyrene as the PAH reference substance.

A study of suspended particulate matter and PAHs in indoor and outdoor air was reported by Ando, Tamura, and Katagiri (18). They developed a new portable sampler that could separately collect particles with aerodynamic diameters of >10, 2-10, and <2  $\mu\text{m}$ . Fine particles with aerodynamic diameters of <2  $\mu\text{m}$  contained high concentrations of PAHs. A gas quantification of 10 PAHs in airborne particulate matter collected near a highway was described by Deshpande and Sarin (19).

A review of the environmental and occupational importance of PAHs was presented by Jacob, Karcher, Belliard, Dumler, and Boenke (20). The occurrence, toxicity, and development of high-purity certified reference materials (seven nitro-

substituted and seven oxygen-containing PAHs: ketones, oxaarenes, and hydroxypolycyclic aromatic hydrocarbons) were reviewed. A high-performance liquid chromatography/fluorescence detection method was validated for the simultaneous quantification of 15 PAHs by Hansen, Olsen, Holst, and Poulsen (11). The method used multiple wavelength shift for simultaneous quantification of different PAHs.

A simplified version of an HPLC method for the determination of PAHs in suspended particles collected from indoor and outdoor air and personal samples was developed by Sisovic and Fugas (12). Low-volume samplers were used, the PAHs were extracted ultrasonically, and the separation of interfering substances between analyses by HPLC was omitted. Three samplers, all of which used polyurethane foam filters, were evaluated for determining PAH concentrations in the breathing zone of coke oven workers by Yang, Li, Heng, and Yi, in a Chinese article (13). Some sampling cartridges were contained in respirators; others were worn on workers' lapels. Data on the concentration of PAHs in air samples from fish smokehouses and meat smokehouses in Denmark were used by Hansen, Poulsen, and Christensen (14) to analyze the extent to which six different volatile PAH compounds could function as markers for the total concentration of six different carcinogenic particulate PAHs. Naphthalene and phenanthrene showed the highest sensitivity as markers for total carcinogenic PAH compounds in air samples from smokehouses. When testing iron foundries, only naphthalene and pyrene were useful as markers for the carcinogens. Hansen, Olsen, and Poulsen (15) did another study of PAHs in air samples from meat smokehouses during cold meat curing and concluded that PAH exposure during cold meat curing might be considered a limited health hazard compared with PAH exposure during hot smoke curing.

A spectral atlas containing UV, IR, fluorescence, mass, and NMR molecular spectra of PAHs has been edited and published by Karcher (16). The atlas also includes vapor pressure, water solubility, octanol-water distribution coefficients, occurrence in the human environment, and health effects (mutagenic and carcinogenic properties). A simple method for the detection of benzo[a]pyrene in workplace air, using thin-layer chromatography with fluorometric detection has been described by Bleichert and Ebert in a German article (17). The analysis of PAHs by synchronous fluorescence spectrometry has been applied to occupational health by Baudot, Viriot, Andre, Jezequel, and Lafontaine (18). About 50 PAHs were studied, and a mixture of 10 PAHs was analyzed and each PAH identified.

An HPLC method was developed for the quantification of the PAHs chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, and benzo[ghi]perylene in the particulate matter of indoor air by Risner and Conner (19). Samples were collected on 1- $\mu\text{m}$  pore size Fluoropore membrane filters, extracted with acetonitrile, and then analyzed on a polymeric octadecylsilane, silica-based column with fluorescence detection, using a mobile-phase gradient of water and acetonitrile. Recoveries were excellent and relative standard deviations ranged from 5.0 to 9.6, for a method capable of detecting <1 ng/m<sup>3</sup>.

Ares (20) has developed an algorithm, UVDECODE, for direct extraction and analysis of environmental PAHs using derivative UV spectrophotometry. The algorithm can be applied to a direct extract of the airborne particulate sample with a single solvent such as cyclohexane. Results are comparable in accuracy and precision to those obtained by chromatography or direct extraction techniques. A fully automated system, comprising a liquid chromatograph coupled on-line to a gas chromatograph by means of a loop interface, has been constructed by Oestman, Bemgaard, and Colmsjoe (21) for cleanup and analysis of PAHs. The PAHs were isolated by LC and then the PAH fraction was transferred to the GC, where the PAH analysis was completed. This system, compared with ordinary off-line LC cleanup followed by GC analysis, has a 50-100-fold increase in sensitivity, yielding a detection limit for individual PAHs of a few nanograms per sample, using flame ionization detection. The technique was demonstrated with samples of urban air and used automobile lubricating oils.

May, Benner, Wise, Schuetzle, and Lewtas have completed a review of the standard reference materials (SRMs) from the National Institute of Standards and Technology (NIST)

Table I

chemical	technique <sup>a</sup>	ref (lang) <sup>b</sup>
acetaldehyde	HPLC/UV	I1
	GC	I9 (R)
acetic acid	GC-FID	I1
acetone	GC	I3 (R)
	GC	I9 (R)
		I24
	GC-FID	L8
	GC-FID	L18
	GC-FID	I1
acrylonitrile		I43
alcohols		I1
aldehydes	GC-FID, GC/MS	I1
aliphatic amines	GC-FID	I43
aliphatic hydrocarbons		I43
alkylbenzenes		I23 (G)
allyl chloride		I51
aluminum and its compounds	(review)	I1
aminoethanol compounds (method II)	IC	I6 (G)
ammonia, liq., for oxygen and water		I5 (R)
anilines: 2-bromo-6-chloro-4-nitroaniline, N,N-dihydroxyethyl-m-chloroaniline, N-(hydroxyethyl)-N,β-cyanoethylaniline, N-ethyl-N,β-cyanoethyl-m-toluidine	GC	
arsenic and its compounds	(review)	I52
arsine		I107 (J)
asbestos, bulk	stereo polarized microscope with dispersion staining	I1
asbestos, fibers	TEM	I1
barbituric acid and sodium salt	photometry/derivative	I10 (R)
barium stearate	AA	I8 (R)
benzaldehyde	UV	I4 (B)
		I12 (R)
benzene	GC/MS	I7 (G)
	GC-FID	L18
benzyl alcohol		I12 (R)
	headspace GC; HPLC/UV	I11 (J)
beryllium and its compounds	(review)	I53
bismuth and its compounds	(review)	I54
boron hydride (B <sub>2</sub> H <sub>6</sub> )		I107 (J)
bromine		I23 (G)
1,3-butadiene	GC	I13
1-butanethiol	GC-FPD (sulfur mode)	I1
n-butanol	GC	I14 (R)
	GC-FID	L18
isobutanol	GC	I14 (R)
2-(2-butoxyethoxy)ethanol	GC-FID	L18
butyl acetate	GC-FID	L18
butyl cellosolve acetate	GC-FID	L18
butyric acid	GC/anilides	I2
cadmium	multiple methods; review	I15
	anodic stripping voltammetry	I46 (CH)
cadmium and its compounds	(review)	I55
cadmium stearate	AA	I8 (R)
carbon dioxide		I91
carbon tetrachloride	EPA method 18	I18
carboxylic acids	Me esters/Cap. GC/MS	I16
cellosolve	GC-FID	L18
cellosolve acetate	GC-FID	L18
chemical warfare agents: GB, GD, HD, VX		I37
chlordane	GC-ECD	I1
chlorinated dibenzo-p-dioxins (CDDs)	simult steam distn/solvent extractn	I9
chlorinated dibenzofurans (CDFs)	simult steam distn/solvent extractn	I96
chlorinated hydrocarbon solvents	Cap. GC/MS	I19 (J)
chlorinated organic compounds: (volatile) in water	headspace GC	I17 (J)
	solvent extraction	I17 (J)
	distillation	I17 (J)
chloroform	EPA method 18	I18
chlorothalonil (pesticide)	HPLC/UV	I20
	GC	I21
chromium and its compounds	(review)	I56
chromium(VI) compounds	derivatization/vis absorp spectrophot	I1
cobalt and its compounds	(review)	I57
copper and its compounds	(review)	I58
cumene	GC-FID	L18
cyanoacetic acid	HPLC/UV	I1
	HPLC/UV	I22
cyclohexane	GC-FID	L18



