



# The use of conifer needles as biomonitor candidates for the study of temporal air pollution variation in the Strasbourg region



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## H I G H L I G H T S

- *Pinus nigra* and *Cedrus atlantica* were used as the biomonitor candidates for environmental pollution in Strasbourg.
- Both conifer species accumulated pollutants in the same order of magnitude.
- PAHs were the most accumulated pollutant by the conifer needles.
- The highest accumulation was obtained in mild weather in which no precipitation was observed.

## A R T I C L E I N F O

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## A B S T R A C T

The continuous emission of polluting chemicals into the atmosphere requires the implementation of monitoring of ambient air quality. The use of vegetation for environmental monitoring can be considered as a simple monitoring technique by providing a cheap and accessible matrix. In this study, needles of two conifers (*Pinus nigra* and *Cedrus atlantica*), were used for the consecutive biomonitoring of multipollutants such as pesticides, polychlorinated biphenyls, and polycyclic aromatic hydrocarbons (PAHs) in an urban area in Strasbourg (France). The extraction was performed by accelerated solvent extraction, solid-phase extraction, and solid-phase microextraction and was followed by gas chromatography coupled to tandem mass spectrometry and liquid chromatography coupled to tandem mass spectrometry analyses. The results obtained for conifer samples collected in 5 successive weeks (April 09–May 07, 2015) show a similar variation of the different types of pollutants. A pollution peak was observed during the second week of analysis, and the concentration of all pollutants then decreased to complete disappearance at the end of the sampling period. PAHs were the most concentrated with a total concentration of about 35.87 ng g<sup>-1</sup>, and naphthalene was, among these pollutants, the most concentrated with a total concentration of about 15.1 ng g<sup>-1</sup>. The analysis of meteorological data during this period suggests that the results correlated with climatic conditions that widely vary during this period of the year. The results show that the concentration peak was obtained when no precipitation was detected.

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## 1. Introduction

The continuous emission of hazardous chemicals into the atmosphere makes it important to estimate their concentrations to determine the quality of ambient air. In fact, the undesirable effects

of environmental pollution have been widely reported (Barker et al., 2002; Terzano et al., 2010; Rodríguez et al., 2016; Malmqvist et al., 2017; Wing et al., 2017). According to the World Health Organization (WHO), about 3.7 million people die each year because of the toxic effects of this pollution (2014).

The use of pesticides to protect crops from various adverse effects can lead to serious environmental problems because of the persistence of pesticide residues in various environmental matrices (Sharma et al., 2014). Furthermore, pesticides can be

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often accumulated in the environment, leading to several harmful disruptions, and hence, most of them have been banned worldwide (Zheng et al., 2016). This is particularly the case for organochlorine pesticides (OCPs) that have been widely used worldwide because of their agricultural and industrial advantages (Xu et al., 2010). The use of OCPs is subjected multiple regulations because of their toxicological effects, mainly endotoxic, neurotoxic, and carcinogenic, especially dichlorodiphenyltrichloroethane (DDT) and Hexachlorocyclohexane (HCH), and their use has been either banned or strictly limited in many countries (van den Berg, 2009; Jit et al., 2011).

Moreover, among the various, organic atmospheric pollutants, polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) are a series of hazardous organic pollutants of special concern because of their environmental persistence. These pollutants are widespread in the environment because of the multiplicity of their sources (Sarria-Villa et al., 2016). PAHs are mainly emitted into the atmosphere through combustion processes; they are produced as by-products of an incomplete combustion of organic matter, volcanic eruption, forest fires, and vehicle emissions (Pongpiachan et al., 2013; Abdel-Shafy and Mansour, 2016; Elorduy et al., 2016; Li et al., 2016). In fact, PAHs, because of their atmospheric persistence, are a great concern, because they are known for their carcinogenic and mutagenic properties (Kamal et al., 2014). In addition, the most toxic PAHs are those with five or more benzene rings, known as high-molecular-weight PAHs (HMW PAHs). They are considered as the most toxic, mutagenic, and carcinogenic. Especially in urban areas, they are responsible for several respiratory diseases and cancer of skin, lungs, and bladder in humans (Alagić et al., 2016; Elorduy et al., 2016; Li et al., 2016). PCBs are artificial organic chlorinated compounds widely used as fluids in transformers and capacitors. They were widely produced and used until they were banned by the United States Congress in 1979 and by the Stockholm Convention on Persistent Organic Pollutants in 2001 (Porta and Zumeta, 2002). The lipophilic nature of these compounds contributes to their high potential of bioaccumulation in food chain (Chamkasem et al., 2016). Because of the toxicity of these entire class of compounds, many countries abided the convention to limit the damage caused through uncontrolled use of these persistent organic pollutants (POPs) (Elabbas et al., 2013).

