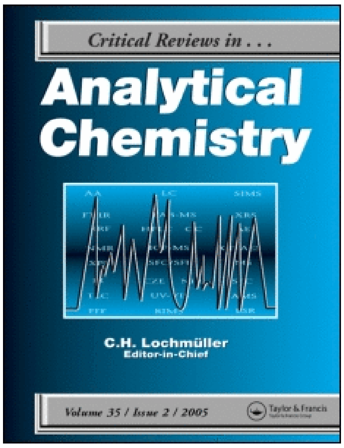

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Analysis of Organic Compounds in Air: A Review

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ABSTRACT: Methodology for the analysis of organic pollutants in air has developed rapidly during recent years. The most recent analysis methods are presented. The current status of the analytical methods used in the determination of organic pollutants in air are shown.

KEY WORDS: air pollution, analysis, PAHs, halocarbons, VOCs, SVOCs, amines, isocyanates, aldehydes.

I. INTRODUCTION

Growing emphasis on environmental monitoring has encouraged the development of more rapid and less expensive methods of analysis for toxic pollutants.

In ambient, organic pollutant analysis among the critical point have to be considered are good sampling technique together with an appropriate analysis method. Different sampling techniques are reported, such as canister sampling, bag sampling, diffusive sampling are used. Air sample collection using solid sorbent, such as Tenax, or Carbopack B, combined with subsequent gas chromatography (GC) separation after preconcentration is also in common use for analyzing trace gas. Solid phase microextraction (SPME) is a new technique, which is shown to be a viable alternative to sampling airborne compounds.

In the analysis of environmental samples, the first problem one generally encounters is sample preparation. Often too diluted or complex, samples need to undergo a series of specific treatments to make them compatible with analytical techniques and to ensure effective separation and detection. Many enrichment techniques have been described, which vary according to the type of pollutant and the analysis method. For GC analysis, adsorption on porous materials followed by thermal desorption, preconcentration in a cryotrap and followed by flash-heating into the GC column or by solvent elution and the subsequent injection of an aliquote of the solution are, for instance, used to analyze volatile organic substances in air. Pollutants adsorbed on solid matter are desorbed by Soxhlet or supercritical extraction and concentrated by evaporation of the solvent. Another preconcentration method consists of trapping volatile air pollutants in a reaction liquid containing a derivation agent.

GC is by far the most widely used tecnique in the analysis of organic compounds in air using several detector such as flame ionization (FID), electron capture (ECD), or mass spectrometry (MS).

Fast and on-line screening methods in analytical chemistry play an ever-increasing role in industry, medicine and environmental sciences. In addition to fluorescence, photoemission, plasma emission and Raman's spectrometry, photoacoustic spectrometry belongs to the techniques where signal formation is related to the incident light intensity. Hence the use of laser sources is extraordinarily advantageous because they allow remote sensing of low concentrations of gases in the atmosphere from distances up to several kilometers.

Simultaneous analysis of components have become increasingly important in the last few years. Sensors arrays are preferred over chro-

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matographic techniques because no laborious sampling processes (e.g. extraction, derivatization) are required; the separation itself is not so reproducible, the chromatographic techniques need high-quality materials (e.g., solvents, inert gases), and are time consuming. Furthermore, sensor arrays have the advantage of combining good sensitivity and selectivity at relatively low cost. Additionally, electrochemical sensors constitute an important group in environmental analysis for simultaneous detection in array competitive with quantitative chromatographic analysis techniques. The importance of microelectrode arrays and electronic noses has increased in the last few years. Their development makes it possible to consider them, separately, as main classes of sensor arrays. Very often, the sensor arrays contain ion selective, membrane electrodes, gas sensors, and biosensors.

In general, the use of state-of-the-art procedures and the development of new and novel approaches to analytical problem solving requires an adequate knowledge of the existing literature. In this paper we show the most recent methodologies for the determination of organic micropollutants. The compounds have been grouped in volatile or semivolatile species. However, some compound groups, such as aldehydes, polycyclic aromatic compounds, halocarbons, isocyanates and amines, have been separately described because their characteristic analysis.

II. ANALYSIS OF VOLATILE ORGANIC COMPOUNDS

Volatile organic compounds (VOCs) comprise an important group of pollutants commonly present in air. Some of these compounds are potent carcinogens or mutagens at very low concentrations.

Gas chromatography combined with either a FID or MS is the method most often chosen for the determination of VOCs in air. As VOCs are found in trace concentration in air and the sensitivity of analytical methods currently available for the determination of organic trace pollutants, it is often insufficient to measure low environmental concentrations directly, a preliminary enrichment step is required.

A. Sampling and Extraction

Diffusive sampling has been used for about 20 years in occupational hygiene to measure concentrations of potentially harmful compounds in work place air. Owing to its simplicity in use, from viewpoints of both the occupational hygienist performing the measurements and the workers involved in personal air sampling, the technique is considered to be a valuable alternative to the traditional pumped sampling. An inherent advantage is their low costs. Hafkenscheid and Mowrer[1] investigated organizational details and results of an international intercomparison of tube-type diffusive samplers for the monitoring of volatile hydrocarbons (HC) in ambient air. Periago et al.[2] evaluated environmental levels of aromatic HC in gasoline service stations using diffusive samplers and GC-FID detection. The same detector was used by Mowrer et al.[3] that monitored diffusive samplers of C_6-C_9 VOCs collected in urban air; based on passive sampling during 7 days. Harper et al.[4] validated a diffusive sampler for methyl-ter-buthyl ether (MTBE) according to a published US National Institute for Occupational Safety and Health (NIOSH) validation protocol. Bates et al.[5] explained the difficulties encountered when trying to quantitatively recover certain VOCs from ambient air using high uptake rate diffusive samplers. The solution proposed by the authors combines the versatility of Radiello cartridges with a popular auxiliary tube.

Passive sampling can be achieved with badge or tube devices. Generally, tube samplers are exposed for longer periods of time than badges. A passive sampling[6,7] method has been developed for acetic and formic acid in ambient air. The procedure involves collection of the vapours in Pames diffusion tubes and analysis by ion chromatography. Ion exchange chromatography using a Dionex DX199 ion chromatograph was used to determine the concentration of acetic and formic acid in corrosion products from underside of lead roofs on historic buildings by Edwards et al.[8] Monn and Hangartner[9] measured VOCs in air during 2 weeks, using a passive sampling device. Methods for determination of cyclic organic acid anhydrides (OAAs) in industrial air by GC are reviewed by Jonsson et al.[10] A prototype-passive sampler for VOCs was evaluated by Otson and Cao[11] using charcoal as sorbent, CS_2 as extractant, and selected ion mass detection.

Sampling by solid sorbent has some advantages, as, for instance, it is less costly than canister and it is easier to transport. Sample enrichment on solid sorbent such as charcoal followed by thermal desorption is widely used to analyze HC or halocarbons in air. Bemgard et al.[12] described the theory of cylindrical denuder tubes as samplers for nonreactive VOCs in air. Risse[13] described an analytical method for HC by GC using a denuder sampling system with charcoal. A dynamic system for producing test atmospheres of 2,4-dinitrophenol through the evaporation of solid materials is described. The use of impingers and denuder tubes as sampling devices for 2,4-dinitrophenol in the gas phase, was developed by Oms-Molla and Klockow.[14] Qin et al.[15] developed a simple method for the trace determination of methanol, ethanol, acetone and pentane by preconcentration on solid sorbents followed by GC-FID. Bartulewicz et al.[16] used common sorbents, such as activated charcoal and silica gel, to preconcentrate the samples prior to GC analysis. The advantages of CS_2 as desorbent from the activated charcoal in the determination of styrene were reported by Bartulewicz et al.[17] and Begerow et al.[18] A mathematical model to quantitatively unify the desorption of styrene from active-C by CS_2 was reported by Prosek.[19] Johnson et al.[20] studied five alternative adsorbents to charcoal, such as Ambesorb XE-340, Tenax GR, Anasorb 747, Carbosieve SIII, and Kureha Beades Activated charcoal. Each adsorbent was subjected to screening and dynamic spiking/recovery test using 22 target VOCs prior to GC-MS analysis. VanOsdell et al.[21] reported about VOC removal at low contaminant concentrations using granular-activated carbon. Porous polymer sorbents such as Tenax are selectively inert and hydrophobic and normally have large surface areas. Among different sorbent Tenax has been used for the preconcentration of VOCs because of its excellent properties of trapping pollutants along with high thermal stability, which in turn facilitates thermal desorption. Several authors[22,23,24,25,26,27] measured VOCs in air using Tenax as solid sorbent and a thermal desorption cold trap (TCT) with GC-FID detection. Polyurethane foam/Tenax cartridge was used to collect aromatic hydrocarbons, chlorinated phenol, guaiacols from air. The analytes were determined by GC-MS and GC with electron capture detection (ECD).[28]

The chromatographic behavior of VOCs could be modelled using a Langmuir isotherm.[29] The Langmuir isotherm equation was studied in the absorption of organic molecules from air onto Tenax GC by MS detection.[30]

Other sorbents were evaluated as alternatives to Tenax. Munari et al.[31] utilized a gas analyzer for the determination of VOCs in air. The analyzer consisted of a gas chromatograph linked to a sampling device that incorporated a cooled fused-silica microtrap filled with Tenax GR or Carbotrap B. Brown[32] reported a project involving 12 participants to evaluate the useful alternatives to Tenax sorbent, using test compounds representing very volatile and very polar compounds. Chromosorb 106 is recommended for the sampling of more volatile and polar organic vapours encountered in work place atmospheres. The relative performance of Tenax TA, Chromosorb 106, and Carbotrap for the long-term diffusive sampling of some typical VOC was studied by Kilic and Ballantine.[33] Four OAAs were sampled using Tenax and Amberlite XAD-2 solid sorbents and determinate by GC-FID by Jonsson et al.[34] Anasorb CMs was evaluated for the sampling of VOCs by Baya and Siskos,[35] the results were compared with those obtained for Tenax. Bartulewicz et al.[36] sampled air through a tube filled with two sections of Chromosorb 102; phenol was desorbed from each section with benzene and analysed prior to back-extraction with NaOH 0.1 *M*. Popp and Paschke[37] reported a new 80-μm Carboxen-polydimethylsiloxane (PDMS) fiber for solid phase extraction (SPE) for enrichment of VOCs from water and air. Pham-Tuan et al.[38] studied a novel preconcentration technique for online coupling to high-speed narrow-bore capillary portable micro GC to calculate enrichment factors of n-alkanes and VOC.

McComb et al.[39] developed a microextraction of VOCs using the inside needle capillary adsorption trap (INCAT) device.

Thermal desorption is a simple and very sensitive technique, commonly used in combination with GC-FID detection for determination of VOCs.[40,41,42,43,44,45,46] Helmig[47] studied artifact-free preparation, storage, and analysis of solid adsorbent cartridge used in the analysis of VOCs in air. Bertoni and Tappa[48] described the use of thermal desorption with light adsorbents for the determination of VOCs as air pollutants at the ppb levels. The desorption traps were made of glass tubes filled with Carbotrap or Carbopack C or Tenax TA. Gee and Sollars[49] used a sampling bag containing portable sampling equipment. Each bag contained sampling apparatus for collection of VOCs. A low-flow pump was connected to a stainless steel thermal desorption tube packed with two adsorbents: Carbopack B, designed to trap VOC between C_5-C_{10} and Carbosieve SIII that retained lighter-molecular-weight VOCs (C_2-C_5). Analyses of VOCs were performed by thermal desorption/capillary GC with FID for quantification and an ion trap detector (ITD) used to assist in compound identification. Castellnou et al.[50] analysed HC (C_2-C_8) by GC-FID in airborne samples. These authors compared refrigerated (at –10°C) and ambient temperature cartridges containing a combination of packing materials. The material collected was analyzed by thermal desorption-cryofocussing-GC (desorption at 300°C and cryofocussing trap at 15°C).

