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Guide to portable instruments for assessing airborne pollutants  
arising from hazardous wastes

Guide sur les instruments portatifs pour l'évaluation des polluants contenus dans l'air  
en provenance des sites de décharge de déchets dangereux

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ORGANISATION INTERNATIONALE  
DE MÉTROLOGIE LÉGALE

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INTERNATIONAL ORGANIZATION  
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## Foreword

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**GUIDE to PORTABLE INSTRUMENTS  
for ASSESSING AIRBORNE POLLUTANTS  
ARISING from HAZARDOUS WASTES**

**1. Scope**

1.1. This Document provides definitions and guidelines for selecting portable instruments to measure airborne pollutants at hazardous waste sites. A brief description of several types of instruments, including some of their important metrological and technical characteristics, is given.

1.2. This Document also provides background and literature references on the application of these instruments. Emphasis is placed on methods and requirements for testing and calibrating instruments. Specifics of sampling methods and of instrument type evaluation and approval are not addressed. This Document is not intended to be a Recommendation for legal pattern approval testing, initial and subsequent verification, or metrological control of the instruments covered.

Note: Some of these portable instruments may become the subject of future OIML Recommendations. Transportable and mobile instruments for field measurements are not covered except for a brief discussion of their applicability as given in point 2.

1.3. The general application of portable instruments at hazardous waste sites is described. Then, information is provided for the following instruments: (a) compound-specific instruments, (b) chemical detector tubes, (c) total hydrocarbon (THC) analyzers, (d) gas chromatographs (GCs), (e) infrared (IR) analyzers, and (f) dust monitors. Table 1 lists examples of field portable and transportable and mobile instruments.

Note: Air sampling pumps and instruments for measuring radioactivity that may be required in this area of application are not covered in this Document.

Table 1  
Instruments for field measurements of hazardous wastes

Portable instruments	Transportable and mobile instruments
Chemical detector tubes	Atomic absorption spectrometers
Compound specific instruments	Fourier transform infrared spectrophotometers
Dust monitors	Gas chromatographs
Gas chromatographs	Gas chromatographs/mass spectrometers
Infrared spectrophotometers	Inductively-coupled plasma emission spectrometers
Total hydrocarbon analyzers	Liquid chromatographs
	Multistage mass spectrometers (MS-MS)
	X-ray fluorescence spectrometers

- 1.4. This Document is intended for government officials responsible for developing measurement regulations for hazardous waste site activities and for those responsible for making or overseeing actual field measurements. It should also be useful for nonregulatory organizations with similar responsibilities. This Document should be applicable for undergraduate, graduate, or continuing education on this subject. In addition, it should be of assistance to instrument manufacturers.
- 1.5. Specific measurement methods associated with field measurements are not covered in this Document. A potential user, however, should select and apply a field instrument for assessing airborne pollutants after consideration of the following points: (a) that pollutants to be measured are known or unknown, (b) that potential interferences of other gases with a measurement of compounds of interest are taken into account, and (c) that the instrument is calibrated at appropriate intervals with specific gases associated with the pollutants to be measured.
- 1.6. This Document does not address requirements and tests for instruments that may be necessary for personnel safety. Users, therefore, should determine that such instruments meet the intrinsic safety and labeling requirements according to national regulations.

## **2. Application**

- 2.1. Metrological procedures have been documented for environmental analytical purposes [1,2]. Pollution from hazardous wastes represents a special type of measurement problem because of the generally unknown nature of the compound(s) to be analyzed, the episodic nature of emissions, and the multiphase nature of the problem. Assessing airborne pollutants at hazardous waste sites using multimedia sampling techniques has confirmed the difficult nature of such monitoring [3]. Similar problems are encountered in instrumentation used to analyze hazardous waste material and contaminated water and soil.
- 2.2. The use of portable instruments, particularly real-time or near real-time instruments, for monitoring at hazardous waste sites has been specified in some national guidelines. For example, in the United States, these guidelines have been issued by the U.S. Environmental Protection Agency (EPA) and recommended by the U.S. National Institute for Occupational Safety and Health (NIOSH). In particular, requirements for the use of a portable THC vapor monitor based on photoionization (PID) or flame ionization (FID) detection are outlined [4,5]. Requirements for personal protection equipment for instrument operators, as well as site sampling protocols, are also provided in the literature [3-5] and in national regulations [6].
- 2.3. The current practice is to use portable instruments that can be taken directly onto a hazardous waste site. In addition to the THC analyzers, such instruments include GCs, IR analyzers, chemical detector tubes, compound-specific instruments, and dust monitors. Some information about the metrological and technical performance characteristics of these instruments is usually provided by manufacturers.

