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Development of a hollow fibre liquid-phase micro extraction method coupled with capillary electrophoresis/mass spectrometry for determining nitrophenolic compounds from atmospheric particles

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Nitrophenolic compounds present in the atmosphere gained a lot of attention as they are known for their negative effect on human health as well as for their phytotoxity being a cause for forest decline. Moreover, nitrophenols have the ability to absorb light in the range of near ultra violet to visible light, thus they are also contributing to the so-called brown carbon. Most of the available methods for determining nitrophenols in particulate matter are using organic solvents for extraction. Those methods are not applicable if one wants to focus only on the water-soluble fraction. Therefore, a method using a three-phase hollow fibre liquid-phase micro extraction (HF-LPME) was developed to enrich nine nitrophenolic compounds (2-Nitrophenol, 3-Nitrophenol, 4-Nitrophenol, 2-Methyl-4-nitrophenol, 3-Methyl-4-nitrophenol, 4-Nitrocatechol, 2,6-Dimethyl-4-nitrophenol, 2,4-Dinitrophenol, 3,4-Dinitrophenol) from aqueous extracts of atmospheric particles. Analysis was performed by capillary electrophoresis coupled with electrospray ionisation mass spectrometry (CE-ESI-MS). The background electrolyte composition was optimised to a 20 mM ammonium acetate buffer at pH 9.7 containing 15% methanol (v/v). Persistent peak tailing during electrophoretic separation was observed for 4-Nitrocatechol. Flushing the capillary with Ethylenediaminetetraacetic acid (EDTA) prior sample injection strongly improved the peak shape. Four extraction parameters (composition of organic liquid membrane, pH of acceptor phase, salting out effect, extraction time) and their effect on the analyte recoveries were examined. The HF-LPME consisted of 1.8 mL sample solution kept at pH 2 as donor phase and 15 μl 100 mM aqueous ammonia solution as acceptor phase inserted into a hollow fibre. Dihexyl ether was used to form a supported liquid membrane inside the pores of the hollow fibre. As a result low detection limits in the range of nmol L-1 were achieved and the developed method was found to be competitive with more established methods. Recoveries between 11 and 90% of aqueous standard solutions could be obtained with enrichment factors between 10 and 100. Interday and intraday repeatability were tested and found to be in an acceptable range for most compounds (6-15% and 7–10%, respectively). However, they were higher for 4-Nitrocatechol (59% and 48%) and 2-Nitrophenol (17% and 35%). The developed method was successfully applied to exemplary samples of atmospheric particulate matter from field experiments. Five out of nine investigated nitrophenols could be determined in atmospheric particles collected at the rural research station Melpitz (Saxony, Germany). The obtained results indicate that out of the investigated compounds 4-Nitrophenol is present at the highest concentrations followed by 4-Nitrocatechol. Furthermore, it was found that nitrophenols are present in higher concentrations in winter than in summer samples indicating that sources are more likely available in winter time.