Table I (Continued)

chemical	technique <sup>a</sup>	ref (lang) <sup>b</sup>
cyclopentadiene		I30 (R)
cyclopentane	GC-FID	L18
diazomethane		I2
decane	GC-FID	L18
dibenzo- <i>p</i> -dioxin	(review)	I28
dibenzofuran	(review)	I28
dibromochloropropane		I23 (G)
dichloroethane	GC	I3 (R)
dichloromethane		I24
1,3-dichloropropene (fumigant)		I25
dicyclopentadiene		I30 (R)
diethyl ether		I24
dimethylcadmium	polarography in HCl solns	I26 (R)
dioxins	GC/MS	I29 (D)
dipentamethylene thiuram tetrasulfide	HPTLC	I27
endrin	GC/Ni-63 ECD	I1
ethanol	GC	I9 (R)
	GC-FID	L17
	GC-FID	L18
ethyl acetate	GC	I3 (R)
	GC-FID	I9 (R)
ethylamine	derivative/colorimetry	I31 (CH)
ethylbenzene	GC-FID	L18
ethyl butanoate	GC	I9 (R)
ethyl formate	GC	I9 (R)
ethyleneamines: diethylenetriamine, ethylenediamine, triethylenetetramine	HPLC/UV	I1
ethylene glycol ethers and acetates: 2-ethoxyethanol, 2-ethoxyethyl acetate, 2-methoxyethanol, 2-methoxyethyl acetate		I32
ethylene oxide		I34
2-ethyltoluene	GC-FID	L18
3-ethyltoluene	GC-FID	L18
fibers	phase-contrast microscopy	I1
formaldehyde	GC-FID	I1
	UV	I4 (B)
		I34
	fluorescence by UV	I119
	derivatization/fluor.	I119
	2,4-dinitrophenylhydrazine/impinger/spectro (for inhalable dust/fibers)/chromotropic acid	I119
	filter (as above)/2,4-dinitrophenylhydrazine/perchloric acid/HPLC	I120
	silica gel/2,4-dinitrophenylhydrazine	I121 (J)
Freon 11	GC	I114 (P)
furfural	polarography	I35 (R)
furfurole	UV	I4 (B)
furfuryl alcohol	GC-FID	I1
gallium	oscillo/polarography	I36 (CH)
gallium and its compounds	(review)	I59
germanium and its compounds	(review)	I60
glutaraldehyde	GC-FID	I1
glycidol	photometric and GC	I38 (R)
glycol ethers	multidimensional GC/MS	I33
gold and its compounds	(review)	I61
halocarbons (chlorofluorocarbons): CFC-11, CFC-12, CFC-113, CGC-114, CFC-115, Halon-1211, Halon-1301, Halon-2402	GC-ECD	I39 (J)
halogenated, purgeable organics in fish, sediments, and water	(review)	I40
halothane		I34
heptane	GC-FID	I19
2-heptanone	GC-FID	I19
herzine (rubber)	GC	I3 (R)
heteroaromatic hydrocarbons	GC/MS	I7 (G)
hexamethylene diisocyanate (HDI) and HDI prepolymer		I42 (F)
hexane		I24
	GC-FID	L17
	GC-FID	L18
hydronaphthalenes		I43
hydrogen cyanide	visible absorption spectrophotometry	I1
hydrogen sulfide	Dositox analyzer	I41 (G)
4-hydroxy-4-methylpentanone	GC-FID	L18
indium and its compounds	(review)	I62

Table I (Continued)

