



Non-target analysis of household dust and laundry dryer lint using comprehensive two-dimensional liquid chromatography coupled with time-of-flight mass spectrometry



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HIGHLIGHTS

- First non-target analysis of household dust and dryer lint samples.
- First application of LC × LC-ToF MS for indoor environmental analysis.
- Establishment of rapid identification and confirmation approaches.
- Traditional and novel environmental contaminants were found in house dust and dryer lint.

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ABSTRACT

Household dust and laundry dryer lint are important indoor environmental matrices that may have notable health effects on humans due to chronic exposure. However, due to the sample complexity the studies conducted on these sample matrices until now were almost exclusively on the basis of target analysis. In this study, comprehensive two-dimensional liquid chromatography coupled with time-of-flight mass spectrometry (LC × LC-ToF MS) was applied, to enable non-target analysis of household dust as well as laundry dryer lint for the first time. The higher peak capacity and good orthogonality of LC × LC, together with reduced ion suppression in the MS enabled rapid identification of environmental contaminants in these complex sample matrices. A number of environmental contaminants were tentatively identified based on their accurate masses and isotopic patterns, including plasticizers, flame retardants, pesticides, drug metabolites, etc. The identity of seven compounds: tris(2-butoxyethyl) phosphate, tris(2-chloropropyl) phosphate, n-benzyl butyl phthalate, dibutyl phthalate, tributyl phosphate, triethyl phosphate and N, N-diethyl-meta-toluamide was confirmed using two-dimensional retention alignment and their concentrations in the samples were semi-quantitatively determined.

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1. Introduction

The indoor environment has increasingly gained attention as an important source of human exposure to environmental contaminants, such as polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), organophosphate flame retardants (OPFRs), poly- and perfluoroalkyl substances (PFASs), plasticizers and pesticides (Björklund et al., 2009; Carlsson et al., 1997; Harrad et al., 2008,

2006; Rudel et al., 2003). The exposure in the indoor environment can be as much as 1000-fold higher compared to the outdoor environment, due to relatively longer residence time, poorer ventilation and slower degradation of contaminants (Smith, 1988). The pollution in the indoor environment is believed to be able to contribute to respiratory diseases, cancer, neuropsychological disorders, etc (Bornehag et al., 2004; Samet et al., 1988, 1987). The potential adverse health effects are expected to be more severe for infants and children due to their frequent hand to mouth contact, resulting in higher intake of dust which can contain high amounts of contaminants, indicating a higher vulnerability.

Dust is one of the most frequently studied matrices in the indoor environment (Bornehag et al., 2005; Mannino and Orecchio, 2008;

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Shoeib et al., 2005; Wilford et al., 2005). It is a mixture of fibers, dead skin cells, bugs, soil particles, residues of furniture, electronics and other domestic consumer products. Due to the complexity of the matrix, studies on indoor dust are almost exclusively limited to targeted analysis of a specific group of compounds, and based on gas chromatography coupled to mass spectrometry (GC-MS) or liquid chromatography coupled to mass spectrometry (LC-MS) (Canosa et al., 2007; Mannino and Orecchio, 2008; Shoeib et al., 2005; Van den Eede et al., 2011). A method for non-target screening of environmental contaminants in indoor dust using comprehensive two-dimensional gas chromatography (GC \times GC) coupled to time-of-flight mass spectrometry (ToF-MS) was described by Hilton et al. (2010). Using such a comprehensive approach, not only the compounds reported to be present in the dust sample by the National Institute of Standards and Technology (NIST) were detected, but also the presence of several other compounds, e.g. biphenyls and sulfur, nitrogen, and oxygen analogs of PAHs, was observed.

Comprehensive two-dimensional liquid chromatography coupled to time-of-flight mass spectrometry (LC \times LC-ToF MS) is an emerging analytical technique that has shown its strength in resolving complex environmental matrices such as wastewater treatment plant (WWTP) effluents (Haun et al., 2013; Ouyang et al., 2015, 2016). The major advantage of LC \times LC in environmental analysis compared to one-dimensional LC is the greater peak capacity, multi-selectivity and, therefore, the reduced matrix effect resulting in ion suppression in the MS interface. Furthermore, high resolution MS and two-dimensional retention time alignment strongly support the identification of unknown environmental contaminants. Therefore, LC \times LC-ToF MS is a very useful tool for non-target screening of complex environmental samples.

As an alternative matrix for house dust, laundry dryer lint may be used as a proxy for human exposure. The dryer lint contains synthetic fibers, cotton, human hairs and probably residues of laundry detergent. Several studies have been conducted to investigate lead, polybrominated diphenyl ethers (PBDEs), bisphenol A and dioxins in laundry dryer lint (Berry et al., 1993; Loganathan and Kannan, 2011; Mahaffy et al., 1998; Stapleton et al., 2005). However, similar to indoor dust, non-target analysis of this matrix is expected to significantly add to the knowledge on the occurrence of (toxic) chemicals in laundry dryer lint.

