Investigation of the Emission of Monocyclic Aromatic Hydrocarbons from a Wastewater Treatment Plant at Lausanne (Switzerland) by Differential Optical Absorption Spectroscopy (DOAS)

Rodrigo Jiménez, Tiziana Iannone, Hubert van den Bergh and Bertrand Calpini
Air Pollution Laboratory (LPAS)
Swiss Federal Institute of Technology (EPFL)
CH-1015 Lausanne, Switzerland
rodrigo.jimenez@epfl.ch

Dieter Kita
Thermo Environmental Instruments Inc.
Franklin, MA 02038, USA

ABSTRACT

Recent research works show that wastewater treatment plants may significantly contribute to the total emission of volatile organic compounds (VOC) in urban areas. In order to investigate this contribution, a measurement campaign was carried out during September 1999 in the Lausanne area. The concentrations of monocyclic aromatic hydrocarbons (MAH) over a wastewater treatment plant at Lausanne were monitored by differential optical absorption spectroscopy (DOAS) and gas chromatography (GC-FID). A flame ionization detector (FID) was used for monitoring the overall concentration of non-methane hydrocarbons (NMHC). The local meteorological conditions were also measured. The concentrations of benzene, toluene, ethylbenzene, xylenes, 1,2,4-trimethylbenzene and phenol over the wastewater treatment plant as measured by DOAS and GC-FID are compared. In addition, based upon the results of a recent field experiment, the impact of the oxygen (O_2) interference on DOAS measurements of aromatic compounds is addressed. The MAH concentration time series and its relation to that one of NMHC, as well as to the meteorological conditions, are presented. The VOC release and production phenomena involved in the wastewater and sludge treatment processes are analyzed using complementary measurements of aromatic compounds in wastewater and gas processing streams.

Keywords: air pollution, wastewater, sludge, incineration, emission, non-methane hydrocarbons (NMHC), monocyclic aromatic hydrocarbons (MAH), differential optical absorption spectroscopy (DOAS), gas chromatography (GC), flame ionization detector (FID)

INTRODUCTION

Photochemical pollution is a widespread air quality problem of major concern nowadays. It is caused by the photo-oxidation of volatile organic compounds (VOC) in the atmosphere. Wastewater and solid waste treatment operations have been identified as significant sources of VOC [1-2]. For instance, it is estimated that 6% of the total VOC emitted in the Chicago area comes from its wastewater treatment plants [2].

Monocyclic aromatic hydrocarbons (MAH) are an important fraction of VOC. They account for about 30% (w/w) of all non-methane hydrocarbons (NMHC) in urban air [3] and usually are the second most important VOC fraction emitted by waste treatment operations [1]. In addition, MAH are toxic or even carcinogenic as benzene, and likely precursors of secondary aerosols [3].

This study is the result of a short measurement campaign aimed at investigating the emission of VOC, and particularly of monocyclic aromatic hydrocarbons (MAH), from a wastewater and sludge treatment facility at Lausanne, Switzerland (Vidy STEP). Despite some measurements were made in wastewater and process gas streams, this investigation was focused on fugitive emissions of MAH from sludge treatment operations. Path averaged measurements of MAH obtained by differential optical absorption spectroscopy (DOAS) were instrumental in this purpose.

The spectral interference of oxygen is the main problem for DOAS measurements of MAH [3]. In order to overcome this difficulty, an alternative approach was devised and applied previous to this investigation for obtaining a set of instrument-specific oxygen reference spectra from plain open-air spectra.

DOAS measurements coupled to meteorological measurements indicate that fugitive MAH stratify vertically in the sludge treatment area. Ambient air samples analyzed by gas chromatography with flame ionization detection (GC-FID) show that a horizontal gradient exists as well. Possible sources of fugitive emissions of VOC, particularly of MAH as detected by DOAS, are the volatilization of VOC already contained in sludge during one or several handling operations previous to incineration, and unintended pyrolysis [4] during the sludge incineration process (on site produced VOC source).

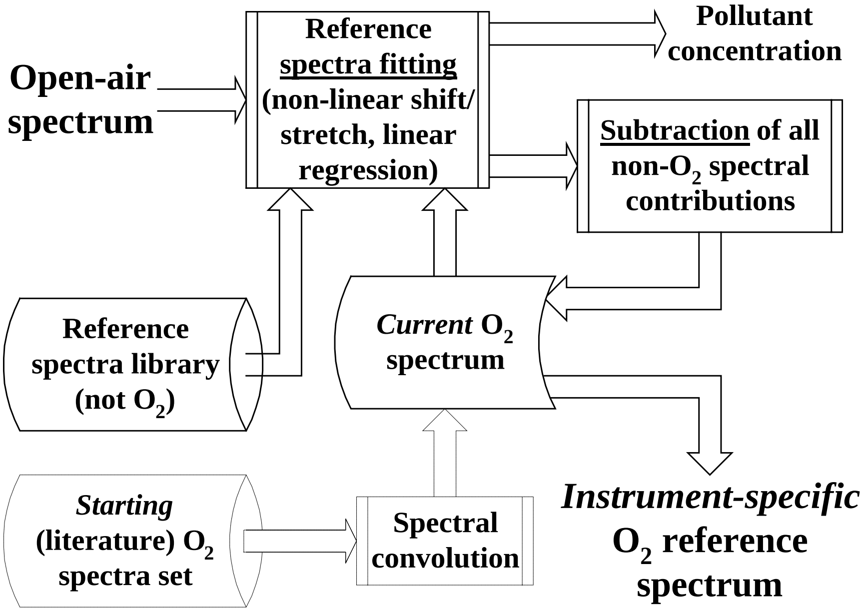
At the average wastewater flow rate of Vidy STEP, the maximum volatilization rate of MAH contained in wastewater is estimated in about 500 Kg per day.

Further investigation will be required to quantify and elucidate the fugitive MAH release phenomena.