chemical	technique <sup>a</sup>	ref (lang) <sup>b</sup>
iron and its compounds	(review)	I63
isoamyl acetate	GC	I9 (R)
isobutanol	GC-FID	I18
isocyanates	HPLC of urea derivative/electrochemical and UV detection	I1
isobutyraldehyde	GC	I9 (R)
2-propanol	GC	I9 (R)
		I34
	GC-FID	L17
	GC-FID	L18
ketones (method I)	GC-FID	I1
		I43
lanthanides	(review)	I64
lead	anodic stripping voltammetry	I46 (CH)
		I47 (F)
lead and its compounds	AA	I44
	(review)	I65
lead in paint films	(review)	I45
lead stearate	AA	I8 (R)
lithium and its compounds	(review)	I6
lysine	PC	I48
magnesium and its compounds	(review)	I67
manganese	XRF (EDX and WDX)	I49
manganese and its compounds	(review)	I68
mercury	AA, cold vapor	I1
mercury and its compounds	(review)	I69
mesitylene	GC-FID	L18
metals, trace, in atmosphere	(review)	I50
methanol	GC	I9 (R)
methomyl (insecticide)		I87
1-methoxy-2-propanol	GC	L18
methyl acetate	GC-FID	L18
methyl acrolein	GC	I9 (R)
methyl cellosolve	GC-FID	L18
methyl ethyl ketone	GC	I9 (R)
	GC-FID	L17
	GC-FID	L18
methyl isobutyl ketone	GC-FID	L17
	GC-FID	L18
methyl methacrylate	GC-FID	I1
4-methyl-penten-2-ol	GC	I116 (R)
2-methyl-1-propanol	GC-FID	L18
methylene chloride	GC	I3 (R)
	GC-FID	L18
molybdenum and its compounds	(review)	I70
mutagens		I43
nickel and its compounds	(review)	I71
niobium and its compounds	(review)	I72
nitrated polycyclic aromatic hydrocarbons (PAHs)		I4
nitroaromatics (mutagens)	mutagenic/GC	I88
nitrosamines	GC-TEA	I1
N-nitrosodiethanolamine		I89 (G)
nitrogen dioxide		I91
nitrous oxide		I34
	GC-ECD	I90 (J)
octane	GC-FID	L18
organic acids	GC/anilides	I2
oxygen		I91
ozone		I92 (G)
painter's naphtha (VM&P naphtha)	GC	I114 (P)
	GC-FID	L18
PCBs (polychlorinated biphenyls)	simult steam distn/solvent extraction	I95 (G)
pentachlorophenol	HPLC/UV	I1
		I43
	Cap. GC/ITD	I93 (G)
	immunochemical	I97
pentamethylene glycol	GC	I112 (R)
pentane	GC-FID	L18
perchloroethylene	EPA method 18	I18
pesticides	simult steam distn/solvent extraction	I95 (G)
phenylurea derivatives		I94 (R)
phosphine	change in electrical resistance in sampling film	I98
		I107 (J)
phthalates		I43
platinum group metals and their compounds	(review)	I73
plutonium	leaching from soil with conc HNO <sub>3</sub> or HF	I99
polycyclic aromatic hydrocarbons		I43
propionate (dalapon)		I100 (R)

Table I (Continued)

chemical	technique <sup>a</sup>	ref (lang) <sup>b</sup>
propionic acid	GC/anilides	I2
propyl acetate	GC-FID	L17
propylene glycol dinitrate	ethyl acetate extraction/GC	I101
quartz in coal mine dust	IR	I1
radon and its decay products	(review)	I102
	Alpha-spectrometry	I103 (G)
	(review)	I104 (G)
	Alpha-counting/spectrometry	I105
ribavirin	HPLC/UV	I1
selenium	AA	I106 (P)
selenium and its compounds	(review)	I74
silane (SiH <sub>4</sub> )		I107 (J)
silica, respirable crystalline	X-ray polymer diffraction	I1
silver and its compounds	(review)	I75
silver stearate	AA	I8 (R)
styrene		I47
	personal pressure device/gen atm sampling	I108
	(review)	I109 (G)
	GC-FID	L18
sulfur dioxide	IC	I1
	fuchsin-HCHO/photometry	I110 (R)
	net acidity method	I111
	UV-fluorescence analyzer	I111
	(review)	I76
tantalum and its compounds	(review)	I77
tellurium and its compounds	GC-FID	L18
tetrachloroethylene	GC	I112 (R)
tetraethylene glycol	(review)	I78
thallium and its compounds	(review)	I82
thorium and its decay products	Alpha-spectrometry	I103 (G)
thoron daughters	Alpha-counting/spectrometry	I105
	(review)	I79
tin and its compounds	(review)	I80
titanium	GC	I3 (R)
toluene	GC	I9 (R)
		I34
	GC-FID	L17
	GC-FID	L18
toluene diamines (2,4- and 2,6-)	HPLC/UV	I1
toluene diisocyanate (TDI)	oscillographic method	I113 (CH)
1,1,1-trichloroethane	GC	I114 (P)
	GC-FID	L18
1,1,1-trichloro-4-methyl-4-penten-2-ol	GC	I115 (R)
1,1,1-trichloro-4-methyl-3-penten-2-ol	GC	I115 (R)
trichloroethylene	EPA method 18	I18
	GC-FID	L17
	GC-FID	L18
trichloroisocyanuric acid	HPLC/UV	I22
1,2,3-trimethylbenzene	GC-FID	L18
1,2,4-trimethylbenzene	GC-FID	L18
trimethylolpropane phosphate (TMP-P)		I116
tungsten and its compounds	(review)	I81
turpentine compounds	GC	I9 (R)
uranium, its compounds and decay products	(review)	I82
valeraldehyde	GC-FID	I1
vanadium and its compounds	(review)	I83
welding fumes	PIXE	I117
m-xylene	GC-FID	L17
	GC-FID	L18
o-xylene	GC-FID	L18
p-xylene	GC-FID	L18
yttrium and its compounds	(review)	I84
zinc and its compounds	(review)	I85
zinc dibenzylthiocarbamate	HPTLC	I27
zirconium and its compounds	(review)	I86

<sup>a</sup> Technique abbreviations: AA, atomic absorption spectrophotometry; Cap., capillary; ECD, electron capture detector; EDX, energy-dispersive X-ray fluorescence spectroscopy; FID, flame ionization detector; FPD, flame photometric detector; GC, gas chromatography; HCHO, formaldehyde; HPLC, high-performance liquid chromatography; HPTLC, high-performance thin-layer chromatography; IC, ion chromatography; ITD, ion trap detector; Me, methyl; MS, mass spectrometry; Ni-63, nickel-63 isotope electron capture detector; PC, paper chromatography; PIXE, particle-induced X-ray emission spectroscopy; TEA, thermal energy analyzer; TEM, transmission electron microscopy; UV, ultraviolet detector; WDX, wavelength-dispersive XRF; XRF, X-ray fluorescence spectroscopy. <sup>b</sup> Languages (other than English): B, Bulgarian; CH, Chinese; CZ, Czech; D, Dutch; F, French; G, German; J, Japanese; P, Polish; R, Russian; S, Spanish.

for SRM 1649 (urban dust/organics), SRM 1650 (diesel particulate matter), and SRM 1597 (complex mixture of PAHs from coal tar) (J22). These SRMs were originally developed

to assist laboratories in validating analytical procedures for the determination of polycyclic organic compounds in complex mixtures.

