

REVIEW ARTICLE

# Daily measurement of organic compounds in ambient particulate matter in Augsburg, Germany: new aspects on aerosol sources and aerosol related health effects

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## Abstract

Several epidemiological studies have shown that in the human population ambient particulate matter (PM) is associated with adverse health effects. Little is known, however, about the relative effects of aerosol constituents. Since 2002, diurnal samples of ambient PM<sub>2.5</sub> were analysed by automated methods for the quantification of particle-associated organic compounds (POC). Data on chemical composition have been investigated in epidemiological and biological effect studies. As a result of these studies, the associations found between PAH concentration and symptoms of myocardial infarction survivors suggest a major influence of combustion sources on cardiovascular health effects. The correlations found between formation of reactive oxygen species and the presence of specific organic compounds suggests an important influence of biomass combustion particles in PM<sub>2.5</sub>-associated oxidative stress.

**Keywords:** Ambient aerosol; particulate organic matter; health effects; reactive oxygen species; cardiovascular effects

## Introduction

In its recent air quality guidelines the World Health Organization stated that the 'evidence on airborne particulate matter (PM) and its public health impact is consistent in showing adverse health effects at exposures that are currently experienced by urban populations' (WHO 2006). Ambient air pollution has been associated with cardiovascular disease exacerbation in association with daily elevations of ambient particles (Peters et al. 2001, Pope et al. 2006). Hospitalizations for myocardial infarction were more frequently observed on days with high fine and ultrafine particle concentrations (Forastiere et al. 2005, Lanki et al. 2006) and re-hospitalization was more frequent in myocardial infarction survivors

in associations with fine and ultrafine particle number concentrations (von Klot et al. 2005). The major effect of PM on the pulmonary system is the exacerbation of inflammation. The generation of oxidative stress by metals or organic compounds is a central mechanism by which PM contributes to inflammation (Li et al. 2008). In addition there is growing concern that ambient PM increases the risk of lung cancer (Møller et al. 2008).

Current air quality standards are based on the mass measurements of particles (e.g. EPA 2004). It is under discussion whether physical properties like particle number concentrations or surface areas would be better measures, as these metrics are more strongly correlated with effects in toxicological assessments than particle mass (Peters et al. 2001, Pope et al. 2006). On the other

hand there is increasing discussion on the importance of chemical composition and biological properties of PM (e.g. formation of reactive oxygen species, ROS) for the adverse health effects (Viana et al. 2008). However, little information is available on the influence of chemical composition on cardiovascular or pulmonary disease exacerbation. At present the available evidence seems insufficient for drawing general conclusions on this matter. In particular, there is a lack of epidemiological and toxicological data on specific organic PM components (de Kok et al. 2006).

This paper focuses on actual investigations on the role of the organic constituents in PM and on PM-induced effects which were made possible by the development and implementation of a technology for daily monitoring of organic compounds associated with ambient PM.

## Sampling, parameters and analytical focus

Ambient urban air particulate matter (PM<sub>2.5</sub>) has been sampled on a daily basis in Augsburg, Germany since August 2002. In December 2004 the sampling site was moved from a monastery garden (about 5000 m<sup>2</sup>) within the old town (Schnelle-Kreis et al. 2007) to the central aerosol measurement site in Augsburg, which was set up for a better characterization of the urban background aerosol. This measurement site is operated by the Helmholtz Zentrum München. Parallel to sampling PM<sub>2.5</sub> mass and number concentrations were determined semicontinuously. Since January 2005 additional methods for characterization of, for example, size fractionated number concentrations and total active surface have been applied. Data on chemical composition (particulate sulphate and nitrate, black smoke, organic and elemental carbon mass concentrations) are measured semicontinuously (Cyrys et al. 2006).

