This thesis is focused on optimization of preparation of aqueous environmental samples for determination of persistent pollutants – perfluorinated organic acids, which are potentially dangerous for living organisms. The goal of the thesis is optimization of SPE conditions for preconcentration of these compounds for their determination by GC-MS. Perfluorinated organic acids with carbon chain lenghts of C_6-C_{12} were selected as analytes. Effect of sample pH value, effect of type and elution solvent volume, addition of indifferent salt and ion pair reagent have been studied during the optimization. The results show that the extraction efficiency depends on analyte carbon chain length and reaches values from 75 to 110 % for C_6 – C_8 and 55 to 95 % for C_9 - C_{12} acids. The overall increase in extraction efficiency was more pronounced for acids with shorter chain length (for C_6 up to ten times), whereas for long chain acids the improvement was only moderate.