

Monitoring and Reporting VOCs in Ambient Air

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1. Introduction

Volatile organic compounds (VOCs) are part of the large hydrocarbon family, a vast array of aliphatic, aromatic hydrocarbons, their halogenated derivatives, alcohols, ketones and aldehydes. VOCs have a property of conversion into vapour or gas without any chemical change. They are highly reactive hydrocarbons and participate in atmospheric photochemical reactions. Some of them have negligible photochemical activity; however they play an important role as heat trapping gases in atmosphere. VOC of both primary and secondary origin in ambient air have immense importance as they have direct as well as indirect effects on climate change, ecology and human health.

Many VOCs are of natural origin while many owe their existence to anthropogenic activities. Natural sources of VOCs include forests, termites, oceans, wetlands, Tundras and volcanoes. Estimated global emission rate of biogenic VOCs is 1150 Tg yr⁻¹ (Guenther et. al., 1995).

The anthropogenic sources of VOCs consist of vehicular emissions, petroleum products, chemicals, manufacturing industries, painting operations, varnishes, coating operations, consumer products, petroleum handling, auto refinishing, cold clean degreasing, printing inks, dry-cleaning etc.

In presence of oxides of nitrogen and sunlight, VOCs form ozone and other products. Oxidation of VOCs by reaction with hydroxyl radicals is the main removal process. The oxidation of complex organic molecules leads to the fragmentation, production of a range of reactive free radicals and more stable smaller molecules such as aldehydes. VOCs are cause of concern firstly due to its role in formation of ground level ozone and smog and secondly due to some of them being carcinogenic, mutagenic and teratogenic in nature. Adverse effects of ozone on human health, crop viability and yields are well documented. Wide range of VOCs, imply wide range of reaction rates, which means large range of transport distances. Many VOCs have low reactivity and thus long atmospheric life times and can be classified as Persistent Organic Pollutants (POPs). Some VOCs are Hazardous Air Pollutants (HAPs) by virtue of their toxicity.

International concerns regarding VOCs arise due to their ability of long range transport, distribution and accumulation in various components of environment, their toxic nature and significant contribution from natural sources. Ambient air monitoring of VOC is aimed to control or avoid adverse impacts on humans and ecology. This should also result in knowledge of types and category of VOCs in terms of photochemical ozone creating potential of VOCs, concentrations of VOC species, their dispersion routes and fate in

environment. However there are many issues embedded in measurement of ambient VOCs, starting from the definitions of VOCs to monitoring and reporting protocols. In this chapter we will discuss the various methods in practice for defining, sampling, analysing and reporting of VOCs. The chapter also stresses the need for a uniform method to deal with VOCs for the purpose of comparison and better understanding.

2. Definitions of VOCs

Definition of VOCs vary according to context. A very general definition is "VOCs are organic substances which are volatile and are photo chemically reactive". Below are the definitions of VOCs used by various organisation.

UNECE (United Nations Economic Commission for Europe):

All organic compounds of anthropogenic nature, other than methane, those are capable of producing photochemical oxidants by reacting with nitrogen oxides in the presence of sunlight are VOCs

WHO (World Health Organisation):

World Health Organisation has defined VOC Based on 'Boiling Point' range, "Any organic compound" will be denoted as very volatile organic compound (VVOC) if the boiling point is in the range of $< 0^{\circ}\text{C}$ to up to 50°C ; and the will be denoted as volatile organic compound (VOC) if the boiling point falls within the range from 50°C - 100°C up to $240 - 260^{\circ}\text{C}$.

ASTM (international standards developing organization):

Any compound of carbon evaporating under specific test conditions; water and exempt volatile solvents (methylene chloride, p-chlorobenzotrifluoride, acetone, volatile methyl siloxanes) are not included as VOC.

ISO 16000-6 (International Organization for Standardization):

Any organic compound in the indoor air of homes, offices and public buildings as well as organic compounds, which emit from construction materials and are detected in the test chamber"

- This definition relates to Indoor Air Quality

European Union (Directive 2001/81/EC):

"volatile organic compounds" and "VOC" mean all organic compounds arising from human activities, other than methane, which are capable of producing photochemical oxidants by reactions with nitrogen oxides in the presence of sunlight".

