

Simultaneous determination of chemicals in dust samples - an automated fractionation method

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During the past decades dust has been increasingly investigated in exposure studies since it has been identified as a relevant carrier of contaminants (HILTON & JONES 2010). The main classes of organic chemicals in dust are flame retardants (FRs), fluorinated compounds (PFASs), organophosphate esters (OPEs), phthalate esters (PEs), polychlorobiphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), pesticides and synthetic musks (MERCIER & GLORENNEC 2011). Given the fact that a broad range of compounds are present in dust, we developed an analytical method for the simultaneous determination of the aforementioned classes of contaminants. Ultrasonication extractions with mixtures of hexane and acetone at different percentages were tested to evaluate the most efficient extraction condition for the selected compounds.

The extraction was followed by a purification step in order to reduce the matrix complexity, to obtain a better signal/noise ratio, avoid fast injection needle clogging, and enhance the lifetime of the LC and GC columns. The purification was carried out by separating the extract into four fractions of increasing polarities using a Gilson GX-271 ASPEC™ system. Silica SPE cartridges (particle size 40µm, 60 Å pore diameter, 500mg/3mL) were used with a non-destructive clean-up (ethyl acetate and methanol) to ensure that target chemicals were not degraded. The solvents used in the fractionation were hexane, with a polarity index (P.I.) of 0.1, n-butyl chloride (P.I. 1.0), dichloromethane (P.I. 3.1) and a mixture of ethyl acetate:methanol (1:1) (P.I. 4.8). The fractions were analysed by GC- and LC-MS.

The developed extraction and fractionation method was validated by recovery tests and the feasibility of the procedure was assessed through the analysis of a standard reference material for dust, SRM 2585, which contains a considerable number of organic contaminants.

The application of the method was finally tested on real dust samples from vacuum cleaner bags.

Future research will involve the suspect and non-target screening of the fractions by GC and LC coupled to high resolution time of flight mass spectrometry. Both ESI and APCI sources will be used. The identification of the compounds will be performed by means of the "Multi target screening analysis" instrument software in which the identification of compounds is based on accurate mass, isotopic pattern and retention time.

The developed method is sensitive and selective for the simultaneous determination of various classes of contaminants and can be performed in a relatively short time while keeping the procedure simple.

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