

Analysis of Perfluorinated Chemicals in Sludge: Method Development and Initial Results

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Abstract. A fast, rigorous method was developed to maximize the extraction efficacy for ten perfluorocarboxylic acids and perfluorooctanesulfonate from wastewater-treatment sludge and to quantitate using liquid chromatography, tandem-mass spectrometry (LC/MS/MS). First, organic solvents were tested for extraction efficiency, including acetonitrile (ACN), methanol (MeOH), isopropanol (IPA), tetrahydrofuran (THF), and 50/50 ACN/MeOH (v/v). Among the extractants tested, 50/50 ACN/MeOH yielded the best results for our combined criteria of extraction efficacy and solvent-handling convenience. Second, chemical pretreatment prior to solvent extraction was tested with sodium hydroxide (NaOH), potassium hydroxide (KOH), hydrochloric acid (HCl), and potassium persulfate (K₂S₂O₈). Pretreatment with NaOH and HCl effectively recovered additional PFCs from the sludge, but KOH and K₂S₂O₈ digestion were less effective than no pretreatment. Third, cleanup methods were investigated with solid-phase extraction using HLB (hydrophilic-lipophilic balanced) and WAX (weak-anion exchange) stationary phases, and with ion-pairing. The HLB stationary phase yielded a slight edge over the other two cleanup strategies in terms of recoverable PFCs and chromatographic separation. Finally, the appropriateness of isotopically labeled PFCs for quantitating unlabeled PFCs using isotopic dilution in complex sludge extracts was evaluated by comparison to results obtained with the standard-addition method. A National Institute of Standards and Technology (NIST) domestic sludge (CRM 2781) was analyzed using our finalized method and compared with previously reported results.