US EPA Definition of VOC - Volatile Organic Compounds

"Volatile organic compounds (VOC)" means any compound of carbon, excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and ammonium carbonate, which participates in atmospheric photochemical reactions

3. Estimation of VOCs

Any technique of measuring VOCs must consist of three major components namely,

- the means of sampling of air sample
- the means of extraction, and
- the means of detection of the target analytes;

3.1 Methods of sampling

The first step of monitoring of VOCs is the collection of air sample. Different methods of VOC sampling in air are practiced worldwide for this purpose (Demeestere et. al., 2007).

- *Active sampling* is done by drawing a specified volume of the air using a pump through an adsorbent tube with a constant, usually low air flow rate. In some cases sampling can be done for long term where the collection of air sample is done as average samples intermittently over a long period, or sampling at a very low air flow rate.
- *Grab sampling* is done for a very short periods of time (10-30 seconds) generally using evacuated polished stainless steel or aluminium canisters. However, evacuated canisters can also be used for time integrated sampling over minutes to days using a suitable flow restrictive inlet. The VOCs are enriched afterwards in the laboratory. The lesser sensitivity of this technique limits its use for microenvironments such as indoor air or occupational environment studies since fairly high concentrations are required.
- *Passive sampling* consists of adsorbents normally contained in a thin tube. The cross section of the tube and the distance between the opening of the tube and the adsorbent surface determines the sampling rate of the passive sampler.

Instruments with photoionization detector (PID) are often used for measuring total VOCs (TVOCs) in air directly. These are also known as combustible gas meter or explosivity meter. These instruments are not chemical-specific, they measure the total organic or flammable vapor concentrations in the air mass under consideration. The detection limits of these instruments are typically higher in the parts per million range.

There are more sophisticated methods in practice which measure chemical specific VOCs in the air sample. The primary step of the methods requires pre-concentration of the target VOCs. There are two pre-concentration methods widely used which are cryogenic trapping and adsorptive sampling either in passive or active mode.

3.1.1 VOC pre-concentrations on solid sorbents

Both Active and passive sample pre-concentration is done on solid adsorption badges and packed tubes. Enrichment of VOC onto solid sorbents, either by active or passive sampling is a well established sample preparation technique for VOC in air.

Passive sampling: In passive (diffusive) sampling, sorbent material is exposed to air for a period in the order of days. Tube type or badge type samplers are used for sorbent holding in passive sampling.

Active sampling: Active sampling occurs by pumping air through a bed of sorbent(s) in a tube, at a rate typically in the range of 10–100 mLmin⁻¹ range for a period in the order of minutes or hours. VOC enriched sorbents are typically desorbed by thermal desorption or chemical desorption. A recent work reported in Kolkata in a microenvironment namely in passenger cars used active sampling followed by chemical desorption for sample pre-concentration (Som et al., 2007).

Cryogenic sample pre-concentration: In this process an air flow is passed through a cooled tube usually filled with glass beads. Sample preparation in this method is typically done with the aid of cryogenic fluids where trapping temperatures kept between –150 to –170°C. Recent applications where this technique is combined with canister sampling are reported for analysis of indoor environments (Hsieh et al., 2006).

3.2 Method of extraction

After pre-concentration of VOCs they need to be desorbed for further analysis. There are many extraction techniques presently in practice such as chemical and thermal desorption, Solvent extraction, Solid phase micro-extraction etc.

3.2.1 Chemical desorption

Subsequent to pre-concentration of VOCs in solid sorbent, especially activated charcoal, desorption is done using some organic solvent. Carbon disulfide (CS₂) is widely used for this purpose; Methanol and acetonitrile are also used by some researchers. Typically 1-2ml of solvent is used for desorbing target VOCs from the exposed solid sorbent. It is kept for 45 - 60 min with occasional stirring for desorption.