MEASUREMENT OF MONOCYCLIC AROMATIC HYDROCARBONS BY DOAS

The differential optical absorption spectroscopy (DOAS) technique has been successfully used for the detection of diverse atmospheric trace gases since the late 70s [5]. A comprehensive description of this technique can be found elsewhere [5]. On the other hand, its application for the detection of monocyclic aromatic hydrocarbons (MAH) has not been without problems [6-11], despite the fact that most MAH exhibit relatively sharp absorption bands in the 240-300 nm wavelength region [7,12]. Besides the simultaneous presence several MAH in the air volume observed, the major difficulty for DOAS measurements of MAH is the spectral interference of oxygen [3]. At this wavelength range, oxygen displays a very complex spectral structure due to the overlapping of 4 band systems with extremely narrow (some picometers) absorption transitions and the spectral interference of the O_2-O_2 and O_2-N_2 systems [3]. While a very high spectral resolution would be required to resolve these bands, the spectral resolution of a typical DOAS system is some angstroms. At this resolution, the oxygen bands overlap, leading to an apparent deviation of the Beer-Lambert-Bouger (BLB) law. The overall result is that at low resolution, oxygen spectra become a non-linear function of concentration, optical path and pressure. In these conditions, a different oxygen reference spectrum would be theoretically required for each location and path length. In practice, a suitable set of *instrument-specific* oxygen reference spectra is required instead on a single oxygen absorption cross section, as it is case for any other light-absorbing compound.

Figure 1. Procedure used for the recovery of instrument-specific oxygen reference spectra from open-air spectra.



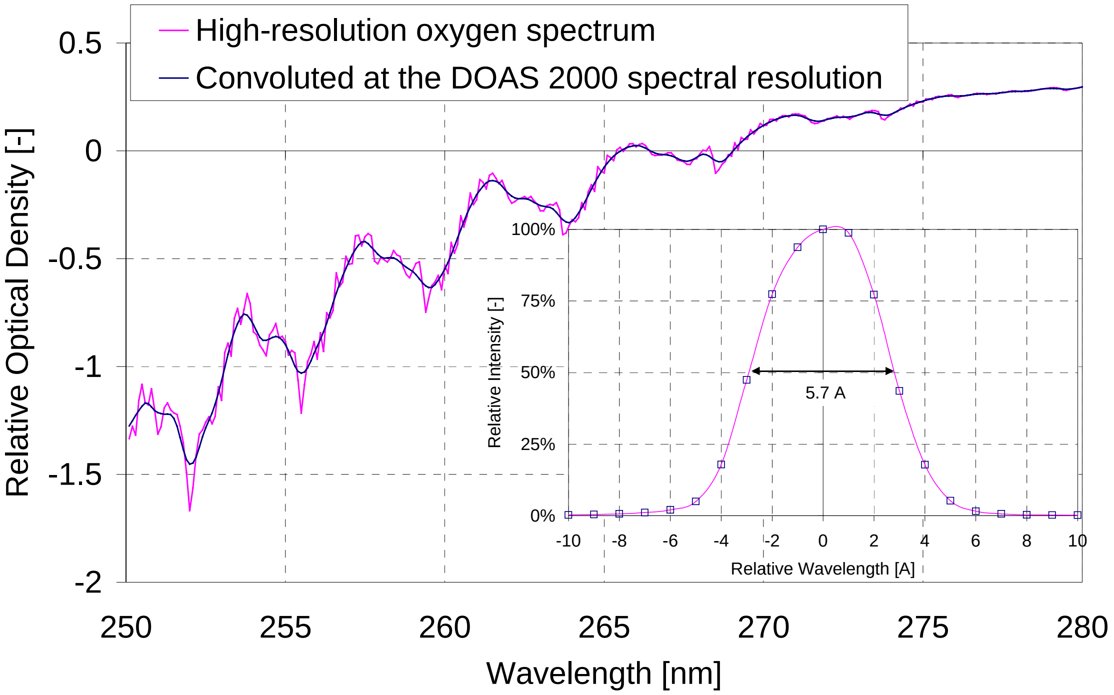
In addition, the spectral accuracy and resolution of a DOAS spectrometer must be high enough to allow resolving between the very resembling spectral features of similarly substituted MAH (mono-, di-, tri-substituted), such as those between toluene and ethylbenzene, and among the xylene isomers [7,12].

In order to provide a solution for the oxygen interference problem, we developed and tested an alternative procedure for the recovery of instrument-specific oxygen reference spectra from plain open-air spectra (see Figure 1). This recovery procedure is summarized as follows:

- Open-air spectra in the wavelength region 240-280 nm were recorded at different path lengths from 120 to 1100 m during a long integration time (30 min) in order to assure a high signal to noise ratio. As explained below, the fact the air masses observed were relatively polluted simplified instead of making more difficult the oxygen spectra recovery process.
- A set of oxygen spectra at relatively high resolution (0.15 nm FWHM), obtained in laboratory conditions by Volkamer, Etzkorn, Geyer and Platt [3], was used as *starting* reference spectra for the retrieval of instrument-specific oxygen reference spectra from plain open-air spectra. For this purpose, Volkamer's oxygen spectra were previously convoluted to the spectral resolution of the DOAS instrument used for the field measurements herein reported (DOAS 2000®, Thermo Environmental Instruments Inc.). Figure 2 shows the instrumental function of the DOAS 2000® spectrometer (0.57 nm FWHM) together with a high-resolution oxygen spectrum and the resulting convoluted spectrum.
- The spectral contributions of ozone, sulfur dioxide and a suitable set of MAH (benzene, toluene, ethylbenzene, o-, m-, p-xylene, 1,2,4-trimethylbenzene and phenol) were fitted to and subtracted from each open-air spectrum. Volkamer's spectra were used as dummy wavelength functions in the recovery process; i.e. a best-matching Volkamer's spectrum was found for each open-air spectrum from which an instrument-specific oxygen spectrum was recovered.
- In all cases, the ozone concentrations calculated by the recovery method were in close agreement with field measurements. The calculated concentrations of MAH were as well within the expected concentration range in the measurement location (Grenoble, France). A subset of the recovered oxygen spectra is shown in Figure 3.
- Each new instrument-specific oxygen spectrum was tested as fitting reference for the same open-air spectrum from which it was recovered (second iteration). For every recovered oxygen spectra, the pollutant concentrations retrieved in the second-iteration were in close agreement to those originally obtained when using Volkamer's spectra. This shows that the recovery process converged, thus no additional iterations were carried out.