## BIOLOGICAL MONITORING

Biological monitoring is another way to monitor personal exposure in the workplace. Suspected exposures resulting from inhalation, dermal absorption, and oral ingestion can be monitored in exhaled air, body fluids, and tissues, respectively. In many cases metabolites of contaminants produced by the various metabolic pathways can be monitored to determine exposure and biological half-lives. While biological monitoring is a good index of personal exposure, it sometimes requires invasive techniques for sampling, special handling of samples for storage, transportation, and preservation for stability. In addition, careful monitoring records, scheduling of sample collection, and documentation of medical records must be followed in order to interpret results. Despite these requirements, biological monitoring methods were cited most often in review articles from abstract searches conducted for this article, indicating their popularity for industrial hygiene monitoring.

One of the major advantages of biological monitoring is that it is capable of measuring internal exposure to various contaminants. There are three types of measurements used to evaluate internal dose: (1) the concentration of the contaminant itself in various biological media, (2) the concentration of its biotransformation products (metabolites) in the same media, and (3) the determination of nonadverse biological changes that are the results of the reaction of the body to exposure. Generally, the presence of a chemical contaminant or its metabolite in blood, urine, tissues, and/or expired air gives a good index of exposure. The American Conference of Governmental Industrial Hygienists (ACGIH) has established biological exposure indices (BEIs) for a number of contaminants that are known to be hazardous when they are inhaled, absorbed, and ingested by the body (*K1*). Because of the complex biological matrix of samples collected from internal body fluids, tissues, and waste products, special extraction, digestion, and other separation techniques must be used to concentrate and isolate samples for analyses. Preparation of these samples is a key to successful detection and quantitative analyses. Since the last Industrial Hygiene review article was written, most of the improvements in new monitoring methods have involved better separation techniques, derivatization reactions, and increased analytical sensitivity. Both classical wet chemical and instrumental analyses continue to be used for a majority of the monitoring methods.

Biological monitoring of exposure can be divided into two main categories based on whether the contaminant is an inorganic or organic substance. Metals and organometallic contaminants are the primary inorganic substances monitored, while solvents comprise the organic substances monitored. The organic solvents can further be subdivided into classes and subclasses based on functional groups, derivatives, and products such as pesticides, insecticides, and herbicides. Clinical analyses of hormone levels, enzyme activity, and mutagenic and carcinogenic activity also were considered and cited as another important part of biological monitoring.

Atomic absorption, graphite furnace, electrothermal, and inductively coupled plasma emission spectroscopy continue to be the methods of choice for measuring metals in biological samples. Because of their increased sensitivity, specificity, and reproducibility, these methods were repeatedly cited in literature reviews for measuring lead, cadmium, zinc, antimony, arsenic, chromium, and nickel (*K2-K14*). Cold vapor absorption spectroscopy was still utilized for mercury (*K15-K17*). Electrochemical techniques such as potentiometry, polarography, and conductivity were also noted as alternative methods for measuring some metals (*K18-K22*). Ion-selective electrode analysis of bromide, fluoride, and cyanide and ion chromatography of alkaline earth and transition metals were other methods frequently cited (*K23*). Of the metals most frequently analyzed, lead was first, followed by chromium, cadmium, and mercury. Lead was also the most popular metal analyzed in blood and urine samples.

Gas and liquid chromatography were the primary methods of analysis reported for biological monitoring of organic solvents and their metabolites in blood and urine samples. The most frequent aliphatic solvent monitored was hexane and its metabolite, 2,5-hexanedione (*K24-K26*). Benzene, toluene, xylene, styrene, phenol, and aromatic amines and

their respective metabolites were the primary aromatic solvents monitored in urine samples (*K27-K29*). Several methods for monitoring chlorinated solvents (*K30*), isocyanates (*K31-K33*), and specific metabolites from pesticides (*K34*), insecticides, and fungicides (*K35*) were also reported. Improved methods for measuring thiocyanate in blood and urine were cited in a number of papers studying cyanide exposure and metabolism (*K36*). In another paper, biological monitoring of exposure to *N,N*-dimethylformamide was studied by monitoring urinary excretion of methylformamide, formamide, and *N*-acetyl-*S*-(*N*-methylcarbamoyl)cysteine (*K37, K38*). Improved specific enzyme assays for measuring the effects of lead exposure by monitoring blood  $\delta$ -amino acid dehydrase activity (*K39*) and urine *N*-acetyl- $\beta$ - $\alpha$ -glucosaminidase activity were reported (*K40*). Additional assays for monitoring exposure to carcinogens and mutagens using an in vivo micronucleus test in blood (*K41*) and a bacterial test in urine (*K42*) were some of the latest developmental methods reported. Finally, an updated complete guide for biological monitoring methods can be found in the *NIOSH Manual of Analytical Methods*, third edition, fourth supplement, published in 1990 (*K43*).

## PASSIVE DOSIMETERS

**1. Introduction.** Cao and Hewitt have written a review (*L1*), concerning the application of passive samplers to the monitoring of low-concentration organic vapors in indoor and ambient air. In it, they mention Brown's definition (*L2*) of diffusive samplers as being devices which are capable of taking samples of gas or vapor pollutants from the atmosphere, at a rate controlled by a physical process such as diffusion, through a static air layer or permeation through a membrane, but which do not involve the active movement of the air through the sampler. The first quantitative descriptions of passive samplers were published nearly 20 years ago, and several reviews of passive sampling techniques have been published (*L3-L9*). (Some of these were published in earlier Industrial Hygiene reviews, but have been included here for completeness, as referenced in Cao and Hewitt's review, (*L1*). The earlier reviews dealt mainly with the application of passive samplers to workplace monitoring.)

**2. Badges.** A film badge biosensor for hazardous environmental chemicals has been described by Case and Crivello (*L10*). It mimics a biological response mechanism related to tumor initiation in carcinogenesis and is used as a first alert hazard indicator for monitoring situations in which the offending chemical agents may not be known. The badge is activated for air monitoring by slight moistening or is dipped directly into contact with a liquid or soil sample, causing a color development. Its response to over 130 organic and inorganic chemicals has been evaluated, with a high correlation to carcinogens, especially polycyclic aromatic hydrocarbons, aromatic amines and anilines, phenolics, heavy metals (Cd(II) and Cr(VI)), aldehydes, acrylics (acrylonitrile or acrylamide), oxidants, hydrazines, triazine herbicides or chlorinated pesticides, and similar compounds. Sensitivities as low as 10 nmol have been reported.