3.2.2 Thermal desorption

A very effective method of extraction from solid sorbent is thermal desorption. In this non-destructive method the sampling tube with exposed solid sorbent is subjected to high temperature typically in the range of 200°C to 380°C depending upon the characteristics of the solid sorbent. The target VOCs are desorbed and collected for further analysis. Cryogenic focus trap is often used followed by thermal desorption of target compounds for their collection. Alternatively they can be used directly for subsequent analysis.

3.2.3 Solvent extraction

In this method dissolving airborne VOC in an appropriate liquid solvent for pre-concentration can be achieved using impingers and denuders. Derivatization reagents can also be added converting the analytes in reaction products more suitable for subsequent separation and/or detection. This method is of particular use for the analysis of carbonyl compounds in air, with 2,4-dinitrophenylhydrazine (DNPH) the most widely used derivatization agent, forming hydrazones which can be analysed via HPLC and UV-vis or fluorescence detection (Dutta et al., 2009). Alternatively, DNPH impregnated cartridges of silica gel, florisil or octadecyl silane are widely used for collecting carbonyl compounds from air (Fujita et al., 2003, Hell'en et al., 2006). After elution with acetonitrile a very low detection limits (LODs) can be achieved by HPLC-MS analysis (Hell'en et al., 2006). Although the sensitivity of these methods are mostly sufficient, they are time consuming and not easily applicable for continuous measurements. However, Motyka et al. (2006) described a novel combined wet denudation-chemiluminescence method, based on the Trautz-Schorigin reaction, allowing continuous determination of formaldehyde in air with a LOD of 0.6 µg m⁻³.

3.2.4 Solid phase micro-extraction based techniques

SPME is based on the equilibrium partitioning of target analytes between the sampled matrix and a stationary phase, coated on a fused silica fiber. Among the liquid-like and solid polymeric stationary phases available in a wide variety of polarity. As an alternative to thermal desorption, Barro et al. (2004) introduced the combined use of adsorbent cartridges and SPME for analysis of trace levels of chlorobenzenes in air. If automated, the adsorbent/HS-SPME method allows high-throughput analysis of VOC at the sub ngm⁻³ level.

3.2.5 Membrane extraction

In this method analytes are transferred from a donor to an acceptor phase through a single or multi-membrane device, where distinction can be made between non-porous and (solvent impregnated) porous membranes. Membrane extraction techniques are suitable for passive sampling over extended periods, e.g. for monitoring indoor air quality. An obstacle, however, is the necessity to calibrate each compound individually. (Zabiegala et al., 2003; 2006)

3.3 Method of analysis

A number of advanced analytical techniques are engaged for analysis of chemical specific VOCs subsequent to sampling and extraction. Gas Chromatography (GC) among them is most widely used separation technique where the target compounds are separated inside a column containing liquid stationary phase adsorbed on to the surface of a inert solid packing material by a inert gaseous mobile phase. Temperature of the column is controlled for obtaining a good separation and resolution of the analytes. The analytes are separated depending upon their various physical properties like polarity, molecular weight, structure etc. There are plenty of detectors that are used as a common practice such as Flame ionization (FID), Thermal conductivity (TCD), Electron capture (ECD), Nitrogen-phosphorus, Flame photometric (FPD), Photo-ionization (PID), Hall electrolytic conductivity. All the detectors have specific target pollutant group but the most accurate and versatile detector is the Mass detector. This detector has one specific advantage over the others that it can identify the unknown compounds as well. Other chromatographic methods are also used for some specific group of VOCs like for analysis of carbonyls. High performance liquid chromatography (HPLC) is employed followed by solvent extraction.

4. Challenges in monitoring VOCs

Measurement of VOCs in ambient air is often difficult, because of the variety of VOCs of potential concern, the variety of potential techniques for sampling and analysis, and the lack of standardized and documented methods.