Several authors determined VOCs in air by thermal desorption GC/MS.[51,52,53,54,55,56] An improved strategy for measurement of VOCs in air based on cooled sampling and analysis by thermodesorption GC-MS/FID was developed by Haunold et al.[57] Koch et al.[58] studied the removal of water form multibed carbon traps with adsorbed polar VOCs. A stainless-steel column was packed with Carbopack C, Carbotrap B and Carbosieve SIII. Each sorbent in the column was separated by silanized glass wool. A second similar column was prepared using Carboxen 569 in place of Carbosieve SIII. The columns were subjected to elution in a thermal desorption unit followed by GC-MS detection. The use of GC-MS system to chemically identify of pentachloronitrobenzene in ambient air extracts was summarized by Thompson et al.[59] Seke and Onda[60] presented a method for the adsorption-desorption sampling of VOCs in air and the determination by GC-MS, using multiple bed packing of sorbents such as Carbopack B and Carboxene 100.

Other authors[61,62,63,64] used a **SPME** for the determination of VOCs in air by GC ion trap MS (ITMS). The same procedure was reported by Duffy and Nelson[65] for benzene and 1,3-butadiene determination in accordance with the US EP TO-14 method. An interesting alternative at traditional thermal desorption is carried out by means of microwave irradiation.[66,67]

Cryogenic preconcentration is frequently used for VOC measurement with GC systems.[68,69,70] A refined cryogenic sampling method and a modification of a commercial thermal desorber for automatic analysis of atmospheric sulfur compounds with a gas chromatography coupled to the sulphur chemiluminiscence detector is reported by Chen and Lo.[71] Giarroco et al.[72] continuously monitored C_2-C_{10} VOCs in air using cryogenic trapping with capillary-GC-FID analysis. Zielinska et al.[73] reported on C_2-C_{12} monitoring with sampling VIA SUMMA stainless steel canister, cryogenic preconcentration, and (GC/IRD/MSD) for qualitative identification and GC-FID for quantification of hydrocarbons and HPLC for identification and quantification of carbonyl compounds. Huang et al.[74] quantified toxic HC target compounds in engine exhaust and air by aluminium oxide porous-layer open-tubular capillary GC-MS using isotopically labeled internal standards. The samples were collected in SUMMA electropolished stainless-steel canisters. Analytes were transferred in He as carrier gas to a Teckmar 6000 thermo desorbed, collected in a cryotrap and injected for GC by desorption at 200°C.

Moschonas and Glavas[75] studied the effect of a addition of a graphitized carbon black trap to a glass beads trap on the cryoconcentration of some non-methane HC in ambient air with special emphases on trapping of C_2 HC and desorption of xylene. Consecutive stainless-steel tube traps were used, the first packed with nonsilanized glass beads and the second with the same packing proceeded by Carbotrap carbon black. Desorbed HC were analyzed by GC.

Several authors studied the use of an online membrane extraction microtrap GC system.[76,77] Zhu et al[78] reported an experimental arrangement comprising a catalytic incinerator, a membrane module, a microtrap and GC/FID detector for continuosly monitoring of VOCs emission.

B. Chromatography

Habram et al.[79] reported a dual capillary column **GC** for the determination of trace HC in ambient air. Kolb[80] analyzed quantitative traces of VOCs in air, water, and soil using equilibrium headspace GC. The simultaneous analysis of VOC and halogenated HC was demonstrated using a dual column/dual detector arrangement with ECD and FID; results from electrolytic conductivity and MS detection were also given. A comparison of flame ionization and ozone chemiluminiscence for the determination of atmospheric HC was carried out by Marley and Gaffney.[81] Etiope[82] evaluated a micro gas-chromatographic technique for environmental analyses of CO_2 and C_1-C_6 alkanes. The portable GC instrument contains two micro GC modules, each fitted with an oven and a micromachined thermal conductivity detector. Other papers reported on the analysis of VOCs by programmed temperature vaporizer (PTV) followed by GC-FID detection.[83,84,85,86]

Colleman et al.[87] discussed a new chromatography-based continuous emission monitoring system (CEMS) for the measurement of VOC and hazardous air pollutants (HAPs) in less than 4 min. This system circumvents the quantification problems encountered by other analyzers such as FIDs, nondispersive infrared (NDIRs), and photoionization detectors (PIDs) because it measures each solvent individually. Unlike Fourier transform infrared (FTIR) and mass spectrometry (MS), which require the attention of highly trained personnel, this GC-CEMS performs error-free analyses under actual manufacturing conditions, with minimal intervention by a control-room operator.

In a effort to develop a new autoGC system for Photochemical Assessment Monitoring Station (PAMS) that does not rely on a permeable membrane dryer for water management, several authors[88,89,90] investigated a combination of uncooled multisorbent traps and a stirling engine closed-cycle cooler, that reduces the injection volume for GC separation and allows better resolution.

Means of testing for ozone precursors are discussed by Brenner and Mueller,[91] including automated measuring stations for CO_2, N oxides, SO_2, VOC, and aromatic compounds.

The development of **mass spectrometry** to monitoring hazardous air pollutants in real time has generated great interest. Several studies have relied on conventional methods for characterizing compounds in air.[92,93,94,95,96] Mass spectrometry is generally labor-intensive, time-consuming, and expensive. It is also unsuitable for monitoring polar VOCs. These limitations have provided the impetus for the introduction of new monitoring systems, based on mass-spectrometry and direct air-sampling interfaces, including membrane introduction quadrupole MS[97,98,99] and ion trap mass spectrometry.[100,101,102] The complex photochemical transformation of biogenic aromatic HCs such as isoprene and also of anthropogenic aromatic HCs is an important source of carboxylic acids in the troposphere. The identification of unknown carboxylic acids can be difficult. Chien et al.[103] described the development of a method to analyze airborne carboxylic acids using derivatization with pentafluorobenzyl bromide (PFBr) followed by capillary GC/ITMS analysis. The author reported for the first time the employment of pentafluorobenzyl alcohol and methane as a chemical reagent to facilitate molecular weight determination of PFBr derivatives of carboxylic acids.

The use of selected ion monitoring (SIM) principles is used by Wessen and Schoeps[104] to analyze microbial volatile organic compounds in indoor air. Tokuhara et al.[105] determined aldehydes and organic solvents by automated thermal desorption combined with GC electron impact mass-spectrometry detection (GC-EIMS) operating in SIM mode. Sauerbier et al.[106] determined VOCs in indoor air and their effects on personnel. After trapping on Amberlite XAD, the VOC are determined By GC-EIMS detection.

Several VOCs were analyzed by Hiatt[107] by using vacuum distillation VD coupled with GC/MS.

Because the concentrations of volatile organic sulfur compounds (VOSCs) in ambient air are usually very low, very sensitive and selective analysis methods are needed. To achieve the required sensitive level, preconcentration of the sample is necessary before analysis. However, these techniques entail certain problems. Many of these problems can very effectively be minimized by using on-line or on-site analysis techniques.

Membrane inlet mass spectrometry (MIMS)[108] is one of the most promising analytical techniques for this purpose.

C. Spectroscopy

FTIR remote sensing measurement is becoming increasingly popular for the remote detection of airborne VOCs. The excellent selectivity of infrared spectroscopy allows a single instrument to be used for the detection of a variety of compounds. Since the physical collection of a sample is not required FTIR remote sensors offer capabilities for monitoring large areas that cannot be covered adequately with conventional fixed-point gas sensors. Bangalore et al.[109] used a FTIR remote sensing measurement to implement the automated detection of trichloroethylene (TCE) vapor in the presence of a variety of infrared background signatures. Chu et al.[110] investigated the FTIR spectra of several VOCs.

Johnson et al.[111] studied the application of surface-enhanced infrared absorption spectroscopy as a sensor for VOCs.

Infrared differential absorption lidar (IR DIAL) techniques allow remote sensing of gases in the atmosphere from distances up to several kilometres. A field test was carried out with two nearly identical coherent CO_2-laser-based DIAL system by Ritcher et al.[112]

In the past, a number of papers have been published dealing with evanescent-field fiber-optic sensors that detected absorption spectra of HC in the near-infrared (NIR) and middle-infrared (MIR) regions, respectively. In the MIR hydrocarbons have characteristic vibration spectra but fiber-optic sensing requires special MIR-transmitting fibers made from chalcogenide or silver halide glasses, which is not in accordance with the low-cost requirement for sensors. In contrast to the problems encountered in the operation with MIR-laser, laser diodes with wavelengths in the NIR region are easy to operate at room temperature, and another advantage is the availability of robust glass fiber for these wavelengths. Recently, a photoacoustic application using NIR laser diodes has been presented.[113] Sneider et al.[114] studied the possibility of combining external cavity diode laser with a photoacoustic detector for high-sensitivity monitoring of gases such as acetylene and methane. The gaseous sample is held in a closed photoacoustic cell and changes in pressure in the cell were measured with a microphone. A long-path, multimode, integrated optical sensor for non-polar organic compounds is described that measures directly their evanescent wave absorbance spectra in the NIR range by Buerk et al.[115]

One of the problems in both these techniques is that the strong absorption bands of water in the NIR and MIR can overlap HC absorption line. In the UV region many HC have strong absorption bands due to electron transitions. The evanescent field of an optical waveguide structure can be used to obtain sensitive chemical sensors with an interaction length of several centimeters. Schowotzer et al.[116] present the first results of evanescent UV absorption spectroscopy with sensitized silica fibres in air and in water containing small traces of toluene, naphthalene, or fluorene as test substances

Doleman et al.[117] mixed poly(vinyl acetate) (I), poly(methyl methacrylate), and their mixtures with carbon black to fabricate arrays detectors for measurements of air polytest.

An alternative technique is the **Differential Optical Absorption Spectroscopy** (DOAS). The advantages of DOAS are the capability of *in situ* measurements, the absence of wall losses, the high sensitivity, and good time resolution of the order of few minutes. Brocco et al.[118] measured in urban air aromatic HC by means of DOAS and automatic chromatograph (VOC analyzer). Several authors[119,120] monitored monocyclic aromatic hydrocarbons (MAHs) in urban air with DOAS technique

Detection and quantification of gaseous air contaminants at low cost is becoming increasingly important especially in the determination of air quality. Kherrat et al.[121] developed a new optical gas sensor, based on an integrated planar wave guide designed as a Mach-Zehnder interferometer with tapers. The interferometer is used as a chemical sensor in which the interaction window is coated with a heteropolysiloxane (HPS) polymer as a thin superstrate film. When organic HC are in contact with the superstrate, the molecules can be absorbed in the polymer and change its refractive index according to the gas concentration.

During the First World War, the United States developed trans-dichloro(2-chlorovinyl)arsine (Lewisite) to be used a chemical weapon. The determination of traces of Lewisite and its decomposition products is critical in environmental recuperation. The development of a methodology for determination of trace levels of Lewisite in ambient air by Flow Injection Analysis (FIA) was reported by Aldstadt et al.[122] Grasdepot et al.[123] utilized a domestic gas sensor with micromachined optical tunable filter for detection of CO and HC. An optical gas analyzer based on optoacoustic filters for monitoring impurities in air was reported by Mazur et al.[124] Yang et al.[125] utilized molecular host siloxane thin films for **surface acoustic wave chemical sensors** for VOCs.

D. Amperometric Sensors

Amperometric electrochemical gas sensors have the advantage of combining good sensitivity at relatively low cost. However, their use is restricted to the detection of gases that are reactive at potentials positive at which oxygen is reduced. Many substances cannot be detected using the amperometric sensors, because they either are too inert (as many halogenated species) or do not react electrochemically in the limited potential window. Oelgeklaus and Baltruschat[126] reported that the detection of many organic compounds such as benzene, its derivatives, and simple halogenated hydrocarbons is possible by means of a Pt-sensor electrode. Baltruschat et al.[127] described a new detection method for less reactive species such as benzene and halogenated HC, which cannot be directly oxidized, which consist of electrochemical sensor cells that comprise a thin Pt or Pd layer sputtered onto a porous Teflon membrane. Josse et al.[128] utilized alternating-current impedance-based chemical sensors for VOC.

Chen et al.[129] described an oscillopolarographic determination of micro-amounts of phenol in environmental samples (water an atmospheric air).

