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Use of gas chromatography-mass spectrometry for the assessment of the contamination caused by small concentrations of nitrophenols in soils and sediments

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Nitrophenols (NPs) are widely distributed environmental contaminants that can be present in soils and sediments due to the degradation of some pesticides (parathion and fenitrothion) or by accidental spilling in ammunition plants or storage places. This communication reports a rapid and sensitive procedure for the determination of the most common NPs in soils by using gas chromatography coupled to mass spectrometry (GC-MS) as the analytical technique. Ultrasound assisted extraction (UAE) was employed for the extraction of the NPs from the soil samples to an organic solvent. Next, the resulting UAE extracts were submitted to dispersive liquid-liquid microextraction (DLLME) for achieving an effective preconcentration. DLLME is an easy-to-carry out, environmentally friendly separation technique involving minimal amounts of organic solvents. Since the volatility of NPs is low, as a previous stage to the GC-MS measurement the compounds were derivatized using a simple "in-situ" acetylation procedure. The main parameters affecting the UAE stage, as well as the DLLME and derivatization steps, were investigated looking for maximum analytical signals. The optimized procedure provided extraction recoveries in the 72-86% range, with precision values (expressed as relative standard deviation, RSD) $\leq$ 12%, and detection limits ranging from 1.3 and 3.3 ng g-1, depending on the compound. 20 soil and sediment samples, from military, industrial and agricultural areas were analyzed by the studied procedure in order to check its applicability.