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## Review

# On-site monitoring of volatile organic compounds as hazardous air pollutants by gas chromatography

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## Abstract

There are a large number of organic components in air. These components can be classified in six groups. On-site monitoring procedures for two of these groups, oxidant precursors and hazardous air pollutants, are reviewed. For hazardous air pollutants, mainly long-term data are required. Oxidant precursors, however, some of which are very volatile, must be detected and quantified as early and as rapidly as possible. The monitoring techniques differ accordingly. Considerations governing on-site monitoring methods for both purposes are discussed.

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## 1. Introduction

There are a large number of trace organic compounds in air and the quantity of such components originating from human activity is increasing year by year. These components are emitted from industry, automobile exhaust, ag-

riculture, volatile organic solvents, etc., and then mix with the organic components of natural origin. It is very important to be able to detect and quantify these trace organic components in air in order to identify source contributions, causes of photochemical smog formation and effects on human health. The hazards such trace organic components represent depend on the long-term exposure to them. A number of efforts

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to detect these trace organic components in air, determine the concentration levels and define the nature and extent of transformation are listed and reviewed [1–5]. The next stage is to develop continuous monitoring methods for long-term observation of the target components.

## 2. Classification of trace organic components in air

There are long lists of organic components, hazardous air pollutants (HAPs), in environmental air. Many of these components are thought to be carcinogens or mutagens. Many countries have legislation on the maximum admissible concentrations of these components in drinking water, waste water, underground water, environmental water and air, and back this up by monitoring.

It has been proposed that volatile organic components (VOCs) in air should be put into four classes in terms of possible adverse effects: (1) VOC-OXes: VOCs characterized by high photochemical ozone and peroxyacetyl-nitrate (PAN)-creating potentials (mainly alkenes, alkynes and alkanes from C<sub>2</sub> to C<sub>8</sub>, arenes from xylenes to tetramethylbenzenes, terpenes), oxidant precursors. (2) VOC-TOXes: VOCs that are either known to be toxic or cause concern with respect to toxicity (HAPs). (3) VOC-STRATs: VOCs characterized by high ozone depletion potentials (freons, halons, chlorinated hydrocarbons). (4) VOC-CLAIMs: VOCs claimed to have positive or negative greenhouse effects [6].

Two more classes are now added to this list. One is secondary pollutants formed by the photochemical oxidization of hydrocarbons. It is very difficult to identify the original source of aldehydes, free fatty acids, ketones and ethers. Aldehyde, PAN and peroxypropionyl nitrate (PPN) groups can better be classified as secondary pollutants (VOC-FORMs: mainly formed by photochemical oxidization) [2]. The other is semi-volatile organic components [semi-VOCs: changing form according to environment, e.g., air-tight packaging, atmosphere, soil (poly-

aromatic hydrocarbons, pesticides)]. Specifying these two extra groups in the classification will help point efforts to develop monitoring methods in the right direction. It should further be noted that these components are related to their emission sources, and that some of them belong to two or more classes. It is also very important to notice that there is a close relationship between toxic organic compounds in air and in water.

Many monitoring methods (separation and detection procedures) which meet the observation requirements have been developed and examined. A survey of these methods and their applicability is given in Table 1. These methods are also used to complement each other for the purposes of tracking the process of environmental change. But samples need transportation, and time elapses before analysis; consequently, reaction or adsorption often takes place during sample storage [7,8]. The lifetimes of some VOC-TOX components are one day or less [1,2]. The potential change in components such as VOC-OXes within a short time means that a large number of sample analyses is required [9]. In such cases, on-site observation must be the ideal method. Continuous observation of VOC-STRATs and VOC-CLAIMs is also necessary, but in these cases analysis with sample collection by canister or adsorption tube for several times a year, is sufficient [8,10].

This review focuses on the on-site method for monitoring VOCs in air, and examines possible ways of optimizing quantitative analysis for this purpose.