The determination of ethanol, toluene, and *o*-xylene in air was achieved by measuring the transient and steady state conductance signals was reported by Llobet et al.[130]

For a detection and quantification of VOCs at low cost, in a simple, but most flexible, manner, there exist a number of methods such as semiconductor sensors,[131,132,133,134,135,136] piezoelectric crystal-based sensors[137,138,139,140] and biosensors.[141]

A new class of receptor materials for gas sensors for VOC was developed by Buhlmann et al.[142] These plasticized polymeric electrolytes consist of a polymer, a plasticizer, and an organic salt. Diesel fuel and solvent vapours were detected by Dickert et al.[143] in air using a **quartz microbalance (QMB) and a surface acoustic wave (SAW) sensor**. A method for measuring point-source emissions of VOC, acidic vapors and other species is presented by Knaebel and Yeoman.[144]

Abdelghani et al.[145] have evaluated a **surface plasmon resonance (SPR) fiber optic sensor** with chlorinated and aromatic compounds.

III. ANALYSIS OF ALDEHYDES AND KETONES

Aldehydes play an important role in photochemical sources of free radicals in the atmosphere and constitute a source of tropospheric ozone. The most abundant aldehydes in the atmosphere are formaldehyde and acetaldehyde, both know for their toxicity. Formaldehyde is an important industrial chemical, being used extensively in the manufacture of resins and is also used as a bactericide and preservative in goods as diverse as toothpaste, washing-up liquid, and air fresheners. Furthermore, the widespread use of formaldehyde in building materials has given rise to reported indoor formaldehyde concentrations of up to 0.37 to 0.55 ppm in new homes and between 0.12 and 1.6 pp in mobile homes.

A. Derivatization

Several methods are available for aldehyde measurement in air, the most common methods are based on **carbonyl derivatization by 2,4 dinitrophenylhydrazine** (DNPH). Aldehydes

Table 1
Analytical Methods for Volatile Organic Compounds

Species	Sampling/extraction	Detection	Ref.
HC	Diffusive sampling	GC-FID, MS or ITMS	1
Aromatic HC	Diffusive sampling	GC-FID	2
C_6-C_9 HC	Stainless-steel adsorbent tubes diffusive sampler . Thermal desorption.	GC-FID	3
Ethers	Diffusive sampling	GC-FID	4
VOC	Diffusive sampling. Thermal desorption.	GC	5
Acetic and formic acid vapours	Diffusive sampling	Ion chromatography-conductivity detector	6
Acetic and formic acid vapours	Diffusive sampling	Ion chromatography-conductivity detector	7
Acetic and formic acids		Ion-exchange chromatography-chemically suppressed conductivity detection	8
MAH (benzene, toluene and xylene)	Passive sampling device.	GC	9
VOC	Prototype passive sampler	GC-MS	11
Non-reactive VOCs	Cylindrical denuder tubes		12
Aromatic HC	Denuder sampling system with charcoal	GC	13
2,4-dinitrophenol	Impingers and denuder tubes	HPLC-UV	14
Methanol, ethanol, acetone and pentane	Stainless-steel cartridge sampling. Thermal desorption.	GC-FID	15
Alcohols, ethers, esters and ketones	Diffusive sampling (charcoal tubes).Desorption with CS_2	GC	16
Styrene	Diffusive sampling (charcoal tubes).Desorption with CS_2	GC-FID	17
VOC	Diffusive sampling (charcoal).Desorption with CS_2	Dual-column capillary GC with tandem ECD-FID	18
Styrene	Desorption with CS_2.	---	19
VOC	Five adsorbents were evaluated for this application: Ambesorb XE-340, Tenax GR, Anasorb 747, Carbosieve SIII and Kureha Beades Activated charcoal. Petroleum-base charcoal as the second adsorbent	GC-MS	20
VOC	Granular activated carbon	GC-FID	21
C_6-C_{10} HC	Adsorbent sampling (Tenax). Thermal desorption cold trap.	GC-FID	22
HC (C_2-C_{11})	Adsorbent sampling (Tenax-TA). Thermal desorption.	GC-FID	23
VOC	Adsorbent sampling (Tenax-GC)	GC-FID	24
VOC	Adsorbent sampling (Tenax GR/CarbopackB;Carbopack B/Carboxen100 (Carbotrap217). Thermal desorption.	GC-FID	25
Polar VOC	Diffusive sampling. Thermal desorption.	GC-FID	26
VOC	Diffusive sampling, dehumidified and ventilate unit.Thermal desorption.	GC-FID	27
Aromatic HC, chlorinated phenols, guaiacols, and benzenes	Adsorbent sampling (Tenax). Soxhlet extraction.	GC-MS and GC-ECD	28
VOC	Adsorbent sampling (HayeSep Q)	Chromatographic technique	29
HC and oxygenated compounds	Adsorbent sampling (Tenax)	GC-MS	30
VOC	Sampling device which incorporated a cooled fused-silica microtrap filled with Tenax GR or Carbotrap B.	Gas analyzer (gas chromatograph linked to a sampling device)	31
VOC	Adsorbent sampling. Thermal desorption.	GC/MS	32
VOC	Adsorbent sampling (Tenax TA, Chromosorb 106 and Carbotrap) for long-term diffusive sampling	---	33
Cyclic organic acid anhydrides	Adsorbent sampling (Tenax, Amberlite, XAD-2). Bubbler, impingers or glass fibre filter sampling.	GC - FID, ECD, MS	34
VOC	Anasorb CMS compared with Tenax TA. Desorption with acetone.	GC-FID	35
Phenol	Adsorbent sampling (Chromosorb 102). Desorption with benzene.	GC-FID	36
VOC	SPME	GC-FID-ECD	37
n-alkanes C_7-C_{10} and others VOC	Preconcentration device equipped with an open-tubular enrichment column	Portable micro GC	38
VOC	INCAT. Thermal desorption.	GC-FID	39
VOC	Adsorbent sampling. Desorption with CS_2 or methanol/CH_2Cl_2, alternatively thermal desorption.	GC-FID	40
VOC	Adsorbent sampling, cold trap .Thermal desorption unit.	GC-FID, GC-ECD, GC-NPD	41
VOC	Adsorbent sampling (CKTB-1 or Carbochrom B).Thermal desorption.	GC-FID	42
MAH and chlorinated HC	Adsorbent sampling.Thermal desorption cryofocusing.	GC-MS/GC-FID	43
VOC	Adsorbent sampling (Tenax TA). Thermal desorption. Cryotrapping.	GC-FID	44
VOC	Adsorbent sampling (Carbotrap C+ Carbotrap and Carboxen). Thermal desorption.	GC. FID/MS	45
C_2-C_7 HC	Stainless-steel U-tube cold trap. Thermal desorption.	GC-FID	46
VOC	Cartridge sampling . Thermal desorption.	GC	47
VOC	Adsorbent sampling (Carbotrap or Carbopack C or Tenax TA). Thermal desorption	GC	48
VOC	A tube packed at the front end with Carbopack B and at the back with Carbosieve SIII. Thermal desorption.	GC-FID/GC-ITMS	49
HC	Cartridge sampling. Thermal desorption-cryofocussing trap.	GC-FID	50
VOC	Glass tube with adsorbent material (various charcoal-based sorbents or Tenax). Thermal desorption.	GC-MS	51
VOC (from toxic fungi and their mycotoxins)	Tenax TA adsorbent tubes. Thermal desorption.	GC- mass selective detector	52

Table 1 Continued

VOC	Canister sampling. Thermal desorption.	GC-quadrupole MS detection	53
VOC and semi-VOCs	SPE adsorbent. Thermal desorption.	GC-MS	54
VOC	SPME	GC-MS	55
Perfluorohydrocarbons	Tethered air pump system (capillary adsorption). Thermal desorption.	GC-ECD	56
VOC	Adsorption sampling (two separate adsorption tubes in series). Thermal desorption.	GC-MS/FID	57
VOC	Removal water from multibed carbon traps.Thermal desorption.	GC-MS	58
Pentachlorophenol		GC-MS	59
VOC	Automated adsorbent sampling. Thermal desorption.	GC-MS	60
VOC	Portable SPME fiel sampler and a SPME fibre coated with Carboxen/polydimethylsiloxane. Thermal desorption.	GC-ITMS	61
VOC	SPME. Thermal desorption.	GC-MS	62
HC	SPME	GC-FID	63
HC	SPME with poly(dimethylsiloxane)-coated fibres	GC-FID	64
Benzene and 1,3-butadiene	Stainless-steel SUMMA passivated canister	GC-ITMS	65
VOC	A solid adsorbent together with a temperature-programmed desorption system interfaced to a Balzers QMG 420 quadrupole MS via a membrane inlet	MS	66
Benzene and alkylated benzenes	Adsorbent sampling. Microwave desorption	GC-MS	67
Non-methane HC	PTFE cold trap cryofocusing.	GC-FID	68
C_2-C_6 HC	Cryogenic trap	GC-FID	69
Volatile oxygenated HC	Cryogenic trap.	Automated GC	70
Sulfur compounds	Cryogenic sampling. Thermal desorption.	GC-sulfur chemiluminiscence detector	71
VOC y SVOCs	Stainless-steel canisters (for C_2-C_{12} HC); adsorbent sampling (Tenax-TA) (for SVOC C_8-C_{20} HC); C_{18} Sep-Pak cartridges impregnated with DNPH (for carbonyl compounds).	GC-FID; GC/IRD/MSD; HPLC	73
HC	SUMMA stainless-steel canister. Thermal desorption	GC-SIM	74
Non methane HC	Cryoconcentration. Thermal desorption.	GC	75
VOC	On-line membrane extraction microtrap GC system.	GC	76
VOC	On-line microtrap and a sequential valve microtrap	GC	77
VOC	On-line membrane extraction microtrap	GC-FID	78
C_2-C_9 HC	Preconcentration trap.	Dual capillary GC	79
VOC	Split-less sampling with cryofocusing	HS-GC with ECD and FID/ eletrolitic conductivity and MS	80
HC		GC- comparison FID and ozone chemiluminescence detector	81
C_1-C_6 alkanes and CO_2		Portable GC-TCD	82
HC	Adsorbent sampling. PTV. Thermal desorption.	GC-FID	83
VOC	PTV. Thermal desorption	GC-FID	84
VOC	Glass injection port liner filled with activated charcoal from coconut husks. Thermal desorption.	GC-FID	85
HC (C_1-C_6 and C_5-C_{10})	Sorbent tube trap as an injection port liner inside a PTV injection Thermal desorption.	GC-FID	86
VOC and HAP (hazardous pollutants)		GC continuous emissions monitoring system. TCD.	87
VOC	Adsorbent sampling (a mixture of Carbotrap B, Carboxen 1000 and Carboxen 1001). Thermal desorption.	GC with photoionization detector (for aromatic and unsaturated compounds) and an electrolytic conductivity detector (for halogenated compounds) in series	88
Ozone precursor HC	Automated adsorbent sampling, closed-cycle cryofocusing.	GC-FID	89
VOC	Two multisorbent traps (TenaxGR, Carbotrap and Carbosieve SIII). Electric heater for desorption.	GC-ITMS	90
Ozone precursors (CO_2, N-oxides, SO_2, VOC and aromatic compounds)	Automated measuring stations	GC-FID	91
Products containing -COOH, -OH, and -C=O	Carbonyl groups derivatized with PFBHA; carboxyl and hydroxy groups with NO-bis(trimethylsilyl)-trifluoroacetamide.	GC- EIMS	92
HC	Adsorbent and canister sampling.	GC-FID/GC-SIM	93
VOC	Automatic sampler. SFE.	GC-SIM	94
Bis(chloromethyl)ether	Porous polymer sorption tubes (Tenax TA and Tenax GR)	GC-EIMS	95
Oxygenated VOC	Stainless steel canisters.	GC-MS	96
VOC	Automated air collection-concentration-introduction system:dual stainless-steel sample storage canisters. Thermal desorption.	GC-MS	97
VOC	Continuous electro-gas-dynamic flow	Atmospheric-pressure-CIMS.	98
VOC		MIMS method	99
VOC		Ion-store TOFMS	100
VOC	Adsorbent sampling (Tenax).	ITMS	101
VOC	Direct sampling. A comparison between a tubular methyl silicon rubber membrane inlet and an atmospheric sampling glow-discharge ionization source	ITMS	102
Acids and phenols	PTFE bag reactor. Tri-n-octylphosphyne oxide/methylterbuthyl ether extraction. Pentafluorobenzyl bromide derivatization	GC/ITMS	103
Microbial VOC	Sorbent tube. Desorption with CH_2Cl_2	GC-SIM	104