The process of increasing overlap of the narrower oxygen absorption bands due to the oxygen column density increase is clearly seen in the spectra shown on Figure 3. As explained above, this process is apparent and results of observing extremely sharp absorption transitions at low spectral resolution. The practical consequence is that, while the broader transitions are common among spectra at different oxygen column densities, the narrower transitions seem more difficult to predict. Moreover, since this dependence cannot be explained in simple terms, in principle a required oxygen spectrum at a given path length could not be calculated from other oxygen reference spectra by any "interpolation" procedure. In despite of this complexity, such an interpolation procedure was developed and applied (see description below).

Figure 2. Spectra of oxygen ($1.785 \cdot 10^{24}$ molecules/cm^2 column density) as measured [3] at 0.15 nm FWHM spectral resolution ("high resolution") and convoluted to spectral resolution of DOAS 2000® (0.57 nm FWHM). The instrumental function of DOAS 2000 is shown on the right side.



In addition, it was found that at low spectral resolution, even the broader oxygen transitions do not follow the BLB due to the apparent dependence of the oxygen cross section on the optical path length. This dependence is expressed in the following equation.

Equation 1. Differential BLB law applied to oxygen measured at low spectral resolution (non-linear dependence).

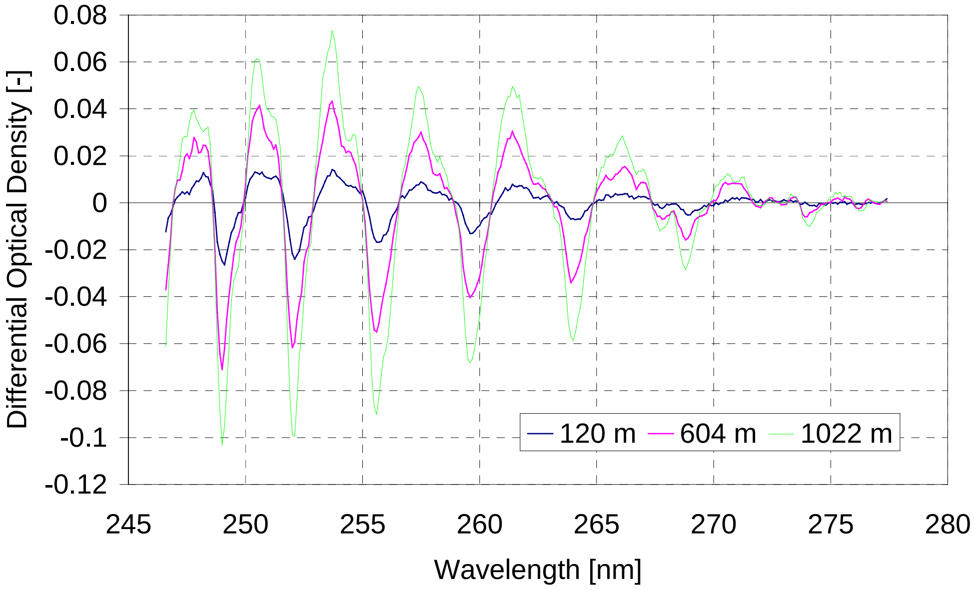
$$D'_{\mathrm{oxygen}}(\lambda, c_{\mathrm{oxygen}}, L) = \sigma'_{\mathrm{oxygen}}(\lambda, c_{\mathrm{oxygen}}, L) \cdot c_{\mathrm{oxygen}} \cdot L$$

where:

λ = wavelength
c = concentration

L = optical path length
σ’ = differential absorption cross section
D’= differential optical density

Figure 3. Instrument-specific oxygen reference spectra recovered from plain open-air spectra recorded at Grenoble, France.



Nevertheless, it was found that using an arbitrarily chosen differential oxygen spectrum as “reference” (norm) spectrum, the dependence of the broader oxygen transitions on the optical path length is mathematically described by the equation below.

Equation 2. Dimensionless (non-linear) BLB equation applied for oxygen measured at low spectral resolution.

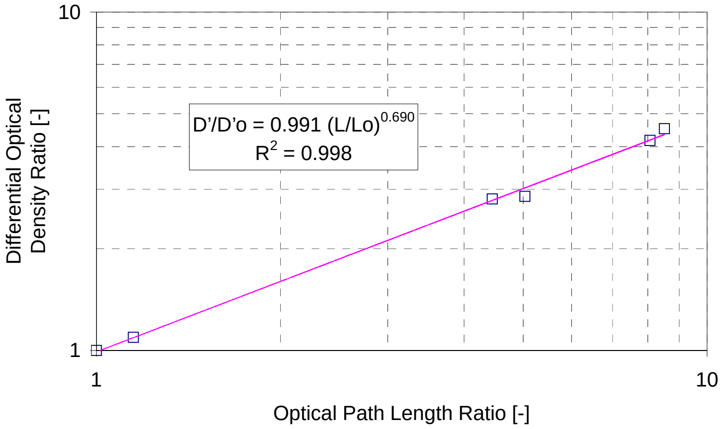
$$\frac{D'_{\text{oxygen}}(\lambda, c_{\text{oxygen}}, L)}{D'_{\text{oxygen}}(\lambda, c_{\text{oxygen}}, L_0)} = \left[\frac{\sigma'_{\text{oxygen}}(\lambda, c_{\text{oxygen}}, L)}{\sigma'_{\text{oxygen}}(\lambda, c_{\text{oxygen}}, L_0)}\right] \cdot \left(\frac{L}{L_0}\right) \approx \beta \cdot \left(\frac{L}{L_0}\right)^{\gamma}$$

where:

β, γ = non-linear BLB equation fitting coefficients
L_0 = optical path length of the “reference” (norm) oxygen spectrum

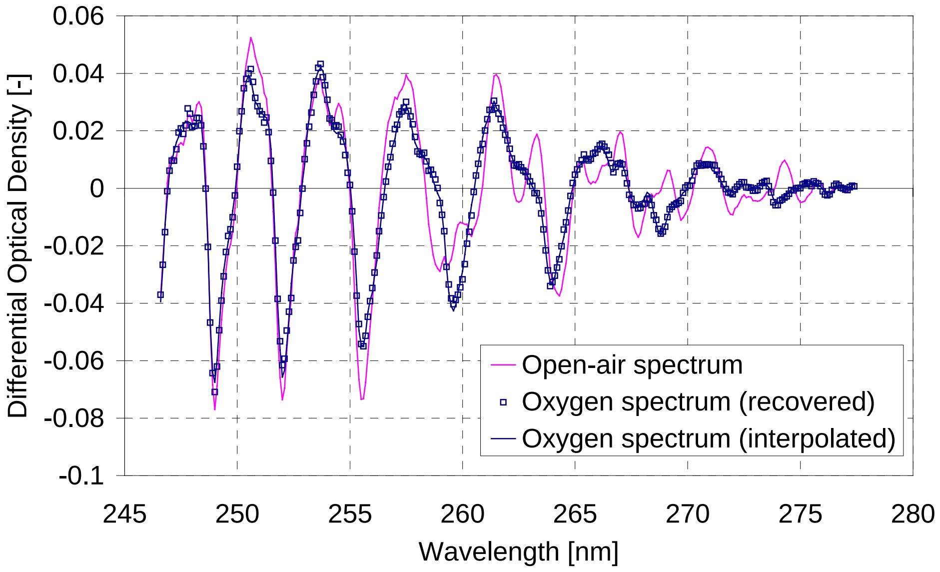
A non-linear regression fitting applied to the recovered instrument-specific oxygen spectra data pairs, (L/L_0, D'/D_0'), gives $\beta \approx 1$ and $\gamma = 0.69$ (see Figure 4). Very similar β and γ coefficients were found for the higher resolution oxygen spectra provided by Volkamer [17].

Figure 4. Non-linear BLB correlation applied to the recovered oxygen spectra.



This finding provided the basis for the development of a fairly successful procedure for oxygen reference spectra interpolation. Figure 5 shows an oxygen spectrum at 534 m optical path length (dots) and the one calculated (line) by interpolation using its two “neighbor” spectra (closest shorter and longer path lengths) as orthogonal basis and their weighting coefficients obtained by multiple linear regression forced to zero. The same procedure was used for approximating an oxygen reference spectrum suitable for the optical path length at Vidy STEP (220 m). It was found that the concentration retrieval was better with this interpolated oxygen spectrum than with any other individual oxygen spectrum available in the new oxygen spectra database.

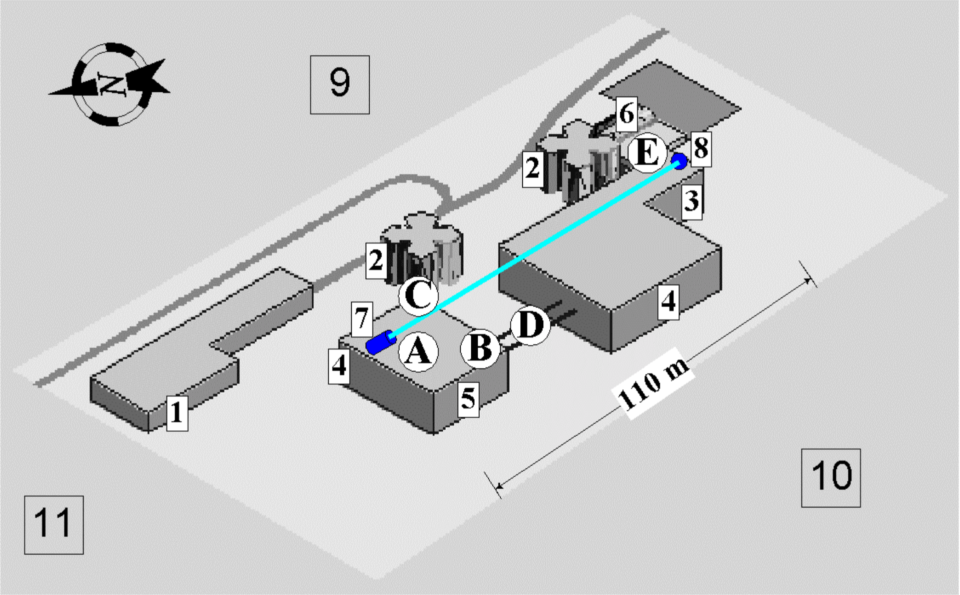
Figure 5. Open-air spectrum (534 m path length) and the oxygen spectrum from it recovered. Interpolated spectrum is shown as well. Note that the oxygen transitions account for a large part of the original spectrum.



EMISSION OF VOLATILE ORGANIC COMPOUNDS FROM A WASTEWATER AND SOLID WASTE TREATMENT PLANT AT LAUSANNE, SWITZERLAND

Concerned by the impact of photochemical pollution in Switzerland, the Swiss Federal Council issued in 1997 a law (OCOV – 814.018) aimed at reducing within 10 years the VOC emission down to the level of 1960, i.e. a 55% equivalent reduction with respect to the highest annual emission level (324,000 tons in 1984) [13]. This law defines VOC as all organic compounds whose normal boiling point is below 240 °C. OCOV – 814.018 establishes a tax of 3 SFr per Kg of VOC emitted (~ 2 US$/Kg), enforceable to all manufacturing processes involving VOC. This new law does not clearly specifies whether public and private wastewater and solid waste treatment facilities are targeted too [13], despite of recent research work showing that these facilities are potential contributors to the emission and ambient concentration of VOC [1-2].

