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Environmental

Supercritical Fluid Extraction of Energetic Nitroaromatic Compounds and Their Degradation Products in Soil Samples

Analytical Chemistry, **77**, 4241-4247 (2005)
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Tags
DB-200, environmental, soil, sludges & sediments

Abstract
This paper explores the use of supercritical fluid extraction (SFE), in combination with various analyte collection strategies, for extracting energetic nitroaromatic compounds and their degradation products from soil samples. The required selectivity has been achieved by a combination of an SFE program and active trapping. Several different collection strategies were tested, using a selection of liquids (methanol, toluene, methyl *tert*-butyl ether, acetonitrile), inert and solid-phase extraction materials (Nexus, Oasis, LiChrolut), and 1-cm liquid chromatography precolumns (porous graphitic carbon, PGC). The best results were obtained using SFE in combination with a PGC precolumn. This setup allows on-line cleanup of the extract, and comparable results were obtained using either GC-ECD or GC-chemical ionization-MS for confirmatory analysis. The time required for a complete analysis was less than 60 min, and only 1 mL of toluene was needed for a 0.5-g representative sample. In contrast, the EPA standard method 8330 required 18-h sonication and 20 mL of acetonitrile for a 4.0-g sample and further time for sample cleanup and HPLC analysis. The method presented here provides method detection limits in the low-nanogram range, with relative standard deviations lower than 7%. The optimized method has been compared and validated with EPA method 8330 in terms of efficiency parameters such as robustness, accuracy (trueness and precision), and capability of detection. The validation demonstrated that the two analytical methodologies give comparable performance for the determination of nitroaromatic compounds, but SFE is superior for analyzing amine degradation products.

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Printed in the UK
August 25, 2013

5991-3385EN

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