Table 1 Continued

Aldehydes and organic solvents	Adsorbent sampling (Tenax). Thermal desorption	GC-EIMS	105
VOC	Adsorbent sampling (Amberlite XAD)	GC-EIMS	106
VOC	distillation and collection in a cryotrap	GC-MS	107
Volatile organic sulfur compounds		GC-MIMS system	108
Trichloroethylene		FTIR remote sensor	109
VOC		FTIR spectra with a HgCdTe detector	110
VOC		Surface-enhanced FTIR spectroscopy sensor	111
VOC	---	IR DIAL sensor	112
Toluene and H_2O vapour		Photoacoustic sensor with NIR laser diodes.	113
Gases (acethylene, methane..)		External cavity diode laser with photoacoustic detector	114
Trichloroethene	---	Integrated optical NIR-evanescent wave absorbance sensor (InGaAs diode-array spectrograph)	115
HC		Optical sensors using UV absorption	116
VOC		Developmet of array sensors	117
Aromatic HC	Adsorbent sampling. Thermal desorption	GC-DOAS	118
Monocyclic aromatic compounds		DOAS	119
HC	Adsorbent cartridge sampling.	GC-DOAS	120
HC		Integrated optical enlarged-field interferometer sensor.	121
Volatile arsenicals: Lewisite	Gas permeation membrane sampling. FIA.	Detector based on the constant-current mode of potentiometric stripping analysis	122
HC and CO		Micromachined optical tunable filter sensor	123
VOC		Optical gas analyzers based on optoacoustic filters. Photodetector.	124
VOC		SAW chemical sensors	125
HC		Potentiodynamic gas sensor (Pt-electrodes)	126
VOC and CO_2		Potentiodinamic gas sensor (Pt- or Pd-electrodes)	127
VOC		Alternating-current impedance-based chemical sensors (Al-electrodes)	128
Phenol	Solvent extraction.	Oscillopolatographic detection	129
VOC (ethanol, toluene and o-xylene)		MOS sensor	130
Ethanol and H_2		A low-power CMOS compatible integrated sensor	131
Propane/butane		MOS sensor	132
Propene		MOS sensor	133
Saturated HC		MOS sensor	134
Saturated HC		MOS with an external catalyst element	135
VOC		Multisensor microsystem	136
Total organic compounds		Quartz crystal piezoelectric sensor	137
Toluene		Modified piezoelectric sensor	138
Olefines		Piezoelectric crystal sensor	139
C_1-C_4 aliphatic alcohols		Piezoelectric quartz sensor, modified with a sorbent film of polyethylene glycol adipate, and Ag electrode.	140
Phenol		Biosensor with tyrosinase.	141
VOC		Plasticized polymeric electrolytes gas sensors	142
HC		QMB and a SAW sensor.	143
VOC, acid vapours and other species	Adsorbent bed (activated carbon)	Gravimetry	144
Chlorinated and aromatic compounds		SPR optical fibre sensor	145

CIMS: Chemical ionization MS. CMOS: ceramic MOS. DIAL: differential absorption lidars. DOAS: differential optical absorption spectrometry. DNPH: 2,4-dinitrophenylhydrazine. EIMS: electron ionization MS. FTIR: Fourier transform IR. INCAT: Microextraction by inside needle capillary adsorption trap. IRD: Infrared detector. Lewisite: trans-dichloro(2-chlorovinyl)arsine. ITMS:ion trap mass spectrometry. MIMS: membrane inlet MS. MOS metal-oxide semiconductor. NPD: nitrogen-phosphorous detector. PTV: programmed temperature vaporisation. PFBHA: O-(2,3,4,5,6-pentafluorobenzyl)hydroxy amine. QMB:Quartz microbalance. SAW: surface acoustic wave. SIM. selected ion monitoring MS. SFE: supercritical fluid extraction. SPE:solid phase extraction. SPME:solid phase microextraction. SPR: surface plasmon resonance. TCD: thermal conductivity detector. TOFMS: time-of-flight mass spectometer.

yield stable hydrazones, which are determined by HPLC with UV detection. Sampling of aldehydes from air can be performed using solutions of DNPH in gas dispersion bottles or preferably solid media, such as cartridges, impregnated with DNPH.[146,147,148,149,150,151,152,153,154,155]

Possanzini and Di Palo[156] compared formaldehyde and acetaldehyde detection by **HPLC with fluorescence detection** using cartridges impregnated with DNPH or 2-diphenyl acetyl-1,3-indandione-1-hydrazone. Both methods were found to be suitable for field experiments.

Levin et al.[157] reported on the preparation of certified reference materials for the monitoring of aldehydes in air by 2,4-dinitrophenylhydrazine (DNPH), including solid hydrazones of formaldehyde, acetaldehyde, acrolein, acetone, and glutaraldehyde.

In order to reduce cost and to allow for continuous, unattended measurement, new automatic instruments for sampling and analysis of aldehydes and ketones from air were described. Schlitt[158,159,160] developed several methods that combine sampling of these substances with (DNPH) derivatization and HPLC analysis. Groempingen and Camman[161] reported the evaluation and automation of a DNPH with HPLC/UV detection for aldehydes and ketones in air.

Schwedt and Kunnecke[162] compared seven methods for analysis of formaldehyde in room air. All compared methods using some form of prior absorption. The Drager cartridge uses reagent of xylene/H_2SO_4 and visual comparison with a scale. The Bio-Check formaldehyde uses two-enzyme reactions and color formation with a tetrazolium salt. Two standard photometric methods are chromotropic acid (CTA), with measurement at 580 nm and acetylacetone, with measurement at 412 nm. Differential pulse polarography can be carried out with LiOH/LiCl or NaOH/EDTA supporting electrolytes. Finally, the HPLC method separates the dinitrophenylhydrazone on a C18 column, with a mobile phase of aqueous 35% acetonitrile and detection at 365 nm, this method is thought to give the best overall performance. Other methods have been described for the determination of atmospheric aliphatic aldehydes by colorimetry.[163] the latter procedures includes reaction with CTA.[164]

Grosjean and Grosjean[165] studied the effect of dry humid air on carbonyl collection efficiency and liquid chromatographic properties of C_1–C_{10} carbonyls using UV, diode array, and MS detection.

Yu et al.[166] developed a passive detecting tube for formaldehyde gas. This tube was filled with 101 white support impregnated with phenolphtalein and 0.05 M-Na_2SO_3 and both ends stoppered with cotton wool and sealed with wax. Color reaction was measured by colorimetry.

B. Gas Chromatography

GC is another technique that can be used for analysis of aldehydes, using several detectors. Many enrichment techniques have been described for GC, such as a preconcentration method consisting of trapping air volatile pollutants in a liquid reaction containing DNPH[167] or adsorption method on porous material.[168] Kartsova et al.[169] sampled air on silica gel cartridges impregnated with PEG-400. The hexamethylenetetramine formed in the eluate was determined by **GC-FID**. Balyatinskaya et al.[170] developed a headspace gas chromatography (HS-GC) separation with FID of toxic organic impurities (alcohols, ketones) from air. Ren and Guo[171] developed a rapid determination of low-molecular-weight aldehydes in air by **GC with thermal conductivity detector (TCD)** for formaldehyde and FID for acetaldehyde and propionaldehyde. Mori et al[172] determined aldehydes in indoor air samples by collection using *o*-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine-impregnated silica gel-solvent extraction-GC-ECD technique. Lehmpuhl and Birks[173] studied a new GC with FID or ECD method for the determination of atmospheric aldehydes and ketones based on C18 cartridge sampling and derivatization with 2,4,6-trichlorophenylhydrazine.

GC-MS appears as a technique of increasing interest for aldehydes determination. Binding et al.[174] determined airborne unsymmetrical aldehydes and ketones by GC-MS. Denuder tubes in the analysis of trace gases were conceived as a method for removing interferences from gas sample and later as means for isolating analytes from the sample, including formaldehyde. Thomas et al.[175] determined formaldehyde by conversion to hexahydrooxazolo[3,4-a]pyridine in a denuder tube with recovery by thermal desorption and analysis by GC/MS. The analysis and stability of aldehydes and terpenes stored in an electropolished canister were investigated by Batterman et al.[176] over 16 days, the canister was sampled and the analytes were determined by cryogenic preconcentration, GC, and MS. The applications of impact mass spectrometry (IMS) for trace gas analysis in work place and environmental sample was carried out by Liess.[177]

Formaldehyde emitted form wood and steel furniture was collected by adsorption, derivatized with acetylacetone and determined by UV and GC-ion trap MS.[178]

C. Micellar Electrokinetic Capillary Chromatography

The determination of formaldehyde, acetaldehyde, and acetone in the atmosphere by micellar electrokinetic capillary (MEKC) chromatograph was carried out by Lin et al.[179]

D. Amperometric Detectors

Most of the methods available for formaldehyde determinations are based on the formation of a color reagent, as mentioned above, but they have chemical interference and generate large amount of toxic wastes. Detection of formaldehyde by **amperometric detector** after chromatographic separation would appear to be an appealing option for a sensitive and selective detection. The chromatographic ion-exclusion/ion-exchange separation of formaldehyde from other polar organic molecules in air sample extracts containing aqueous sodium hydrogensulfite, and the selective detection by amperometry at a silver electrode was carried out by Shi and Johnson.[180] The polarographic method can be considered as an alternative to photometry and HPLC, Alter et al.[181] analyzed formaldehyde for this procedure. Chang and Xie[182] determined formaldehyde in ambient air using Girard’s reagent T (hydrazone)-coated glass fiber filters and adsorption voltametry.

Hori et al.[183] determined low concentration (10 to 120 ppb) of formaldehyde in air by means a detector and discriminator tube. The detector tube acted as a pH indicator.

E. Sensors

Gas-phase sensing has been dominated by **nonbiological sensors**, such as electrochemical, semiconducting and pellister-type sensors. Amperometric and potentiometric gas sensors are generally limited to a narrow range of electroactive gases. Semiconducting gas sensors suffer from a lack of specificity and have high power consumption demands. Woo et al.[184] detected formaldehyde using a portable high-throughput liquid absorption air sampler (a constant-drive syringe pump and mixing-chamber apparatus). Orlik et al.[185] studied the properties of ceramic semiconductor sensors based on tin(IV) oxide-antimony(III) oxide. The sensor responds to CO, methane, and formaldehyde in air. In recent years, the utility of small liquid drops and thin films has increased for sampling. In biphasic systems, such as collection of a gaseous analyte or solvent extraction, the analyses can be carried out *in situ* or on-line. Pereira and Dasgupta[186] reported a fiber optic/light-emitting diode (FO/LED)-based system that relies on the 3-methyl-2-benzothiazolone hydrazone (MBTH) chemistry for a portable inexpensive drop sensor that measures HCHO. It is the first sensor to use sequential reagent addition in a drop.

A need exists for gas sensors with low power consumption that are selective for unreactive gases and vapors. **Biosensors** have the advantage of high inherent selectivity, but to date have been mainly confined to aqueous media and certain organic solvents. Very few biosensors have been demonstrated for direct gas-phase monitoring. Vianello et al.[187] realized potentiometric detection of formaldehyde in air by an aldehyde dehydrogenase. Denninson et al.[188] developed biosensors capable of detecting low levels of formaldehyde and ethanol vapor. Both biosensors are based on dehydrogenase enzymes, which produce reduced nicotinamide adenine dinucleotide as product of the oxidation.

