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Analytical Instrument Performance Criteria

Field-Portable Spectroscopy

Kevin Ashley, Column Editor

Field-portable methods for monitoring airborne contaminants and toxins in the workplace have received increasing attention in recent years. A number of portable monitors for airborne contaminants have been available commercially for many years. However, new developments may provide for on-site compliance monitoring, which has heretofore been more the exception than the rule. The ability to conduct measurements on-site in the occupational setting offers significant advantages. Field-portable methods are often desired so that decisions regarding worker protection, engineering controls, etc., can be made quickly. The capability for rapid decision making offered by on-site monitoring can help save costs, and also offers a means to assess, and thereby prevent, worker overexposures to toxic substances in a timely manner. Field-based monitoring is especially useful for applications in the construction industry, in agriculture, and in other industries where jobs may be short-term and the workforce is transient. On-site techniques can also be beneficial in instances where short-term monitoring is desired. In this report, field-portable spectrometric techniques are covered, and some applications are described. Due to limited space here, a general overview is presented; more specific information is available by consulting the literature referenced herein.

Portable Gas and Vapor Analyzers

While many commonly used portable gas and vapor analyzers are based on electrochemical or electrical measurement,(1−3) some rely on spectrometric means. Direct-reading instruments that rely on the use of infrared (IR), fluorescence, luminescence, or colorimetry have proven to be most popular.(2−5) Other spectrometries have also been used for on-site real-time gas and vapor monitoring. Some portable instruments (e.g., IR) allow for multi-gas detection, while others are designed for the measurement of single species of interest such as carbon monoxide (CO) or ozone (O_3). Table I summarizes direct-reading spectrometric devices that are commonly used in the industrial hygiene field for monitoring gases and vapors.

Most portable infrared gas analyzers are non-dispersive IR instruments that ordinarily require a plug-in power source.(2,3,6) Some dispersive instruments have also been introduced.(2) Battery-powered instruments have been produced, but these are generally species-specific; for instance, an IR photometer for monitoring carbon dioxide (CO_2) over a wide concentration range relies on nickel-cadmium (Ni-Cd) batteries as an optional power source. Portable multi-gas IR analyzers are becoming more popular, as their applicability is enhanced through interfacing of the instrument with spectral libraries via computer. A wide variety of organic and inorganic gases can be monitored semi-quantitatively or, in some cases, quantitatively. For example, portable IR monitors for CO are able to quantitatively measure this dangerous compound in the parts per million (ppm) range, where such concentration levels are potentially hazardous to life and/or health.

Field-portable Fourier transform infrared (FTIR) instruments are now available,(7,8) and a national voluntary consensus standard has been published

TABLE I
Portable spectrometric techniques for monitoring gases and vapors

Instrumental technique	Applicable analyte(s)	Comments
Infrared photometers	CO, CO_2, NO_x, N_2O, SO_2, hydrocarbons, fluorocarbons, etc.	Detection limits: sub-ppm to few percent range; single-species or multi-gas devices
Colorimetric detection	Formaldehyde, toluene diisocyanate, HCN, Cl_2, H_2S, SO_2, NO_x, etc.	Sub-ppm detection limits for most species; specific for certain target analytes
UV/visible photometers	Hg vapor, O_3, SO_2, NO_x, NH_3, organic vapors, etc.	Sub-ppm to ppm detection limits; analyte-specific
Chemiluminescent detectors	O_3, NO_x	Highly sensitive and selective; detection limits: $\sim$10 ppb
Photometric analyzers (includes devices based on flame photometry, fluorescence, other)	CO, SO_2, sulfur compounds, halogenated hydrocarbons, phosphorus compounds, etc.	Measurement in ppb to ppm range, depending on type; single- and multi-species devices

that describes a portable FTIR method for determining gaseous compounds.(9) IR and FTIR monitoring allow for real-time or near real-time measurement of numerous toxic gases and vapors, and have applications in many occupational settings. An advantage of IR or FTIR monitoring is that sample preparation is minimal, and gases and vapors may be monitored following sampling by using a suitable sampler; alternatively, no samples may need to be obtained at all (depending on the application). Open-path FTIR for real-time in situ monitoring of airborne gaseous pollutants has become popular for remote sensing, and also offers promise for applications in occupational settings.(10,11) A new IR spectral database standard, National Institute of Standards and Technology (NIST) SRD 79, has been made available.(12) This NIST IR database contains absorption coefficient data for 21 hazardous air pollutants and provides for quality assurance for quantitative FTIR open-path in situ measurements of these species.