A number of trace organic components of air are present at extremely low concentration levels. This means that a very powerful method is required, especially for VOC-OX and VOC-TOX measurement. Capillary gas chromatography (GC), with sensitive and selective detection, can meet the requirements. In the development of the system, the most important aspects are the selection of the detector and the sample volume appropriate to the analysis. Table 2 shows the appropriate detectors, together with estimates of minimum sample volumes for long-term observation (10 times higher than the detection limit).

Table 1  
Classification of VOCs and observation procedure (sample collection–separation–determination)

Classification		On-site monitoring	Sampling/analysis
VOC-OX		TD–GC–FID TD–GC–MS	Canister TD
VOX-TOX	Halogenated hydrocarbon	TD–GC–PID–ECD (+FID) TD–GC–PID–ELCD TD–GC–MS	Canister TD
	Oxidized hydrocarbon	TD–GC–MS	Canister TD
VOC-FORM	PAN, PPN	SL–GC–ECD CT–GC–ECD	CT
	Aldehyde	NA	Reaction–GC–FTD Reaction–GC–MS Reaction–HPLC–UV
Semi-VOC		NA	SPE–GC–MS PUF–GC–MS
VOC-STRAT		NA	Canister–GC–ECD
VOC-CLAIM		SL–GC–FID (methane) TD–GC–FPD (dimethyl sulfide)	Canister

TD = Adsorption–thermal desorption; SL = direct injection by sample loop; CT = cold trapping; Reaction = collection by chemical reaction with reagent; SPE = solid phase extraction; PUF = polyurethane foam collection; NA = not available.

### 3. Problems and key points in the development of on-site monitoring methods

High-resolution gas chromatography is a powerful technique suitable for on-site monitoring for trace and complex volatile organic components in air (VOC-OX and VOC-TOX). However, there are some problems to be solved concerning the optimization of the analysis system. Sample collection and the introduction procedure are key aspects governing the construction of automated systems.

Liquid nitrogen or a similar coolant is commonly used for sample collection and sample injection into a capillary gas chromatograph for gaseous sample analysis. (1) A large proportion of sample collection and concentration is done with liquid nitrogen or liquid argon [11,12]. (2) Liquid nitrogen or liquid carbon dioxide is used for cryogenic focusing during introduction of the sample into the capillary column. There are two

techniques: (a) a cryogenic focusing device is attached in front of an analytical column [13–16]; (b) the GC oven is operated at sub-ambient temperature (whole-column cryogenic focusing) [17–25]. In both techniques, plugging problems are caused by water or carbon dioxide in air samples.

When both easy maintenance is required and long-term observation is the objective, liquid nitrogen and liquid argon are not desirable as coolants because of their rapid loss by evaporation. The system developed must be simple in design and readily optimizable. These problems will be solved if the sample is collected and concentrated on an adsorbent, thermally desorbed by rapid heating, then concentrated on a stationary phase. With these techniques the use of coolant would be omitted. Reaction of collected sample with ozone is known to take place on the surface of adsorbent [7,26]. This should be taken into account in system evaluation.

Table 2  
Appropriate detectors for on-site monitoring and detection limits

Detection system	Detection limit (estimated sample volume for 0.1 ppb level detection)	Stability /week	Special property	Applicability
Flame ionization detection (FID)	0.1 ng (100 ml)	± 2%	Proportional response to carbon number, very stable. Good for quantitative detection	VOC-OX VOC-TOX VOC-CLAIM (CH <sub>4</sub> )
Mass spectrometry (MS)	1 pg/SIM mode (10 ml) 0.1 ng/SCAN mode (100 ml)	± 10%	Poor sensitivity in scan mode. Highly selective. Low-molecular-mass hydrocarbons difficult to detect	VOC-OX VOC-TOX VOC-FORM
Electron-capture detection (ECD)	1 pg (CCl <sub>4</sub> ) (1 ml)	± 3%	Highly sensitive to over 3 chlorine atoms in molecule. No destruction	VOC-TOX VOC-FORM(PAN) VOC-STRAT
Electrolytic detector (ELCD: halogen mode)	1 ng (1000 ml)	± 5%	Proportional response to the number of chlorines, bromines. Stable	VOC-TOX
Photoionization detector (PID)	0.1 ng (benzene) (100 ml)	± 5%	Selective response to aromatics unsaturates. No response to alkanes. No destruction	VOC-OX VOC-TOX
Flame photometric detector (FPD)	10 pg (sulfur) (50 ml)	± 5%	Non-linear response. Sulfur-, phosphorus-selective	VOC-CLAIM

Nevertheless, an adsorption/thermal desorption system will be preferable to a liquid coolant system for continuous monitoring in a short period because of its simple handling.