- 2.4. In some emergencies and accidents, hazardous pollutants may be released into the environment, and may involve one or more chemicals in various concentrations and physical states. Such releases may have either short-term or long-term effects and involve one or more of the environment's media: soil, surface water, groundwater, and air [7]. Examples of accidents that could involve such releases include: train derailments, truck and ship accidents, storage tank spills and leaks, chemical and petroleum facility and vessel fires, and chemical warehouse fires. Although some of these examples do not fit the usual definition of "hazardous waste sites", the same portable instruments have been demonstrated to be effective in both general applications.
- 2.5. At hazardous waste sites, the handling of unknown chemicals in various conditions may be required and, therefore, may potentially result in an emergency. A pollutant release usually involves one or more chemical compounds in concentrations ranging from nonhazardous to acutely hazardous levels. Hazardous waste sites, therefore, may adversely affect the surrounding environment in one or all of the exposed media (soil, surface water, groundwater, and air). Containment and remedial action at hazardous waste sites may range from on-site chemical treatment to off-site disposal of materials. The following are examples of sites where various types of containment and remediation may be required: unlined lagoons, above ground storage tanks, below ground storage tanks, above ground or buried drums, chemical spills, incinerators, soil contamination sites, illegal sanitary landfills, abandoned chemical plants and warehouse facilities, illegal river and bay dumping, and mineral tailings from mining operations.
- 2.6. Portable instruments can assist in determining if acute concentrations of chemicals are present in the environment, and some types (for example, THC analyzers, GCs, IR analyzers, and dust monitors) may be capable of identifying the presence of low level pollutants [8]. While such instruments may not always be capable of identifying specific compounds or making repeatable concentration measurements in field applications, they are extremely useful in locating the environmental origin of the pollutants. This measurement capability is usually also helpful in determining the extent of evacuation zones, sampling plans, occupational and public health medical surveillance, public access, and containment and remedial actions. When equipped with a data recording device, real-time analyzers may be used for measuring long term time-weighted average concentrations.
- 2.7. Transportable instruments are often used to supplement the analysis obtained by portable instruments at hazardous waste sites including emergency or accidental chemical releases. Some hand-portable instruments have detection limits for specific compounds below 0.5 ppm. Such limits may not be realizable in the field in ambient air, which may contain a variety of other compounds. Therefore, the hand-portable instruments are most useful when determining the presence or absence of a specific compound, or total hydrocarbon and dust concentrations. Transportable instruments, however, are usually capable of measuring concentrations at a detection limit well below 0.5 ppm under most field conditions, and are very useful for spot-check measurements to either identify or verify the possible pollutant. The disadvantage of using transportable instruments in the field as compared to portable instruments is that they are usually bulky and require nonportable sources of power and utilities.

2.8. Since airborne pollutants may have a direct impact on both occupational and public health and safety, it is extremely important that the sampling-analytical turnaround time should be as short as possible in most cases. Measurement results are often needed quickly on-site to coordinate operational cleanup and remedial activities and to implement timely occupational health, public health, and safety activities. Analytical results obtained in a mobile laboratory, therefore, may be more advantageous than those from an off-site operational laboratory in support of field monitoring activities. The analyst and other laboratory professionals involved could also assist in providing advice on sampling and precautions in carrying out field work. If rapid on-site analytical information is not required, then samples may be analyzed off-site at a stationary laboratory that could be operated on a 24-hour basis, if necessary. These laboratories would be expected to yield higher repeatability and accuracy of analysis because of the higher quality of the instruments available for sample preparation, handling, and analysis.

2.9. Thus, the requirements for the applications noted in points 2.1 through 2.8 may be satisfied as follows: samples taken and analyzed off site using laboratory-based instruments, samples taken and analyzed at the hazardous waste site using transportable instruments mounted in a mobile laboratory, or samples taken and analyzed on site using hand-portable instruments.

2.10. The portable instruments covered in this Document are generally designed for detection of pollutants around the concentration range of the personal exposure limit (EL). In order to be effective in personnel monitoring, such instruments would require having, or being associated with, a data recording device.

Note: Exposure limit (EL) means the maximum acceptable concentration (MAC or MAK) in the workplace of a hazardous airborne pollutant and is expressed as a time-weighted average (TWA) over an eight-hour workday. Several other terms synonymous with MAC, including "threshold limit value" (TLV) and "permissible exposure limit" (PEL), are frequently used in national regulations and recommendations. It should be noted that EL values may vary significantly because each one may be based on different toxicological, epidemiological, chemical and regulatory considerations [9-11].

### **3. Terminology**

Note: A primary source of definitions of terms used in metrology is reference [12]. A list of abbreviations used in this Document is given in Annex A. These abbreviations are principally associated with measurements, instruments, and devices.

#### **3.1. Accuracy**

The closeness of the agreement between the result of a measurement and the conventional true value of the measurand.

### 3.2. Calibration

The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system and the corresponding known values of a measurand.

### 3.3. Certified reference material

A reference material one or more properties of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body [13, 14].

### 3.4. Control charts for an instrument

Graphical plots of test results from the same or similar samples or processes with respect to time or a sequence of such measurements, together with the limits within which the measurement values are expected to lie, when the instrument is in a state of statistical control [15,16].

### 3.5. Conventional true value

A value of quantity which, for a given purpose, may be substituted for the true value.

### 3.6. Detection limit

The concentration of a substance that will yield a mean output signal equal to three times the mean short term noise level, as determined on a statistical basis.

Notes: 1) The detection limit is sometimes defined as an output signal equal to some other multiple (e.g., two or ten) of the noise level [2]. The noise level is the random background output signal of an instrument when sampling a blank or clean filtered air.

2) In this Document, the concentration of an airborne pollutant in either a gas or vapor phase is most often expressed in terms of the volume fraction of the pollutant in air: parts-per-million (ppm) [v/v ( $10^{-6}$ )]. Conditions of temperature and pressure shall be specified for conversions to units of mass per unit volume (usually  $\text{mg}/\text{m}^3$ ). Standard conditions may be specified, and for temperature may be  $0^\circ\text{C}$  or  $20^\circ\text{C}$  and for pressure one atmosphere or 101 325 Pa.

### 3.7. Dynamic range of a detector

The range of concentrations over which a detectable change in output signal is produced by an incremental change in concentration of a substance. The lower limit is given by the detection limit, and the upper limit occurs at the point of detector saturation. The value of the dynamic range is expressed by the ratio of the upper to the lower limit and is larger than or equal to the linear range.

### 3.8. Hazardous waste site

A location containing hazardous chemical wastes that can affect or have the potential to affect a larger surrounding area. This applies to any site of uncontrolled hazardous waste dumping, a licensed hazardous waste disposal facility, or a location of accidental hazardous or toxic chemical spills or fires.