In this study, LC \times LC-ToF MS was applied to perform non-target screening of environmental contaminants in indoor dust and dryer lint. In order to achieve a better extraction efficiency of emerging environmental contaminants, a new extraction method based on a previously reported study of PFASs analysis in dust (Fraser et al., 2013) was implemented. Selection of the most interesting samples for LC \times LC-ToF MS was done using an acetylcholinesterase (AChE) inhibition assay.

2. Materials and methods

2.1. Chemicals and materials

Perfluorooctanesulfonic acid (PFOS), perfluorononanoic acid (PFNA), perfluorohexane sulfonate (PFHxS), tris(1,3-chloroisopropyl) phosphate (TDCiPP), tris(2-chloropropyl) phosphate (TCPP), *N,N*-diethyl-meta-toluamide (DEET), dithiobisnitrobenzoic acid (DTNB, Ellman's reagent) and acetylthiocholine iodide (ATC) were purchased from Fluka (Zwijndrecht, the Netherlands). Standards of tris(2-chloroethyl) phosphate (TCEP), tris(2-butoxyethyl) phosphate (TBOEP), tributyl phosphate (TBP), triethyl phosphate (TEP), dicyclohexyl phthalate (DCHP), pirimicarb, isoprocarb, propoxur, varenicline and purified acetylcholinesterase (AChE) from electric eel (*Electrophorus electricus*) were

obtained from Sigma-Aldrich (Zwijndrecht, the Netherlands). Triisobutyl phosphate (TiBP) was purchased from Merck (Darmstadt, Germany). Trimethacarb and 3,5-xylyl methylcarbamate (XMC) were purchased from LGC standards (Teddington, UK). The standards of *n*-benzyl butyl phthalate (BBzP) and dibutyl phthalate (DBP) were purchased from Riedel de Haën (Seelze, Germany). The EPA SRM 2585 house dust was from the National Institute of Standards and Technology (NIST, Gaithersburg, MD, USA). The EPA 531.1 carbamate pesticide calibration mixture was purchased from Restek (Bellefonte, PA, USA). Acetonitrile was HPLC grade supplied by Sigma-Aldrich. HPLC water was obtained from a Milli-Q Reference A+ purification system (Millipore, Bedford, MA, USA). Formic acid added to the eluent was ordered from Fluka.

2.2. Instrumentation

The comprehensive LC \times LC system deployed an Agilent 1100 HPLC binary pump as the first dimension, an Agilent 1290 Infinity UHPLC binary pump as the second dimension together with an Agilent 1100 auto sampler and an Agilent 1290 Infinity thermostatted column compartment (TCC) with a 2-position/4-port duo valve and two sampling loops (80 μ L) installed as the 2D interface (Agilent Technologies, Waldbronn, Germany). The LC \times LC module was controlled and monitored by Chemstation version B.04.03 (Agilent Technologies) with 2D-LC add-on. A ZORBAX Eclipse Plus (1.8 μ m, 2.1 \times 150 mm ID) C18 Rapid Resolution HD column (Agilent Technologies, Santa Clara, CA, USA) was used in the first dimension and a Kinetex pentafluorophenyl (PFP) column (2.6 μ m, 50 \times 4.6 mm ID, Phenomenex, Torrance, CA, USA) in the second dimension. After the second column, in order to adapt an optimal flow for the coupled ToF-MS (Bruker micrOTOF, Bremen, Germany), resolving power \sim 10,000 with an electrospray (ESI) interface, a QuickSplit adjustable flow splitter (Richmond, CA, USA) was applied which directed 20% of the flow to the MS detector. The remaining 80% of the flow was sent to the waste.

2.3. Sample collection

The five dust samples were collected using a Dustream™ dust collector (Indoor Biotechnologies Ltd., Wiltshire, United Kingdom) containing a disposable filter (mesh size 40 μ m) and attached on a household vacuum cleaner tube, from five different families in the region of Uppsala, Sweden. The dust collected was so-called still standing dust, from surfaces little influenced by daily life, e.g. no walking and not containing e.g. bread crumbs and soil. The six dryer lint samples were collected from two households in the Netherlands. The lint was taken gently from the filter sheets of the laundry dryers and then wrapped in aluminum foil before extraction and cleanup.

2.4. Sample extraction and cleanup

To better fit the LC \times LC-ToF MS based method, the extraction and cleanup procedure was carried out using polar solvents methanol and acetonitrile modified from Fraser et al. (2013). Briefly, 50 mg of sample (SRM 2585, house dust or dryer lint) was weighed in a 15 ml disposable polypropylene tube and 5 ml of methanol was added. The tube was vortexed for 1 min, followed by ultra-sonication for 15 min and centrifugation for 5 min at 1500 rpm. The supernatant was transferred to a new polypropylene tube and the same extraction steps were performed on the residue using acetonitrile. The supernatants were combined and filtered over an Envicarb SPE cartridge (Supelco, Zwijndrecht, Netherlands) activated with two portions of 5 ml methanol and acetonitrile (1:1 v/v). The SPE cartridges were rinsed with 0.5 ml methanol and

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