

Polycyclic aromatic hydrocarbons (PAHs) in atmospheric urban area: monitoring on various types of sites

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Abstract The air quality over the Toulouse urban area (France) is recorded daily by the regional “Midi-Pyrénées” atmospheric pollution measurements network (ORAMIP). Relevant data is collected from about 100 analysers spread over more than thirty stations. The regulations covering major indicators of atmospheric pollution (ozone, nitrogen dioxide, sulphur dioxide) have been updated in recent years to include additional compounds like polycyclic aromatic hydrocarbons (PAHs). The ORAMIP, in partnership with the ENSIACET has undertaken background PAH average concentration measurements over the urban agglomeration of Toulouse during spring 2006 for various types of sites (traffic, urban, industrial). The sampling was performed using a low volume air sampler equipped with quartz fiber filters and polyurethane foams. For the two urban sites, total atmospheric concentrations between 12 and 20 ng/m³ have been obtained, whereas for the industrial site the values averaged 22 ng/m³. In addition, and regardless of site,

the average concentrations of benzo[*a*]pyrene, at present the only regulated PAH, were always less than the 1 ng/m³ limit.

Keywords Ambient air quality · Polycyclic Aromatic Hydrocarbons · PAH · Urban air pollution

Introduction

Polycyclic aromatic hydrocarbons (PAHs) usually occur as complex mixtures made up of two or more condensed benzene rings, and the resulting molecules contain only carbon and hydrogen atoms. PAHs are thermodynamically stable and formed during pyrolysis and the incomplete combustion of organic materials in a variety of occupational and environmental settings (Rihs et al. 2005; Unwin et al. 2006; Chen et al. 2007). Several methods of absorption are known both for the general population and workers at risk. They include inhalation of environmental air, ingestion of contaminated foods and drinks and cutaneous absorption (Lodovici et al. 2005; Kazerouni et al. 2001; Storelli et al. 2003; Barranco et al. 2004; Väänänen et al. 2005). Industries where occupational exposure to PAHs is likely to occur include coke ovens and coal tar use, iron and steel works, aluminium casting, foundries, carbon electrodes and carbon black manufacture, asphalt manufacture and use, and many others (Mastral et al. 1996, 1998, 1999; Brandt et al. 2000;

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Binet et al. 2002; Lee et al. 2004; Unwin et al. 2006; Lim et al. 2007). The tertiary sector also contributes to PAH pollution via road traffic (Guillemain et al. 1992; Perico et al. 2001; Wingfors et al. 2001; Mastral et al. 2003).

Epidemiological studies have revealed an increased incidence of cancer, especially of the lung and bladder, among workers occupationally exposed to PAHs (IARC 1983; Steineck et al. 1989; Mumtaz et al. 1996; Pesch et al. 2000). Due to their potential mutagenicity and carcinogenicity, many regulations on PAH emissions have been proposed. A European directive (2004/107/CE) has proposed a maximum permissible risk level of 1 ng/m^3 benzo[*a*]pyrene in ambient air, based on the carcinogenic potential of inhaled particulate PAHs (average calculated for one calendar year of the total contents of the PM10 fraction). Monitoring of PAH is thus obligatory and the member countries had until 15 February 2007 to implement the necessary legislative, statutory and administrative measures to conform with the demands of the Directive. The contribution of benzo[*a*]pyrene in ambient air must be studied by monitoring other Polycyclic Aromatic Hydrocarbons like benzo[*a*]anthracene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, indeno[1,2,3-*cd*]pyrene and dibenz[*a,h*]anthracene.

Sampling of airborne PAHs requires special equipment since those with a lower molecular weight are found mainly as vapour, while those with a higher molecular weight are found in the condensed phase. Consequently, an adequate sampling method must combine a filter for the collection of particulate, and one or more sorbents such as XAD-2 and/or polyurethane foam (Peltonen and Kuljukka 1995; Larsen and Baker 2003; Ciganek et al. 2006; Yang et al. 2007). The most widespread method used for extracting PAHs from the filter and polyurethane foam are extraction by sonication, Soxhlet solvent or accelerated solvent extraction (ASE). Analysis is carried out by gas chromatography (GC) coupled with mass spectrometry, and high performance liquid chromatography (HPLC) coupled with fluorimetry (Peltonen and Kuljukka 1995; Maddalena et al. 1998; Papageorgopoulou et al. 1999; Wingfors et al. 2001; Gutierrez-Daban et al. 2005; Lim et al. 2005; Li et al. 2006).

The aim of our study was to determine the atmospheric concentrations of PAHs for three sites in Toulouse, each with different characteristics (traf-

fic, urban, industrial) and to determine the characteristic PAHs of each site. The whole operation was an exploratory study, the first of its kind in the city.

