



# Compositional variance in extracted particulate matter using different filter extraction techniques



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## HIGHLIGHTS

- Ability to provide exhaustive chemical characterization of single PM extract.
- Significance of directly characterizing extracted PM for toxicological testing.
- Existence of substantial compositional biases between different extraction methods.
- Importance of standardizing PM extraction objectives and procedures.

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## ABSTRACT

Collection and subsequent extraction of particulate matter (PM) from filter substrates is a common requirement for in vivo and in vitro toxicological studies, as well as chemical analyses such as ion chromatography and inductively coupled plasma mass spectrometry. Several filter extraction protocols exist and different laboratories employ different methods, potentially biasing inter-study comparisons. Previous studies have shown significant differences in extraction efficiency between techniques and identified the relevant extraction artifacts. However, a comprehensive inter-comparison of different methods based on the chemical composition of the extracted PM has never been conducted. In the current study, an exhaustive suite of chemical analyses is performed on PM extracted from glass micro-fiber filters using techniques commonly employed in different laboratories: Multi-solvent extraction (MSE) and spin-down extraction (SDE). PM samples were collected simultaneously during field studies conducted in an urban and rural setting using a high-volume PM<sub>2.5</sub> sampler. Results show remarkable compositional variance between the PM extracts for all chemical components analyzed, including metals, water soluble ions, polycyclic aromatic hydrocarbons, non-aromatic organics, elemental carbon and organic carbon. Mass closure was greater than 90% for MSE but deviated substantially for SDE. Detailed retrospective gravimetric analysis of archived SDE samples revealed that a process-based loss of PM mass is the root cause of the differences. These losses are shown to be compositionally biased, both externally between different PM mixtures and internally within a given PM mixture. In combination, the results of this study are the first to demonstrate (i) an exhaustive chemical characterization of a single PM extract, (ii) the significance of directly characterizing the extracted PM used in toxicological studies, (iii) the existence of substantial compositional biases between different filter extraction techniques and (iv) the importance of standardizing filter extraction objectives and procedures to avoid introducing study bias into toxicological studies.

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## 1. Introduction

In air quality science, particulate matter (PM) is commonly sampled from an environment – e.g. the ambient atmosphere,

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smokestacks, building interiors and laboratory generated exhaust streams – by drawing air across a filter, or some other type of substrate, to separate the PM from the gases. PM collected on filters can then be analyzed for composition using various analytical techniques and/or toxicity using *in vivo* and *in vitro* systems. In almost all cases, the PM must be removed, or extracted, from the filter prior to analysis. Depending on the objective, the filter extraction process can be exhaustive – i.e., maximizing the amount of total PM removed from the filter – or selective, i.e. extracting only certain PM components or compound classes.

For toxicological studies, the primary objective of filter extraction is to conserve, as much as possible, the physical and chemical properties of the PM as it originally existed in the atmosphere or exhaust stream – including particle size, number concentration, morphology and individual particle compositional and structural integrity – so that the results of these studies are representative of true population exposure. Currently, the most widely applied filter extraction technique involves sonication in ultra-pure water followed by lyophilization to remove the water and recover dry PM (Devlin, 2009; Bowser, 2009). The PM is then suspended in the delivery vehicle and sonicated and/or vortexed immediately prior to instillation or aspiration. Extraction efficiencies – i.e. the mass of PM removed by extraction relative to the mass of PM collected on the filter – on the order of 60–70% are commonly reported for this technique and this efficiency may be compositionally biased. Recently, an exhaustive, multi-solvent extraction (MSE) technique including sonication, liquid–liquid extraction, selective filtration and solvent removal was introduced, resulting in extraction efficiencies consistently exceeding 90% (Bein and Wexler, 2014).