From the point of view of system design, the use of liquid carbon dioxide is a possibility. For the design of the rapid monitoring system, the most important point is minimization of the sample volume in such a way that the target components can be reliably detected. This is the fundamental difference between the rapid and long-term monitoring systems in terms of design requirement.

#### 4. On-site monitoring system designed for VOC-OX

The purpose of this system is the observation of change within a short time; thus rapid analysis, as soon as possible after sampling, is required. The ideal would be continuous observa-

tion. But the mean concentration based on hourly analysis will be sufficient to provide the information required for emission source monitoring, estimation of photochemical reaction or research into long-range transportation.

##### 4.1. Selection of the detector and GC separation

Flame ionization detection (FID) is the preferable because the target components range from ethane (C<sub>2</sub>) up to C<sub>13</sub> hydrocarbons, and the major components are aliphatic and aromatic. The FID response relative to the number of carbons in the molecule is easy to calibrate by relative response. The system with MS as the detector was applied to the non-methane organic gas analysis method (recording >C<sub>5</sub> hydrocarbons for the assessment of automobile exhaust gas). The disadvantage of such a system in air quality monitoring, however, is the difficulty in obtaining quantitative data for <C<sub>5</sub> hydrocarbons. It is very hard to design one single on-site

monitoring system to detect and quantify trace components such as alcohols, aldehydes, esters, ketones, etc. The GC–FID system design will differ depending on the target component range, from  $C_2$  to  $C_9$  or  $C_2$  to  $C_{13}$  analysis. An aluminium-plot column used for  $C_2$  isomers separation over room temperature has been described [27]. A well-known shortcoming of this column is the change in the retention time of unsaturated components caused by the presence of water. Sample and carrier gas have to be dried, whereby the identification of the component may be missed. Membrane-type dryers such as nafion (Perma pure dryer) will be better than the use of desiccants (e.g.,  $Na_2CO_3$ ,  $K_2CO_3$ ,  $CaCl_2$ ,  $Mg(ClO_4)_2$ ,  $P_2O_5$ , etc.), which, when used as dryers for air samples, melt on contact with the desorbed water or solve the sample into the desorbed water. It is better to use a molecular sieve or similar desiccant to dry the carrier gas. Also, some components disappeared in the column during separation (e.g., 1,1,1-trichloroethane converted to 1,1-dichloroethene and HCl). Single-column separation is very easy to operate, but separation up to  $C_{15}$  hydrocarbons is difficult without sub-ambient temperature operation. Column switching or a multi-dimensional system will be required for  $C_2$  to  $C_{13}$  analysis [28].

The practical system design proposed is as follows. (1)  $C_2$  to  $C_9$  separation: sample drying–concentration by adsorbent–thermal desorption–aluminium-plot column separation–temperature programming. (2)  $C_2$  to  $C_{13}$  separation [28]: sample drying–concentration by adsorbent–thermal desorption–methyl silicone pre-column–aluminium-plot column separation–temperature programming–column switching. These systems can also operate in long-term monitoring. The sample volume (100–500 ml) is small enough to ensure that the detection limit is below 0.1 ppb.

#### 4.2. Sample concentration

The adsorbent trap design recommended for a wide range of hydrocarbons is useful for multi-stage trap design, e.g., TENAX–activated aluminium–activated charcoal or graphitized car-

bon–molecular-sieving carbon. It will be better to dry the sample before adsorption.

### 5. On-site monitoring system designed for VOC-TOX

The purpose of this system is to observe the average exposure of mankind to harmful air components. The average concentration is important rather than change within a short time. In this context, analysis frequencies of about twice per day will be sufficient.