A Czech patent by Vaneczek and Waldman relates the preparation of activated carbon tablets for chemical dosimetry, by bonding refined carbon with a fused polyethylene (*L11*). A dosimeter with microporous membranes laminated on opposite sides of a microporous support has been described in a U.S. patent by Miksch (*L12*). Michal has developed a new type of soaked wafer dosimeter which includes a gas-permeable, weblike wafer made of a material inert to polyamines, soaked in an absorptive medium. It is called a "zero diffusion path gas dosimeter", and is described in a U.S. patent (*L13*).

A German patent by Wulf (*L14*) includes information about a dosimeter which has at least three layers of poly(ethylene terephthalate) dress lining fabric. Two layers were glued together with very narrow strips of solvent-free adhesive and cut into patches for mounting in commercial photographic slide mounts, sandwiching between them a filter paper impregnated with an indicator or reactant solution. Sampling of gaseous pollutants with an INRS diffusive sampler has been described in a French article by Delcourt, Guenier, and Muller (*L15*). Another German patent, by Kieseles and Tewes, relates the use of dosimeters with reusable electrochemical

measuring cells for monitoring gaseous or dissolved substances. Applying a voltage to the cell electrodes, which are separated by an ion exchange membrane, converts products formed with the electrolyte reagent into secondary products by redox reaction (L16).

Two desorption solvent additives have improved the desorption of polar compounds from activated charcoal in charcoal tubes and in passive diffusive samplers. A desorption solvent of carbon disulfide containing 10% amyl alcohol was found by Kenny and Stratton (L17) to be an excellent desorption solvent for ketones, acetates, alcohols, and hydrocarbons. Beck, Stock, and Whitehead used 5% of 2-(2-butoxyethoxy)ethanol in carbon disulfide (L18) to improve the desorption of 42 polar compounds from charcoal tubes and diffusion badges. See the table in section I for a list of the chemicals referred to in refs L17 and L18.

**3. Tube-Type Passive Samplers.** According to May (L19), diffusive samplers developed in recent years have depended on two essentially different techniques of evaluation: adsorptive collection and subsequent laboratory evaluation, and the use of reagent layers, as in diffusion tubes, giving a direct reading. Charcoal-filled diffusive samplers are desorbed with solvents (e.g., CS<sub>2</sub>) and the desorbate is analyzed by gas chromatography. Diffusion tubes are marked in units of ppm-hours for the contaminant analyzed. The dose, indicated by the length of stain on the tube, is divided by the number of hours the tube has been exposed to the contaminant to determine the concentration in ppm. Comparison measurements with active systems have shown good correlation for diffusive samplers. Bertoni, Canepari, Rotatori, Fratarcangeli, and Liberti have run validation tests on a thermally desorbable double-layer, tube-type passive sampler filled with two different graphitized carbon blacks to determine the influence of air velocity and direction, concentration, and sampling time on sampling rate (L20). They used a special apparatus to generate dynamic artificial atmospheres, making possible the simultaneous sampling of 48 tubes at two different air velocities. Studies were conducted at concentrations to the ppb level.

A Japanese article (L21) by Tanaka et al., contains a method for estimating the service life of a respirator cartridge, using a passive colored tube containing silica gel impregnated with chromium oxide and sulfuric acid placed in the middle of a respirator cartridge containing activated charcoal. Break-through time for the cartridge and stain onset time for the tube as a function of toluene or ethyl acetate vapor concentration (100–1000 ppm) were examined. The stain of the tube was an effective indicator for estimating the break-through time or time to change the cartridge.

**4. Validation.** Another article, by Guild, Myrmel, Myers, and Dietrich (L22), considers the value of a bilevel validation approach for a series of chemically related compounds in a homologous series. Full NIOSH protocol validation is done for the lowest molecular weight member of the series. If that member cannot be validated, the next higher member will be tested. Then the higher members of the series will be tested for analytical recovery, measured sample rate and capacity, and the effects of interferences by other chemicals present—thus the term, "bilevel validation". Cassinelli, Hull, Crable, and Teass (L23) developed the NIOSH protocol for the evaluation of passive monitors, and the Health and Safety Executive (HSE) in Great Britain developed a protocol for assessing the performance of a diffusive sampler (L24). Brown, Saunders, Fields, et al. developed a proposal for a harmonized approach to the evaluation of diffusive samplers in a paper presented at the 1989 American Industrial Hygiene Conference (L25). Guild et al. (L22) mentioned that there are some differences in the protocols developed by NIOSH and the HSE, but the two agencies are currently working together to develop a protocol that is mutually satisfactory. Both protocols cover all parameters that can affect sampling accuracy: concentration, sampling rate and capacity, sample time, reverse diffusion, air velocity, monitor orientation, humidity, temperature, interfering compounds, and storage stability, including personal and area monitoring and comparisons with the results of other monitoring methods.

A computer model for calculating the effective uptake rates of tube-type diffusive air samplers has been developed by Van den Hoed and Van Asselen (L26). Calculated uptake rates show good agreement with values determined experi-

mentally under various conditions of concentration and exposure time. The model is particularly useful for sampling multicomponent atmospheres and has been field tested successfully.

## SPECIFIC CHEMICAL DOSIMETERS

Chemical passive dosimeters utilize reagents to form derivatives which are subsequently analyzed. Many chemicals have been monitored over the past four years, using such techniques. They have been listed in tabular form in Table II.

## SAMPLING STRATEGY AND QUALITY ASSURANCE/QUALITY CONTROL

Griepink and Vandendriessche (N1) have written a chapter titled "Is Everything Under Control?" in the Royal Chemical Society's publication, *Clean Air at Work*. They make the valid point, that "Accuracy cannot be bought from a supplier; it is the result of working with due consideration and care, both at the stage of organizing the laboratory and at the stage of performing the measurements". Their program, aimed at improved quality of measurements, was spurred on by an interlaboratory comparison of dioxin analyses in fly ash at four laboratories which used the same sets of calibrants. Each laboratory had a wide scatter of results, with the ratio of highest result for a given dioxin to the lowest result for the same dioxin being 6 or 7 for each laboratory!

Clayton and Davis have prepared a review on ambient air sampling strategies for environmental pollutants, including sampling considerations, siting of samplers, and interpretation of results for pollutants of industrial and environmental interest (N2). Grosjean described the assessment of exposure to chemical agents in air at the workplace for comparison with exposure limit values and discussed measurement strategy in a draft proposal prepared by Working group 1 of CEN TC 137. CEN is the Comité Européen de Normalisation (European Committee for Standardization) (N3).