Experimental

Sampling sites

Samples were taken in April and May 2006 at three selected sites named “CCIT”, “Berthelot” and “Eisenhower” (Fig. 1). These three areas represent a traffic site, an urban site and an industrial site respectively.

Air temperature, relative humidity, plus ozone, PM10 and NO₂ concentrations were provided hourly by ORAMIP, and this data collected during each sampling exercise was averaged (Table 1).

Reagents

For all the procedures listed below, we used methylene chloride as the solvent (for residue analysis, >99.8% GC; Fluka).

An EPA 610 standard solution, at around 100 mg/l in methanol/methylene chloride (1:1) for anthracene (ANT), benzo[*a*]anthracene(B[*a*]A), benzo[*a*]pyrene (B[*a*]P), benzo[*k*]fluoranthene (B[*k*]F), chrysene (CHR), indeno[1,2,3-*cd*]pyrene (IP), phenanthrene (PHE), pyrene (PY), at around 200 mg/L for fluoranthene (FL), benzo[*b*]fluoranthene (B[*b*]F), dibenz[*a,h*]anthracene (DB[*ah*]A), benzo[*g,h,i*]perylene (B[*ghi*]P), and fluorene (FLN), at around 1,000 mg/l for acenaphthene (AC), naphthalene (NAP), and 2,000 mg/l acenaphthylene (ACE) was supplied by Supelco. By weighing and by serial dilutions in methylene chloride, solutions with final concentrations between 5 and 30 µg/l have been obtained for standardization.

The following deuterated analogues were used: naphthalene-D8, benz[*a*]anthracene-D12, chrysene-D12 (2,000 mg/l; Supelco). By weighing and by serial dilutions in methylene chloride, a solution containing approximately 2 mg/l of naphthalene-D8 and chrysene-D12 was obtained. This solution was used to spike the PUF and quartz filter after the sampling exercise and used as the extraction standard. Benz[*a*]anthracene-D12 at 2 mg/l in methylene chloride was used as injection standard.

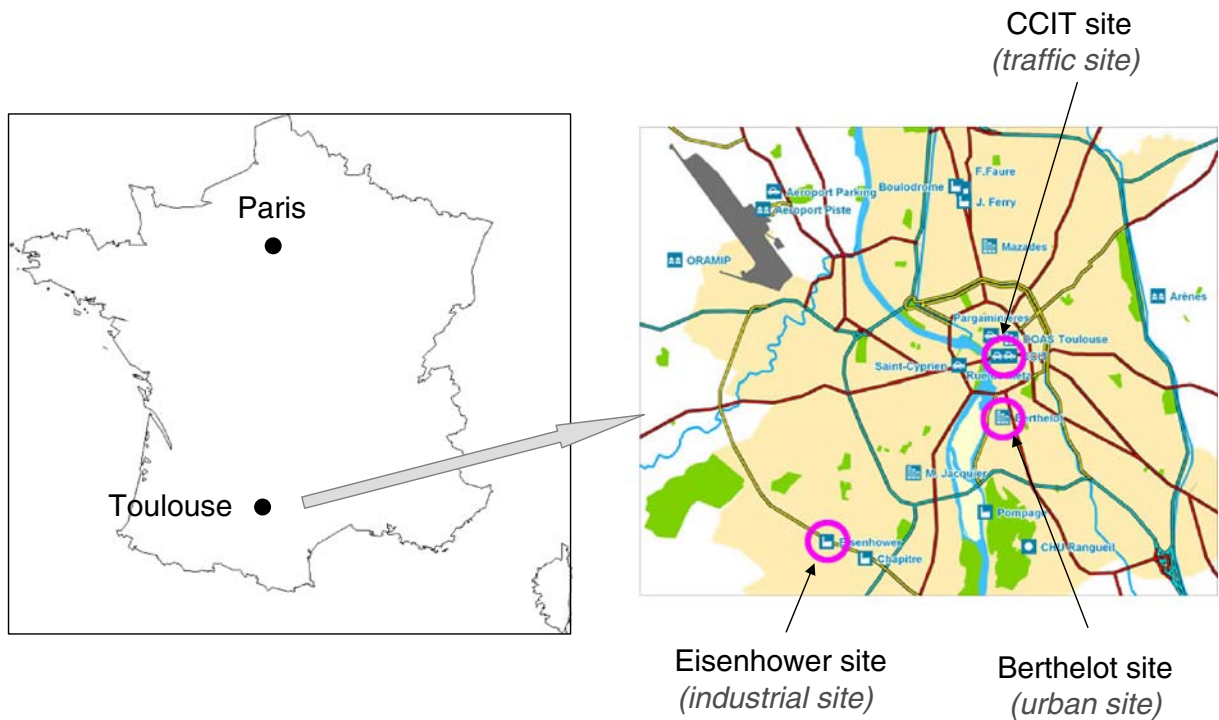


Fig. 1 Localisation of the sampling sites

Air sampling

Atmospheric samples were collected using a low volume air sampler (Partisol Plus, Ecomesure) equipped with quartz fiber filters (diameter 47 cm,

Whatman) and four polyurethane foams (PUF, 40 mm diameter, 20 mm thick, Ecomesure) inserted in a ChemComb cartridge. Particles with a diameter <10 μm (PM₁₀) were collected. Samples between 24 and 72 m³ were collected at a flow rate of 1 m³/h.