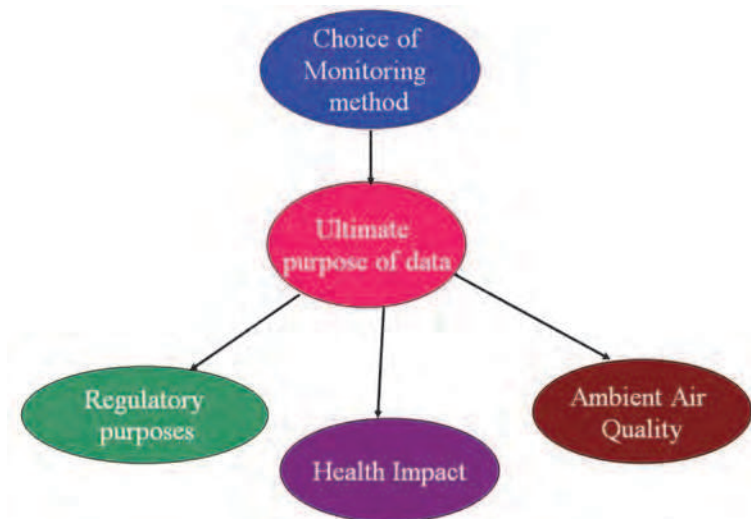


Fig. 1. Selection of monitoring method depending upon the use of data.

Volatile Organic Compounds (VOCs) present a particularly unique testing dilemma since there are a large number of different compounds defined as VOCs. The process of accurately and consistently measuring the quantity of total VOCs emitted is a concern to industry, researchers and regulatory agencies. Measurement of VOCs can be divided into two categories

- Source Emissions
- Ambient Air

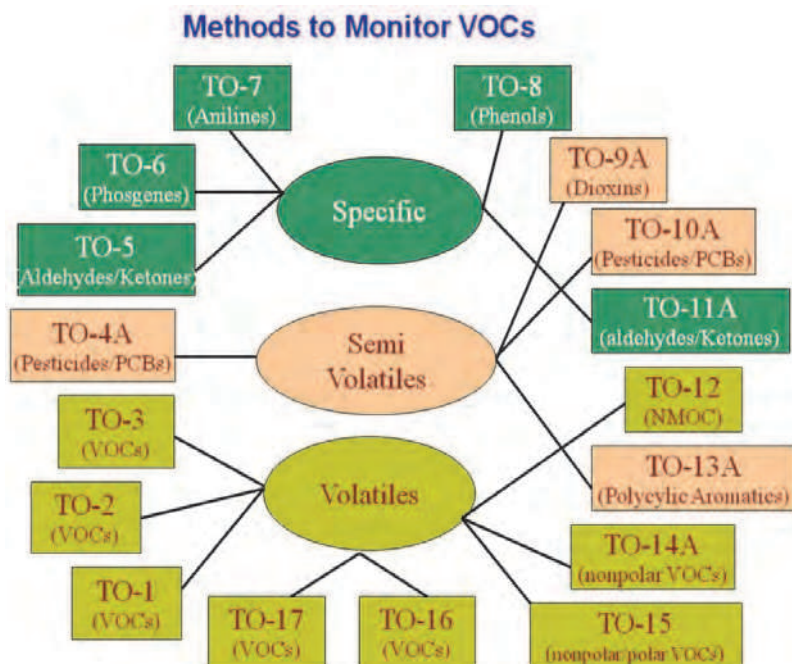


Fig. 2. Selection of monitoring method depending upon the pollutant type.

4.1 Source emission monitoring

There are three primary mechanisms for evaluating VOC emissions.

- Material Balance
- Emission Factors
- Emission Testing

All of above can be used for source emissions and reporting format depends on the compliance requirement.

USEPA has promulgated number of methods for VOC measurement which is applicable to source emission monitoring. Below is tabulated (table 1) the list of such methods along with respective application.