#### 5.1. Selection of the detector and GC separation

Much effort has been put into air quality and water quality analysis [3,11–25,29–33]. Since the target components are similar in both cases, it is possible to apply the separation and detection system used for water quality analysis direct to air quality analysis [34].

The target components are chlorinated or aromatic hydrocarbons; the recommended detector must provide selective and sensitive detection. MS is suitable for such a purpose. This has the further advantage of compatibility with multiple detection systems: PID–electron-capture detection (ECD), PID–FID (TO-14 method, U.S. EPA air quality analysis) [35], or PID–ELCD (method 502.2, US EPA water quality analysis) [36]. With the exception of a few freons, most of the target components listed in the TO-14 method are included in Method 502.2 (see Table 3). Thus, it is possible to analyse for VOC-TOX components under the same conditions as employed in purge and trap analysis if the sample volume is optimized. The advantage of ELCD compared with ECD is the relative response proportional to the number of chlorine or bromine atoms in a molecule. The sensitivity of ELCD is less than those of FID. A larger sample (>1 l) is required in order to obtain better detection. Fig. 1 shows the chromatogram of an air sample.

Analysis conditions for water quality using MS for detection can also be easily applied to air quality analysis. The breakthrough volume of the

Table 3

Volatile organic components listed according to EPA Method 502.2 for water quality and (underline) EPA TO-14 Method for air quality analysis

	Component		Component
<u>1</u>	Dichlorodifluoromethane	<u>31</u>	1,2-Dibromoethane
<u>2</u>	Chloromethane	<u>32</u>	Chlorobenzene
<u>3</u>	Vinyl chloride	33	1,1,1,2-Tetrachloroethane
4	Bromomethane	34	Ethylbenzene
<u>5</u>	Chloroethane	<u>35</u>	1,3-Xylene
<u>6</u>	Trichlorofluoromethane	<u>36</u>	1,4-Xylene
7	1,1-Dichloroethene	<u>37</u>	1,2-Xylene
<u>8</u>	Methylene chloride	<u>38</u>	Styrene
9	<i>trans</i> -1,2-Dichloroethane	39	Bromoform
<u>10</u>	1,1-Dichloroethane	40	iso-Propylbenzene
<u>11</u>	<i>cis</i> -1,2-Dichloroethane	<u>41</u>	1,1,2,2-Tetrachloroethane
12	2,2-Dichloropropane	42	1,2,3,-Trichloropropane
13	Bromochloromethane	43	Bromobenzene
<u>14</u>	Chloroform	44	<i>n</i> -Propylbenzene
<u>15</u>	1,1,1-Trichloroethane	<u>45</u>	2-Chlorotoluene
16	1,1-Dichloropropene	46	4-Chlorotoluene
<u>17</u>	Tetrachlorocarbene	<u>47</u>	1,3,5-Trimethylbenzene
<u>18</u>	1,2-Dichloroethane	48	<i>tert</i> -Butylbenzene
<u>19</u>	Benzene	49	1,2,4-Trimethylbenzene
<u>20</u>	Trichloroethene	50	sec-Butylbenzene
<u>21</u>	1,2-Dichloropropane	<u>51</u>	1,3-Dichlorobenzene
22	Dibromomethane	<u>52</u>	1,4-Dichlorobenzene
23	Bromodichloromethane	53	4-Isopropyltoluene
<u>24</u>	<i>cis</i> -1,3-Dichloropropene	<u>54</u>	1,2-Dichlorobenzene
<u>25</u>	Toluene	55	<i>n</i> -Butylbenzene
<u>26</u>	<i>trans</i> -1,3-Dichloropropene	56	1,2-Dibromo-3-chloropropane
<u>27</u>	1,1,2-Trichloroethane	<u>57</u>	1,2,4-Trichlorobenzene
28	1,3-Dichloropropane	58	Naphthalene
<u>29</u>	Tetrachloroethene	<u>59</u>	Hexachlorobutadiene
30	Dibromochloromethane	60	1,2,3-Trichlorobenzene

trap was first tested to establish the purge conditions. It proved to be sufficient for air sample collection. Also, the recovery and the capillary column introduction have been optimized. High resolution was achieved by using a narrow-bore capillary column and high-sensitivity detection was achieved by means of whole-sample introduction into the column. In this case, cryogenic focusing is recommended as a means of obtaining good separation for light hydrocarbons. This procedure will be simplified by miniaturizing the trap (optimized to capillary column) and rapid heating.