3.9. Linear range

The range of concentrations over which the output signal of the instrument is proportional to the concentration of the substance to within a specified percentage, for example  $\pm 5-10\%$ .

3.10. Method

The distinct adaptation of a technique for a selected measurement purpose.

3.11. Primary standard

A measurement standard which has the highest metrological qualities in a specific field.

3.12. Procedure

The set of written directions necessary to use a method effectively.

3.13. Reference material

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

3.14. Repeatability

The closeness of the agreement between the results of successive measurements of the same quantity of a pollutant carried out by the same instrument and under the same conditions of use within a relatively short period of time. Repeatability is synonymous with the term "precision" for an instrument.

3.15. Secondary standard

A measurement standard whose value is fixed by direct comparison with a primary standard.

Note: Most instrument calibrations are performed using secondary standards.

3.16. Statistical control (of measurement)

A means for determining whether the results of measurements using a method, instrument or process over a relatively long time interval are within specified limits established by taking into account the uncertainty and error of measurement.

3.17. Traceability

The property of a result of a measurement whereby it can be related to appropriate standards through an unbroken chain of comparisons.



## 4. Compound specific instruments

### 4.1. General description

4.1.1 A compound specific instrument is designed and calibrated to identify and quantify a specific compound or class of compounds in either gaseous or vapor form in the environment. Toxic gases and vapors may be detectable down to the ppm range with good specificity and rapid response time. Most portable instruments incorporate either electrochemical or semiconductor detection elements and diffusive sampling techniques, that is, those involving diffusion of the pollutant to the detection element [8]. The user should select the instrument type based upon performance appropriate or required for the intended application rather than upon a particular design.

Note: Diffusive or passive sampling utilizes devices that sample at a rate controlled by Fick's first law of diffusion; active sampling utilizes air sampling pumps. Colorimetric instruments are not covered in this Document.

4.1.2. Electrochemical detection elements utilize a variety of principles. The most common elements are galvanic cells, polarographic cells, potentiometric cells, amperometric cells, and potentiostatically-controlled electrode cells. The pollutant may be oxidized or reduced at the working (indicator) electrode. Depending upon cell type, a current or change in cell equilibrium is generated by this reaction in relation to the partial pressure of the pollutant in the environment. Electronics transform the resulting signal to provide a visual and/or audible indication of the pollutant concentration. The specificity and response time depend largely upon the electrode materials and the type of diffusion element or gas porous membrane used. Different designs of electrochemical detection elements may be available from different manufacturers.

4.1.3 Semiconductor detection elements operate on the basis of an interaction between the gaseous pollutant and the semiconductor material that changes in conductivity as the pollutant is absorbed or oxidized on the semiconductor surface. The reaction involved is complex, and the specificity, interferences, and power consumption differ from electrochemical detection elements.

4.1.4. Portable instruments have detection limits in the ppm range. The simplest instruments operate by a threshold-detection alarm; others provide an analog or digital data display; and some have memories that can provide output displays of both the real-time concentration and the time-weighted average concentration values. Minimal drift of the zero, span, and calibration controls is a desirable feature. In many applications, the instrument may also have an "intrinsically safe" requirement [8].

## 4.2. Pollutants detected

The pollutants detected may be inorganic or organic gases or vapors. However, in general, these instruments are used for detecting inorganic gases or vapors because of the higher specificity of the detection elements for inorganic gases (e.g., O<sub>2</sub>, CO, CO<sub>2</sub>, NO, NO<sub>2</sub>, NH<sub>3</sub>, N<sub>2</sub>H<sub>4</sub>, SO<sub>2</sub>, H<sub>2</sub>S, HCN, COCl<sub>2</sub>, etc.). Filters may be used by some manufacturers to increase the specificity for certain pollutants.

## 4.3. Metrological and technical characteristics

4.3.1. An instrument's output signal over its stated linear range should be linear within  $\pm 5\%$ . Its accuracy should be within  $\pm 2$  ppm or  $\pm 10\%$  of its reading, whichever is greater, at a constant temperature. The instrument's repeatability should be within  $\pm 1\%$  of the full-scale (dynamic range) reading, or  $\pm 0.5$  ppm, whichever is greater. The temperature range for operation should be at least from 0 °C to 40 °C.

4.3.2. The detection limit should be between 0.1 and 1.0 ppm, and should be one half or less than the personal EL. The linear range should be from the EL to a minimum of five times that value. For some applications, it may be desirable to use instruments with a linear range exceeding five times the EL.

4.3.3. Calibration should be performed as required using a reference gas standard to provide a concentration at or near the personal EL of the compounds to be measured. It is recommended that calibration should be carried out using two or more concentrations of the reference gas in order to verify the response over the full range of concentrations anticipated in use. The calibration should be performed especially after an instrument has been exposed to potential detector poisons, severe weather conditions, or rough handling.

4.3.4. Compound specific instruments often respond to pollutants other than the specific compounds to be measured or to the compounds which they were designed to detect. Therefore, information should be supplied by the manufacturer indicating the probable air pollutants that may interfere with the detection of the specific compounds to be measured.

Note: For example, an instrument with an electrochemical detector may be labeled as an "H<sub>2</sub>S monitor/alarm", but also may have a significant response to HCN. Materials at hazardous waste sites usually evolve gases of unknown composition; therefore, all gases that may be responsible for an instrument's response under given conditions should be considered. Instruments labeled "toxic gas meters" without information on specificity and interferences shall not be used.

## 5. Chemical detector tubes

### 5.1. General description

5.1.1. A chemical detector tube is a hollow tubular shaped glass body containing one or more layers of chemically impregnated inert material. Detector tubes are normally supplied with both ends fused closed. To use, the fused ends are broken off and a specific volume of air is drawn through the tube with a hand operated aspirating pump. The tube, pump, and other necessary components together form the measuring unit and are supplied by the same manufacturer [17,18].