Figure 6. Scale representation of the sludge handling area at Vidy STEP. Key: [1] Screens, grit chambers; [2] Sludge thickeners; [3] Sludge stockage; [4] Sludge dewatering and incineration; [5] Incineration flue gas cleaning; [6] Biological filter for VOC and odor control; [7] DOAS emitter/receiver (coaxial telescope); [8] DOAS retroreflector; [9] 6-lane highway; [10] Geneva lake shoreline t 200 m; [11] Wastewater treatment area; (A) - (E) GC-FID sampling sites; (B) FID sampling site



In order to investigate the VOC emission from such a facility, an exploratory campaign called STEP was carried out in a wastewater treatment plant at Lausanne, Switzerland (Vidy location). The facility (Vidy STEP) is located at 200 m of the Geneva Lake shoreline and adjacent to a 6-lane highway (see Figure 6). Vidy STEP uses a conventional activated sludge process with fine-bubble diffused aeration for treating the wastewater produced by an equivalent population of 220,000 inhabitants. The facility includes as well process units for the thickening, dewatering (conventional filter presses) and incineration (2 fluidized-bed incinerators) of the sludge produced on site and in neighbor wastewater facilities (57% w/w of the total sludge cake incinerated). Vidy STEP includes as well an incineration flue gas cleaning system (1 cyclone separator and 2 dry electrostatic precipitators for particulate matter removal followed by wet scrubbing with lime and caustic soda solutions) and a biological filtering system for VOC and odor control. A more detailed description of Vidy STEP can be found elsewhere [13].

Table 1 summarizes the analytical instruments used during STEP for the measurement of VOC concentrations in ambient air and wastewater and gas processing streams within the period August 5 - September 10, 1999 (not all measurements performed simultaneously). The FID and GC-FID measurement sites are shown on Figure 6. Additional meteorological data (temperature, pressure, horizontal wind speed and vertical wind speed) from 2 meteorological stations located in the Lausanne area (Chauderon and Plan de Pierre) were provided by SEHL (Lausanne's Environmental Authority).

Table 1. Summary of analytical techniques and measurements used and carried out during STEP. DOAS, GC-FID and FID measurement sites are shown on Figure 6. All dates refer to 1999.

Technique	Apparatus	Phase / Site	Period	Comments
DOAS	DOAS 2000®, Thermo Environmental Instruments Inc.	Ambient air (optical path)	03-10.09	220m optical path length, 15 min integration time
GC-FID	ETA 8521®, DANI 86.10	Ambient air (Sites A to E); Raw incineration flue gas; Biofilter (inlet, outlet)	07-09.09 (ambient air); 23.08 –02.09 (gas processing streams)	Routine calibration tank BTEX mixture (1-3 ppm ± 5%), Scott Specialty Gases
FID	FID 3-100 ®, JUM	Ambient air (Site B)	06-10.09	Calibration tank: Propane (85 ppm ± 2%), SL Gas
SPME-GC-FID	HP 5890 HP 5971-A	Wastewater (inlet, outlet)	02-08.08	

Volatilization, whether by free diffusion from stagnant wastewater or forced by aeration (stripping) during the wastewater treatment process, was expected to be an important source of fugitive emissions of MAH. In order to assess this emission area source, MAH contained in wastewater at the inlet (only liquid phase) and outlet of the wastewater treatment process were sampled by solid phase micro-extraction (SPME) and analyzed by gas chromatography with mass spectroscopy (GC-MS) (see Table 2). These measurements confirmed the hypothesis: depending on the efficiency of the volatilization process, up to 500 Kg/day of MAH could be emitted from the activated sludge area.

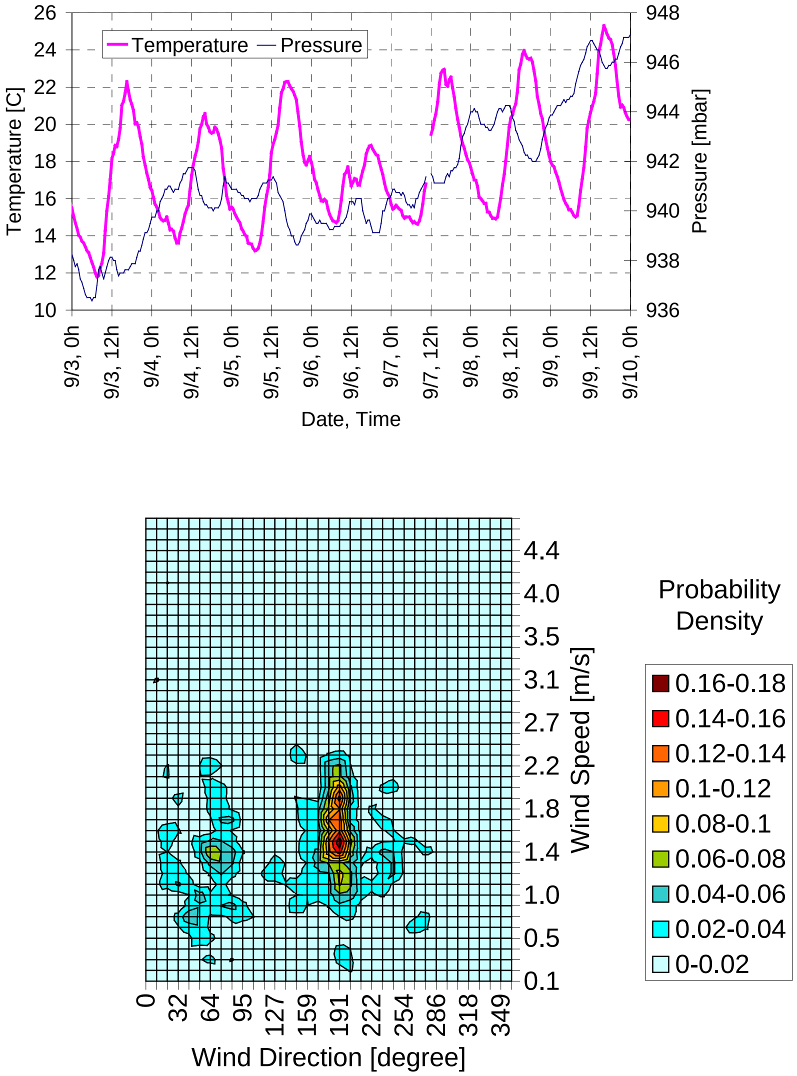
Table 2. Inlet and outlet concentrations of MAH in wastewater at Vidy STEP as measured by SPME-GC-MS. Daily SPME samples (3 duplicates) were taken at 10:00-16:00 during the period August 2-8, 1999. Vidy STEP treated 94,600(±18%) m^3/day of wastewater during this period. All average concentrations are within 10% standard deviation.