Recently, the Japanese water quality regulation method adapted its procedure of purge and trap-capillary GC-MS. This now has direct

sample introduction into the capillary column with or without cryogenic focusing.

Depending on the total sample volume and its water content, water in the air often causes plugging. This problem has been overcome by the purge and trap method. The purge gas was saturated by water vapor, and when TENAX was used as a trap adsorbent material, the trapped sample could be introduced into the capillary column. Recently, U.S. EPA Method 524.2 was extended (revision 4) to analyse 83 target components [32]. Fig. 2 shows how some oxygenated hydrocarbons in air can be analysed for VOC-TOX components under the same conditions (the identification of some of the peaks is given in Table 4).

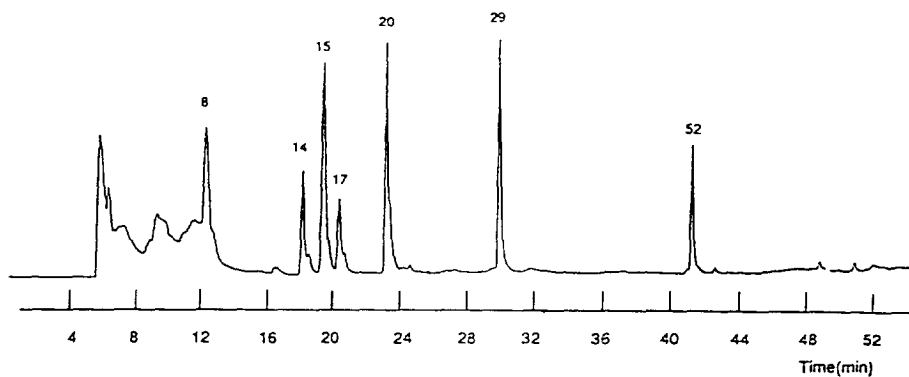


Fig. 1. Chromatogram of halogenated organic hydrocarbons in air obtained via adsorption–thermal desorption–capillary column–GC–ELCD. Analytical conditions: 1-l sample collected on TENAX GC trap at 0°C; desorption temperature 250°C, 2 Quadrex 502 columns 75 m × 0.53 mm I.D.; column temperature profile 40°C, 10 min hold, raised at 4°C/min to 230°C. Numbered peaks show components detected according to the list in Table 3. PID–ELCD dual detection system can be easily applied.