5.1.2. The chemicals contained within the packing material undergo a chemical reaction when exposed to an airborne pollutant. The exposure produces a color change during the sample intake of each pump stroke. After a prescribed number of pump strokes, corresponding to a specific air volume, the concentration of a pollutant is indicated by the discoloration length on a calibrated scale printed on the detector tube, if it is a length-of-stain type. The scale values for a specific detector tube type are calibrated for its contents, volume, and specific pollutants and for a specific air delivery from its pump; therefore, detector tubes and pumps of different manufacturers may not be interchangeable. For many pollutants, the color change is distinctive; however, the presence of other interfering gases and vapors may cause some color diffusion. Manufacturers should provide information on this possible effect.

5.1.3. In another type of commercially available detector tube, detection depends on color comparison, or color matching, of detector tubes. With this type, a specific number of pump strokes is needed until the color of the indicating layer matches the color of a comparison layer, or of a color standard, which is a part of the detector tube. Therefore, the number of pump strokes, equivalent to a specific air sample volume, that provides a match with a given standard concentration color is then used to calculate the concentration of the airborne pollutant. Alternatively, some detector tube types utilize a fixed number of pump strokes together with multiple color standards. With these types, the user may need to extrapolate between colors of the scale to determine the concentration of the pollutant.

5.1.4. A simultaneous direct-reading detection tube system is available for determining unknown compounds at hazardous waste sites. This instrument consists of several detection tubes, which are sensitive to different pollutants, mounted in parallel. During a measurement, an air sample is drawn simultaneously through the assembly of detector tubes of a system. Color changes are then noted in specific detection tubes of the system at the end of the sampling period; therefore, many compound or compound-class analysis results can be obtained in a short time period [19]. Some manufacturers of such detection tubes utilize a sequential multiple tube decision logic in their hazardous materials characterization kits.

## 5.2. Pollutants detected

5.2.1. Chemical detector tubes respond to the presence of airborne inorganic and organic gases and vapors. Particles also may be detectable to some extent, for example aerosols of both liquid and solid particles.

5.2.2. With a simultaneous direct-reading or a sequential decision logic detector tube system, it is possible to detect several different classes of chemicals, such as ketones, esters, halogenated hydrocarbons, and some important inorganic gases (e.g., ammonia or acid gases like sulfur dioxide and others).

## 5.3. Metrological and technical characteristics [17,18,20]

5.3.1. The measuring range of detector tubes is broad and includes concentrations beginning far below 1 ppm and up to some volume percentage.

5.3.2. More than 200 different types of detector tubes are commercially available, and, therefore, a wide variety of more than 350 different hazardous organic and inorganic gases and vapors may be measured.

5.3.3. In general, the accuracy of detector tubes of a specific type is expected to vary from lot to lot and among manufacturers. The manufacturer shall provide an accuracy statement for detection of specific chemical classes with each lot of detector tubes.

5.3.4. Length-of-stain detector tubes have a concentration (length) scale printed on the surface of the tube. The scale may directly indicate concentration units or length units, such as mm. If the concentration is not directly printed on the tube, then the manufacturer shall provide a table or chart giving the concentration corresponding to the length of stain or color change. The scale shall be calibrated by the tube manufacturer with known concentrations of reference gases [21-24].

5.3.5. A detector tube shall be stored under the environmental conditions specified by the manufacturer and shall be used on or before the date printed on the tube, or its package, and discarded after that date.

5.3.6. Calibration of the detection tube at a hazardous waste site by the user is usually not necessary, since the detector tube can be regarded as a "preserved" analytical device. Each detection tube is normally designed for a single use, although it is possible to use some tubes more than once during the same day. With some detection tubes, it is also possible to calibrate the stain length after a measurement if both ends are sealed immediately and then checked with a known concentration of reference gas in a laboratory.

5.3.7. For field calibrations, known concentrations of reference gases should be made available in pressurized containers [13,14].

5.3.8. When using a simultaneous direct-reading detector tube system [19], the air flowrate through each tube shall be calibrated using a measuring device such as a soap bubble flow meter or a differential sensing device that has been calibrated against a soap bubble flow meter [8].

5.3.9. Chemical detector tubes often respond to pollutants other than the specific compounds to be measured or to the compounds which they were designed to detect. Therefore, information should be supplied by the manufacturer indicating the probable air pollutants that may interfere with the detection of the specific compounds to be measured. Users should consider any information on accuracy provided by the detector tube manufacturer in interpreting measurement results.

## **6. Total hydrocarbon analyzers**

### 6.1. General description

6.1.1. Total hydrocarbon (THC) analyzers used at hazardous waste sites principally employ either a FID or PID [8,25,26].

6.1.2. A portable instrument using a FID consists of a hydrogen fuel supply with a regulating system, a continuous sampling system, a combustion and ionization chamber, a detector electrode, and an analog or digital signal output display. Some instruments may also contain a pressurized air cylinder with a regulating system to enhance combustion and may also have a portable strip chart recorder. The instrument is normally battery (dc) powered and may operate for a minimum of 6-8 hours on a fully charged battery. Some may also have adaptors that allow operation on alternating current (ac) power. The sampling system is designed for vapor analysis only.

6.1.3. A portable instrument using a PID consists of a low pressure gas discharge lamp, an ionization chamber (with a collection and acceleration electrode), a sampling pump, a signal amplifier, and an output device for displaying the concentration. The instrument is normally battery (dc) powered and may operate for a minimum of 6-8 hours on a fully charged battery. Some may also be adaptable to ac power.