PM<sub>2.5</sub> samples for detailed organic speciation are collected with a sequential low volume sampler on quartz fibre filters. Until December 2004 chemical analysis of particle-associated organic compounds (POC) was done by direct thermal desorption-gas chromatography-time of flight mass spectrometry (DTD-GC-TOFMS). With this method a broad range of non- and semipolar compounds had been analysed (Schnelle-Kreis et al. 2005). Compounds which are emitted by a single or a few known sources can be used as molecular tracers for these sources: e.g. hopanes are constituents of all mineral oil- or coal-based fuels and lubricants. Dehydroabietic acid methyl ester is specific for the combustion of coniferous wood, whereas polycyclic aromatic hydrocarbons (PAH) and their oxidized homologues (O-PAH) are emitted from all processes of incomplete combustion (Schnelle-Kreis et al. 2007). In 2005 the analytical method was expanded by a two-

stage automated derivatization method for the analysis of polar compounds. In the first stage polar compounds are converted to their trimethylsilyl derivatives by reaction with MSTFA (*N*-methyl-*N*-trifluoroacetamide) directly on the filter. In the second stage MSTFA is added to the carrier gas during thermal desorption. The extended method now allows analysis of a broad variety of POC ranging from non-polar alkanes to polyols such as 1,6-anhydro- $\beta$ -D-glucopyranose (levoglucosan, a specific tracer for biomass combustion) in one analytical run.

For the study on the relationship with ROS formation (see below) the PAH and O-PAH were analysed by gas chromatography/high-resolution mass spectrometry (GC/HRMS). Details of this method are given by Liu et al. (2006).

Here we focus on PAH, O-PAH and the source specific tracers mentioned above (Tables 1 and 2). Like all other products from incomplete combustion, the PAH showed very strong seasonal variation with maximum concentrations in the winter months and lowest concentrations during summer. The ratio of the highest and lowest concentrations (monthly averages) were in the range of 30–50 for the more stable PAH such as benzo-fluoranthenes (BBKF), benzo[e]pyrene (BEP), benzo[ghi]perylene (BGH) and up to 110 for the more reactive PAH like benzo[a]anthracene (BAA) and benzo[a]pyrene (BAP) (Schnelle-Kreis et al. 2007). As shown in Figure 1 the day to day variation of PAH concentrations during winter were in the same range. Some hopanes like 17 $\alpha$ (H),21 $\beta$ (H)-30-norhopan (29ab) and 17 $\alpha$ (H),21 $\beta$ (H)-hopan (30ab) can be used as molecular tracers for lubricating oil emissions from traffic. These hopanes showed the least seasonal variation in concentration of all compounds investigated. On average their concentrations were two- to five-fold higher in winter months than in summer months. The tracers for biomass (levoglucosan, analysed since 2005) or wood combustion (dehydroabietic acid and its methylester) showed highest concentrations during the heating periods. Surprisingly the average concentrations were only five- to ten-fold lower during summer.

## Results

The established long-term monitoring of POC can be used to investigate aerosol-related atmospheric processes and the impact of different sources on ambient PM (Schnelle-Kreis et al. 2007). The investigation of the detailed chemical composition on health effects is possible via epidemiological studies (Küpfer et al. unpublished data). Furthermore parameters which are suspected to cause biological effects, such as the potency of

**Table 1.** Concentration ranges of ambient particles and selected particle-associated organic compounds in PM<sub>2.5</sub> samples during the winter 2003/2004 (15 October 2003 to 12 February 2004).

Substance	Abbreviation	Unit	Minimum	Maximum	Mean	Median
PM <sub>2.5</sub> (TEOM)	PM <sub>2.5</sub>	µg m <sup>-3</sup>	6.2	37.7	17.5	16.8
Number concentration (CPC)	PNC	cm <sup>-3</sup>	4100	32 700	14 600	13 200
Fluoranthene	FLU	ng m <sup>-3</sup>	0.13	12.6	1.6	1.2
Pyrene	PYR	ng m <sup>-3</sup>	0.10	13.1	1.7	1.2
Benzo[a]anthracene	BAA	ng m <sup>-3</sup>	0.04	7.78	1.1	0.7
Crysene	CRY	ng m <sup>-3</sup>	0.24	15.1	2.6	2.0
Sum of benzo[a]fluoranthenes	BBKF	ng m <sup>-3</sup>	0.36	14.8	3.5	2.9
Benzo[e]pyrene	BEP	ng m <sup>-3</sup>	0.11	3.90	1.0	0.7
Benzo[a]pyrene	BAP	ng m <sup>-3</sup>	0.08	7.10	1.3	0.8
Indeno[1,2,3-cd]pyrene	IND	ng m <sup>-3</sup>	0.03	3.22	0.5	0.4
Benzo[ghi]perylene	BGH	ng m <sup>-3</sup>	0.03	5.44	0.8	0.6
Fluoren-9-one	FLU-O	ng m <sup>-3</sup>	0.11	26.0	2.2	1.0
Anthracen-9,10-dione	ANT-DO	ng m <sup>-3</sup>	0.27	5.96	1.5	1.1
Cyclopenta[def]phenanthren-4-one	CYC-O	ng m <sup>-3</sup>	0.04	7.72	1.2	0.7
Benzo[a]fluoren-11-one	BAF-O	ng m <sup>-3</sup>	0.04	3.80	0.8	0.5
Benzo[b]fluoren-11-one	BBF-O	ng m <sup>-3</sup>	0.08	4.65	1.0	0.7
17α(H),21β(H)-30-norhopan	29ab	ng m <sup>-3</sup>	< LOQ	1.70	0.4	0.3
17α(H),21β(H)-Hopan	30ab	ng m <sup>-3</sup>	< LOQ	0.96	0.2	0.2
Dehydroabietic acid methylester	DHAM	ng m <sup>-3</sup>	< LOQ	38.1	3.7	2.2