Table 1 Air temperature, relative humidity, ozone, PM10 and NO₂ average concentrations

Place	Identifier	Sampling start	Sampling end	O ₃ (μg/m ³)	PM 10 (μg/m ³)	NO ₂ (μg/m ³)	Average temperature (°C)	HR(%)
CCIT (traffic site)		04/10/06 (12 h30) ^a	04/11/06 (12 h30)	Not measured	17.1	53.2	13.5	71.8
	T2	04/11/06 (13 h)	04/13/06 (13 h)		21.5	58.7	10.7	63.6
	T1	04/15/06 (8 h)	04/18/06 (8 h)		15.0	35.5	13.4	71.6
Berthelot (urban site)	U2	04/19/06 (9 h30)	04/21/06 (9 h30)	66.0	21.5	24.3	17.1	58.7
	U3	04/21/06 (12 h35)	04/24/06 (12 h35)	67.4	18.7	16.0	18.9	68.1
	U1	04/24/06 (12 h50)	04/25/06 (12 h50)	66.5	18.4	16.0	18.6	66.2
Eisenhower (industrial site)	I2	04/26/06 (8 h30)	04/28/06 (8 h30)	66.0	13.6	24.3	15.0	77.0
	I3	04/29/06 (9 h30)	05/02/06 (9 h30)	67.4	18.8	16.0	14.0	54.8
	I1	05/02/06 (9 h50)	05/03/06 (9 h50)	66.5	23.9	16.0	16.9	54.5

The data collected during each sampling session is averaged

^a Not analysed because of the presence of water in important quantity on foam/filter (phenomenon relative to the meteorological bad conditions during sampling)

Prior to sampling, PUF plugs were cleaned by Soxhlet extraction with methylene chloride (a first extraction (24 h) followed by a second one (24 h) with pure methylene chloride). After this conditioning, the foams were wrapped in aluminium foil and left overnight in a fume cupboard to allow the solvent to evaporate. They were then transferred and stored in a desiccator.

Filters were placed in Petri dishes and kiln-fired at 500°C overnight, then these dishes were wrapped in aluminium foil and stored in a desiccator.

After sampling, PUF and filters were transported to the laboratory in an icebox. Stringent precautions were taken during sampling and storage to avoid possible contamination. Field blanks were obtained by using PUF and filters which were stored and transported in the same bowls as those which were sampled. PAH extractions were made within 2 days.

Extractions of blank PUF and filters showed no PAH contamination above the quantification limits.

Pre-treatment and analysis of samples

After sampling, a PAH-D solution was added on PUF plugs and filters. PAH eluting between naphthalene and acenaphthylene were corrected by naphthalene D8 and compounds eluting between fluoranthene and benzo [*g,h,i*]perylene by chrysene-D12.

PUF plugs and filters were both extracted by ASE 300 (Dionex) with methylene chloride at a temperature of 100°C and a pressure of 140 bar (one cycle). Extracts were collected in glass bottles.

Samples were concentrated to 1 ml by evaporation under a stream of nitrogen at atmospheric temperature and pressure. At the end of the procedure, 100 µl of the benzo[*a*]anthracene D-12 solution was added to each sample in micro test tubes which were hermetically sealed and stored at -28°C until analysis. Benzo [*a*]anthracene D-12 was used as internal standard.

Analyses were performed by Gas Chromatography (HP 5890 Series II) coupled with Mass Spectrometry chromatographic column (HP 5971 A) with a DB-5MS 30 m×0.25 mm, 0.25 µm thickness (J&W Scientific). Chromatographic conditions were as follows: injector temperature 300°C; injection in splitless mode (1 min); thermal program: 5 min at 100°C, ramp 20°C/min up to 180°C (3 min), ramp 15°C/min up to 260 °C (5 min), ramp 10°C/min up to 300°C (10 min); carrier gas helium, 1 ml/min, injection volume 0.5 µl, temperature transfer line 300°C. The chromatogram was obtained in the selected ion monitoring (SIM) mode from characteristics shown in Table 2.