6.1.4. Additional useful components for a THC analyzer include an audible alarm, a portable strip chart recorder, and a gas sample dilution system to increase the dynamic range.

### 6.2. Pollutants detected

6.2.1. A FID-THC analyzer may be used to detect almost any volatile organic compound. In monitoring at hazardous waste sites, however, two broad classes of organic compounds will not effectively be detected: (a) those compounds whose personal ELs are below the detection limits of the instrument and (b) those compounds having very few carbons and an equal or greater number of oxygen atoms, such as formaldehyde or formic acid, which either will not be detected or will be detected with poor sensitivity.

6.2.2. Pollutants detected by a PID-THC analyzer depend on its lamp energy. An instrument with a UV source having a wavelength output corresponding to 11.7 eV will respond to all aliphatic hydrocarbons except methane, and to all olefinic and aromatic hydrocarbons. It is particularly useful for the detection of chlorinated hydrocarbons such as carbon tetrachloride and chloroform, commonly found at hazardous waste sites. An instrument equipped with a UV source with a peak wavelength output corresponding to 10.2 eV will respond to all unsaturated hydrocarbons (benzene, ethylene, etc.), aliphatic hydrocarbons with more than 4 carbon atoms, and chlorinated hydrocarbons that have a double bond (e.g., trichloroethylene or perchloroethylene). It will not respond to the major components of air, such as nitrogen, oxygen, and carbon dioxide, or to methane, ethane, or propane, since these molecules have ionization energies greater than 12 eV. The 10.2 eV and 10.6 eV systems are the most useful for screening and have the highest sensitivity. An instrument equipped with a UV source with a peak wavelength output corresponding to 9.5 eV is more selective and would have a higher relative response to aromatic hydrocarbons, amines, and mercaptans than for a 10.2 eV instrument. Lamps of different energy output can be used in combination as interchangeable units with an instrument to determine whether certain classes of compounds are present. For example, a high response using an 11.7 eV lamp compared to that using a 10.2 eV lamp may indicate the presence of chlorinated hydrocarbons, such as carbon tetrachloride or chloroform, which will only respond when using the former lamp. Similarly, a high response using a 9.5 eV lamp relative to that using a 10.2 eV lamp may indicate the presence of aromatic hydrocarbons or amines. Photoionization based instruments will also detect inorganic compounds such as ammonia, iodine, phosphorus trichloride, chlorine (11.7 eV lamp), etc. Since the air sampling probes of these instruments are generally not heated, they will not respond to chemicals which are not volatile at ambient temperatures. In addition, the ionizing energies of the major components of pure air are all higher than 12 eV and, therefore, will not be detected by a UV lamp.

6.2.3. Neither a FID-THC nor a PID-THC instrument is compound specific. Both respond to all compounds that may be ionized by a flame or an ultraviolet lamp.

### 6.3. Metrological and technical characteristics

6.3.1. A flame ionization detector works on the principle of the combustion of organic molecules in a hydrogen flame and then the collection of the resultant ions on an electrode. In general, the FID is more sensitive to hydrocarbons than to any other chemical class. Its sensitivity decreases for similar molecules in which a carbon is replaced by nitrogen, oxygen, or halides. However, when the ratio of carbon to non-carbon reaches 4 or greater, the response of the FID becomes similar to that for hydrocarbons [25- 26].

6.3.2. A FID-THC analyzer should be calibrated with methane, benzene, or other reference gas standards as appropriate. It should be zeroed electronically or with unpolluted air. Calibration plots of known concentrations versus instrument output usually consist of 3 to 5 points over the expected concentration range. For known pollutants on site, the unit can be calibrated directly with those compounds. Calibration data should be repeatable to within  $\pm 5\%$  during a workshift. A FID-THC analyzer used on waste sites should have a linear response from 0.10 to 1000 ppm (v/v-methane). The detection limit for methane should be approximately 0.10 ppm.

6.3.3. A PID-THC analyzer is calibrated by first setting the electronic zero by turning the instrument to the standby position and adjusting the zero potentiometer control device until the meter reads zero. This is the first calibration point. One or two other calibration points can be obtained by evaporating a known volume of benzene or toluene in a chamber of known volume of air to produce a gas of known concentration, by using a reference gas standard, or by using a dynamic vapor generation system [21-24,27]. The detector gain (span) control is used to adjust the detector response until it displays the actual reference value. The repeatability of this method should be between 5 % and 10 % and depends on the skill of the operator. Response factors can sometimes be used to estimate the concentrations of related chemical species to within  $\pm 15$ -20 %. If more accurate values are required, then the actual compounds to be measured should be substituted for benzene or toluene. For a quick check of the instrument response and sensitivity, a pressurized cylinder of a reference gas standard can be used in the field. A useful reference standard is one containing 10-100 ppm of isobutylene in air. If the instrument is to be used at high humidities, then air of the approximate humidity level should be mixed with the reference gas standard, since high humidity will depress the detector signal [27].

6.3.4. A PID-THC analyzer for most compounds should have a detection limit in the range of 0.1 to 1 ppm. In general, its linear range should extend over three orders of magnitude beginning at the detection limit. The response time should be less than 5 seconds to reach a reading of 90 % of its final indication.

## **7. Gas chromatographs**

### 7.1. General description

7.1.1. A portable GC used at hazardous waste sites is commonly equipped with one or a combination of the following detectors: FID, PID, ECD (Electron Capture), and AID (Argon Ionization) [28]. The TCD (Thermal Conductivity Detector) is sometimes used but is not covered in this section [8].

