ABSTRACT

A method employing solid-liquid extraction with methanol and solid-phase extraction (SPE) clean-up step using Supelco Supelclean™ ENVI™-Carb 3 mL cartridges (0.25 g graphitized carbon adsorbent) followed by gas chromatography – mass spectrometry with negative chemical ionization (GC-NCI-MS) has been optimized and applied for determination of ultratrace concentrations of C_6 – C_{12} perfluorinated carboxylic acids (PFCAs) in soil samples. A sophisticated multifactorial statistic method, response surface methodology, employing 1/16 fractional factorial design and the face centered central composite design as well has been applied to find the significant parameters which influence the extraction procedure of PFCAs and SPE clean-up step and to set the optimum extraction and clean-up levels of eight parameters evaluated yielding the maximum extraction recovery of all PFCAs. The analyte extraction recoveries and the limits of detection and quantification have been obtained. The recoveries of individual PFCAs were within a range from 85 to 100 % for analyte spiked concentration level of 1.1 ng g^{-1} and within a range from 91 to 107 % for analyte spiked level of 2.1 ng g^{-1}. The values of limits of detection were 1.9 – 3.0 pg $g^{-1}$and limits of quantification 6.4 – 10.1 pg g^{-1}. This analytical method has been tested on determination of C_6 – C_{12} perfluorinated carboxylic acids in soil samples collected from nine different geographical locations in Prague and villages in the close neighbourhood. The concentrations determined were of the order of tens of pg g^{-1} and C_8 – C_{12} perfluorinated acids, except from C_9, occurred most often.

Subject words

Analytical chemistry, separation methods, gas chromatography

Keywords

Perfluorinated carboxylic acids, soil, gas chromatography