The quantification limits (S/N=10) for a sample of 24 m³ (1 m³/h for 24 h) gave the following

Table 2 Analytical characteristics

Compound	Abbreviation	Retention time (min)	Number of aromatic rings	Quantitation ion	Confirmation ion
Naphtalene	NAP	6.7	2	128	127
Naphtalene d8	NAP d8	6.6		136	
Acenaphthylene	ACE	10.1	2	150	152
Acenaphthene	AC	10.4	2	153	154
Fluorene	FLN	11.7	2	165	166
Phenanthrene	PHE	14.4	3	178	176
Anthracene	ANT	14.6	3	178	176
Fluoranthene	FL	17.2	3	202	200
Pyrene	PY	17.7	4	202	200
Benzo(<i>a</i>)anthracene	B(a)A	21.1	4	228	226
Benzo(<i>a</i>)anthracene d12	B(a)A d12	21.05		240	
Chrysene	CHR	21.2	4	228	226
Chrysene d12	CHR d12	21.15		240	
Benzo(<i>b</i>)fluoranthene	B(b)F	25.3	4	252	250
Benzo(<i>k</i>)fluoranthene	B(k)F	25.4	4	252	250
Benzo(<i>a</i>)pyrene	B(a)P	26.3	5	252	250
Indeno(1,2,3- <i>cd</i>)pyrene	IP	30.2	5	276	138
Dibenzo(<i>a,h</i>)anthracene	DB(ah)A	30.3	5	278	139
Benzo(<i>g,h,i</i>)perylene	B(ghi)P	31.3	6	276	173

concentrations: 0.06 ng/m³ for IP, DB(ah)A and B(ghi)P and 0.03 ng/m³ for the other PAHs.

Transport and laboratory blanks were processed along with the samples collected on each expedition, and were obtained from PUF and filters that were left in the same fridge and icebox as the actual sampling materials. Blank levels of individual PAHs were normally very low and in most the cases undetectable. In addition, recoveries of PAHs were determined by adding a standard amount of a known PAH to a blank filter and to a blank foam. The average recoveries of the 16 PAHs varied from 80% for NAP, ACE and AC to 96% for the others.

Two standard solutions containing 120 ng of ACE and 100 ng of B(a)P were injected into four PUF and four quartz fibre filters respectively. They were kept at 4°C and extracted after different periods of time (24, 48, 72 h and a week). Three tests were carried out for each different period (Table 3). The level of PAH recuperation decreases significantly when it is stored for more than 3 days. In practice, all extractions were made within 2 days of sampling.

Principal component analysis (PCA)

Principal component analysis is a multivariate statistical method used in environmental science to reduce the dimensionality of a data set (Mai et al. 2003; Terzi and Samara 2005; Yang et al. 2007). PCA is a projection method that helps to visualize all the information contained in a data table. Its principle is to find the directions in space along which the distance between data points is the largest. It transforms the original variables, which may possess a significant correlation, into a set of uncorrelated orthogonal variables, called principal components (PCs; Gambaro et al. 2004). PCs are ranked so that each one carries more information than any of the following ones. The relationship between samples can be presented graphically as a score plot of the PCs. Two samples can be described as similar if they have close values for most

Table 3 Recuperation levels of ACE and B(a)P after stocking foams or filters at 4°C for 24, 48, 72 h and 1 week

	24 h (%)	48 h (%)	72 h (%)	A week (%)
ACE	95	95	80	50
B(a)P	99	95	94	80

variables, which mean close coordinates in the multidimensional space and vice versa.

In our study PCA was used to analyze the different compositions of the PAHs in each sampling site. For the PCA, the concentrations of the 16 PAHs were used as the multiple dimensions. PCA was carried out with Unscrambler v. 7.5 (Camo ASA, Norway).

Results and discussion

Results for the CCIT (traffic) site

We can see that in general the PAH concentrations, (i.e. the sum of particulate and vapour fractions) vary from 0.06 to 4.57 ng/m³ included (Fig. 2a).

The main PAHs emitted are PHE, FL, PY and ACE. The first three of these are characteristic of diesel vehicle exhaust gas, while FL and PY are also characteristic of waste gases from domestic heating. The ‘marker’ PAHs from petrol-engined vehicles (B(a)P, B(ghi)P and IP) have been detected at concentrations between 0.16 et 1.26 ng/m³ included.

The concentrations for the samples taken between 11 and 13 April (48 h) are higher than those for the period 15 to 18 April (Fig. 2a). This is because, the 11, 12 and 13 April were weekdays (Tuesday to Thursday) whereas the 15, 16 and 17 April (72 h) were the Easter weekend (Saturday to Monday). Traffic is obviously much greater in Toulouse on weekdays than over the Easter weekend, highlighting the ‘temporal variable’, with concentrations varying as a function of the day of the week.

The six PAHs characteristic of car exhaust emissions [PHE, FL, PY, B(a)P, B(ghi)P, IP] account for 47% of the total PAH concentration. ACE accounts for 14% (Fig. 3a).