7.1.2. A portable FID, PID, ECD or AID gas chromatograph includes a column, a gas or gas/liquid sample injection system, a carrier gas handling system, and a portable strip chart recorder, integrator, or data processing system. The unit should preferably incorporate an oven or heat transfer device to control column temperature (see the Note in point 7.3.1). An instrument may be ac or dc powered. The carrier gas may be high purity air, hydrogen, helium, nitrogen, or argon. Sometimes the instrument may be designed to only be used as a THC analyzer.

### 7.2. Pollutants detected

7.2.1. Pollutants detected are the same as with a FID or PID THC analyzer; however, the GC column separates compounds so that identification and quantification of the individual components may be possible.

7.2.2. A portable GC may operate isothermally when powered by an incorporated battery; however, temperature programming may generally be performed only if powered by alternating current. In either case, the selection of appropriate column length and packing materials is an important consideration in maximizing column efficiency. Conditions are usually selected for retention times of 10 minutes or less, when possible, for samples in the vapor phase.

7.2.3. For halogenated pollutants, the ECD may be the best detector to choose to detect low concentrations. At hazardous waste sites, non-halogenated and halogenated substances frequently occur together. Under such circumstances, a combination of two detectors (PID/FID, AID/FID, PID/ECD, FID/ECD, or AID/ECD) would provide the best measurement.

### 7.3. Metrological and technical characteristics

7.3.1. A calibration of a FID-GC, PID-GC, or AID-GC may be accomplished by injecting known concentrations of a reference gas standard and recording the retention times for identification of various compounds. The reference gas standard used shall be a certified reference standard or equivalent [13,14]. The peak area or peak height is recorded and plotted against known concentrations of the reference gas. The column type and its temperature and sample size are also recorded. In general, 3 to 5 calibration points over the expected sampling range should be sufficient. Calibration should be performed with several different compounds simultaneously. An instrument should have a repeatability for retention times and areas within  $\pm 2\%$  for a gas reference standard when operated under stable conditions.

Note: The requirement for a repeatability of  $\pm 2\%$  for retention times shall be satisfied in either of two ways: (a) preferably by maintaining constant column temperature and carrier gas flow rate or (b) by injecting immediately before and immediately after each chromatographic analysis, a reference gas standard - a series of n-alkanes or compounds similar to those to be measured and which bracket the retention times of the compounds to be measured.

7.3.2. In the field, a calibration check should be made with a reference gas standard as in point 7.3.1. At a hazardous waste site, a strip chart recorder shall be used along with a peak integrator (unless the integrator also produces a chromatographic tracing), because a chromatographic tracing shall be available for review and for the record of the measurement.

7.3.3. The linear range and detection limit for benzene, respectively, should at least have values for the indicated detectors as follows: FID-GC:  $10^5$  and 0.1 ppm (vol); PID-GC:  $10^4$  and 0.01 ppm (vol); AID-GC:  $10^3$  and 0.01 ppm (vol).

7.3.4. A portable GC may be equipped with microprocessors with programmed retention time and detector response values for specific compounds to be measured. Such instruments shall also be calibrated prior to and during field measurements according to point 7.3.1.



7.3.5. Gas chromatographs often respond to pollutants other than the specific compounds to be measured or to the compounds which they were designed to detect. Therefore, information should be supplied by the manufacturer indicating the probable air pollutants that may interfere with the detection of the specific compounds to be measured.

## **8. Infrared spectrophotometers**

### 8.1. General description

8.1.1. A portable IR spectrophotometer, commonly called an IR analyzer, consists of an IR light source, a wavelength- dispersive element, variable slits, an IR sensitive detector (e.g., triglycine sulfate or lithium tantalate), a sampling pump, a sample cell, and an analog or digital readout device. Additional useful components include a microprocessor for storing calibration information, a portable strip chart recorder, and a metal bellows pump for transporting the airborne sample or reference gas standards to the sample cell.

8.1.2. IR analyzers operate on the principle of measuring the absorption of energy by a gaseous sample component of interest at one or more specific wavelength(s) over a given pathlength, for which the change in energy is proportional to concentration of that sample. The infrared radiation causes the vibration of specific molecular bonds and consequently absorption at discrete wavelengths. As the pathlength within a sample cell of an instrument becomes longer, its detection limit becomes lower. For air monitoring, the instrument may have a sample cell with either a fixed or variable pathlength [8].

### 8.2. Pollutants detected

Pollutants detected are those compounds that have absorption bands in the spectral region of 2.5 to 25 micrometers. Certain airborne pollutant compounds at hazardous waste sites may be difficult to detect in the IR region because of interference from atmospheric CO<sub>2</sub>, CO, or H<sub>2</sub>O. The minimum resolution of an instrument should be of the order of 1 % at its operating wavelength.

### 8.3. Metrological and technical characteristics

8.3.1. A portable IR analyzer used to detect a known or suspected compound at a hazardous waste site is usually calibrated by the closed-loop method, which uses a recirculating air pump [29]. The instrument is first adjusted to its maximum pathlength, for the best sensitivity, and to the wavelength corresponding to a specific absorption band of the compounds to be measured. The latter information may be obtained from reference data, or can be verified by scanning with a reference gas standard having a known concentration of the chemical compound of interest. The instrument is then adjusted to zero readout using appropriately filtered air, or pure air, as the gas sample. Measured volumes of a reference gas standard of a single compound are then injected into the gas transport system. The change in energy absorbed is recorded. Since IR spectrophotometry is a non-destructive test method, additional amounts of the reference gas standard may be injected to calibrate over the expected concentration range to be monitored. Interference by other compounds with the measurement of specific compounds of interest can be calculated. The data for calculations are obtained by adding known concentrations of such interferent gases with the compounds of interest in the gas transport system and measuring the change in absorbance at the detection wavelength of the compounds to be measured as a function of the concentration of the interferent compounds.