The correct determination of VOC emission rates is dependent on multiple factors. One factor is the ultimate purpose of the data. If testing is performed for regulatory purposes, then the regulating agency must define the pollutant and the reporting units. Usually regulating agencies require knowledge of total VOC emission, control efficiency of control equipments and concentration limits. Commonly used methods are M18, M25 and M25A. These methods suffer from limitations of detection technology and in all the methods accept M18 TVOC is reported as "Carbon" or "Methane". M18 uses FID & MS as a detector. M18 is excellent for speciating individual compounds, testing for Toxic Air Pollutants (TAPs) and Hazardous Air Pollutants (HAPs), for mass emission tests when there are only a few VOCs in the gas

Method number	Title/application
USEPA M18	VOCs by GC analysis, sampling and on-line systems
USEPA M21	VOC leaks (fugitive emissions).
USEPA M25a	Gaseous VOC concentration by Flame Ionisation Detector
USEPA M25b	Gaseous VOC concentration by infrared analyser
USEPA M25c	Non-methane organic carbon in landfill gases
USEPA M25d	VOC of waste samples
USEPA M25e	Vapour Phase organic concentration in waste samples
OSW M0010	Semi VOCs by GC after sampling with a modified Method 5 (MM5) train
OSW M0011	Aldehydes by sampling with a MM5 train and 2,4-DNPH. Analysis by GC-M5 or HPLCUV
OSW M0030.	Sampling with a 2-tube VOST and analysis by GC. For medium volatility VOCs
OSW M0031	Sampling with a 3-tube VOST, and analysis by GC. For medium to very high volatility organic compounds.
OSW M0040	Bag sampling for high volatility, high concentration compounds.
USEPA M106	Determination of VCM (Vinyl Chloride Monomer)
USEPA M107	VCM of in-process waste water samples
USEPA M107a	VCM of solvents
USEPA M204A	VOCs in liquid input stream
USEPA M204B	VOCs in captured stream
USEPA M204C	VOCs in captured stream (dilution technique)
USEPA M204D	Fugitive VOCs from temporary total enclosure
USEPA M204E	Fugitive VOCs from building enclosure
USEPA M204F	VOCs in liquid input stream (distillation)
USEPA M305	Potential VOC in waste
USEPA M307	Emissions from solvent vapour emissions
USEPA M308	Sampling for methanol with a heated system. Analysis by GC-FID
USEPA M318	Extractive FTIR for measuring emissions from the mineral wool and wood Fibreglass industry.
USEPA M310A	Residual hexane
USEPA M310B	Residual solvent
USEPA M3201C	Residual n-hexane in EDPM rubber
USEPA M312A	Styrene in SBR latex (GC)
USEPA M312B	Styrene in SBR latex by capillary GC
USEPA M312C	Styrene in SBR latex produced by emulsion polymerisation
USEPA M320	Vapour-phase, organic and inorganic emissions by extractive FTIR

Table 1. Key promulgated USEPA methods for VOC monitoring

stream and for characterising a gas stream. Method 18 utilizes gas chromatography to separate the VOC compounds from each other and from other interferences in the gaseous stream. The detector used in this method is specifically calibrated for each VOC compound present using known standards to develop response factors and linear operating ranges for the method. This method is capable of providing true results in terms of individual VOC components which when totalled provide a total VOC concentration. The main advantage of this method is that results are reported "as VOC".

Method 25 measures TGNMO (total gaseous non-methane organic) by first separating the VOC components from methane, carbon monoxide and carbon dioxide. The remaining VOC compounds are chemically converted to methane molecules, which are quantitatively measured by a FID (flame ionization detector). This method provides a measurement of the VOC composition in terms of its carbon content. This method normalizes the response factor for individual VOC components since the carbon in each component is converted to methane before performing the quantification. This method has detection limit of about 50 ppm carbon cannot be used on many outlets where the concentration is often considerably less than that. This method reports VOC "as Carbon".

Method 25A is an instrumental method in which the VOC is introduced into a FID chamber without first separating the VOC components. The FID is calibrated with a standard gas such as methane or propane and the method results are often reported in terms of the calibration gas used (e.g., "as propane"). The main problem with this method is the variation of the FID response to VOC components other than hydrocarbon compounds. VOC compounds containing oxygen or halogen atoms may differ as much as two-fold in FID response from similar hydrocarbon compounds. This method is sensitive to low concentrations, relatively easy to use, low cost and may be converted to "as VOC" results for simple gas streams where the composition is known.