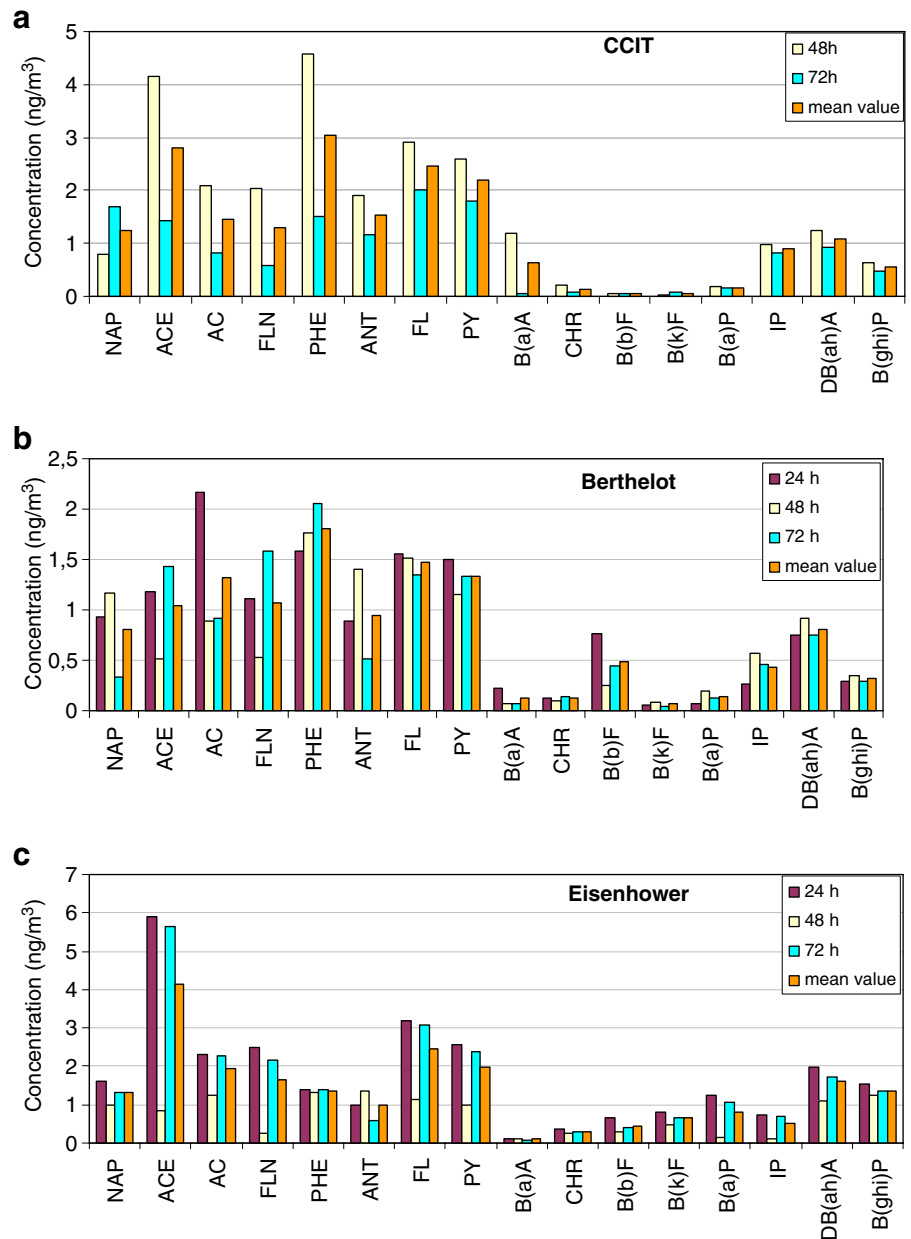
Results from the Berthelot (urban) site

The atmospheric PAH concentrations found at this site were between 0.06 and 2.16 ng/m³ included (Fig. 2b).

The main PAHs emitted are ACE, AC, FLN, PHE, FL, PY.

PHE, FL and PY are characteristic of diesel vehicle exhausts while as stated previously FL and PY are also characteristic of waste gases from domestic heating. The sources for the ACE emissions are varied and correspond to different sorts of combus-

Fig. 2 Variations in the individual PAH compound concentrations by site (ng/m^3) (a: traffic site; b: urban site; c: industrial site)



tion. We can see that the six compounds account for almost 65% of the PAHs found at the site (Fig. 3b).

Results from the Eisenhower (industrial) site

The atmospheric PAH concentrations are between 0.08 and 5.9 ng/m^3 (Fig. 2c).

The main PAHs emitted at this site are ACE, AC, FL, PY and account for more than half of the total PAH site concentration (Fig. 3c). The first two

compounds, mainly present in the gaseous phase, correspond to assorted industrial combustion, whereas FL and PY, as was seen previously, are characteristic of diesel engine exhausts. The presence of a domestic waste incinerator nearby (SETMI) explains the presence of PHE, PY, CHR, B(b)F, B(k)F, DB(ah)A. It should be noted that the samples taken during the Bank Holiday weekend (29 April to 2 May, 72 h) do not have lower concentrations than those for the other days.