8.3.2. In the field, the instrument is adjusted to the same wavelength and pathlength as that used for its calibration to measure a specific compound of interest. Air samples are then drawn in through the gas transport system, and the change in energy absorbed is compared to the calibration curve for the compound to be measured. Field measurements should have a repeatability of  $\pm 10\%$ .

Note: A significant change in instrument response to specific compounds to be measured may occur for a change in relative humidity and carbon dioxide concentration. Therefore, the calibration described in 8.3.1 shall be performed under such conditions that simulate the expected field conditions to the extent possible.

8.3.3. For detecting unknown organic compounds, the instrument may be operated at 3.4 micrometers, which is the C-H bond characteristic wavelength, and at the maximum possible sample-cell pathlength. In the field, the instrument response under these conditions should be reported as equivalents of concentrations of hexane or methane according to the calibrated response of the instrument to these compounds over the measured concentration range using the closed-loop method.

8.3.4. An IR analyzer yields more specific results than those obtainable using a THC analyzer. The detection limit for a single compound to be measured in air should be between 1-10 ppm (v/v). Selection of the wavelength of the compounds to be measured is most important in minimizing interferences, which are additive for gas samples. The detection limit shall be determined for each compound to be measured in the presence of potential interfering compounds.

8.3.5. An IR analyzer often responds to pollutants other than the specific compounds to be measured or the compounds which they were designed to detect. Therefore, information should be supplied by the manufacturer indicating the probable air pollutants that may interfere with the detection of the designated specific compounds to be measured.

## **9. Dust monitors**

### 9.1. General description

9.1.1. Portable, direct-reading dust monitors commercially available fall into three categories: quartz piezoelectric oscillators, beta-ray attenuation instruments, and light scattering instruments [8].

- 9.1.2. A quartz piezoelectric oscillator consists of a point-to-plane electrostatic precipitator that deposits the airborne dust on one of the two electrodes of a quartz crystal. The crystal is caused to oscillate at its natural resonant frequency by means of associated electronics. A gradual decrease in the crystal's output frequency results from dust deposited on its surface, thus providing a measure of the mass concentration of the sampled airborne dust. A small diaphragm pump, particle size selective precollector, a crystal cleaning mechanism, batteries, and ancillary electronics are part of the instrument.
- 9.1.3. A beta attenuation mass monitor consists of a particle size selective precollector to retain the non-respirable fraction, a particle collection stage (e.g., jet-to-plate impactor, or filter), a beta-ray emitting radioactive source (e.g., carbon-14), a beta detector (e.g., Geiger-Muller tube), pulse counting and processing circuitry, a pump, and batteries. First a reference beta count is usually obtained through the clean collection substrate; next dust is collected for a determined period of time; and finally a count of beta particles passing through the dust deposit is made. The mass concentration of dust in the air is computed from the ratio of the measured final to initial beta counts and from the sampled air volume (collection area and mass attenuation coefficient of the beta radiation are instrument constants). Limitations in sensitivity, time response, operational flexibility, and high cost of beta attenuation instruments have resulted in a reduction of their use in the U.S.A.; in the European Community, they are applied principally for ambient monitoring.
- 9.1.4. A light scattering instrument consists of an illumination source (usually a light emitting diode such as GaAs or GaAlAs), a scattered light detector (typically a silicon detector), collimating and receiving lenses, associated electronics, and a battery. Some instruments incorporate a pump and particle size selective precollector while others sample passively, based on convective and circulatory air currents. The nephelometric type of this instrument is appropriate for hazardous waste site monitoring. It is based on measuring the intensity of light scattered from a defined light source by various concentrations of dust particles in a defined volume.
- 9.1.5. Additional useful components for these instruments are a small portable data storage device and a strip chart recorder. Other such components are a membrane filter for concurrent dust sample collection for subsequent analysis, a reference light scattering element, and an adapter for filtering the air for checking and setting zero response.

## 9.2. Pollutants detected

- 9.2.1. Dust monitors respond to the presence of airborne particles, that is, aerosols of both liquid and solid particles.

9.2.2. Because dust monitors are not species specific, interferences from sources other than those of interest may occur; therefore, at hazardous waste sites, careful consideration should be given to sampling locations and background measurements. Often the use of a multiplicity of spatially dispersed monitors operating concurrently may be required to ensure meaningful measurements.

### 9.3. Metrological and technical characteristics

9.3.1. Quartz crystal piezoelectric oscillators cover the particle size range of about 0.01 - 5  $\mu\text{m}$ , whereas light scattering instruments respond to particles between 0.1 and 10  $\mu\text{m}$ . The particle size range of a beta-ray attenuation instrument depends on its method of collection: 0.5 to 10  $\mu\text{m}$  when using an impactor device and no specific lower particle size limit when using a filtration monitor. Some of the dust monitors include particle precollectors designed to limit the dust concentration measured to the so-called respirable fraction; that is, with a 50 % size cutoff of either 3.5  $\mu\text{m}$  or 5.0  $\mu\text{m}$ .

Note: The design requirements for precollectors differ in the U.S.A and in the E.C. In the United States, the requirements are to limit progressively the collection so that 50 % of the particles of 3.5  $\mu\text{m}$  aerodynamic diameter and 100 % of the particles of 10  $\mu\text{m}$  diameter are not collected. In the European Community, the respective limits are 5  $\mu\text{m}$  and 7.1  $\mu\text{m}$  [11]. Both criteria are recognized in standards of the International Organization for Standardization (ISO) [30].