Table 2
Analytical Methods for Aldehydes and Ketones

Species	Sampling/extraction	Detection	Ref.
C_1-C_9 carbonyls	Cartridge sampling. DNPH derivatization.	LC-UV	146
Aldehydes	Sampling intercomparisons: pumped and/or diffusive sampling. Derivatization with DNPH.	HPLC ; monitor photoacoustic detector and an automatic colorimetric analyser; on-line GC with photo-ionisation detector; on-line GC-FID	147
Acetaldehyde	Modified diffusive sampler. DNPH-coated filter.	HPLC-UV	148
C_1-C_5 carbonyls	Cartridge sampling. DNPH derivatization. Extraction with acetonitrile.	HPLC-UV	149
Carbonyls	Cartridge sampling. DNPH derivatization.	LC-UV	150
Aldehydes and ketones	Adsorbent sampling. DNPH derivatization.	HPLC-UV	151
Formaldehyde	Cartridge sampling DNPH derivatization.	HPLC-UV	152
Aldehydes; aromatic HC	For aldehydes: silica cartridges coated with DNPH. For HC:cartridges sampling (Tenax TA). Thermal desorption.	HPLC-UV for aldehydes; GC-FID for aromatic HC	153
Carbonyls	C_{18} cartridge sampling. DNPH derivatization.	HPLC-UV	154
Carbonyls	C_{18} and silica gel cartridge sampling. DNPH derivatization.	LC -UV	155
Formaldehyde and acetaldehyde	Cartridge sampling	HPLC-fluorescence detection	156
Aldehydes	Coated filters with DNPH or DNPH solutions.	HPLC-UV; LC-TSP-EIMS.	157
Aldehydes and ketones	Automatic sampling: autoinjector for HPLC modified with conical mini-impingers. DNPH derivatization.	HPLC-UV	158
Aldehydes and ketones	Modified mini-impinger on-line with HPLC. DNPH Derivatization.	HPLC-UV	159
Formaldehyde	Miniaturised impinger mounted directly in the autoinjector. DNPH derivatization.	HPLC-UV	160
Formaldehyde	Comparison methods using prior absorption.	Colorimetry; differential pulse polarography; HPLC and spectrophotometry.	162
Formaldehyde	A sensitive tape containing silica gel and impregnated with a solution containing hydroxylamine sulfate, methyl yellow, glycerol and methanol.	Indicator tapes for colorimetry in automatic detector	163
Formaldehyde	Impingers.	Chromatropic acid method (NIOS-3500method)	164
Formaldehyde	Passive detecting tube	Colorimetry	166
Formaldehyde	A bubbler with H_2O. DNPH derivatization.	GC-ECD	167
Formaldehyde	SPME. DNPH derivatization.	GC-ECD	168
Formaldehyde	Cartridge sampling.	GC-FID	169
Ketones and alcohols	Adsorbent sampling.	HS-GC-FID	170
Low molecular weight aldehydes	Direct injection.	GC-TCD/FID	171
Aldehydes	Adsorbent sampling (PFBHA-impregnated silica-gel). Solvent extraction with benzene.	GC-ECD	172
Aldehydes and ketones	Cartridge sampling. 2,4,6-trichlorophenylhydrazine derivatization	GC-ECD compared with GC-FID	173
Aldehydes and ketones	Adsorbent sampling (DNPH-modified Chromosorb P AW).	HPLC-UV/GC-MS	174
Formaldehyde	Denuder tube with hexahydrooxazolo(3,4-a)pyridine, adsorbent sampling (Tenax TA). Thermal desorption and PTV injection.	GC-EIMS	175
Aldehydes and terpenes	Electropolished canisters, cryogenic preconcentration	GC-MS	176
Diborane, arsine, formaldehyde, nitrobenzene, phenol and hydrogen sulfide		by GC-IMS	177
VOC	Adsorption into distilled water-acetone derivatization	GC-ITMS for VOC. UV for formaldehyde.	178
Formaldehyde, acethaldehyde and acetone	Sampling in a DNPH solution. Extraction with CS_2.	MEKC	179
Formaldehyde	Impingers.	Ion- exclusion and ion-exchange chromatography with pulsed amperometric detection	180
Formaldehyde		Differential pulse polarography	181
Formaldehyde	Girard's reagent T-coated glass fibre filters	Adsorptive voltametry	182
Formaldehyde		Detector tube (acted as a pH indicator)	183
Formaldehyde	A portable high-throughput liquid absorption air sampler	Colorimetry	184
Formaldehyde, methane and CO		CMOS sensor	185
Formaldehyde	Drop collector	In-situ colorimetry	186
Formaldehyde	Pumped sampling (glass coil)	Potentiometric detection sensor	187
Formaldehyde and ethanol		Dehydrogenase biosensors	188

CMOS: ceramic metal-oxide semiconductor. DNPH: 2,4-dinitrophenylhydrazine. EIMS: electron ionization MS.HS: headspace. IMS: ion mobility spectrometry. ITMS:ion trap mass spectrometry. MEKC: micellar electrokinetic capillary chromatography. PTV: programmed temperature vaporisation. PFBHA: O-(2,3,4,5,6-pentafluorobenzyl)hydroxy amine. SPME:solid phase microextraction.TCD: TSP: thermospray.

IV. ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS

The term “semivolatile organic pollutants” (SVOCs) was coined by the US Environmental Protection Agency (EPA) to designate compounds that are not sufficiently volatile to be analyzed by purge and trap GC. The compounds range in volatility from dichlorobenzenes to benzo[*ghi*]perylene. The standard methods to determine SVOCs in air are very cumbersome and costly, involving adsorption on conventional adsorbents, for example, XAD-2 resin and polyurethane foam (PUF), recovery by Soxhlet extraction, evaporative concentration, followed by injection of only a small fraction of the final liquid sample. The inefficient utilization of the sample puts an increased demand on, volume sampled, accumulation flow rate, and concentrator size. Large amounts of ultrapure solvents are required and the adsorbents, which normally cannot be reused, are subjected to extensive precleaning procedures to ensure artifact-free analysis. With the longer term aim of replacing the sample preparation procedures with convenient concentrating and thermal desorption steps, thick-film silicone rubber traps were constructed in a novel multichannel configuration by Ortner and Rohwer.[189]

A. Sampling and Extraction

Many different methods have been used to sample and analyze SVOCs. The simplest sampling method is to draw air through a filter (for particles) followed by a sorption tube (for the gas phase). Thermal desorption analysis has been used both to analyze vapor phase SVOCs collected on Tenax[73,190] and to analyze particle associated SVOCs collected on filters.[191]

Solid phase extraction (SPE), thermal desorption, and GC-MS[54,192,193] or GC-FID detection[194] were used for determination of SVOCs in environmental material.

A multitude of methods has been published for the sampling of terpenes, based on adsorption on polymers like Tenax followed by thermal desorption-gas chromatography (TD-GC). Due to their unsaturated character, terpenes react readily with ozone. During the sampling, ozone may react with the trap material resulting in major alterations of the measured terpene patterns. Calohirou et al.[195] tested many of these potential ozone scrubbers, using an artificial system.

B. Chromatography

Dataur et al.[196] studied the improvement of analytical techniques for the determination of monoterpenes in atmospheric samples by water removal on potassium carbonate and calcium chloride. The analysis of monoterpenes or isoprene was carried out by GC-FID.

Larsen et al.[197] describe the results of a interlaboratory comparison with special attention drawn to the discussion of potential error sources common for the analysis of terpenes in air.

The use of MIMS for detection of SVOCs in air has two important advantages: sample enrichment without sorbent preconcentration and reduction of interferences. Cisper and Hemberger[198] demonstrate that rapid, on-line sensitive detection of SVOCs in air is easily accomplished by using membrane introduction ion trap mass spectrometry.

V. ANALYSIS OF POLYCYCLIC AROMATIC HYDROCARBONS

Polycyclic Aromatic Hydrocarbons (PAHs) are products of incomplete combustion of carbon compounds. They are ubiquitous in the environment and are important in the assessment of the air quality situation, first because of their potential risk to humans (toxicity, carcinogenic, and mutagenic effects are attributed to many compounds of this class), and second their influence on chemistry in the troposphere. They constitute an important environmental hazard. Furthermore, PAHs promote the soiling of building surfaces through deposition of airborne particulate matter. Thus, analysis of these compounds is an important task.

Due to the extensive amount of data suggesting the hazards of these compounds, many PAHs are on the EPA priority pollutant list. Significant levels of PAHs are found in the atmosphere but are more prominent in polluted urban areas.

Table 3
Analytical Methods for Semivolatile Organic Pollutants

Species	Sampling/extraction	Detection	Ref.
SVOC and VOC	SPE. Thermal desorption	GC-MS	54
VOC y SVOCs	Stainless-steel canisters for C_2-C_{12} HC; Adsorbent sampling (Tenax-TA) for SVOC C_8-C_{20} HC; cartridges sampling (with DNPH) for carbonyl compounds.	GC-FID; GC/IR/GC-MS; HPLC-UV	73
SVOC	Thick film silicone rubber traps	GC-FID; PTV inlet -GC-MS	189
Oxygenated terpenes	Adsorbent sampling onto stainless-steel tubes (TenaxTA). Cartridge sampling with DNPH for carbonyls. Thermal desorption.	GC-EIMS; HPLC-UV	190
SVOC	Adsorbent sampling (TenaxTA). Thermal desorption.	GC-FID/GC-FTIR/GC-EIMS	191
SVOC and VOC	HS-SPME .	GC-MS	192
SVOC (PAH)	Coupled subcritical water extraction and SPME.Thermal desorption.	GC- SIM	193
SVOC	Automated flow through system with a SPME poly(dimethylsiloxane) fibre.	GC-FID	194
Terpenes	Adsorbent sampling onto stainless-steel tubes (TenaxTA). Thermal desorption.	GC-EIMS	195
Monoterpenes	A preconcentration/thermodesorption module packed with Tenax TA (the moist vapour was passed through two desiccant tubes and K_2CO_3 or $CaCl_2$ to remove H_2O).	GC-FID	196
Terpenes	Sampling comparison	GC-MS/GC-FID	197
SVOC	Direct analysis	MIMS	198

DNPH: 2,4-dinitrophenylhydrazine. EIMS:electron ionization MS. FTIR:Fourier transformed IR. HS:Headspace. PTV: programmed temperature vaporisation. SIM:selected ion monitoring MS. SPE:solid phase extraction. SPME:solid phase microextraction.

PAHs are lipophilic substances, that is, nonpolar; however, polar functional groups can be introduced in PAHs by incomplete combustion in diesel engines or by chemical reactions with oxygen, sulfur, or nitrogen atoms forming PAH derivatives with a higher polarity. The substituted PAHs have been identified in diesel exhausts and atmospheric aerosols, in forest fires, industrial processes, domestic heating, or natural sources

Due to their wide range of vapor pressures, some atmospheric PAHs exist exclusively in the gas phase and others as adsorbed particulate matter. The volatility of these organic compounds controls their transport in the workplace and the environmental in general.

A. Sampling and Extraction

Correct air sampling of air pollutants is associated with a series of problems such as insufficient adsorption, reevaporation, and chemical destruction of already collected material. Due to their reactivities and volatilities, losses of PAHs during sampling are almost unavoidable. In addition, the concentrations of PAHs in air are low, and many of these compounds are unstable and volatile. These phenomena add significant error to detection of PAHs by air sampling. They have delayed a routine program on the determination of pollutants in ambient air for a long time.

The sampling methods used to detect and quantify PAHs in the environment are usually different from those applied in work places. In the first case the low concentrations (ng/m^3), and the special interest on the particulate matter, require the use of **a fiber glass filter** with high flow pumps using different detectors such as GC-MS[199,200] or GC-FID.[201]

The use of **XAD-2** is widely used for sampling, Zhu et al.[202] sampled air to determine PAHs on XAD-2. Extracts containing the PAHs were analyzed by HPLC.