4.2 Ambient air monitoring

Measurement of VOCs in ambient air is often difficult, because of the variety of VOCs of potential concern, the variety of potential techniques for sampling and analysis, and the lack of standardized and documented methods. Measuring TVOC concentrations does not provide reliable indication of potential health impacts of air pollution. In case of ambient air monitoring of VOC is aimed to control /avoid adverse impacts on humans and ecology must result in knowledge of

- Types of VOCs
- Concentrations of VOCs
- Their dispersion routes
- Their fate in environment
- Category of VOCs in terms of photochemical ozone creating potential
- VOCs in terms of their health end point

VOCs that are of importance due to their toxicity are listed in Table 2.

Very little guidance is available for the determination of toxic organic compounds in ambient air. As a result, while monitoring VOCs as air pollutant one is required to develop his own monitoring strategies, including selection of monitoring methods, sampling plan design, and specific procedures for sampling, analysis, logistics, calibration and quality control and averaging time. The choice of monitoring method for ambient measurement of VOCs depends on the ultimate purpose of the data such as:

- regulatory purpose
- Health impact
- Impact on Climate change
- Impact on ecology

Compound	Category
acrylamide	cat.2 carcinogen and mutagen
acrylonitrile	cat.2 carcinogen
azinphos-methyl	very toxic
benzene	cat.1 carcinogen
benzo(a)anthracene	cat.2 carcinogen
benzo(b)fluoranthene	cat.2 carcinogen
benzo(k)fluoranthene	cat.2 carcinogen
benzo(j)fluoranthene	cat.2 carcinogen
benzo(a)pyrene	cat.2 carcinogen, mutagen and teratogen
butadiene	cat.2 carcinogen
carbon disulphide	cat.2 teratogen
1-chloro-2,3-epoxypropane	cat.2 carcinogen
chloroethene (vinyl chloride)	cat.1 carcinogen
dibenzo(a,h)anthracene	cat.2 carcinogen
1,2-dichlorethane	cat.2 carcinogen
dichlorvos	very toxic
dieldrin	very toxic
diethyl sulphate	cat.2 carcinogen and mutagen
dimethyl sulphate	cat.2 carcinogen
dnoseb	cat.2 teratogen
endosulfan	very toxic
endrin	very toxic
1,2-epoxypropane	cat.2 carcinogen
2-ethoxyethanol	cat.2 teratogen
2-ethoxyethyl acetate	cat.2 teratogen
4,4'-methylenebis(2-chloroaniline)	cat.2 carcinogen
4,4'-methylenediphenyl diisocyanate	very toxic (inhalation)
nitrobenzene	very toxic
2-nitropropane	cat.2 carcinogen
phenol	very toxic (inhalation)
phorate	very toxic
phosgene	very toxic
polychlorinated biphenyls	IARC group 2A
2-propen-1-ol	very toxic (inhalation)
pyrene	very toxic
1,1,2,2-tetrachloroethane	very toxic

Table 2. List of VOCs Identified to be of Importance due to their Toxicity

USEPA has compiled compendium of methods to monitor VOCs and toxics in ambient air (Table 3). It gives description of technical aspects of the available methods. However none of these methods recommend any averaging period. Literature shows that concentration of VOCs have been reported as ppmv/ ppbv and also microgram per meter cube for samples collected as grab sample or for time periods varying from few seconds to hours.

Compendium Method No	Type of Compounds	Determined Sample Collection Device	Analytical Methodology
TO-12	Volatile organic compounds	Tenax® solid sorbent	GC/MS
TO-22	Volatile organic compounds	Molecular sieve sorbent	GC/MS
TO-32	Volatile organic compounds	Cryotrap	GC/FID
TO-4A	Pesticides/PCBs	Polyurethane foam	GC/MD
TO-52	Aldehydes/Ketones	Impinger	HPLC
TO-62	Phosgene	Impinger	HPLC
TO-72	Anilines	Adsorbent	GC/MS
TO-82	Phenols	Impinger	HPLC
TO-9A	Dioxins	Polyurethane foam	HRGC/HRMS
TO-10A	Pesticides/PCBs	Polyurethane foam	GC/MD
TO-11A	Aldehydes/ketones	Adsorbent	HPLC
TO-122	NMOC	Canister or on-line	FID
TO-13A	Polycyclic aromatic	Polyurethane foam	GC/MS
TO-14A	Volatile organic compounds (nonpolar)	Specially-treated canister	GC/MS and GC/MD
TO-15	Volatile organic compounds (polar/nonpolar)	Specially-treated canister	GC/MS
TO-16	Volatile organic compounds	Open path monitoring	FTIR
TO-17	Volatile organic compounds	Single/multi-bed adsorbent	GC/MS, FID, etc.