These pollutants need to be monitored to assess the state of outdoor environment. In fact, the use of active sampling for environment monitoring was the method of choice for many years. This sampling method uses a pump running at a defined low rate to aspirate air through a filter and/or an adsorbent bed (Tuduri et al., 2012). Even though these methods are well known for their reliable data, they also suffer from numerous drawbacks such as expensive solvents and equipment, and much time and energy consumption (Wang et al., 2009). As an alternative for these pumps, passive samplers were developed. In this technique, pollutants adsorb on a chemical substrate such as polymeric resin, polyurethane foam, and thin films of ethylene vinyl acetate (Farrar et al., 2005; Hazrati and Harrad, 2007; Wang et al., 2009; Tuduri et al., 2012). These samplers permit environment monitoring without pumping and therefore without harmful sounds and energy requirement. Furthermore, a natural substance such as vegetation can be a perfect substitute for both passive and active samplers; this method is known as biomonitoring. Biomonitoring permits to estimate the environmental pollution through a natural substance, i.e., vegetation, thereby allowing qualitative and quantitative assessment of the presence of pollutants. Moreover, vegetation is especially an advantageous functional matrix for the assessment of these airborne pollutants (Ratola et al., 2014). Indeed, the use of vegetation for environment monitoring remains the cheapest, most

available, and simplest matrix for atmospheric monitoring (Klánová et al., 2009). In addition, many studies reported the use of vegetation as the passive samplers of POPs and chemical compound in the atmosphere to identify the pollution sources and to determine local and global contamination patterns (Schulz et al., 1999; Gerdol et al., 2002; St-Amand et al., 2009; Ratola et al., 2010).

Among the different vegetation species, conifer needles can play an important role as the passive samplers. Conifers can accumulate pollutants over the years; they are widespread and can be found over a large and poorly accessible area (Romanič and Krauthacker, 2007; Ratola et al., 2014). Furthermore, conifer needles are characterized by a high affinity toward low or medium polar compounds because their high wax leaf content, which allows them to retain pollutants over the years. Different studies show that conifer needles are effective in the monitoring of pesticides (Ratola et al., 2014) and organic semi-volatile pollutants such as PAHs (Ratola et al., 2006; Amigo et al., 2011), PCBs (Grimalt and Van Drooge, 2006; Al Dine et al., 2015), and OCPs (Hellström et al., 2004).

The extraction of pollutants from such matrix needs particular attention, because the aim is to obtain a full recovery of the analytes without co-extraction or involuntary compounds. Several extraction procedures are reported as providing good recovery and efficiency, such as Soxhlet (Ratola et al., 2006), ultrasonic solvent extraction (Tomashuk et al., 2012), or pressurized liquid extraction (Ratola et al., 2006). All these methods are, however, time and solvent consuming and require expensive experiments (Silva et al., 2015).

In the present study, a new method combining accelerated solvent extraction (ASE), purification by solid-phase extraction (SPE) followed by gas chromatography coupled to tandem mass spectrometry (GC-MS/MS) and liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS) was used to analyze 30 non-volatile pesticides, 41 volatile pesticides, 16 PAHs, 22 PCBs and 20 OCPs in one sample. All previous studies focused on the use of GC-MS/MS for the analysis of volatile or semi-volatile pollutants, and none of them have reported the use of the LC-MS/MS for the analysis of non-volatile persistent pesticides. The developed method permits the analysis of about 129 different organic compounds in one single sample. This method was then applied to two different conifer species (*Pinus nigra*, reported by many authors for its passive sampler role (Piccardo et al., 2005; Kalinovic et al., 2016), and *Cedrus atlantica*, (which to our knowledge, has not been reported previously), to study their role as the biomonitors of temporal air pollution variation in the Strasbourg region. It also aimed to demonstrate if the meteorological conditions affect needles contamination given that the study was conducted in early spring 2015 in which the weather broadly varied.

## 2. Methods and materials

### 2.1. Chemical reagents and standard solutions

LC-MS-grade acetonitrile (ACN), Toluene (TOL) from Biosolve (Dieuze, France), HPLC-grade acetonitrile, methanol (MeOH), ethyl acetate (EA), "Fontainebleau" sand from Prolabo (France), silica gel (Merck, Germany) and ultrapure water (Elga system, Antony, France) were used.

Internal standards for LC-MS/MS, namely Carbazepim-d4 (99.3%), Diuron-d6 (99.8%), Pendimethalin-d5 (99%), and Nicosulfuron-d6 (99%), were obtained from CDN isotopes (Quebec, Canada).

Internal standards for GC-MS/MS were Trifluralin-d14, and 4-Nitrophenol-d4 and Naphtalene-d8 (99%), and they were purchased from Sigma-Aldrich (L'Isle d'Abeau, France) and Cambridge Isotope Laboratories (Cluzeau Info Labo, France), respectively.

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