Colorimetric and ultraviolet (UV)/visible spectrophotometric monitors for gases and vapors are widely used in the industrial hygiene field. Most of these monitoring devices (e.g., for ozone, mercury vapor, oxides of nitrogen, ammonia, and sulfur dioxide) function by means of UV absorption.(2,13) Colorimetric monitors ordinarily employ a reaction between a selective reagent and the analyte of interest in order to form a colored complex that can be measured in the visible spectral range. Most commercially available colorimetric and UV/visible photometers require an external power source for their operation. However, there are portable devices that can be operated using battery power (e.g., some mercury [Hg] and sulfur dioxide [SO_2] monitors). Portable, battery-powered devices are useful for personal monitoring, while monitors requiring an external power source are limited to use as area (static) monitors. UV/visible and colorimetric instruments are able to detect most species of interest in the ppm range, and many are equipped with alarms if readings are high. Many of the UV/visible direct-reading instruments offer continuous monitoring capability, with response times of approximately one second for measurements in the ppm range.

Direct-reading chemiluminescent detectors offer a means for measuring ozone (O_3) and oxides of nitrogen (NO_x), with excellent specificity and high selectivity. The operation of chemiluminescent detection involves excitation of O_3 molecules via chemical reaction, and the subsequent detection of photon emission from the excited state species, which may be either an intermediate or a product of the reaction.(14) Ozone may function as a reagent for NO_x analysis or, of course, as the analyte of interest.(15) Luminol has also been used for field-portable monitoring of nitrogen dioxide (NO_2),(16) and ethylene (C_2H_4) has been employed as a reactant for field-portable monitoring of ozone.(17) Due to high power needs, field chemiluminescence instruments generally require an external power source.(2,8)

Photometric analyzers are widely used in the industrial hygiene field for the on-site real-time monitoring of numerous gas and vapor species. The detector is operated via measurement of light emission from a high-temperature H_2 flame.(18) Flame photometric detectors are useful for selective measurement of gaseous sulfur or phosphorus compounds, with detection limits in the parts per billion (ppb) range for these species. The high power required for operation of these devices necessitates the use of an external power source.

Portable photoionization detectors (PIDs) for organic vapors are commercially available.(19) These require less power than do the aforementioned photometric analyzers, and simple hand-held, battery-powered devices can be used to monitor volatile organic compounds (VOCs) in real time. However, for enhanced performance (e.g., minimizing of interferences from more abundant hydrocarbons), PIDs can be used as detectors for portable gas chromatography (GC) instruments.(20)

Fluorescence analyzers are available for monitoring CO and SO_2. A xenon (Xe) or Hg arc lamp is used to excite the analyte species, which give rise to sufficient fluorescence intensity so that detection limits in the few ppb range can be achieved.(2) Photomultiplier tubes are used for detection. Like photometric analyzers, there is a need for high power in order to facilitate source excitation and operation of photomultiplier detectors; therefore, an external power source is required. The performance of the portable fluorimetric SO_2 analyzer has been shown to be equivalent to that of a colorimetric reference method promulgated by the U.S. Environmental Protection Agency (EPA).(8)

Other types of photometric analyzers are commercially available, whereby alternative chemical strategies may be used to produce a spectral signal or color change.(2) Spectral intensity analyzers, for example, can be used for general, nonspecific monitoring of halogenated hydrocarbons. Other types of photometers allow for automated sampling by use of media that undergo a color change upon reaction with target analytes (e.g., such as that already mentioned for monitoring SO_2). Reflectance may be used for the measurement of a variety of species such as ammonia, phosgene, HCN, and arsine, with detection limits in the ppm range. In a few cases, field portability and on-site monitoring applications are enhanced by the use of battery power rather than an external power source.