MAH	**Average Concentration [ppmv]**		**Calculated Maximum Volatilization Rate [Kg/day]**
	Inlet	**Outlet**	
Toluene	1.8	1.5	147.55
Ethylbenzene	0.2	0.0	16.40
Propylbenzene	0.5	0.0	40.75
o-Xylene	0.6	0.5	49.94
m-, p-Xylene	1.0	0.6	81.57
2-Ethyltoluene	0.3	0.0	24.98
1,2-Diethylbenzene	0.0	0.0	0.00
1,4-Diethylbenzene	0.2	0.1	16.30
1,2,4-Trimethylbenzene	1.1	0.2	91.10
1,3,5-Trimethylbenzene	0.8	0.0	65.45
Styrene	0.0	0.0	0.00
Total	**6.5**	**2.9**	**534.04**

The DOAS system operated at Vidy STEP is a monostatic commercial system described in detail elsewhere [14]. The light emission and collection side (coaxial telescope) was placed on the roof of one of buildings where sludge is dewatered and incinerated (see Figure 5). The retroreflector was placed on an adjacent building (sludge stockage). Both buildings are located at the East Side of Vidy STEP (sludge handling operations). The measurement path was thus set for monitoring fugitive emissions from the sludge handling operations (thickening, dewatering, incineration and flue gas cleaning). However, the maximum feasible path length was somehow short for the detection of MAH. The resulting optical path length (220 m) limited the detection of MAH at their lowest concentrations. The detection limits are compound-dependent, for instance about 2 ppb for benzene. Nevertheless, the fact the highway (an important MAH linear emission source) is adjacent to the target area would make very difficult to apply the methodology used by Schafer et al [15] for the estimation of area source emissions. This procedure consists in measuring pollutant concentrations downwind (by open-path monitoring when possible) from which area emissions are estimated by inverse modeling.

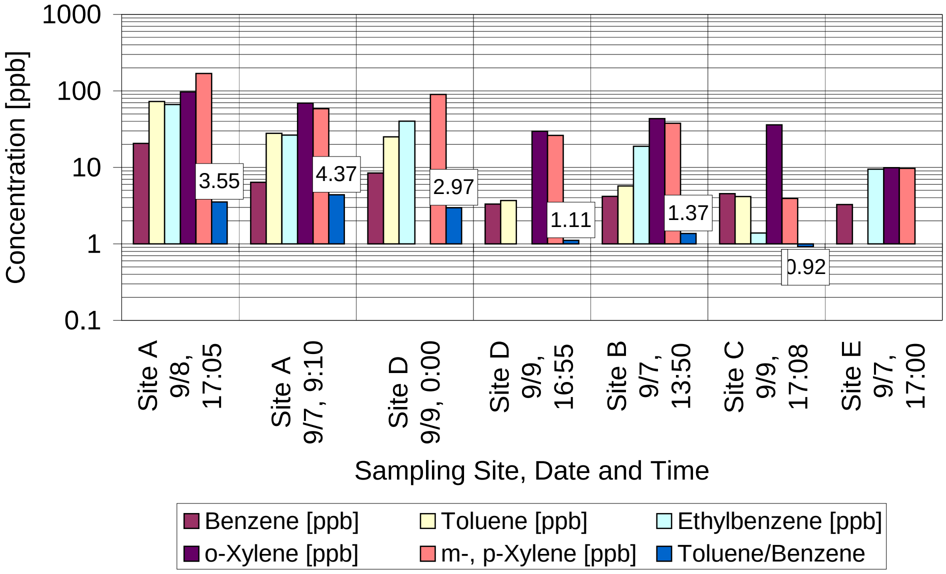
This investigation focuses on the period of intensive DOAS and GC-FID ambient air concentration measurements (September 3-10, 1999). During this period, Vidy STEP treated 90,300(±5%) m^3/day of wastewater and incinerated 62.6 tons/day of sludge cake. Concerning meteorology, the weather was in general good with the exception of the 6th September, partially cloudy. See Figure 7A. This period was characterized by relatively stagnant, anticyclonic conditions. Airflow was probably only local and controlled by valley-mountain wind and lake breeze effects. See Figure 7B. The maximum wind speed (~ 2 m/s) is observed at midday, when comparatively cleaner air masses flow from the Geneva Lake to the Lausanne area. In the morning and in the evening certainly much polluted air masses flow at low speed from north to south (inverse direction).

Figure 7. Weather conditions at Lausanne during the STEP campaign DOAS and GC-FID intensive measurement period (September 3-9, 1999). **Up:** Temperature and pressure time series. **Down:** Wind rose.



The ambient air concentrations of MAH as measured by GC-FID are shown on Figure 8. It is clear that a positive concentration gradient exists from site E to site A (refer to Figure 6). Bearing in mind that most the air samples for GC-FID were made early morning or late afternoon, when air was essentially stagnant, it can be concluded that site A is very likely nearby an area where fugitive emissions are stronger. The measured concentrations are probably linked whether to volatilization of MAH formerly contained in sludge at any of its processing stages or to leaking from the flue gas scrubbing process.

