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液相萃取-高效液相色谱-串联质谱联用测定污泥中的全氟化合物

章涛1, 孙红文1*, ALDER Alfredo C.2, GERECKE Andreas C.3, M LLER Claudia3

1. 南开大学环境污染过程与基准教育部重点实验室, 天津 300071; 2. 瑞士联邦水科学与技术研究所, 杜本多夫 8600, 瑞士; 3. 瑞

Analysis of perfluorinated compounds in sludge by liquid extr performance liquid chromatography-tandem mass spectromet

ZHANG Tao1, SUN Hongwen1*, ALDER Alfredo C.2, GERECKE Andreas C.3, M LLER Claudia3

1. Key Laboratory of Pollution Processes and Environmental Criteria of Ministry of Education, Nankai University Institute for Environmental Science and Technology (EAWAG), Dübendorf 8600, Switzerland; 3. Swiss Federal (EMPA), Dübendorf 8600, Switzerland

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摘 要 全氟化合物是一种新型持久性有机污染物,污水处理厂是其一个主要污染来源。目前还没有建立起一种统一的污泥样品中全氟化方法。本文报道了一种基于液相萃取和高效液相色谱-串联质谱联用技术测定污泥中的7种全氟烷基羧酸及其2种不饱和氟调酸前体物基磺酸及其5种磺酰胺衍生物前体物的方法。实验对萃取剂(甲醇)的pH值、超声萃取温度与时间、洗脱剂体积进行了优化,确定了中性℃下超声萃取10 min, Envi carbon柱净化的前处理方法,并成功地应用于实际污泥样品中全氟化合物的测定。方法的回收率为74%饱和氟调酸除外),线性范围为0.1～20 μg/L (羧酸系列)及0.25～50 μg/L(磺酸系列)内线性关系良好(r2＞0.99),定量限为0.6～3重)。内标物质的使用可有效消除环境基质引起的仪器离子抑制现象,使定量更加准确。

关键词： 液相萃取 高效液相色谱-串联质谱 全氟化合物 活性污泥

Abstract： Perfluorinated compounds are emerging persistent organic pollutants. Wastewater treatment plar reported to be one of their primary sources. However, a standard analytical method for sludge samples has n set up. A novel and rapid analytical method based on the liquid extraction, followed by high performance liqui chromatography-tandem mass spectrometry (HPLC-MS/MS) was set up for 7 perfluorocarboxylic acids (PFCAs) precursors, 6:2 and 8:2 unsaturated flourotelomer carboxylic acids (FTUCAs), and 2 perfluoroalkyl sulfonates (and their precursors, 5 derivates of sulfonamide in sludge. The parameters of methanol extraction, including p sonication temperature and time, and eluent volume were optimized. The extraction method was optimized a extraction under sonication at 40 ℃ for 10 min, and it was successfully used for the analysis of perfluorinated compounds in the sludge samples. The linear calibration curves were obtained in the ranges of 0.1~20 μg/L f and 6:2/8:2 FTUCA and 0.25~50 μg/L for PFASs and their precusors with linear correlation coefficients larger t The recoveries of the target compounds ranged from 74% to 141% (except for FTUCAs) and the limits of quan ranged from 0.6 to 30 μg/kg (dry weight). The use of internal standards can well correct the ion suppression (enhancement) induced by co-eluting components present in the sample extracts, and improve the quantitativ

Keywords： liquid extraction high performance liquid chromatography-tandem mass spectrometry (HPLC-MS perfluorinated compounds activated sludge

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Corresponding Authors: 孙红文

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