Fig. 3 Proportions of the different PAHs found at each site (a: traffic site; b: urban site; c: industrial site)

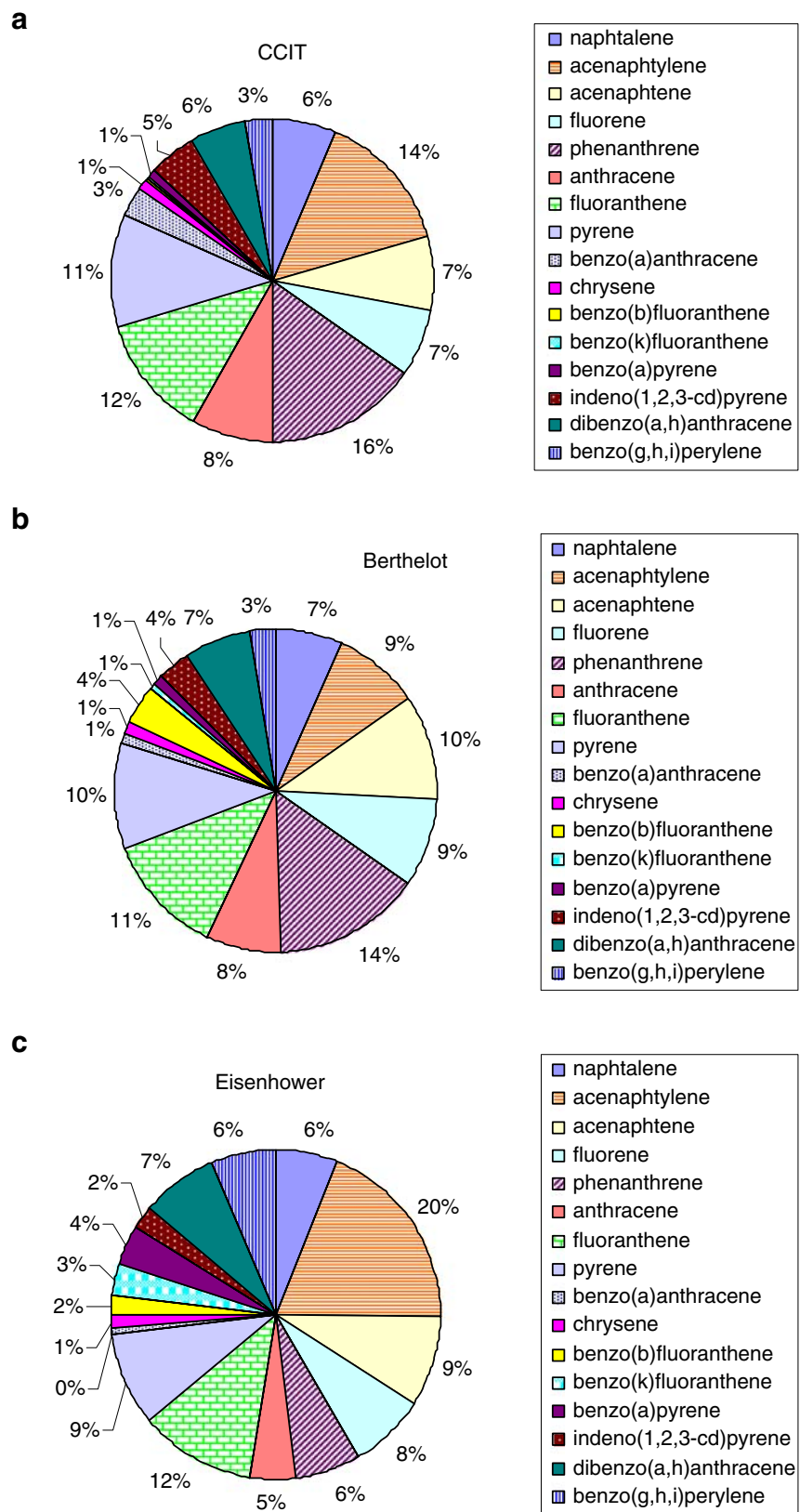
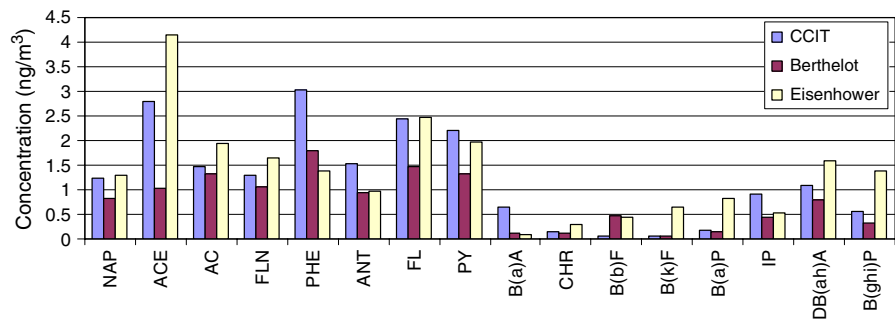


Fig. 4 Average concentrations found at each of the sites (ng/m^3)



Inter site comparisons

Comparing average concentrations, the latter are roughly the same on the CCIT and Eisenhower sites (19.7 and $21.6 \text{ ng}/\text{m}^3$, respectively) whereas they are almost half as low again on the Berthelot site ($12.3 \text{ ng}/\text{m}^3$).