Table 3. List of USEPA Compendium of Methods for VOC / Toxic Air Pollutant Monitoring

5. Issues in reporting of VOCs

Regulations in the World as a common practice are based on limits for mass emissions of total VOC (TVOC) during a year. Most of the inconsistencies are there in reporting the TVOCs. TVOCs concentrations are reported

- as Total Organic Carbon (TOC), which is the concentration of carbon in the gas stream, and usually expressed in milligrams per cubic metre (mg m^{-3});
- as TOC expressed in terms of the equivalent concentration of a specified VOC, such as propane/Toulene, and;
- as the sum of the concentrations of specific, individual VOCs in a sample.

The Total VOC is often referred as total hydrocarbon (THC) or total organic carbon (TOC). But when THC is measured as carbon or as propane then THC cannot be used to represent Total VOC. In real situation gas stream is complex and consist of unknown compounds of variable properties; THCs are reported either as carbon or as propane. When an average MW is not available, the data is often reported on a mass basis 'as carbon' or in terms of

another surrogate. To convert 'as propane' to 'as carbon' the results must be multiplied by 3 as there are 3 carbon atoms in propane

This procedure involves two errors: response factors and total VOC molecular weight.

$\text{gms/hr (as Carbon)} = K * \text{ppm} * 12.01 * \text{Flow rate}$; where K is emission factor

$\text{gms/hr (as VOC)} = K * \text{ppm} * \text{Flow rate} * (\text{MW VOC} / \text{no. C})$

e.g., Benzene is 92% carbon by weight

Ethanol is 52% carbon by weight

$\text{VOC (g/hr)} = \text{THC(g/hr)} / (\text{MW of C in VOC}) / (\text{MW of VOC})$

For Benzene = $\text{THC (g/hr)} / (72 / 78) = \text{THC} / 0.92$

For a mixture of gas stream benzene 25%, ethanol 50% and toluene 25%, $\text{VOC (g/hr)} = \text{THC (g/hr)} / [(0.25) (72/78) + 0.5 (24/46) + 0.25 (84/91)]$

= $\text{THC} / 0.72$

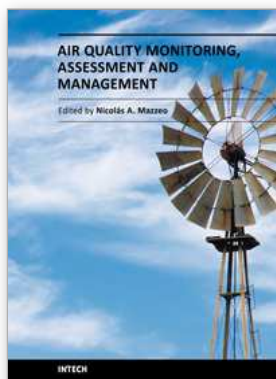
Similarly this issue is present in measurement of ambient concentrations as well.

Thus there is need for development of uniformity in ambient VOC measurement including specific standards, at least for HAPs. Uniform reporting format e.g. TVOC as ppmv & individual VOC as $\mu\text{g}/\text{m}^3$ is also required. Averaging period and uniform detection methodology is to be standardized. Moreover VOC is not to be reported as THC and NMHC.

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Human beings need to breathe oxygen diluted in certain quantity of inert gas for living. In the atmosphere, there is a gas mixture of, mainly, oxygen and nitrogen, in appropriate proportions. However, the air also contains other gases, vapours and aerosols that humans incorporate when breathing and whose composition and concentration vary spatially. Some of these are physiologically inert. Air pollution has become a problem of major concern in the last few decades as it has caused negative effects on human health, nature and properties. This book presents the results of research studies carried out by international researchers in seventeen chapters which can be grouped into two main sections: a) air quality monitoring and b) air quality assessment and management, and serves as a source of material for all those involved in the field, whether as a student, scientific researcher, industrialist, consultant, or government agency with responsibility in this area.

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