LOQ, limit of quantification.

the formation of ROS can be correlated with the organic chemical inventory of PM (Sklorz et al. 2007).

In an initial epidemiological study we investigated the association between concentrations of POC in the PM<sub>2.5</sub> samples and cardiovascular symptoms of myocardial infarction (MI) survivors. As part of the multicentre AIRGENE study (Peters et al. 2007), MI survivors from Augsburg, Germany, recorded the daily occurrence of their symptoms in a diary winter 2003–2004 (15 October 2003 to 10 February 2004). Based on the KORA MI registry (Löwel et al. 2005) a total of 200 MI survivors were recruited. Seventy-four study participants (37%) provided diary data on more than 50 days and reported variable occurrence of symptoms and were therefore considered for analyses. A total of 4221 person-days were analysed for 'avoidance of physically demanding activities due to heart problems' and occurred on 23.7% of the days. Data on 'shortness of breath' was available for 3484 person-days and were recorded on 34.5% of the days. Associations between concentrations of POC in PM<sub>2.5</sub> samples and cardiovascular symptoms of MI survivors were investigated. Analyses were done using generalized estimating equations models adjusting for meteorological

and other time-variant confounders. Effect estimates are presented as odds ratios (OR) together with 95% confidence intervals (95% CI) based on an interquartile range (IQR) increment for the particle air pollution measures. We found an immediate increased avoidance of activities on days with high PAH concentrations (Figure 2, Küpper et al. unpublished data). However, we observed a decreased risk in the avoidance of activities with a lag of 1 day. While we found immediate and 1 day delayed decreases in shortness of breath in association with PAH, results showed an increased risk with a lag of 2 days (Küpper et al. unpublished data). These results indicate that PAH might contribute to the observed increased risk of cardiovascular disease exacerbation. However, PAH may act as unspecific indicators for combustion-related sources.

Both PM<sub>2.5</sub> and PNC were marginally associated with avoidance of activities, but signals were not as strong as the signals for PAH. The high correlation between PAH themselves makes it difficult to attribute the observed associations to specific compounds.

In a study on the oxidative potential, PM<sub>2.5</sub> samples from summer 2005 (21 August 2005 to 12 September

**Table 2.** Concentration ranges of ambient particles and selected particle-associated organic compounds in PM<sub>2.5</sub> samples in summer 2005 (21 August 2005 to 12 September 2005) and correlation with ESR signal ( $n = 20$ ).