New developments in spectrometric gas and vapor monitors have provided for better detection limits and other attributes. Fiber-optic chemical sensors for continuous monitoring have recently been a topic of wide interest,(21) and applications in the measurement of gases and vapors are widespread. Fiber optics have proven to be useful for the design of field-portable devices for optical,(22,23) fluorimetric,(24) and infrared(25) monitoring of species such as VOCs, Hg, and explosive agents. The use of optical sensor arrays for multi-species monitoring is also an area of significant promise.(26) Miniaturization of time-of-flight mass spectrometric devices has allowed for the on-site monitoring of gaseous analytes, with excellent prospects for multi-species monitoring.(27) Hand-held ion-mobility spectrometric (IMS) instruments have found use in industrial hygiene and military applications.(28) In recent work, a hand-held IMS device was used to monitor VOCs on-site in the workplace.(29) The performance of IMS instruments is enhanced when used as a detection scheme following GC separation.(30,31)

Portable Aerosol Monitors

Direct-reading portable aerosol monitors that are used for industrial hygiene purposes are often based on light-scattering or light-attenuation properties (see Table II). The most widely used are light-scattering devices known as aerosol photometers or nephelometers.(3) Light-attenuating photometers are also available, as are other real-time aerosol monitors that are not based on optical techniques.(32) Portable instruments have been developed that are applicable over different aerosol size ranges, and each device has its own benefits and limitations.

Aerosol photometers operate by directing polychromatic light toward an aerosol as it is passed through an optical chamber, and by measuring the light that is scattered at a chosen scattering angle with respect to the incident light beam. Optical aerosol particle counters use a monochromatic light source such as a laser, photodiode, or tungsten filament lamp to illuminate the aerosol sample. For both photometers and optical particle counters, photomultiplier tubes or photodiodes are generally used for detection of the scattered light. Smaller scattering angles are best for the detection of large particles, while a scattering angle of 90° offers maximum sensitivity for small particles.(3) Many factors contribute to the light scattering profile, e.g., wavelength of the incident light beam, size and shape of the particles, refractive index of the particle, density and concentration of the aerosol, and size distribution of the aerosol.(33) Thus, it can be seen that these devices may suffer from numerous limitations that can restrict their applicability for quantitative monitoring; therefore, they are generally not used for compliance monitoring purposes. Nevertheless, they are very useful as on-site screening instruments, and for semiquantitative measurement of aerosol concentrations.

Several popular instruments for estimation of aerosol concentrations in real-time are based on light-scattering methods, and a variety of techniques have been employed to improve their performance.(3,34) Techniques for sampling and analysis have been developed for both "extracted" samples and in situ analysis.(32) Several devices utilize monochromatic IR or near-IR radiation to illuminate the sample, and some employ size-selective devices to isolate the aerosol range of interest. These devices are generally applicable for monitoring aerosols that are about 1.0-μm aerodynamic-diameter or greater, and are, therefore, not useful for the detection of small-diameter aerosols. However, some have been claimed to give accurate aerosol concentration measurements over the range of 0.01 to 100 mg/m^3 for aerosols of 0.1 to 20 μm in diameter.(3)

TABLE II

Direct-reading optical aerosol monitors

Type of monitor	Applicable aerosol size range (aerodynamic diameter)	Comments
Aerosol photometer or nephelometer	0.1 to 1.0 μm	Integral light scattering; aerosols with same size distribution
Optical particle counter	$\sim$0.1 to $>$10 μm	Monochromatic or polychromatic source; light scattering
Condensation nucleus counter	$<$0.01 to 1.0 μm	Particles enlarged for photometric measurement

For measurement of very small aerosols, condensation nuclear counters (CNCs) are usually employed. The CNC functions by actually enlarging the aerosol particles to a size that can be measured photometrically.(3,32) This is usually accomplished by subjecting the ultrafine aerosol to a vapor, and then cooling the mixture to cause super-saturation. In this manner, the aerosol particles operate as condensation nuclei upon which the super-saturated vapor can nucleate and cause the aerosol particles to grow in diameter. The intensity of scattered light can then be used to measure the concentration of the enlarged particles. CNCs are widely used for testing high-efficiency particulate air filters in respirator fit-testing.(35)

With some exceptions, real-time aerosol monitors are survey instruments that can only be used to measure total concentrations of airborne particulates. Furthermore, the size ranges of aerosols that real-time aerosol monitors can measure tend to be limited.(36) Direct-reading, real-time aerosol monitoring instruments generally cannot give any information on the identities of airborne contaminants that may be present in the test aerosol. However, in recent instrument developments, efforts have been made to obtain more species-specific information using real-time optical monitoring. For example, a real-time monitor for respirable particles based on laser light scattering was said to be capable of detecting 0.1 fiber/cm^3 with very short sampling times.(37) The instrument uses a diode array to sense scattered light, and then assigns a particle to a particular class depending on its scattering characteristics. In most direct-reading instrumental applications, knowledge about the specific makeup of the aerosol being monitored is necessary before a survey instrument is employed.