The qualitative distribution of the PAHs emitted varies according to site (Fig. 4). The main PAHs found at the CCIT are automobile pollution markers (PHE, FL, PY, IP), while Eisenhower is characterised by industrial combustion PAHs. Samples from Berthelot have moderate concentrations showing that it is a « typical » urban site

The atmospheric concentration profiles made as a function of the number of aromatic nuclei in the molecules, show an overall decrease as a function of the number of nuclei for each of the sites (Fig. 5).

Comparison of benzo(a)pyrene concentrations

Benzo(a)pyrene is for the moment the only regulated PAH. It is a marker for petrol-engined car and

industrial smelting emissions. Several French sites with different characteristics are shown by way of comparison (Table 4).

The benzo(a)pyrene concentrations found at the Toulouse traffic site are roughly the same as those obtained for the same type of place in La Rochelle (Vallet 2000) and in Marseilles (Airmaraix 2004). This is also true for inter-urban site comparisons. In addition, the concentrations from close to an industrial area at Eisenhower, are within the same range as those found in a study of sites in Saint-Dizier and in Martigues Lavéra ($0.35\text{--}1.5 \text{ ng}/\text{m}^3$) and are still well below the values found at the Neussargues industrial site (wood carbonisation factory).

This comparison allows us to show that the concentrations of benzo(a)pyrene in the Toulouse agglomeration are roughly the same as those found on other French sites and are less than $1 \text{ ng}/\text{m}^3$. However, an important point is that atmospheric PAH emissions show large seasonal variations (Leoz-Garziandia 2003; Mastral et al. 2003). The contribution of domestic heating components is higher in winter when PAH breakdown is lower, which means

Fig. 5 Concentrations of PAH x-rings for each site (ng/m^3)

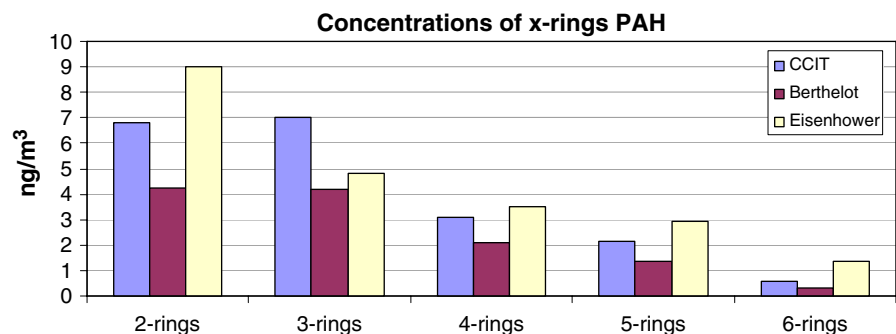


Table 4 Comparison of the B(a)P concentrations from several studies in France

Site et nature	Période	[Sampler] Concentrations (ng/m ³)	References
Boulevard Joffre (La Rochelle; traffic site)	June to August 2000	[HVS] 0.28 [Partisol] 0.35	Vallet 2000
Près d'Arènes (Montpellier; urban site)	05/11/01 05/17/01	[Partisol] 0.20 [Partisol] 0.24	Leoz-Garziandia 2001
Cinq Avenues (Marseille; urban site)	10/18/01 to 01/23/04	[DA80] 0.31	Airmaraix 2004
St-Dizier (Haute Marne; industrial site)	09/24/03 to 10/01/03	[DA80] 0.35	Leoz-Garziandia 2003
Martigues Lavéra (industrial site)	01/19/06 to 02/22/06	[DA80] 1.5	Airfobep 2006
Neussargues (Cantal; industrial site)	10/23/02 to 10/30/02	4.80	Cited in Airfobep 2006
CCIT (traffic site)	April to May 2006	[Partisol] 0.17	This study
Berthelot (urban site)	April to May 2006	[Partisol] 0.13	This study
Eisenhower (industrial site)	April to May 2006	[Partisol] 0.81	This study

that atmospheric concentrations are higher in winter than in summer.