	Unit	Concentration				Spearman R
		Minimum	Maximum	Mean	Median	
ESR signal (unpaired e <sup>-</sup> spins)	10 <sup>5</sup> m <sup>-3</sup>	1.1	10.5	4.6	3.6	-
PM <sub>2.5</sub> (TEOM)	µg m <sup>-3</sup>	3.0	35.3	14.9	15.4	<b>0.49*</b>
PM <sub>10</sub> (TEOM)	µg m <sup>-3</sup>	10.0	44.0	22.4	23.0	<b>0.51*</b>
Black carbon (ethalometer)	µg m <sup>-3</sup>	1.5	3.9	2.6	2.7	<b>0.49*</b>
Number conc. (3–800 nm, SMPS)	cm <sup>-3</sup>	4900	15800	10800	11030	0.17
Fluoranthene	ng m <sup>-3</sup>	0.063	0.301	0.176	0.164	0.12
Pyrene	ng m <sup>-3</sup>	0.072	0.299	0.169	0.163	0.22
Benz[a]anthracene	ng m <sup>-3</sup>	0.022	0.11	0.05	0.049	0.43
Chrysene	ng m <sup>-3</sup>	0.092	0.72	0.19	0.17	0.18
Sum of benzofluoranthenes	ng m <sup>-3</sup>	0.13	1.26	0.32	0.27	<b>0.59*</b>
Benz[e]pyrene	ng m <sup>-3</sup>	0.060	0.24	0.12	0.11	<b>0.49*</b>
Benz[a]pyrene	ng m <sup>-3</sup>	0.039	0.17	0.084	0.079	<b>0.56*</b>
Perylene	ng m <sup>-3</sup>	0.004	0.045	0.016	0.014	<b>0.56*</b>
Indeno[1,2,3-cd] pyrene	ng m <sup>-3</sup>	0.062	0.19	0.12	0.12	<b>0.54*</b>
Dibenz[ah] anthracene	ng m <sup>-3</sup>	0.010	0.029	0.019	0.019	<b>0.64**</b>
Benzo[ghi]perylene	ng m <sup>-3</sup>	0.093	0.23	0.16	0.16	<b>0.56*</b>
Coronene	ng m <sup>-3</sup>	0.041	0.12	0.071	0.066	<b>0.51*</b>
Anthracen-9,10- dione	ng m <sup>-3</sup>	0.11	0.58	0.39	0.41	0.30
Cyclopenta[def] phenanthren-4-one	ng m <sup>-3</sup>	0.010	0.039	0.024	0.024	0.06
Benz[a]fluoren-11- one	ng m <sup>-3</sup>	0.035	0.29	0.11	0.10	<b>0.68**</b>
Benz[b]fluoren- 11-one	ng m <sup>-3</sup>	0.044	0.39	0.15	0.13	<b>0.78**</b>
Benz[de]anthracen- 7-one	ng m <sup>-3</sup>	0.047	0.52	0.17	0.14	<b>0.78**</b>
Benz[a]anthracen- 7,12-dione	ng m <sup>-3</sup>	0.023	0.087	0.050	0.045	0.47*
Cyclopenta[cd] pyren-[4H]-3-one	ng m <sup>-3</sup>	0.005	0.024	0.010	0.009	<b>0.62**</b>
Benzo[cd]pyren- 6-one	ng m <sup>-3</sup>	0.048	0.40	0.14	0.12	<b>0.73**</b>
17α(H),21β(H)-30- norhopan	ng m <sup>-3</sup>	0.09	0.49	0.22	0.19	0.20
17α(H),21β(H)- hopan	ng m <sup>-3</sup>	0.05	0.41	0.18	0.15	0.20
Docosanoic acid methylester	ng m <sup>-3</sup>	< LOQ	0.17	0,07	0,07	<b>0.55*</b>
Tetracosanoic acid methylester	ng m <sup>-3</sup>	< LOQ	0.16	0,07	0,07	<b>0.61**</b>
Hexacosanoic acid methylester	ng m <sup>-3</sup>	< LOQ	0.09	0,04	0,03	<b>0.59*</b>
Dehydroabietic acid	ng m <sup>-3</sup>	9.3	122	23	11	<b>0.80**</b>
Levoglucosan	ng m <sup>-3</sup>	36	293	123	126	<b>0.78**</b>

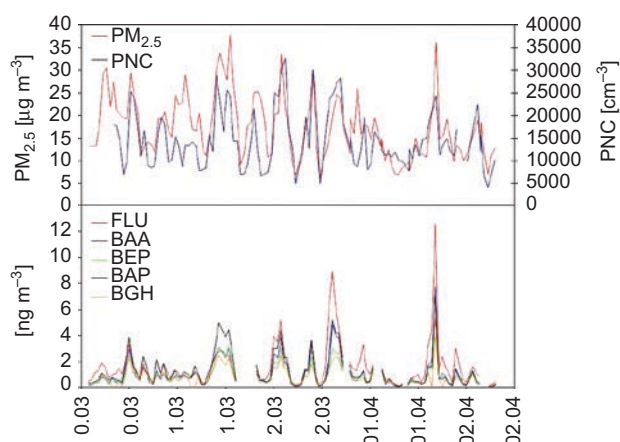
\*Significant ( $p \leq 0.05$ ); \*\*highly significant ( $p \leq 0.005$ ). LOQ, limit of quantification.