9.3.2. The overall mass concentration range in air covered by a quartz piezoelectric instrument is 0.01 - 10  $\text{mg}/\text{m}^3$  with a dynamic range of 100 and a detection limit of 0.01  $\text{mg}/\text{m}^3$ . The overall range covered by a light scattering instrument of the nephelometric type is 0.002 - 200  $\text{mg}/\text{m}^3$  with a dynamic range of 1000 and a detection limit of as low as 0.001  $\text{mg}/\text{m}^3$ . The mass concentration range for a beta monitor is 0.1 - 50  $\text{mg}/\text{m}^3$  with a dynamic range of 20 with an impactor device and is 0.01 to 10  $\text{mg}/\text{m}^3$  with a dynamic range factor of 50 to 100 with a filtration device.

9.3.3. The response of direct-reading dust monitors depends on several particle parameters: (a) particle size for instruments based on the quartz piezoelectric oscillator principle and (b) particle size, shape, and index of refraction for light scattering instruments. Especially for instruments of the latter type, calibrations should be carried out with reference dust standard that is representative of field conditions. A gravimetric reference measurement obtained by means of concurrent filter collection may be appropriate for both types.

9.3.4. Some nephelometric instruments incorporate reference standards for calibration, or span checks, based on insertable light scattering elements. Beta attenuation instruments often include a calibration absorber.

9.3.5. The instrumentation for gravimetric reference measurements requires the following: an inertial precollector (only if the respirable particle size fraction is to be measured); a filter holder with a membrane-type filter; a pump; a flowmeter and flow controller; and a laboratory scale capable of measuring filters up to 100 mg with a repeatability of  $\pm 0.05$  mg, or better.

9.3.6. Another consideration for calibration is the maintenance of a reference dust standard monitor in a mobile or stationary laboratory to which all field monitors may be compared using an aerosol generator.

Note: Instrument calibration is not always feasible in the field because the concurrent filter collection necessary for the gravimetric determination of the dust concentration may not provide a sufficient sample for accurate weighing at a hazardous waste site. In such cases, a laboratory calibration facility should be available. It should include a stable aerosol generation apparatus (for example, a small fluidized bed generator with a deionizing column) and a mixing and sampling chamber to produce dust or aerosol samples for simultaneous calibration of the filter sampler and monitor. The reference dust produced by such a facility should have properties similar to that expected of dust at the hazardous waste site. If specific information about that dust is unavailable, or if information is unavailable about the generated aerosol, a reference dust standard should be used for calibration [12,13].

9.3.7. All surfaces of a piezoelectric oscillator and optical components of an instrument shall be cleaned periodically, and the instrument shall be re-adjusted to zero response according to the manufacturer's specifications.

9.3.8. A dust monitor can be checked routinely with respect to zero concentration response by purging them with clean filtered air. Some light scattering instruments have internal air purge capabilities. Especially for a nephelometric type instrument, each instrument's zero stability should be determined to assess its repeatability and overall accuracy of measurements.

## **10. General procedures for calibration and testing**

10.1. All portable instruments involved in measuring airborne chemical releases should be calibrated, tested, and maintained prior to use according to the manufacturer's recommendations.

10.2. Operators should consider if an instrument can function properly under various extreme field atmospheric and operational conditions, such as low and high temperatures; clean and dusty environments; low and high humidity; rain and snow; and high and gusty winds. The degree to which these operating conditions may affect instrument performance should be specified by the manufacturer. The guidelines for specifying environmental tests provided in International Document OIML D 11 "General Requirements for Electronic Measuring Instruments" [31] may be helpful.

10.3. Operators should consider the following means of field checking the response of an instrument for monitoring pollutant emissions from both hazardous waste sites and emergency or accidental releases.

#### 10.3.1. Reference gas standard

A small pressurized cylinder of a reference gas standard, which is available commercially and traceable to a certified reference material [13,14], should be available for all field work. Such reference gas would permit periodic routine checks of an instrument as well as performance verification when the field concentration of a particular target pollutant changes significantly.

Note: Care should be taken to minimize the exposure of personnel to harmful reference gases, such as benzene, used for testing and calibration.

#### 10.3.2. Field prepared reference gases

A reference gas standard may be unavailable for field testing. An alternative would be to place a known amount of a highly volatile organic compound such as toluene into a container of known volume [21-24]. Such field-prepared reference gases also may be prepared from more than one compound as required. A field-prepared reference gas can furnish qualitative information only, since quantitative data on concentrations would depend on knowing the temperature and pressure at which such a reference gas was used.

#### 10.3.3. Basic operational check

Under some circumstances, a reference gas will not be available or cannot be prepared for determining the field operational condition of an instrument. In such cases, a material or compound should be used that can produce a positive instrumental response before use. The following are examples of materials or compounds that are often available for such purposes: gasoline, felt tip pens, cigarette smoke, and vehicle exhaust. This type of basic operational check instead of the recommendations of points 10.3.1 or 10.3.2 shall be used only as a last resort. Unless the instrument has been checked using the procedures described in points 10.3.1 or 10.3.2, the quantitative data obtained in field measurements shall not be given credibility.

10.4. The procedures and results of calibration and verification shall be recorded in a log for each instrument. This log should contain enough detail on an instrument's history of operation to develop adequate maintenance schedules. An instrument shall be calibrated after maintenance and repair.

10.5. A control chart should be kept for each instrument in order to determine if its performance upon calibration is within statistical control [15,16].

## ANNEX A

### LIST OF ABBREVIATIONS

AID	Argon Ionization Detector
ECD	Electron Capture Detector
EL	Exposure Limit
FID	Flame Ionization Detector
GC	Gas Chromatograph
MAC or MAK	Maximum Permissible Concentration
PEL	Permissible Exposure Limit
PID	Photoionization Detector
THC	Total Hydrocarbon Analyzers
TLV	Threshold Limit Value
TWA	Time-Weighted Average

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