Chemical composition studies, however, require a suite of analytical techniques given the chemical complexity of PM and each technique measures a certain class of compounds; e.g. metals via Inductively Coupled Plasma Mass Spectrometry (ICP-MS), inorganic ions via Ion chromatography (IC) or polycyclic aromatic hydrocarbons (PAHs) via Gas Chromatography Mass Spectrometry (GC-MS). In this case, the goal of filter extraction is to selectively extract certain compounds while minimizing co-extraction of potential interfering species to eliminate matrix effects – i.e. in complex multi-component systems, the presence of certain components can interfere with the detection of others, either synergistically or antagonistically – and thus each analytical technique typically requires its own filter extraction protocol. For example, trace element analysis via ICP-MS requires an initial organic solvent extraction followed by acid digestion using a strong acid (Herner et al., 2006). The initial organic solvent extraction is necessary for most combustion generated aerosol and/or secondary organic aerosol (SOA) since (i) the trace metals are typically encapsulated by layers of organic compounds and (ii) most organic compounds are hydrophobic and thus are not likely removed from the filter to any significant degree by water alone. Once the organic layers are removed, acid digestion dissociates the metal oxides and salts, bringing the metal ions into solution for analysis. Similarly, there are several different sample preparation protocols for molecular speciation of particulate organic carbon via GC-MS that are based on organic solvent extraction followed by post-extraction cleanup steps to dissolve the organics into solution and separate them from the particle matrix (Schauer et al., 1996, 1999; Sheesley et al., 2004; Fine et al., 2001, 2004; Ham and Kleeman, 2011).

The possible existence of toxicological matrix effects – an extrapolation of the idea of chemical matrix effects – is a new concept that is largely unstudied. For particle toxicity, the basic idea is that the sum of endpoint-specific toxicological responses to individual PM components may be different than the response to the composite of those components, i.e. the presence of endpoint-specific toxicologically inert PM components may interfere with

the response to the toxicologically active PM components. This may further depend on the physical form in which the components are present; e.g., dissolved in solution, individual particles or particle aggregates. In this context, toxicological response may vary significantly depending on the filter extraction technique employed. A filter extraction technique designed according to one set of objectives may inadvertently alter the composition of the particle mixture in such a manner as to enhance or inhibit toxicological response relative to another technique designed with a different set of objectives. The current study was designed to test this hypothesis.

Separate filter extraction techniques commonly used in different laboratories and designed with different sets of objectives were used to extract ambient PM collected simultaneously from an urban and rural sampling site using high-volume PM<sub>2.5</sub> sampler systems. The extracted PM was exhaustively characterized both chemically and toxicologically using a suite of analytical techniques and toxicological endpoints. A comprehensive inter-comparison of the filter extraction techniques based on the chemical composition of extracted PM is presented in what follows. Results from the toxicological studies are published separately (Van Winkle et al., *in press*). Overall, and to the authors' knowledge, this is the first study to (i) provide an exhaustive chemical characterization of a single PM extract, (ii) analyze the same PM extracts as used in subsequent exposure studies and (iii) inter-compare different filter extraction techniques in terms of the chemical composition of extracted PM.

## 2. Methodology

### 2.1. PM sampling

Field studies were conducted simultaneously during winter 2011 at two separate sampling sites representing an urban and rural environment using PM<sub>2.5</sub> high-volume sampler systems (Tisch Environmental Inc., TE-6070V-2.5-HVS) equipped with PM<sub>10</sub> size selective heads (Tisch Environmental Inc., TE-6001), operating at a flow rate of 40 cfm and loaded with aluminum foil substrates for collecting the coarse PM fraction ( $PM_{10-2.5} = 2.5 < D_{p50} < 10 \mu\text{m}$ ) and Teflon coated borosilicate glass microfiber filters (Pall Corporation, TX40H120WW-8X10) for collecting the fine PM fraction ( $PM_{2.5} = D_{p50} < 2.5 \mu\text{m}$ ). Aluminum foil substrates were pre-baked at 500 °C for 24 h and glass microfiber filters were pre-cleaned via successive sonication in milli-Q H<sub>2</sub>O, dichloromethane (DCM) and hexane (Hx). Field blanks were included for all studies. The **urban** sampling site was located on the rooftop of a two story building at the northeast corner of T St. and 13th St. in downtown Sacramento, CA, surrounded by a mixture of residential, commercial and industrial sources and within a quarter mile of a major freeway interchange. The **rural** site was situated on top of a single story laboratory in the southeast corner of the Center for Health and the Environment complex on the south campus of U.C. Davis and surrounded by agricultural lands. PM<sub>2.5</sub> filter samples and field blanks from both sites were extracted using two different filter extraction techniques detailed below and the extracts subjected to an exhaustive chemical characterization using a range of analytical techniques, as discussed later.

### 2.2. Filter extraction techniques

In total, five different filter extraction techniques commonly employed by different groups, including the US Environmental Protection Agency, were prescreened via qPCR analysis of THP-1 monocyte cell line response to the extracted PM samples using a six panel assay, including inflammatory mediators and PAH

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