A quality assurance/quality control plan was developed by Pritchett, Mickunas, and Kurlick (N4) for a trace atmospheric gas analyzer (TAGA 6000E mass spectrometer/mass spectrometer) used in monitoring indicator chemicals in ambient air during the Love Canal Emergency Declaration Area habitability study. Objectives defined for the study included those directly related to the instrument's performance, setting criteria for accuracy, precision, detection limits, sensitivity decay, and sampling efficiency, plus other objectives concerned with the comprehensiveness of documentation and sampling.

The Health and Safety Executive interlaboratory quality control scheme for isocyanates has been described by Bagon (N5). The scheme is called AQUA (analytical quality assurance) and has been applied to the Health and Safety Executive's method MDHS 25 (organic isocyanates in air: laboratory method using 1-(2-methoxyphenyl)piperazine solution and HPLC). A special measuring chamber has been developed by Bauer, Dahmann, Fricke, Herwald, and Neidhart, where real welding procedures could be performed to determine the concentration of chromium(VI) at welding workplaces, in a study of quality control of measuring procedures by interlaboratory tests (N6).

Freeman has investigated quality control/mark quality assurance and the determination of formaldehyde in stationary source emissions and ambient air, both indoor and outdoor, by the TO-5, TO-11, and CARB 430 methods. He found that accurate results require attention to details not described in the published methods: e.g., keep purified 2,4-dinitrophenylhydrazine reagent and samples at 4 °C, select commercially cleaned bottles, validate them prior to use, and minimize exposure of the reagent to the ambient air. Sep-Pak cartridges work well as a sampling medium for the determination of low levels of formaldehyde (N7).

A method for evaluating function is defined as the expected value of the analysis as a function of the true content of the analyte in the samples, according to Brind, Inge, and Holst (N8). The analytical technique investigated was inductively coupled plasma atomic emission spectrometric analysis for iron, manganese, and titanium from metal-spiked filter samples. Certification limits were set, based on the square



Table II

chemical	reagent, absorbent, adsorbent	technique <sup>a</sup>	ref (lang) <sup>b</sup>
acetaldehyde	enzyme badge	color comparison or reflectometry	M28
acetic acid	porous PVC disks enzyme badge	color comparison or reflectometry	M13 (G) M28
acetone	water		M1
amines, aromatic and primary	1,2-naphthoquinone-4-sulfonic acid	colorimetry	M2
amines, low-mw, aliphatic	cellulose filters impregnated with H <sub>3</sub> PO <sub>4</sub> , or gas washing bottles/HCl	HPLC	M3
amines, volatile, primary and secondary: allylamines, butylamine, diethylamine, dimethylamine, ethylamine, isopropylamine, methylamine	filter impregnated with naphthylisothiocyanate	HPLC	M4
ammonia	fluorescamine on paper substrate coated with sodium lauryl sulfate	fluor.	M5
benzene	carbon-filled substrates diffusive sampler passive sampler	GC	M6 (G) M37 M51
carbon disulfide	carbon-filled substrates	GC	M7 (F)
carbon monoxide		colorim	M8
	Y-type zeolite, Na ion partially exch with Zn ion	thermal desorp, CH <sub>4</sub> form., GC	M9 (J) M10
chlorine	0.1% sulfamic acid soln neutrally buffered 10-mM KI soln porous PVC disks	Cl ion-specific electrode IC	M11 M12 M13 (G)
chlorine oxide	neutrally buffered 10-mM KI soln	IC	M12
dimethylhydrazine, unsymmetrical	citric acid soln	NIOSH colorimetric or coulometric	M23
ethanol	enzyme badge	color comparison or reflectometry	M28
formaldehyde	glass wool/20% diethanolamine distilled water	colorimetry 2-hydrazinobenzothiazole/ spectro	M14 (CH) M15
	filter paper/NaHSO <sub>3</sub> soln filter paper/2,4-dinitrophenylhydrazine (DNPH)	chromotropic acid/spectro desorb w/CH <sub>3</sub> CN; gradient HPLC/UV	M16 M17
	fiber glass/DNPH enzymes	color comparison or reflectometry	M1 M28
	glass fiber filter paper/DNPH diffusive sampler passive monitor		M32 M37 M41
heptane	silica gel/DNPH	methanol desorb/HPLC	M53
hexane	passive sampler		M51
hydrazine; derivatives	carbon-filled substrate	CS <sub>2</sub> Desorb/GC	M19
	polyester sheet coated w/citric acid soln in methanol		M20
hydrazine			M21
hydrogen chloride	p-(dimethylamino)benzaldehyde or vanillin modified NO <sub>x</sub> sampler colored silver complex; removal of Ag <sup>+</sup> as AgCl; complexing agent/pH indicator in a polyacrylamide gel	colorim	M22 M24
hydrogen fluoride	porous PVC disks filter paper/Na <sub>2</sub> CO <sub>3</sub> review	color chart	M25 (J)
hydrogen peroxide	enzymes	color comparison or reflectometry	M13 (G) M26 (G) M27
hydrogen sulfide	detector tube		M28
mercury	hydrar diffusion tubes	length of stain	M29
methyl acetate	silica gel		M30
methylhydrazine			M31
	p-(dimethylamino)benzaldehyde or vanillin	colorim	M21 M22
nitrogen dioxide	glass fiber filter paper/triethanolamine filter paper/triethanolamine effect of temperature on personal samplers Amaya-Sugiura passive sampling method modification diffusive sampler		M32 M34 (J) M35 M36
	κ-carrageenan gel/Saltzman's reagent; succinic acid buffer soln	spectro	M37
nitrogen oxides (NO <sub>x</sub> )	CrO <sub>3</sub> /triethanolamine		M38 (J)
organic solvents	passive organic vapor dosimeters		M33
organophosphonates (using DMMP as a simulant for Sarin)	Tenax	thermal desorp/GC	M39 M49
ozone	diffusive sampler		M37