Sturaro et al.[203] compare two different trapping systems to determine PAH in the workplace, a polytetrafluoroethylene filter coupled with a XAD-2 sorbent and a Carbotrap 150 cartridge. The double sampler method was more efficient for the condensed ring aromatic compounds analyzed, while the Carbotrap 150 is more suitable for the aromatic fraction present in the form of vapors.

Others authors[204,205] used a sampling head with glass fiber filters for retaining the particle-bound PAH fraction, a **PUF** to trap the medium volatile fraction, XAD-2 for the volatile fraction, and a second foam plug. The extracts were analyzed by GC-isotope dilution MS. Other sampling media include PUF,[206] a Whatman Quartz microfiber filter,[207,208,209] or the use of two consecutive Teflon-coated glass filters.[210]

Others authors[211,212] compared the closed-face 25-mm Gelman total dust sampler (TDS) and a different personal sampler designed at the Institute of Occupational Medicine (IOM) in order to meet the criteria for inhalable dust and PAHs. The samples were analyzed by GC-FID. The authors used a mass selective (MSD) for identification of PAHs compounds.

Butterfield et al.[213] used a **solid β-ciclodextrins** (CDs) as a collection medium for extracting PAHs from air. CDs enhance the solubility of PAHs, and their electroneutrality and hydrophilicity prevent interaction with the micelles. Gas-solid interaction of gaseous PAHs with solid CD can be used to decreases the volatility of PAHs during air sampling. However, it is difficult to separate PAHs with large molecular weights, as they cannot be readily included in cyclodextrines. Previous use of bile acids salts (sodium cholate and sodium deoxicholate), as the micellar phase for the separation of hydrophobic compounds such as PAHs proved successful by Dabek-Zlotorzynska and Lai.[214]

The relation between particle size and the atmospheric compounds constitute one of the less well understood aspects. Current ambient air analytical procedures are performed on samples collected by various methods. High volume samplings on filters is susceptible to sampling errors, such as “blow-off”, adsorption of semivolatile compound, and reactions of compounds sampled. An advantage of impactor sampling is the minimization of contact of the sample particles with the air stream. Schnelle et al.[215] used a five-stage high-volume **cascade impactor** and two Berner-type low-pressure cascade impactors to quantify PAHs by HPLC using fluorescence detection.

Measurement of PAHs generally involves the collection of vapor and/or particle-phase samples, followed by extraction and partitioning or fractionation. Liquid-liquid extraction (Soxhlet, others)[216, 217] and subsequent separation by column chromatography, thin layer chromatography (TLC) on glass plates, or by absorption onto special sorbents are widely employed for this purpose. However, such extractions often require large volumes of solvent, which may be toxic and/or expensive, and they often need several hours to even several days to yield acceptable, although often incomplete recoveries of some target analytes. Hence, alternative techniques are needed that are easy to use, fast, safe, and reproducible.

Supercritical fluids becoming attractive alternatives for the conventional liquid solvents used in the extraction of environmental samples. A method for the determination of chlordane and chlorpyrifos at the nanogram-per-cubic-meter level in air by supercritical fluid extraction was developed by Swami et al.[218] Five insecticides and two fungicides were analyzed in the air of green houses using a Tenax sorbent sample tubes and a GC with ECD and nitrogen-phosphorous detector (NPD) by Siebers and Mattusch.[219]

A focused microwave (FMW) extraction of PAHs seem to be a feasible alternative to Soxhlet extraction for many different matrices like marine sediments, soils, and air particulates. Its main advantages are the reduction of the extraction time (10 min) and of the solvent volume (30 mL). The FMW apparatus, working at atmospheric pressure, is easy, rapid, and safe to use. Letellier et al.[220] used a FMW extraction system to extract PAHs from the environmental matrices. Recovery and reproducibility were in agreement with those generated by Soxhlet extraction.

B. Chromatography

Measurement of PAHs involves a high selectivity detection to analyze small samples collected over a relatively short period of time. **GC techniques** were applied to measure PAHs derivatives using different detectors, the most frequently used was a nitrogen-phosphorus (NPD) selective detector,[221] other authors used a GC-FID system[222] and Harner and Bidleman[223] quantified atmospheric levels and particle/gas distribution of polychlorinated naphthalenes (PCN) by gas chromatography. GC/MS techniques are widely used to measure the PAHs.[216,224,225] GC/MS techniques were used to measure PAHs and nitro PAHs using difference sample preparation processes such as collection on a filter, solvent extraction, and fractionation by HPLC, separation by GC and identification and quantification by GC-MS.[226] PAHs were also determinated in the atmospheric environment by collection on a quartz fiber filter; Soxhlet extraction and GC-EIMS detection in selected monitoring.[227,228,229] Carrera et al.[230] analyzed PAHs using the same detection method and a solid-phase disc in the extraction method.

Vincenti et al.[231] developed a procedure for the determination of nitro-PAHs on crude-air particulate and soil extracts by the use of both selective ionization method, such as electron-capture chemical ionization, and specific fragmentation process, in a experimental tandem mass-spectrometry detector (GC-EC-MS). Elimination of purification and fractionation steps is possible using this process. Lopez-García et al.[232] developed an isomer-specific determination of polychlorinated biphenyls (PCB) in air, using an isotope dilution, high-resolution gas chromatography/medium-resolution mass spectrometry method (HRGC/MS).

HPLC is used for determination of PAHs and PAHs derivatives with different detectors. Few data are available on HPLC by UV detection of PAHs.[233] The sample was collected on a glass fiber filter and extracted by Soxhlet, and the separation of PAHs was carried out by HPLC, the detection was performed at 254 nm.

Minoia et al.[234] collected air samples through a glass-wool fiber filters and Supelco ORBO-43 pH absorption cartridges. After extraction the residues were analyzed by HPLC using fluorescence detection and GC-MS. A PAH mixture was separated and analyzed by HPLC on a Bondapak C_{18} or Kaseisorb PO-60-5 column using a fluorescence spectropho-

Table 4
Analytical Methods for Polycyclic Aromatic Hydrocarbons

Species	Sampling/extraction	Detection	Ref.
PAH	Glass-fibre filters connected to a cylindrical ampoule with XAD-2. Soxhlet extraction.	TLC compared with GC-EIMS	199
PAH	Glass fibre filter. Soxhlet extraction	GC-EIMS	200
PAH	Glass fibre filters. Soxhlet extraction.	GC-FID	201
PAH	Adsorbent sampling (XAD-2) or Millipore prefillers for PAH in particulates	HPLC-fluorescence detection	202
PAH	Adsorbent sampling (PTFE filter coupled with XAD-2 sorbent and a Carbotrap 150 cartridge). Thermal desorption.	GC- SIM	203
PAH(naphthalene,alkylnaphthalenes,fluorene,phenant	Glass fibre filter for retaining the particle-bound PAH fraction, a PUF plug to trap the medium volatile fraction, XAD-2 resin for the volatile congeners and a second foam plug.SPE.	GC-isotope dilution MS	204
Volatile and particle-bound PAHs	Glass fibre and a PUF filter sampling	GC-MS	205
PAH	Pollutants suspended on PUF. Extraction:microwave-assisted process was compared with Soxhlet extraction	GC-EIMS	206
Nitro- and hydroxy-PAH	Quartz Microfibre filter sampling. Soxhlet extraction.	GC-MS	207
PAH	SFE of quartz microfibre filters. Soxhlet extraction	LC-GC-AES and LC-GC-ITMS	208
PAH and alkyl-PAH (and s-heterocyclic compounds)	Quartz fibre filters. Ultrasonical extraction.	GC- SIM	209
PAH	Two consecutive Teflon-coated glass filters.Soxhlet extraction	HPLC-fluorescence detection	210
Pyrene	Inhalable aerosol sampler and a total dust sampler. Ciclohexane extraction.	GC-FID	211
Dust ; particulate, volatile & semivolatile PAHs	Comparison of the closed-face 25 mm Gelman total dust sampler and the IOM inhalable sampler	GC-FID/GC-MS	212
Volatile PAHs	Glass fibre filter with CDs	Fluorescence emission and absorption spectroscopy	213
PAH	Pressure injection samples	MEKC	214
PAH	Two systems: i) a five-stage high-volume cascade impactor. ii) two Berner type low-pressure cascade impactors.	HPLC-fluorescence detection	215
	Soxhlet extraction. HPLC fractionation.	GC-MS	216
Nitro-PAH (nitroarenes and aza-arenes)	Dicholoromethane extraction. Semi-preparative HPLC separation.	TLC and GC-MS	217
Chlordane and chlorpyrifos	Extraction thimbles sampling. SFE	GC-ECD	218
Residues of pesticides	Adsorbent sampling (Tenax tubes).	GC-ECD/NPD	219
PAH	Microwave extraction (open-vessel focussed-microwave system)	GC-EIMS	220
Nitro-PAH (nitroarenes)	Glass fiber filter (Staplex sampler). Soxhlet extraction	GC-MS and capillary GC-NPD	221
PAH;oxy-PAH;alkanes;alkanals;alkanols;sterols;fatt	Filtration sampling. Solvent extraction.	GC/FID and GC/MS	222
Polichlorinated naphthalenes	Hi-vol sampling (double-glass fibre filters + 2 PUF plugs). Soxhlet extraction	GC-FID/NIMS	223
PAH	Soxhlet extraction	GC-MS	224
PAH and alkanes	Lo-vol pumps and running in parallel with either a Charles Magnetron air pump or a large pump fitted with a flow limiting orifice. Solvent extraction.	GC-MS	225
PAH, benz(a)pyrene and nitrated-PAH	Soot collection on a filter. Solvent extraction	GC-MS (aliphatic fraction); HPLC-UV (aromatic fraction)	226
PAH and oxygenated derivates	Quartz fibre filters	GC-EIMS	227
PAH	Quartz fibre filters. Ethanol-benzene extraction.	GC-EIMS	228
PAH and oxygenated derivates	Quartz fibre filter.Soxhlet extraction.	GC-EIMS	229
PAH and organochlorine compounds	SPE under vacuum.	GC-MS	230
Nitro-PAH	Hi-vol Staplex sampler (glass fibre filters). Solvent extraction.	Hibrid tandem MS	231
PAH and aliphatic HC	Glass fibre filter. Soxhlet extraction	HPLC-UV	233
PAH	Glass-wool fibre filters and cartridge sampling. Sonication extraction.	GC-MS and HPLC-fluorescence detection	234
Fluorene,acenaphthalene,chrysene benz(a)antracene		HPLC and polarization synchronous fluorimetric detection	235
PAH	Glass fibre membrane. Sonication extraction.	HPLC-fluorometric detection	236
Nitro-PAH		On-line reduction HPLC/chemiluminiscence detection systems	237
Hydroxy-PAH		LC-MS	238
Nitroarenes and aza-arenes	Semi-preparative HPLC	GC-MS or TLC .	239
Hydroxy-PAH	Extraction with dichloromethane.	Overpresured layer chromatography and TLC on glass plates. Fluorescence indicator. Confirmation by GC-MS.	240
(3-14C)fluoranthene	Glass fibre filter and PUF sampling. Soxhlet extraction.	radioHPTLC	241
PAH	Multichannel silicone rubber trap. Thermal desorption.	GC-REMPI (induced by laser)-MS	242
PAH	i), ii) Direct injection. iii) Glass-fibre filter. Thermal desorption.	i)Photoelectric aerosol sensor. ii)Laser induced fluorimetry (continuous monitoring techniques) iii)GC-MS	243
PAH	Comparison between off-line sampling techniques (extraction with toluene) and an automatic analyser based on a photoelectrical sensor.	GC-EIMS	244
Alcohol and chlorinated HC vapours		Sensor system on the basis of semiconducting polymers	245
PAH		Constant energy synchronous fluorescence	246

AES: Atomic emission spectrometry.CD: cyclodextrin. EIMS: electron ionization MS. .Hi-vol: High-volume. HPTLC: High performance thin-layer chromatography. IOM: Institute of Occupational Medicine ITMS:ion trap mass spectrometry. lo-vol: low-volume. MEKC: micellar electrokinetic capillary chromatography .NIMS: negative ion MS. NPD: nitrogen-phosphorous detector. PUF: polyurethane foam. REMPI: resonance-enhanced multiphoton ionization PTV: programmed temperature vaporisation. SIM. selected ion monitoring MS. SFE: Supercritical fluid extraction. SPE: solid phase extraction. TLC: thin-layer chromatography.

tometer equipped with a polarized device by Zhang et al.[235] and Koyana et al.[236] respectively.