Figure 8. Ambient air concentration of MAH in the sludge handling area as measured by GC-FID. Note that a positive concentration gradient exists between sites A and E.



A close agreement was found between the time trends of MAH (DOAS) and NMHC (FID) measurements (see Figure 9). This correlation leads to conclude that BTEX accounts for approximately a 2% of the carbon of all organic compounds measured at Vidy STEP.

Figure 9. Concentration time series of NMHC as measured by FID and of BTEX as measured by DOAS. Both variables are expressed as carbon (v/v) concentrations. BTEX is the carbon-weighted sum of benzene, toluene, ethylbenzene, o-, m- and p-xylene.

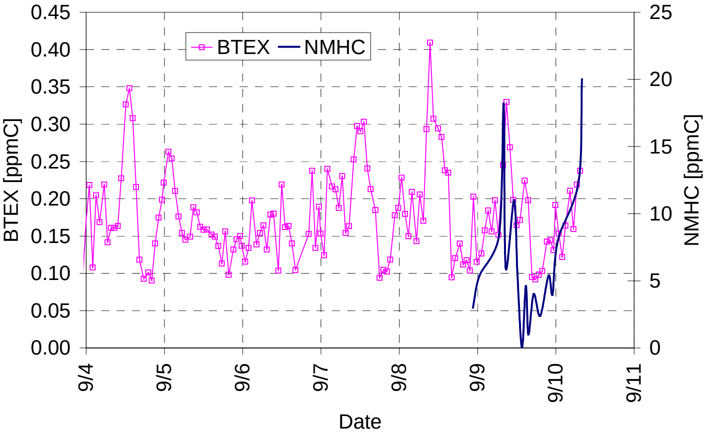
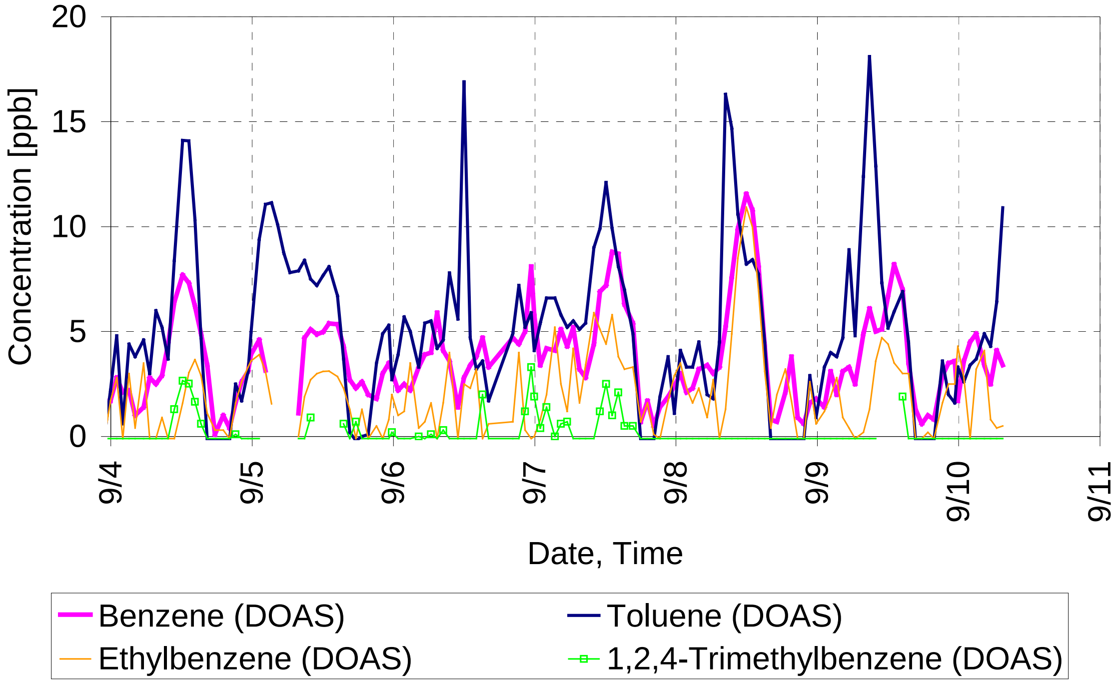


Figure 10. Concentration time series of benzene, toluene, ethylbenzene, and 1,2,4-trimethylbenzene as measured by DOAS.



As shown in Figure 11, DOAS measurements correlate better to GC-FID measurements taken at around midday. This is explained by the fact that the air mass observed is better mixed during that period of the day. The GC-FID measurements corresponding to morning or afternoon samples do not correlate to DOAS measurements not just because of differences in spatial resolution between these techniques [10], but mainly because air is stagnant during these periods of time. Therefore no attempt was made to mathematically correlate the measurements made by these two techniques.

Unexpectedly, peak concentrations of MAH are observed at midday, not during the morning or afternoon as expected if they were related to road traffic and particularly to the linear emission source defined by the adjacent highway. The fact the waste treatment facility is bordered by tall vegetation fences may explain its isolation in term of air mass exchanges. In addition, the ratios of concentration of several MAH to benzene are not in agreement with emission ratios [16] (see table 3). For instance, the average concentration ratio (v/v) of ethylbenzene to benzene is about 0.6, while the expected ratio for emissions from road traffic is around 0.3.

Table 3. Comparison of normalized-to-benzene emission factors [16] and ambient air concentrations of MAH as measured by DOAS at Vidy STEP.

MAH	**Values Normalized to Benzene (v/v)**	
	Ambient air concentrations (DOAS) during STEP	**Emission Factor (Gasoline-powered Road Traffic) [16]**
Toluene	1.27	1.69
Ethylbenzene	0.56	0.25
o-Xylene	0.85	0.34
m-, p-Xylene	1.88	0.77
1,2,4-Trimethylbenzene	0.08	0.29
Phenol	0.08	-

An outstanding correlation was found between the concentration of benzene, toluene, ethylbenzene, o-xylene and 1,2,4-trimethylbenzene as measured by DOAS and the vertical wind speed measured in Chauderon (Lausanne area) (see Figure 11). This leads to conclude that fugitive emissions, accumulated at the ground level of the sludge handling area during a period comprising from late afternoon to early morning, are moved upward by convection during midday. As these polluted air masses rise from the ground level, they cross the DOAS light beam and get detected. No attempt was made to calculate pollutant fluxes, since no local measurements of vertical wind speed were available at the measurement site.

