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A new analytical protocol for the determination of 62 endocrine-disrupting compounds in indoor air

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ABSTRACT

The objective of this study was to develop and validate a new analytical protocol for simultaneous determination of 62 semi-volatile organic compounds in both phases of indoor air. Studied compounds belong to several families: polybrominated diphenyl ethers, polychlorinated biphenyls, hexachlorobenzene, pentachlorobenzene, phthalates, polyaromatic hydrocarbons, parabens, tetrabromobisphenol A, bisphenol A, hexabromocyclododecane, triclosan, alkylphenols, alkylphenol ethoxylates, synthetic musks (galaxolide and tonalide) and pesticides (lindane and cypermethrin). A medium volume sampling system was used to collect simultaneously these endocrine-disrupting compounds (EDCs) from the gaseous and particulate phases. An accelerated solvent extraction method was optimized to obtain all EDCs in a single extract by atmospheric phase. Their extraction from the sorbents and their analysis by liquid and gas chromatography–mass spectrometry (LC/MS/MS, GC/MS and GC/MS/MS) was validated using spiked sorbents (recovery study and analytical uncertainty analysis by fully nested design). The developed protocol achieved low limits of quantification (< 0.5 ng m^{-3}) and low uncertainty values (< 5 ng m^{-3}) for all compounds. Once validated, the method was applied to indoor air samples from four locations (a house, an apartment, a day nursery and an office) and compared to literature to confirm its efficiency. All target EDCs were quantified in the samples and were primarily present in the gaseous phase. The major contaminants found in indoor air were, in descending order, phthalates, synthetic musks, alkylphenols and parabens.

1. Introduction

Over the last decade, evidence has accumulated concerning the potential adverse effects of exposure to environmental chemicals that interact with the endocrine system [1–3]. Humans are chronically exposed to many compounds at low levels in water [4,5], air [6,7], and food [8,9]. Furthermore, adults and children spend much of their time indoors, where the air is often more contaminated than outdoors [10,11]. Various factors can explain the increasing accumulation of contaminants in indoor environments, such as the rapid development of new building materials, furnishings and consumer products and lower air exchange rates for improved energy efficiency [12]. Moreover, air inhalation represents a chronic and passive exposure route for environmental pollutants. Therefore, characterization of indoor air contamination remains an important public health issue.

Many indoor contaminants have been identified [12,13]. Some are endocrine-disrupting compounds (EDCs), which may cause endocrine disorders in animals and humans [14]. Many known or suspected EDCs can be found in the indoor air environment due to their semi-volatility, including flame retardants [15,16], surfactants [17,18], plasticizers [19,20], bactericides [21,22], or synthetic musks [23,24]. Given the health hazard of EDCs and their possible environmental persistence at low doses, it is essential to precisely characterize human exposure to these contaminants.

Several analytical protocols have been developed for the quantification of semi-volatile organic compounds (SVOCs) in indoor air (Table 1). Most of them include an extraction step mainly based on Sohxlet method that may be solvent or time-consuming, or pretreatment steps that increase the sample preparation time (clean-up step) or need high sample amounts (derivatization step). The great majority of these studies were applied to one or two families of EDCs having similar polarities. The only ones that have analyzed a larger number of EDCs employed pretreatment steps or collected sample volumes too small to allow quantification of EDCs at low indoor concentrations (i.e polybrominated diphenyl ethers

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Table 1
Selected analytical protocols for EDCs analysis in indoor air.

Compounds studied	Atmospheric phase studied	Sampling method	Active sampling volume (m^3)	Extraction method	Pretreatment step	Analysis method	Ref.
HBCD TBBPA	Gaseous and particulate separately results in gas+particle	PUF+filter (passive and active sampling)	56	Soxhlet (hex/CH2Cl2, 1/9 v/v)	SPE (silica)	LC/MS/MS (reverse phase)	[6]
104 EDCs (63 detected)	Gaseous+particulate	XAD-2+PUF+filter (active sampling)	12	Soxhlet (hex+6% diethyl ether) shaking (DCM)	Drying with sodium sulfate derivatization	GC/MS	[35,40]
57 EDCs (34 detected)	Gaseous and particulate separately	PUF+filter (active sampling)	20	PLE (DCM)	Derivatization	GC/MS/MS GC/MS	[36]
PCB PBDE	Gaseous	PUF (passive sampling)	–	Soxhlet (hex)	Sulfuric acid+SPE (florisil)	GC/MS	[47]
56 EDCs	Gaseous and particulate separately	XAD-2+filter (active sampling)	136	Depression system (DCM)	SPE (florisil, silica/alumina, acidic silica/silica/alumina)	GC/MS GC/MS/MS LC/MS/MS (reverse phase)	[26]
Phthalates musks (10 detected)	Gaseous	PUF (active sampling)	2	PLE (hex/diethyl ether 95/5 v/v)	–	GC/MS	[37]
Musks	Gaseous	Tenax TA (active sampling)	5	–	–	GC/MS	[38]
Alkylphenols	Particulate	Solid phase extraction disk+filter (active sampling)	14.4	Ultrasonication (acetone)	Derivatization	GC/MS	[41]
PBDE TBBPA	Gaseous and particulate separately	PUF+filter (active sampling)	150	Soxhlet (hex/DCM)	–	GC/MS	[42]
PAH	Gaseous and particulate separately Results in gas+particle	PUF+filter (active sampling)	29	Static extraction (hex/DCM 4/1 v/v)	SPE (acidic silica)	GC/MS	[43]
PAH	Gaseous and particulate separately	XAD-2+filter (active sampling)	28.8	Soxhlet (hex/DCM 50/50 v/v) sonication (hex/DCM 50/50 v/v)	–	HPLC/FLUO (reverse phase)	[44]
PAH PCB PBDE Pesticides	Gaseous	PUF (passive sampling)	–	Soxhlet (DCM)	–	HPLC/FLUO (reverse phase) GC/MS	[45,46]
PBDE PCB	Gaseous and particulate separately results in gas+particle	PUF+filter (passive and active sampling)	2.7	Soxhlet (DCM)	Sulfuric acid SPE (sodium sulfate/aluminum oxide)	GC/MS	[48]
PBDE	Gaseous and particulate separately results in gas+particle	PUF+filter (active sampling)	9.1	PLE (petroleum ether or DCM)	Filtration	GC/MS	[49]
69 EDCs (57 detected)	Gaseous and particulate separately	XAD-2+filter (active sampling)	200	PLE (DCM/MeOH, 2/1 v/v)	–	GC/MS GC/MS/MS LC/MS/MS (reverse phase)	Present study

PLE: pressurized liquid extraction, hex: hexane, DCM: dichloromethane, SPE: solid phase extraction, PUF: polyurethane form, MeOH: Methanol.