An on-line reduction HPLC/chemiluminiscence detection system for nitro-PAHs was developed by Hayakawa et al.[237]

Liquid chromatography-mass spectrometry (LC-MS) is an area of analytical chemistry of increasing importance. Atmospheric pressure chemical ionization (APCI) and pneumatically assisted electrospray (ESP) mass spectrometry (MS) coupled to HPLC have been used for the characterization of hydroxy-PAHs in standard samples by Galceran and Moyano.[238] Results showed that chemical ionization was the main process in APCI, and, in contrast to what happened with ESP, no electrochemical reaction was observed. In order to optimize the response in ESP and to adjust the optimum flow-rate for the coupling of the HPLC system ESP source, the variation of the response flow-rate and the use of a split system were studied by these authors.

TLC, GC, and MS are complementary methods that, when used in parallel, often give good results, especially in the analysis of complex organic mixtures. Several authors combined both techniques: Eskinja et al.[199] remove interfering substances in the cyclohexane extracts using TLC on silica gel G plates. For separation, identification, and determination, TLC separation process and GC-MS were used. Janozska et al.[217] analyzed nitrogen polynuclear aromatic compounds by GC-MS and TLC. The same authors studied[239] the separation of nitroarenes and azarenes present in a dichlomethane extract of airborne particles using HPLC, GC-MS, and TLC.

Modern **high-performance thin layer chromatography** (HPTLC) is an analytical method that fulfills all the requirements for use in qualitative analysis of environmental pollutants. It has become possible to use this method at over pressure (OPLC).[240] Ophoff et al.[241] used a glass fiber filter and three PUF plugs for the qualitative and quantitative determination of the ^{14}C-labeled organic substances by HPTLC.

C. Resonance-Enhanced Multiphoton Ionization

Other methods for the detection of PAHs with the reduction of the time needed for clean up of environmental samples using a system named Resonance-enhanced multiphoton ionization (REMPI) induced by an oxcimer laser has been described by Zimmermann et al.[242]

D. Sensors

Beckmann et al.[243] developed a sensor system for PAHs. The sensor is connected to the spectrometer by optical fibers and can be inserted into a flue to monitor both gaseous and particle-bound PAHs by irradiation with N_2 laser a 337 nm.

Agnesod et al.[244] compared off-line sampling technique with an automatic photoelectric aerosol sensor analyzer to determine PAHs in airborne particulate matter. Comparison of results showed a satisfactory linearity.

A novel polymer-chemosensor microsystem for alcohol and chlorinated HC vapors that can be operated without additional energy consuming heating was presented by Jahnke et al.[245]

He et al.[246] determined PAHs by a synchronous fluorescence system. The method was used for the simultaneous determination of anthracene, benzopyrenes, perylene, and pyrene.

VI. ANALYSIS OF HALOCARBONS

Production of halocarbons by the chemical industry is now restricted under terms laid out in the Montreal Protocol and subsequent revisions. Manufacture and use of specific halocarbons, chlorofluorocarbons (CFCs) (man-made compounds used in refrigeration and cooling systems, foam blowing, aerosol propellants and industrial solvents), halons (bromide-containing compounds used in fire extinguishing), and certain chlorinated solvents is now prohibited because they can participate in reactions that remove the ozone. In addition to, antropogenic sources there are halocarbons know to be biogenically produced, such as CH_3Cl, CH_3Br, which play an important part in stratospheric ozone depletion, and, consequently, there is immense interest in quantitative airflows.

On the other hand, CFCs have been replaced by a group of interim surrogate compounds, the hydrochlorofluorocarbons (HCFCs), which have a labile C-H bond along with lower chlorine and bromine content. Consequently, tropospheric destruction of HCFCs is more rapid than their CFC predecessors, and the ozone depletion potential (OPD) is smaller. HCFCs are in turn being replaced by hydrofluorocarbons (HFCs), which have zero OPD. The following HFCs are the most likely to be used as replacements, HFC 125, 143 a, 134 a, 22,24,124,142b and 123. The monitoring of all these species is important to verify the expected decrease in atmospheric halogen burden as consequence of the Montreal Protocol and to assess the environmental impact of the new substitutes (HCFCs and HFCs).

A. Sampling and Extraction

The use of direct chromatographic analysis is precluded, and sample preconcentration methods are necessary prior to analysis. Methods of preconcentrating ambient air samples normally involve trapping the halocarbons cryogenically followed by GC separation coupled with mass spectrometric detection[43] or ECD.[247,248] However, the ECD is a highly sensitive detector used for direct determination of certain halocarbons without preconcentration.[249,250,251]

A disadvantage to **cryogenic trapping** is that it collects a large amount of water during the sampling, which induces chromatographic difficulties. Bassford et al.[252] eluded this problem by developing a new gas chromatographic method to determine halocarbons at a quantitative level, using a microtrape to adsorb air sample at ambient temperature, followed by thermal desorption directly onto a capillary column.

It is not easy to automate the analytical methods for halocarbons, because they need cryogen or cooling devices for preconcentration of the compound from large volumes of air samples because of their extremely low concentration in the ppt range and low sensitivity to detectors. In addition, they use chemical or physical dryers for water removal in the large volume of samples introduced, which may cause contamination and/or adsorption of target compounds.

Toyoda et al.[253] reported an automatic analytical system for HCFC-22 measurement. By using two columns in series they avoided the use of cryogen and dryer for preconcentration or sample drying. Oxygen was doped into the N_2 carrier gas to sensitize the ECD. It was found that the connection of a normal ECD and an O_2-doped ECD in series makes it possible to measure major CFCs and HCFC-22, respectively, and simultaneously without interfering with each other as mentioned by Sturrock et al.[254] Although the detection limit of HCFC-22 is not low enough for background air monitoring under the current conditions, it can be improved by optimizing the analytical conditions for HCFC-22 measurement alone. Then an improved system can possibly be used even at unattended remote monitoring stations.

B. Gas Chromatography

Toyoda et al. [255] developed an automated **GC/ECD**, as an alternative approach to attain a cryogen-free and dryer-free, high-frequency measurement method that a can be applied to the on-site fields of measurements of halocarbons at remote atmospheric monitoring stations. The analytical system consists of an outdoor-air inlet line, a vacuum line, switching valves, and two gas chromatographs, using two columns in series for each of two groups divided by the volatility of the compounds with three ECDs. It has dual analytical lines, each of which contains a sample loop, a precolumn, and a main column. One is used for the separation of the CFCs, and HCFCs (channel A), and the other for the heavy halocarbons (channel B).

Bruner et al.[256] determined CFCs ($CFCl_3$, CF_2Cl_2, $C_2F_4Cl_2$,CH_3CCl_3, and CCl_4) in Terra Nova Bay with GC-ECD. Air samples were collected in stainless steel canisters whose internal walls had been electro-polished to produce inert surfaces. Samples were analyzed without a preconcentration step. Others authors[257] also used the same collection method.

The measurement of CFCs has become well established as a technique for tracing oceanographic processes. Until recent, seawater analyses have been confined to CFC-11 and CFC-12, using a packed column electron capture—GC system. Recent changes in the CFC use involved increasing the range of transient tracers. To this end other halocarbons have been investigated, such as 1,1,2-trichloro-1,2,3 trifluoroethane (CFC-113) and tetrachloromethane. The CFC-113 used as solvent has an atmospheric growth more important than CFC-11 or CFC-12. The addition of these two news halocarbons as oceanographic tracers involved several problems. Both are less volatile than CFC-11 and CFC-12 requiring an extended analysis time on packed columns systems, with attendant peak broadening, sensitivity loss, and interference from co-eluting compounds such as CFC-113. For these reasons Boswell et al.[258] developed a new chromatographic technique based on a BD-624 megabore column together with a sample trapping/ injection system. In addition, a dual detector configuration increased sample throughput. The new instrument allows resolution of all four CFCs over a wide concentration range.

Lin et al.[259] analyzed chlorinated hydrocarbons pollutants in air by **GC-FID** with solid adsorbents and supercritical carbon dioxide extraction. An ethanolic solution containing 1,2,3-trichloropropane, trimethylenebromide, dichlorobenzene, 1,4-dibromobuthane, and *m*-dibromobenzene (as typical air pollutants) were injected onto solid sorbents (charcoal, Chromosorb 102, Chromosorb 105, and Tenax-TA). After 12 h, the adsorbents were dried to remove ethanol. The standard sample was used for supercritical fluid extraction (SFE) on a self-made apparatus with CO_2 modified with methanol.

Greally et al.[260] utilized GC/FID for the analysis of CFC, halones, and halomethanes. These authors used a silica-based stationary phase for separation of HCFC, which has an advantage over its alumina-based analogue in that labile haloalkanes were not dehydrogenated.

Most of the end-products from degradation of CFC are inorganic acids, salts, CO_2, and trifluoracetic acid (TFA), which is a significant end-product of three of the substitutes HCFC-123, HCFC-124, and HFC-134a. TFA has been detected in environmental water, rainwater, and air. Zehavi and Seiber[261] developed an analytical method for the determination of trace levels of TFA. TFA is derivatized to the volatile methyl trifluoroacetate and determined by automated HS-GC with ECD or manual HS-GC using GC/MS in the SIM mode.

Watanabe et al.[262] used a gas-phase electrolytic conductivity detector applied to GC. A portable GC instrument equipped with the detector described above was used for the determination of volatile chlorohydrocarbons (tricholoroethylene and tetrachloroethylene) in air. The sensitivity of the detector to each compound was dependent on the number of Cl atoms in a molecule of the compound. They used a charcoal tube for sample collection.

C. Sensors

Collins and Rose-Pehrsson[263] developed a **chemiluminiscent chemical sensor** for inorganic and organic vapors (such as a CCl4, CHCl3, and CH2Cl2). The sensors were constructed by immobilization of luminol or *tris*- (2,2′-bipyridyl)ruthenium III between a miniature photomultiplier tube and a polymer-tetrafluroethylene (PTFE) membrane, through which a stream of air was sampled by diffusion. A hydrogel support was used for the luminol sensor utilized to determine chlorinated hydrocarbons; a heated Pt filament incorporated in the glass line leading to the chemiluminiscent cell was used to oxidize the analytes prior to diffusion across the PTFE membrane. A prototype real-time instrument for the determination of trace hydrazine of NH_3 vapour was constructed.

A robust **surface plasmon resonance (SPR) sensor** was fabricated by depositing a silver layer on a section of a silica optical fiber from which the cladding had been removed. The thermally deposited silver was coated with *n*-octadecanethiol to prevent oxidation and fluoroalkylsiloxane polymer. A flow-through detection cell was mounted around the prepared section of the optical fiber. One end of the fiber was illumination with a laser beam at an oblique angle, and the light transmitted through the fiber was detected with a photodiode. Mixtures of chemical vapor and dehydrated air were passed through the detection cell and the changes in transmitted intensity were monitored.