Portable methods for measuring chemical species in captured aerosols ordinarily require that an aerosol sample be prepared and analyzed on-site in the field. In some cases, sample preparation may be minimal, while in other instances, considerable sample treatment may be needed prior to on-site analysis. The option to use a field-portable analytical method depends on the needs of the user. If it is desired to have an analytical result quickly, then it may be necessary to perform on-site analysis. Another possible reason for conducting analysis on-site has to do with the reactivity of target analytes that can exist in the sampled aerosol.(38) Reactive compounds may need to be analyzed quickly in the field using field-portable instrumentation, since the chemical form(s) of the analyte(s) of interest may change if samples are stored for too long a time period (e.g., for subsequent fixed-site laboratory analysis).

Various field-portable spectrometric techniques for on-site determination of heavy metals in collected aerosol samples have been evaluated recently. For instance, portable X-ray fluorescence (XRF) has been used to measure metal species in air filter samples.(39,40) In one study, portable XRF was used to determine metals in filters that were prepared from aerosolized metal oxides.(39) Excellent quantitative results for up to 18 metals were obtained, although detection limits for lead (Pb) and cadmium (Cd) were somewhat high compared to the action levels of interest. Very good correlations were achieved between portable XRF data and results obtained using a laboratory XRF instrument. Thus, portable XRF offers the potential for on-site multi-element monitoring of aerosol filter samples collected in the field. Because the method is non-destructive, the samples can be analyzed subsequently for metals content using a confirmatory technique such as inductively coupled plasma (ICP) spectrometry.

In related studies, modern portable XRF devices have been used to determine lead in filter samples, with a view toward obtaining lower detection limits for this metal. In one investigation, air filter samples were collected from construction sites where lead paint removal activities were undertaken.(41) The lead loading range of the data set was 0.1 to 1500 μg of lead per sample. Portable XRF measurements were conducted on the filter samples using a protocol that accounted for the variability in the density of the aerosol that was deposited on the filters. A National Institute for Occupational Safety and Health (NIOSH) reference technique, graphite furnace atomic absorption spectrometry (GFAAS), was used for confirmatory analysis and method evaluation purposes.(42) For the portable XRF method, a lower detection limit of $\sim$6 μg Pb/filter was determined, and the portable XRF method accuracy was $\pm$16.4 percent. The performance of the portable XRF instrument indicated that the device can be used for the quantitative analysis of lead air filter samples over a wide concentration range. The practicing industrial hygienist can use portable XRF to produce a rapid, on-site determination of lead exposure that can immediately be communicated to workers and help identify appropriate levels of personal protection.

Some other techniques that offer promise for on-site multi-metals spectrochemical analysis include laser-induced breakdown spectroscopy (LIBS)(43) and spark-induced breakdown spectroscopy (SIBS).(44) While the LIBS instrument has recently been commercialized, its cost remains prohibitive. Prototypes of both LIBS and SIBS have been evaluated for their ability to determine a number of heavy metals in air samples. For metals such as lead, LIBS may offer lower detection limits than portable XRF devices.(45) Efforts to make LIBS devices more easily field-portable have focused on the use of fiber optics.(45−47)

Spectrometric methods for the on-site analysis of species present in aerosols following a sample dissolution step have been published. For example, a field-portable method for the determination of airborne hexavalent chromium [Cr(VI)] was recently developed and evaluated.(48) The procedure employed ultrasonic extraction(49,50) in order to solubilize Cr(VI) in test samples. Subsequently, solid-phase extraction using strong anion exchange was used to separate Cr(VI), which is anionic under basic conditions, from Cr(III) and other metal cations. Following elution of the isolated Cr(VI), trace concentrations of this species were measured using the diphenylcarbazide method(51,52) by means of a field-portable, battery-powered spectrophotometer. The field-portable method performed equivalently to fixed-site reference laboratory methods used to determine Cr(VI).(53) It is expected that additional field-portable spectrometric measurement methods for more analytes will become more widely used in the future for industrial hygiene monitoring.

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EDITORIAL NOTE: This column is an update of material previously published within a chapter of the *Encyclopedia of Analytical Chemistry*.(54) Kevin Ashley, Ph.D., is with the U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, 4676 Columbia Parkway, Mail Stop R-7, Cincinnati, Ohio 45226-1998 (USA); telephone: (513) 841-4402; fax: (513) 841-4500; e-mail: kashley@cdc.gov. This article was prepared by U.S. Government employees as part of their official duties and, legally, may not be copyrighted in the United States of America.