Table II (Continued)

chemical	reagent, absorbent, adsorbent	technique <sup>a</sup>	ref (lang) <sup>b</sup>
perchloroethylene	passive sampler		M51
phosgene (COCl <sub>2</sub> )	porous PVC disks		M13 (G)
polycyclic aromatic hydrocarbons (PAHs)	diffusive sampler		M40
radon (indoor)	Alpha track detectors, charcoal canisters, Electret monitors	α-particle counting	M41
	activated charcoal canisters		M42
	personal monitor (in homes)		M43
	personal monitor (in homes)	γ-ray detector	M44
	Electret ionization chamber	Electret	M46 (G)
	(electrostatically charged Teflon disk = the Electret)	voltage change	
radon and decay products (daughters)	combined dosimeter		M45 (G)
	side-by-side Alpha-detectors (one chamber with a diffusion filter)	α-particle counting	M47 (G)
sulfur dioxide	diffusive sampler		M37
	passive dosimeters: (1) filter paper/sodium tetrachloromercuriate/silicone interphase; (2) filter paper/sodium tetrachloromercuriates (3) tetrachloromercuriate soln and silicone membrane		M48 (S)
toluene	carbon-filled substrates	GC	M6 (G)
	diffusive sampler		M37
volatile organic compounds (in indoor air)	passive sampler, using hexafluorobenzene as a tracer for ventilation rates		M50
	diffusive passive sampler		M52
(in indoor and outdoor air)	passive sampler		M51
xylene	diffusive sampler		M37
m-xylene	carbon-filled substrates	GC	M6 (G)

<sup>a</sup> See footnote a in Table I for technique abbreviations. <sup>b</sup> See footnote b in Table I for language abbreviations.

root of the relative mean square error. An overview of the Centers for Disease Control quality assurance program, which focuses on data quality assessment procedures for serum dioxin/furan measurements, was presented by Turner, Patterson, Isaacs, and Alexander (N9). Studies included a U.S. Air Force Vietnam veteran ranch hand, NIOSH chemical workers, and data from Seveso, Italy. The same authors reviewed laboratory quality assessment procedures for serum dioxin and furan measurements (N10).

Quality assurance initiatives in Europe have been reviewed by Herve-Bazin (N11), referring to the adoption of the international standards in the ISO 9000 and EN 45000 series. Since formal accreditation of laboratories may not be sufficient to guarantee that results obtained by different laboratories would be the same on identical samples, the laboratory should participate in a program of measurement audits, proficiency testing or interlaboratory comparisons specified by the accreditation body. This paper concentrates on the analysis of prepared samples that are generally supposed to represent air samples at the workplace under real conditions.

Jackson and West (N12) have shared experiences with the Workplace Analysis Scheme for Proficiency (WASP) used in Great Britain at the Occupational Medicine Hygiene Laboratory of the Health and Safety Executive. Performance is assessed in terms of a quantitative performance index, which is a function of the total error of a participant's results. Approximately 30% of the laboratories tested have shown a lack of consistency from round to round which reflects poor internal quality control procedures. They make the statement that "There is an emerging consensus that proficiency testing is the single most effective means of establishing mutual confidence between analytical laboratories".

Quality control and assurance in hair analysis related to arsenic in drinking water in Hungary has been described by Bozsai (N13). Hair samples were collected from a lowland area, where the natural arsenic content in deep well water was 50–200 µg/L. The highest accumulation was among young children, 3–6 years old. Another quality control scheme, in Spain, has been detailed by Bartual (N14), involving the analysis of lead and chromium(III) in occupational hygiene samples over the first four years of operation. An accreditation program for laboratories analyzing lead in air was suggested. (Note: The American Industrial Hygiene Association has established an Environmental Lead Laboratory Accreditation Program.)

Laboratories in the United Kingdom are accredited by the National Measurement and Accreditation Service, according to Firth (N15). In this article, another chapter in *Clean Air at Work*, he states that it is extremely important that the accrediting bodies in different countries, particularly within the European Economic Community, set very similar criteria for accreditation, which "will reduce barriers to trade between countries and simplify procedures and reduce costs for organizations which need to have measurements carried out".

In Belgium, the Bureau of Reference (BCR) program (N16), as described by Vandendriessche, includes projects to improve the accuracy of the determination of volatile organic compounds in workplace air by personal monitoring techniques based on reversible sorption. One method which has been certified by this program is the analysis of aromatic hydrocarbons on Tenax. Similar work is going on for aromatic hydrocarbons on charcoal, and for chlorinated hydrocarbons, using thermal and solvent desorption.

## INDOOR AIR QUALITY

Indoor air monitoring has become a major area of concern in the field of industrial hygiene. With the advent of diseases such as "sick building syndrome" has come the need to monitor the indoor environment for various noxious odors and gases that emanate from building materials, equipment, carpets, and other office supplies. Also included are natural gases and manmade contaminants concentrated within confined work areas with inadequate or poorly maintained ventilation systems. Because of the complex nature and matrix of indoor air contaminants, several review papers have been published on sampling and analytical procedures that attempt to separate and identify these unknown contaminants (O1, O2). The primary contaminants identified so far include volatile organic compounds, formaldehyde, nicotine, carbon monoxide, carbon dioxide, and some polynuclear aromatic hydrocarbons. Most of these compounds are derived from manmade products, while another primary contaminant, naturally occurring radon, produced from the decay products of uranium and radium distributed in trace amounts in the earth's crust in the ground, has been extensively monitored (O3, O4). In addition, formaldehyde and nitrogen dioxide (O5) and even some pesticide monitoring studies have been reported. For each of these different indoor air contaminants, specific sampling strategies and monitoring devices have been re-

ported. For radon gas, charcoal adsorption and  $\alpha$ -particle tracking were the primary monitoring techniques reported (O6). The most frequent monitoring procedure used for volatile organic compounds was passive dosimetry followed by gas chromatography of the desorbed compound (O7). This procedure, when combined with a gas chromatograph equipped with a mass spectrometer, gave even better analytical capability to identify many of the unknown contaminants present.

## FUTURE NEEDS AND TRENDS

Industrial hygiene chemistry has concerned itself with personnel and area monitoring for many years, and rightly so, since our major concern is the occupational health of the working population. This work must go on, at an accelerated pace, if we are to protect our workers from the effects of exposure to harmful chemicals. As analytical techniques, instruments, and methods drive detection limits lower and lower, regulators will undoubtedly continue to lower the permissible exposure limits, putting greater constraints on engineering design for new manufacturing facilities and laboratories. As exposure limits are lowered, sampling and analytical methods are being evaluated at lower concentrations. The original sampling and analytical method evaluation protocol is being revised by NIOSH in the United States, according to Kennedy and Abell (P1). They give a summary of key experiments proposed for the revised evaluation protocol for analytical recovery, sampler capacity/breakthrough time, sample stability, and precision and accuracy (bias).