Table 5
Analytical Methods for Halocarbons

Species	Sampling/extraction	Detection	Ref.
Halocarbons (chlorinated C_1- and C_2-HC) and MAH	Chrompack desorbed off-line (Carbopack B and Carbosieve SIII).Thermal desorption and cryofocusing.	GC- SIM and GC-FID	43
Halocarbons, C_1-C_4 organic nitrates	Bag sampling . Preconcentrated on fused silica beads at -180°C. Desorption at 30 °C. Cryofocusing.	GC-ECD	247
Halocarbons, HC	Stainless-steel canisters. Cryogenic trap for preconcentration and cryofucusing	GC-ECD for halocarbons and GC-FID for hydrocarbons	248
Volatile halogenated HC (VHC)(halohydrocarbons)	Passive sampler (Charcoal). Extraction with m-xylene	GC-ECD	249
Organochlorine compounds	Hi-vol sampling (PUF and glass fibre filters). Soxhlet extraction.	GC-ECD	250
Halocarbons (methyl iodine (iodomethane); 3 CFC; CCl_4; $CHCl_3$)	Trap sampling (Porasil C). Thermal desorption.	GC-ECD	251
Halocarbons	Microtrap with Carbotrap and Carboxen. Back-flushing desorption.	GC-ECD	252
HCFC	Automatic analytica system: an outdoor air inlet line, a vacuum line, valves conected with precolumn of chromatograph	Automated GC-oxygen-doped ECD	253
Halocarbons	System with an outdoor-air inlet line, a vacuum line, switching valves and two GC with three ECD. The system required no cryogen for preconcentration or any method of water removal	Automated GC-ECD	255
CFC (CFC13, CF2Cl2, C2F3Cl3, C2F4Cl2, CH3CCl3, CCl4)	Stainless-steel canisters (electro-polished internal walls).	GC-ECD	256
CFC(12,114,114a, 113), dicholoromethane), halones (1301 and 1211)and bromomethane	Stainless-steel canisters.	GC-ECD	257
Halocarbons	A purge-and-trap system with Unibeads 25. Thermal desorption.	GC-ECD	258
1,2,3-trichloropropane, trimethylenedibromide, dichlorobenzene, 1,4-dibromobuthane, m-dibromobenzene	Standard injection onto solid adsorbents (Charcoal, Chromosorb 102, Chromosorb 105, Tenax-TA). SFE with CO_2 and methanol	GC-FID	259
Halocarbons		GC-FID	260
TFA (trifluoroacetic acid)	Glass denuder tube sampling. Extraction: alternative to GC/MS for high salt samples: acidifying the concentrated sample with sulfuric acid, extracting with diethyl ether and reextracting with dilute aqueous bicarbonate	automated HS-GC-ECD or manual HS-GC-SIM	261
Volatile chlorinated HC	Charcoal tube sampling.	Portable GC-ECD	262
Chlorinated HC (CCl_4, $CHCl_3$, CH_2Cl_2); hydrazine; NH3		Chemiluminiscent chemical sensor	263
Chlorinated HC(trichloroethylene, CCl_4;Chloroform; methylene chloride)	Sensor on line	SPR sensor. Detector: photodiode	264
Halon alternative agents		LIBS	265
CCl_4		glow discharge AES	266

AES: Atomic emission spectrometry. Hi-vol: High-volume. HS:headspace. LIBS: Laser-induced breackdown spectroscopy. PUF: polyurethane foam. SIM: selected ion monitoring MS. SFE:supercritical fluid extraction. SPR: surface plasmon resonance

Chlorinated hydrocarbons/air mixtures (trichloroethylene, carbon tetrachloride, chloroform, and methylene chloride) produced an increase in transmitted intensity.[264]

D. Others Methods

As consequence of prohibition of the halons, alternative compounds appear such as C_2F_5H, CF_4, CF_3H, and CF_2H_2. The detection of the alternative agents is of practical concern in order to monitor their concentration and to ascertain their effect on atmospheric chemistry. **Laser-induced breakdown spectroscopy** (LIBS) is investigated by Williamson[265] as a method for the detection of halon alternatives. The emission of fluorine, which is liberated in a plasma, is used for the analysis. The ultimate goal is the application of this technique in a several hostile fire suppression testing environmental.

Kim et al.[266] developed a **gas-jet glow discharge atomic emission spectrometry** to eliminate toxic contaminants or harmful gases in airflows. The CCl_4 was chosen as the toxic pollutant owing to its similar molecular structure with respect to the final target samples such as phosgene ($COCl_2$) and diphosgene ($ClCOOCCl_3$) and CCl_4 itself, which show serious toxicity effects.

VII. ANALYSIS OF AMINES AND ISOCYANATES

Isocyanates are known occupational hazards. They are reactive compounds that cannot feasibly be analyzed as such. For the last 25 years the determination of isocyanates has been based essentially on **derivatization with various amine reagents** due to the high reaction rate between isocyanates and amines. Several authors[267,268,269] described methods for the determination of aromatic isocyanates in air using dibutylamine (DBA) as the derivatizing reagent followed by LC and UV and ESP-MS detection.[270,271] DBA was found to be a robust reagent reacting fast with the isocyanate groups and without know interferences.

Tinnenberg et al.[272] studied toluene diisocyanate (TDI) air levels at two polyurethane-factories using the 9-*N*-methyl-aminomethyl-anthracene (MAMA) reagent.

A reversed phase-grade elution HPLC method for the separation of 1-2(2-pyridyl)piperazine (1-2pp) of the four diisocyanates was developed by Wang.[273]

Schlitt[274] developed a new automatic analyzer for the determination of diisocyanates and formaldehyde in the atmosphere, which combines air sampling and derivatization of the contaminants to stable hydrazones and urea derivatives.

Tryptamine was evaluated as a reagent for derivatizating hexamethylene diisocyanates by Rudzinski et al.[275] In two separate studies, impinger sampling using 1-(2-methoxyphenyl) piperazine in toluene and solid sorbent sampling with a tryptamine-coated XAD-2 resin were compared with impinger sampling with tryptamine in DMSO.

Ethanolamines are used extensively in industry as corrosion inhibitor for pH adjustment. The ethanolamines involve health hazards as they act as respiratory irritants and sensitizing agents. Various techniques are used for determination of ethanolamines; however, only a few methods for the determination simultaneously are described. Sollenberg[276] described an easily operated and low-cost method using isotachophoresis (ITP) for the determination of ethanolamines in metalworking fluids on air samples collected in workrooms.

Aromatic amines were determined as their ***N*-dimethylthiophosphoril derivatives** by GC/NPD.[277]

The use of test methods for determining various organic and inorganic substances is urgent for economic reasons and because methods are of great importance in environmental analysis for determining organic contaminants. For these reasons Evgen'ev et al.[278] studied the applicability of **chlorodinitro derivatives of benz-2,1,3-oxodiazole** in selective test methods for determining toxic amino compounds in air and aqueous media, using visual and spectrophotometric detection of signals.

Tran and Gao[279] developed a novel **integrated acoustooptic tunable filter (IAOTF) and an erbium doped fiber amplifier (EDFA)** for the determination of monomethylhydrazine.

The determination of traces of triethanolamine in air by **GC-MS** was studied by Giachetti. [280]

VIII. CONCLUSIONS

Diffusive samplers are very practical tools for air quality monitoring especially if extensive campaigns or personal monitoring are planned. The accuracy and precision of the methods have been established by parallel sampling and by intercomparison with other methods. The passive samplers can be used to evaluate the suitability of storage and display environments, and test the efficiency of mitigation strategies. For the monitoring of hydrocarbons, tube diffusive samplers analysed by thermal desorption in combination with GC provide an optimum compromise between economy (reusable), ecology (solvent--free desorption), and detectability (absence of solvent peaks).

Tenax sobent has been used because of its excellent properties of trapping pollutants along with high thermal stability, which in turn facilitates thermal desorption. Hydrocarbons in the C_8 to C_{20} range when collected on a Tenax cartridge seem to be more stable after storage than when collected in the canisters. Chromosorb 106 is recommended for the sampling of more volatile and polar organic vapors. However, a number of other sorbents are also satisfactory for sampling the majority of compounds. These sorbents included Carbotrap and Carbopack B. Anasorb CMS is a promising sorbent for the sampling of VOCs. The combination Anasorb CMS and Tenax TA showed good results and needs further examination for the sampling of other single VOCs or mixture in different proportions.

A dry purge method is very effective in removal of water adsorbed on sorbents such as Carbosieve S-III and Carboxen-569, and deserves to be used widely, especially for the determination of polar organic compounds in air.

Sorption on the activated charcoal followed by desorption with CS_2 is the reliable method for determination of different classes of oxygenated compounds in emissions.

The develop of MS for monitoring hazardous air pollutants in real time has generated great interest. The analysis is facilitated by introduction of new monitoring systems, based on MS and direct air sampling interfaces. MS, especially when coupled with GC, has potential for use as a field-monitoring technique for detecting vapor-phase organic compounds.

A CG-CEMS was demonstrated to perform reliably in a factory setting over a long period of time. This system circumvents the quantification problems encountered by other analyzers. The autoGC is suitable for monitoring hazardous air pollutants by using a multiadsorbent concentrator tube.

The multitude of sensor elements and their new multistep design allows a high degree of versatility to tailor the sensing performance according to the analytical needs. Remote sensors do not require physical collection of samples. FT-IR remote sensor offers capabilities for monitoring large areas that cannot be covered adequately with a conventional fixed point. UV absorption in the evanescent field of polymer-clad silica fibers can be used for the realization of low-cost hydrocarbon monitoring. An NIR laser is easy to operate at room temperature and another advantage is the availability of robust glass fiber for these wavelengths. It was shown by field experiment that coherent infrared differential absorption lidars can be used as remote sensors for a broad range of atmospheric gases. Many organic solvents, which are not reactive in the potential window accessible in amperometric sensors, can be detected by using an appropriate potential program to a Pt electrode.

The use of DOAS system in an advanced monitoring station should be considered an important improvement of the existing network in order to upgrade their responses toward an effective strategy for pollution control.

The simplicity and sensitivity of the SPR fiber optic sensor are promising. This device can be used either as an optical fiber refractometer or as a chemical vapor sensor. The potential of the technique is far from being fully explored.

Chemical derivatization coupled with gas chromatography and mass-selective detection provides an effective means of monitoring several different compounds in air.

Thermal desorption analysis has been used to analyse vapour phase SVOCs collected on both Tenax or filters. The use of MIMS has two important advantages: sample enrichment without sorbent preconcentration and reduction of interferences.

Table 6
Analytical Methods for Isocyanates and Amines

Species	Sampling/extraction	Detection	Ref.
Isocyanates and amines	Impingers. DBA and ET derivatization	LC-ESP-MS	267
Monomeric and polymeric isocyanates	Impingers. DBA and ET derivatization	LC-UV	268
Isocyanates and amines	Impingers. DBA and ET derivatization.	LC-UV	269
Low molecular weigh aliphatic isocyanates	Impingers. DBA derivatization	LC-ESP- MS/ GC-chemical ionization with amonia, monitoring positive ions	270
Aliphatic isocyanates	Impingers. DBA derivatization	LC-ESP- SIM	271
Toluenediisocyanate	Impingers. MAMA or DBA derivatization.	LC-UV/LC-MS	272
Diisocyanates	Glass fibre filter sampling. 2-PP	HPLC-UV	273
Diisocyanates and formaldehyde	Miniimpingers. 2-PPderivatization for diisocyanates; DNPH-cartridge for carbonyl compounds.	HPLC-fluorescence detector (for diisocyanates); HPLC-UV for carbonyl compounds	274
Isocyanates	Tryptamine derivatization in an impinger and on XAD-2	HPLC with fluorescence detector/ electrochemical detector	275
Ethanolamines		Isotachophoretic method	276
Amino compounds		Indicator tubes. Visual, spectrophotometric and HPLC-UV	278
Monomethylhydrazine		NIR spectrometer based in AOFT and Er-doped fibre amplifier	279
Triethanolamine	Adsorbent tube sampling (silica gel). Solvent extraction.	GC-EIMS	280

AOTF: acoustic-optic tunable filter. DBA: dibutylamine. DNPH: 2,4-dinitrophenylhydrazine. EIMS: electron ionization MS. ESP: electrospray. ET: ethylchloroformate. MAMA: 9-N-methyl-aminomethyl-anthracene. NIR: near infra-red. 2-PP:1-(2-pyridyl)piperazine. SIM. selected ion monitoring MS.

The analysis of PAHs require the use of fiber filters with high flow pumps using different detectors such as GC-MS or GC-FID because of their low concentrations.

Alternative extraction techniques are need of that are easy to use, safe, and reproducible. The use of CDs as a collection medium for extracting PAHs from air enhances the solubility of PAHs. Supercritical fluids or FMW extractions seem to be feasible alternatives.

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