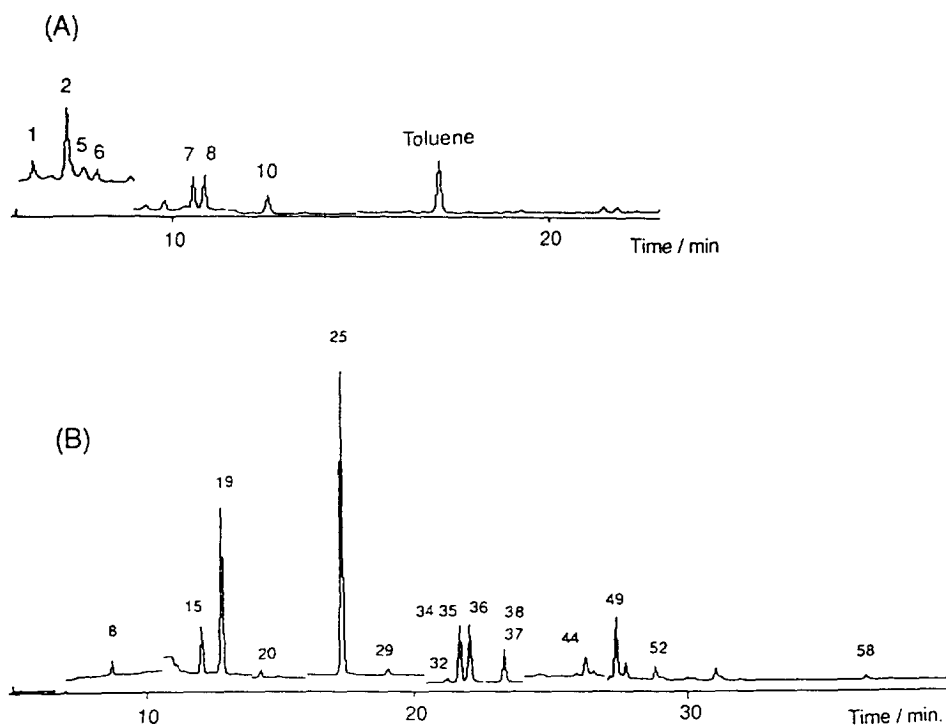


Fig. 2. Chromatogram of VOCs in air obtained via adsorption–thermal desorption–capillary column–GC–MS (SIM mode). The chromatogram is shown as a reconstructed total ion. (A) A 60-ml sample concentrated on TENAX GC at room temperature. Oxygenated components were selected. Target components are listed in Table 4. Numbered peaks show components detected in air according to the list in Table 4. (B) A 100-ml sample concentrated on TENAX GC at room temperature. Target components are listed in Table 3. Numbered peaks show components detected in air, according to the list in Table 3. Analytical conditions: desorption temperature 280°C, Halo-Matics 624 column (Quadrex) 30 × 0.25 mm I.D.; column temperature profile 40°C, 6 min hold, raised at 4°C/min to 200°C; short thick-film methylsilicone column placed in front part of analytical column as a focusing column; this part was cooled by carbon dioxide to about –50°C.

Table 4

Target components list for oxygenated hydrocarbons in air; detailed GC conditions shown in Fig. 2A

No.	Component	No.	Component
1	Methanol	7	2-Butanone
2	Ethanol	8	Ethyl acetate
3	Acetonitrile	9	2-Methoxyethanol
4	Acetone	10	1-Butanol
5	Diethyl ether	11	4-Methyl-2-pentanone
6	2-Propanol	12	Dimethylformamide

To correct the MS sensitivity, the addition of an internal standard is recommended. This technique is commonly used for water quality analysis and will apply to air quality analysis. An internal standard was fed into each sample as shown in Fig. 3. The internal standards used were 1,4-difluorobenzene, d5-chlorobenzene and bromochloromethane [37]. Fluorobenzene is used as an internal standard for water quality analysis [36]. One of the drawbacks of this system is that it is expensive.

### 5.2. Special considerations for long-term observation

It is very important to determine long-term exposure in order to estimate the hazard caused by trace organic components. Extending the analysis interval to once or twice a day will simplify the analysis and reduce the number of data from which the average concentration is obtained, but then the sample collection procedure and/or storage became very important. If

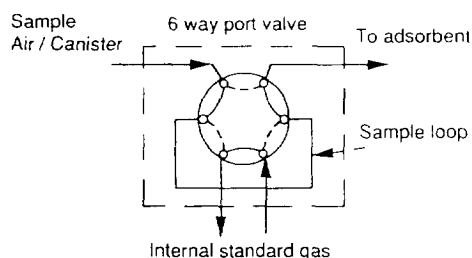


Fig. 3. Flow diagram of internal standard addition for VOCs analysis using GC-MS. During sample collection, internal standard gas was fed into the trap from the sample loop at regular intervals via a six-port injection system.

the sample intake remains in the trap for such a long time, the collected volume will be huge, excessively large for MS analysis. In such cases, some of the collected sample can be split off into the second trap, so that the sample volume is optimized [37]. The sample stored in the canister can easily be used to optimize the sample introduction volume. The flow of stored sample through the trap in each analysis will determine the optimum sample volume (canister sampling was used for field sampling under the TO-14 method). If storage is for less than one day, the artifact will be eliminated. Large amounts of sample will be required if MS is operated in the scan mode; sample volume will be much smaller if ion-trap MS is used as the GC detector [38].

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