2005) were analysed additionally for ROS formation. One aliquot of each filter was used for electron spin resonance (ESR) spectroscopic measurements, described in detail by Briedé et al. (2005). Briefly, after addition of the spin trap 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) and ascorbic acid, ROS formation was measured directly on the PM-containing filters. For statistical analysis Pearson and Spearman rank correlation coefficients between the ESR signal the concentrations of POC and other aerosol characteristics were calculated. We found high correlations of the concentrations of five to seven ring PAH and four to five ring oxidized PAH with the formation of ROS (Sklorz et al. 2007, Table 2). Correlations were most pronounced for some PAH monoketones like the benzofluorenones, which are not yet reported in literature to be redox-cycling active. But also for some PAH, significant or highly significant correlation to ROS

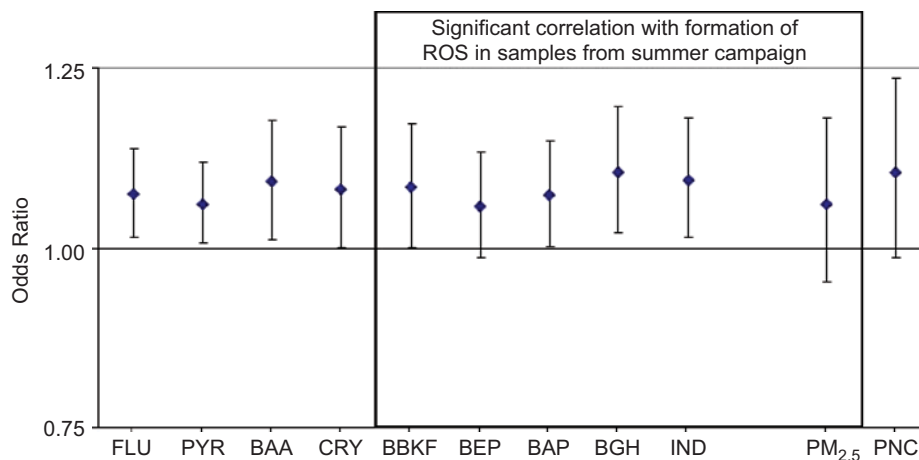
formation capacity was found. The associations of ROS formation with some methylesters from long chain alcanoic acids and even higher correlations with some specific molecular tracers for coniferous wood combustion (dehydroabietic acid) and biomass combustion (levoglucosan) can be explained by the high correlation of PAH and oxidized PAH with wood combustion tracers. No correlations were found with molecular tracers (Schnelle-Kreis et al. 2007) for fossil fuel consumption like the hopanes. This finding suggests a major influence of biomass combustion emissions to the formation of ROS from ambient PM<sub>2.5</sub> even in summer with low activity of domestic heating. The results also suggest that the effects observed in the epidemiological study may depend in part on the oxidative stress potential.

## Conclusions and outlook

Automated methods for the analysis of POC on a daily basis have been developed and applied in epidemiological and biological effect studies. The long-term period of data lasting from 2002 to 2009 enables not only evaluation of sources and processes but also epidemiological and biological effect studies. As a result of these studies, the associations found between PAH concentration and symptoms of MI survivors suggest a major influence of combustion sources on cardiovascular health effects. The correlations found between formation of oxygen radicals and the presence of specific organic compounds suggests the important influence of biomass combustion in PM<sub>2.5</sub>-associated ROS formation. Further research on the relationship between radical formation, epidemiological impact and presence of specific O-PAH and POC are likely to provide a better understanding of the relationship between the source-dependent chemical composition of PM and the toxicological risks associated



**Figure 1.** Daily mean of ambient particle concentrations and particle associated polycyclic aromatic hydrocarbons (PAH) during winter 2003/2004 (15/10/2003–12/02/2004). Top: particle mass and number concentrations. Bottom: PAH concentrations. For abbreviations see Table 1.



**Figure 2.** Associations between concentrations of polycyclic aromatic hydrocarbons as well as ambient particle concentrations and the symptom 'Avoidance of activities' on the same day. Odds Ratio per increase in interquartile range. For abbreviations see Table 1.

with PM exposure. In addition to the described technology now established in Augsburg for routine daily monitoring of organics, novel on-line aerosol mass spectrometry techniques may be applied for an even higher time resolved analysis on PM associated compounds such as PAH (Bente et al. 2006).

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