Finally, it is important to mention that very high concentrations of MAH (up to 0.1% v/v of benzene and from 10 to 200 ppm for other MAH) as measured by GC-FID were found in the incineration flue gas stream (previous to gas cleaning operations). It is likely that such elevated quantities of MAH are produced inside the sludge incinerators by unintended pyrolysis [4]. In this case, at least a fraction of the VOC emitted by Vidy STEP would be produced on site.

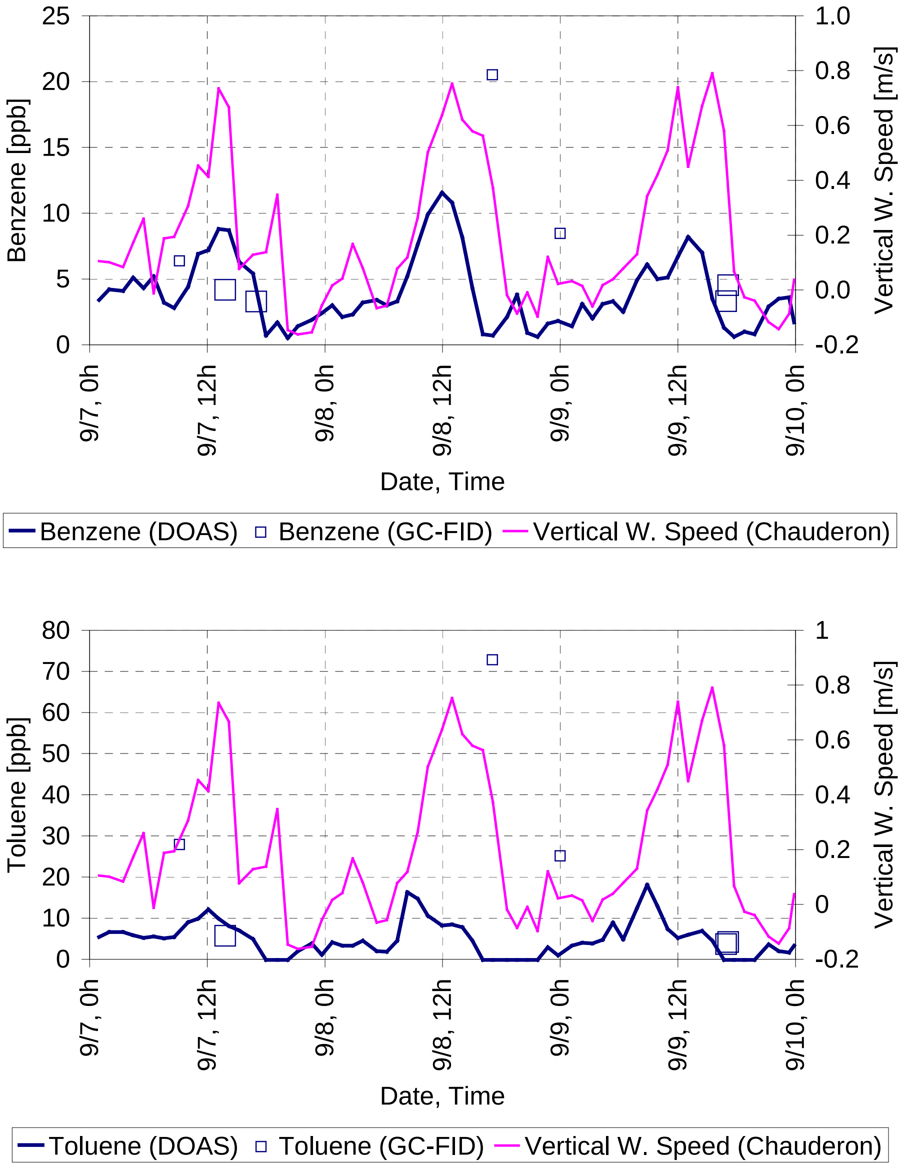
CONCLUSIONS

A new, alternative approach for the recovery of oxygen spectra from open-air spectra allows obtaining instrument-specific oxygen reference spectra sets. In addition, it was found feasible to interpolate these spectra within a limited range of oxygen column densities in order to improve the quality of the retrieved concentration time series.

DOAS is a very versatile technique for the real-time, simultaneous monitoring of monocyclic aromatic hydrocarbons in regard of its time resolution and detection limits. At the optical path length of this study (220 m), the time trends of m- and p-xylene are not in agreement with those of benzene, toluene, ethylbenzene and 1,2,4-trimethylbenzene. The retrieval of m-and p-xylene is subject of further investigation.

The joint analysis of concentration time series and meteorology would lead to indicate that fugitive emissions of MAH accumulate in stagnant conditions at the ground level and are moved upwards by natural convection around midday.

Figure 11. Time series of benzene (up) and toluene (down) as measured by DOAS and vertical wind measured at Chauderon (Lausanne area).



Despite there is sound evidence that MAH detected by DOAS are emitted inside the sludge handling area perimeter, at this point of the investigation, it is still not possible to apportion the measured concentrations of MAH in ambient air to any particular source of emissions within the sludge handling area. Possible sources of fugitive emissions of VOC are the flue gas scrubbing system and volatilization from sludge.

Concentrations of benzene as high as 0.1% were detected in the incineration flue gas before the gas cleaning operation. Stack measurements are thus required to determine the strength and time variability of this emission point source.

Emission of up to 500 kg/day could be possible if all the MAH contained in wastewater were volatilized. DOAS could be used as well to monitor this volatilization process and its dynamics.

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