As more companies achieve globalization of their markets and manufacturing operations, a greater sharing of information and agreement among nations concerning exposure limits, sampling techniques, laboratory measurement techniques, instrumentation, and field applications will be needed. Performance requirements for measuring methods have been described in an article by Zloczynski (P1). The program and status of the European Committee for Standardization Working Group, CEN/TC 137/WG2, are described, and the first Draft European Standard issued by this working group has been issued as prEN 482 (March 1991). This is a general standard that will be used as an umbrella specification for all subsequent standards for workplace air measurements. The main ideas of the general standard are described, followed by a view on further activities in preparing European standards for diffusive samplers, sampling pumps, sampling tubes, and detector tubes.

A relatively new arena for the industrial hygiene chemist is in monitoring the air we breathe in our communities, in the buildings where we work, and outside the perimeters of our plants and laboratories. Sampling, instrumental, and analytical techniques for these types of results will require longer sampling times with passive samplers capable of sampling low concentrations of organic vapors. Thermal desorption and ultrasonic desorption will need to be further developed to include passive samplers other than the tube-type passive samplers which are best served by this technique today. Cao and Hewitt (L1) have suggested that a validation procedure for the application of passive samplers to low-concentration sampling should also be developed. They have suggested that the samplers can be calibrated by exposing them to relatively high concentrations (1 or 10 ppm) for relatively short time periods for environmental monitoring, but further work is required to ensure the validity of this technique. Firth (P3) assessed future trends in assessment and measurement of hazardous chemicals in air, based upon an international symposium held in Luxembourg, Sept. 9-13, 1991, included in the book *Clean Air at Work*, referenced several times in this review. In a discussion of exposure limits, the point was made that the error in a given result may be  $\pm 30\%$ . If so, a 5-ppm exposure result could actually be 3.5-6.5 ppm. Legislative bodies which set these limits have not taken this into account. Practically speaking, our example indicates that, to exceed a 5-ppm exposure limit, the result would have to be above 6.5 ppm, and conversely, to be below the limit, the result would have to be below 3.5 ppm to be 95% certain that the limit had or had not been exceeded. Currently, a result of 5.1 ppm would exceed the limit, whereas a result of 4.9 ppm would not, if the method error was not considered. This area of concern needs to be clarified.

Sampling is a major contributor to the uncertainty in monitoring results and must be validated with an acceptable protocol. An international protocol for sampling and analytical technique validations is needed. Performance standards for analytical methods rather than the development of standard methods allow a flexible approach within different laboratories for the analysis of samples which have been collected by standard techniques (P3). Laboratory accreditations and proficiency testing procedures are being set up, and mutual acceptance of such accreditations between countries is taking place.

Qualitative assessment of areas can be made, using simple field methods, in many cases. These techniques and instruments, ranging from colorimetric methods to portable instrumentation, need to be expanded to other chemicals. The development and improvement of quantitative field instruments is underway. Appropriate remedial action can take place more rapidly and effectively when these instruments are available and trained technicians are available to use them properly. Future trends in the instrumentation arena will lead to the development of "black boxes", which contain expert systems enabling the instrument to think for itself, checking and correcting various functions, according to Miller (P4). Production of the test gas at the sensor will allow automatic calibration.

Most of the work reported in this review pertains to air monitoring. A large segment of the abstracts reviewed related to biomonitoring, i.e., analyzing chemicals or their metabolites in blood, urine, or breath. Another area of concern is the absorption of toxic chemicals through the skin. So, air monitoring, biological monitoring, and surface contamination monitoring are all subjects for further research. Sensitization of workers to various chemicals is an area of concern, since it can lead to allergies or even death in extreme cases. The development of techniques for sampling and analyzing these sensitizing agents alone and in mixtures is another area of research need.

## CONCLUSIONS

As the boundaries between the workplace, the community, and global environments have merged, so will the demand to monitor and control unwanted exposures to environmental air contaminants. First, these contaminants need to be sampled, trapped, and identified for subsequent analyses. Next, once identified, they will be quantitatively measured with some of the most sophisticated and rigorous analytical procedures developed by our analytical chemists today. Hopefully, the results of the analyses will enable industrial hygienists to identify the sources and emissions of these contaminants so that appropriate control measures can be employed to reduce or even eliminate exposure all together. For the time being, the field of industrial hygiene chemistry will continue to rely on innovative analytical techniques as they are developed and apply this science to evaluate potential harmful exposure (Q1). Gas chromatography and high-performance liquid chromatography will still continue to be the most widely used analytical techniques for industrial hygiene samples. Microprocessors and the electronics industry seem to be on the cutting edge of many of the latest developments in automated analytical chemistry, while laser technology seems to be closing in on some of the most sensitive detection systems. There are many challenges ahead in the field of industrial hygiene analytical chemistry, particularly as we shift from existing solvent processes to less toxic alternative substitutes. Continuous improvement of our environment also brings with it the need for additional air monitoring to keep exposures as low as reasonably achievable. These new monitoring methods must be validated for accuracy and precision (A1). Field tests and round robin tests need to be conducted to satisfy quality assurance and good laboratory practices. As new technologies evolve and older ones are improved or phased out, new and better monitoring methods will be needed to evaluate exposure.

Braithwaite et al., (Q2) in their summary report for the international symposium, *Clean Air at Work*, listed several pertinent conclusions and recommendations pertaining to industrial hygiene:

(1) Need more simple, easy, and cost-effective methods, including diffusive methods.

(2) Need more direct-reading, real-time, portable monitoring instruments.

(3) Need more collaboration in the determination of the performance of all types of measuring instrumentation, particularly dust-monitoring instruments.

(4) Quality assurance and laboratory accreditation programs should be harmonized or mutually recognized.

(5) The national databases should be brought under an international umbrella as far as possible.

(6) Measurement quality and databases need to be coordinated at community (European Economic) level (reviewer's note: at international level).

(7) Problems and strategies with regard to monitoring indoor air quality need to be more precisely defined and strategies evolved to deal with them.

(8) Need to determine whether indoor air quality should be controlled by air limit values, by controlling source emissions, or by both.

(9) Discrepancy between indoor air quality criteria and industrial workplace limit values needs to be explained, since the values differ by several orders of magnitude, but are based on similar toxicity and epidemiology data.

(10) To avoid duplication of effort, information, expertise, and resources must be shared at an international level.

They conclude their remarks, and this review concludes, with this statement: "The ultimate objective is to protect the working population, through research, information and practical guidance and the promotion of better health and hygiene practices. In particular, we should address our efforts to the small and medium-sized firms, and to the training of the new generation of occupational hygienists and analysts". To this statement, we can add a strong "AMEN!"

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