Principal component analysis

PAH concentration data of each site were analysed by PCA. The first two PCs accounted for 76% of the explained variance in the original data set. Figure 6 plots scores on the first two PCs to visualize the differences in the profile of PAHs. Samples could be

discriminated between three groups (A, B and C) by PC1 (51%). Urban samplings (U) with a lower PAH concentration are found in the Group A as T2 sampling which was made during the WE. Because of the decrease of traffic, the sampling T2 shows the characteristics of an urban site. Sampling I2 is not included in the group B because of the two different air masses it represents. Group C seems to be related to the traffic sites. Let us note that PC2 accounted for 25% of the explained variance describes the variation in PHE and B

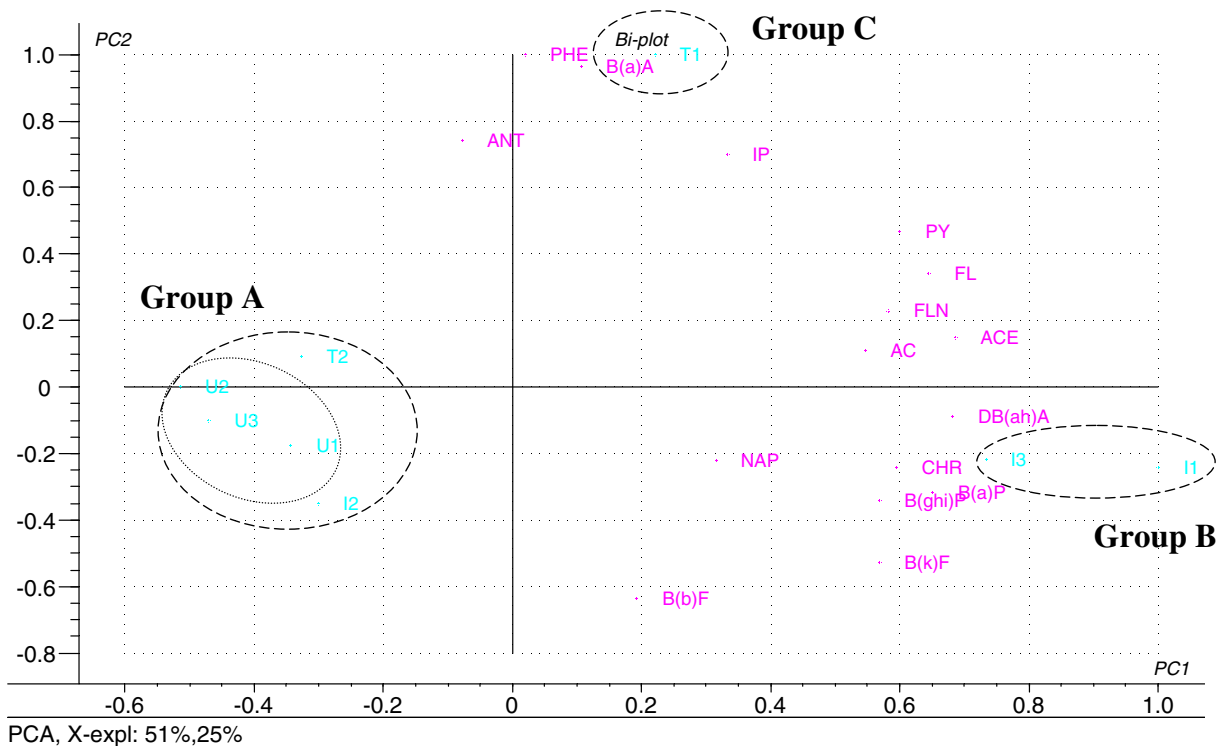


Fig. 6 Score plot of the first two PCs on PAH profiles (U urban site, I industrial site, T traffic site (cf Table 1))

(a)A and separate efficiently group C between the two other ones. The plot show that the samples of Group B have strong CHR, B(a)P and DB(ah)A intensities.

Summary and conclusion

The objective of this sampling campaign was to study the concentrations of sixteen PAHs at three representative sites in the Toulouse agglomeration: that of the CCIT which is an urban or so-called « automobile traffic » site, Berthelot which is a « typical » urban site, and that of Eisenhower, a site close to an industrial area.

The atmospheric PAH concentrations found at these three sites of the Toulouse agglomeration in April and May 2006, mirror those concentrations found in most European cities, which at present have total concentrations of around 25 ng/m³ in winter and 5 ng/m³ in summer (Masclat et al. 2005). Our results for the two urban sites in fact give concentrations between 12 and 20 ng/m³ included, and for the site close to an industrial area the total atmospheric concentrations are an average 22 ng/m³.

In addition, regardless of site, the average benzo(a)pyrene concentrations, at present the only PAH actually regulated, are always less than the 1 ng/m³ limit.

Nonetheless, in view of the seasonal variations in atmospheric PAH concentrations, it would be interesting to continue this study particularly during the winter period.

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