

**Microscale solid-phase microextraction probe for *in situ* analysis of perfluoroalkyl substances and lipids in biological tissues using mass spectrometry**

Yunyun Yang,<sup>\*a</sup> Jiewei Deng,<sup>\*b</sup> Yaohui Liu,<sup>a</sup> Kaili He,<sup>a</sup> Zhangmin Xiang<sup>a</sup> and Tiangang Luan<sup>b</sup>

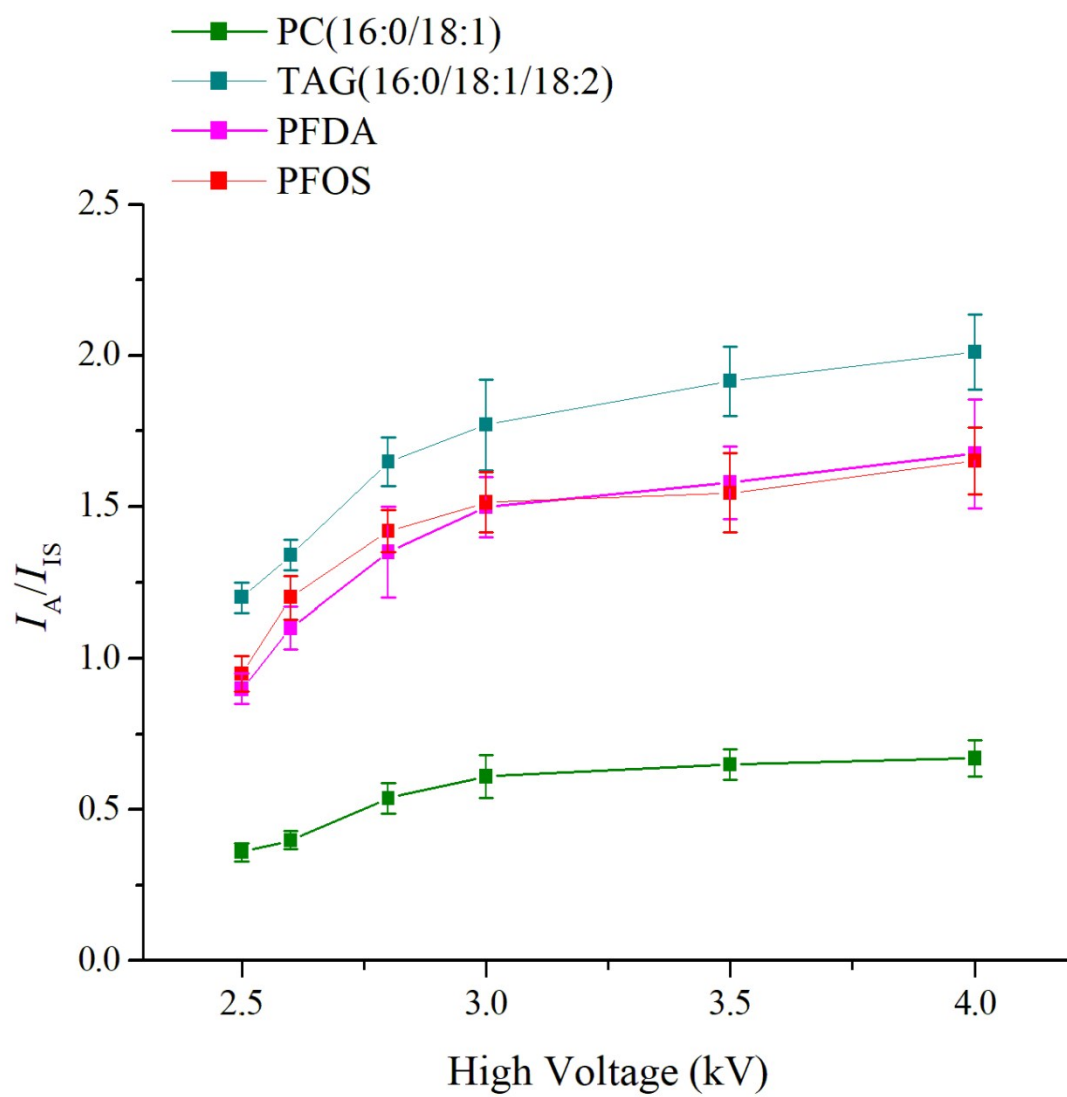
<sup>a</sup>Guangdong Engineering and Technology Research Center for Ambient Mass Spectrometry, Guangdong Provincial Key Laboratory of Emergency Test for Dangerous Chemicals, Guangdong Institute of Analysis (China National Analytical Center Guangzhou), 100 Xianlie Middle Road, Guangzhou 510070, China.

<sup>b</sup>State Key Laboratory of Biocontrol, South China Sea Bio-Resource Exploitation and Utilization Collaborative Innovation Center, School of Life Sciences, Sun Yat-Sen University, 135 Xingangxi Road, Guangzhou 510275, China.

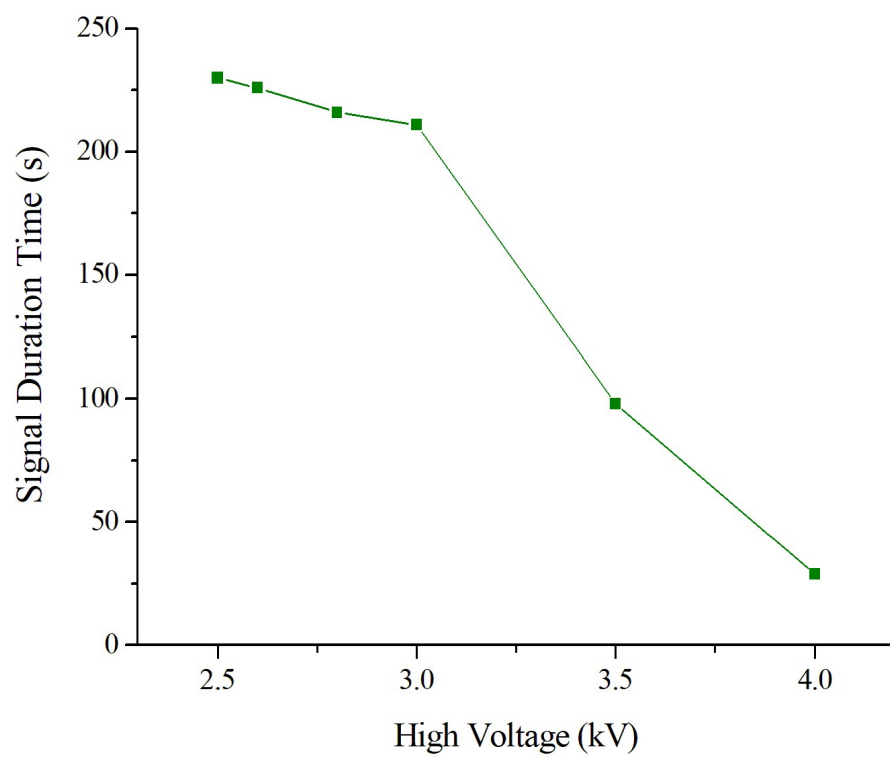
Corresponding author:

E-mail: yy\_yang@vip.126.com.

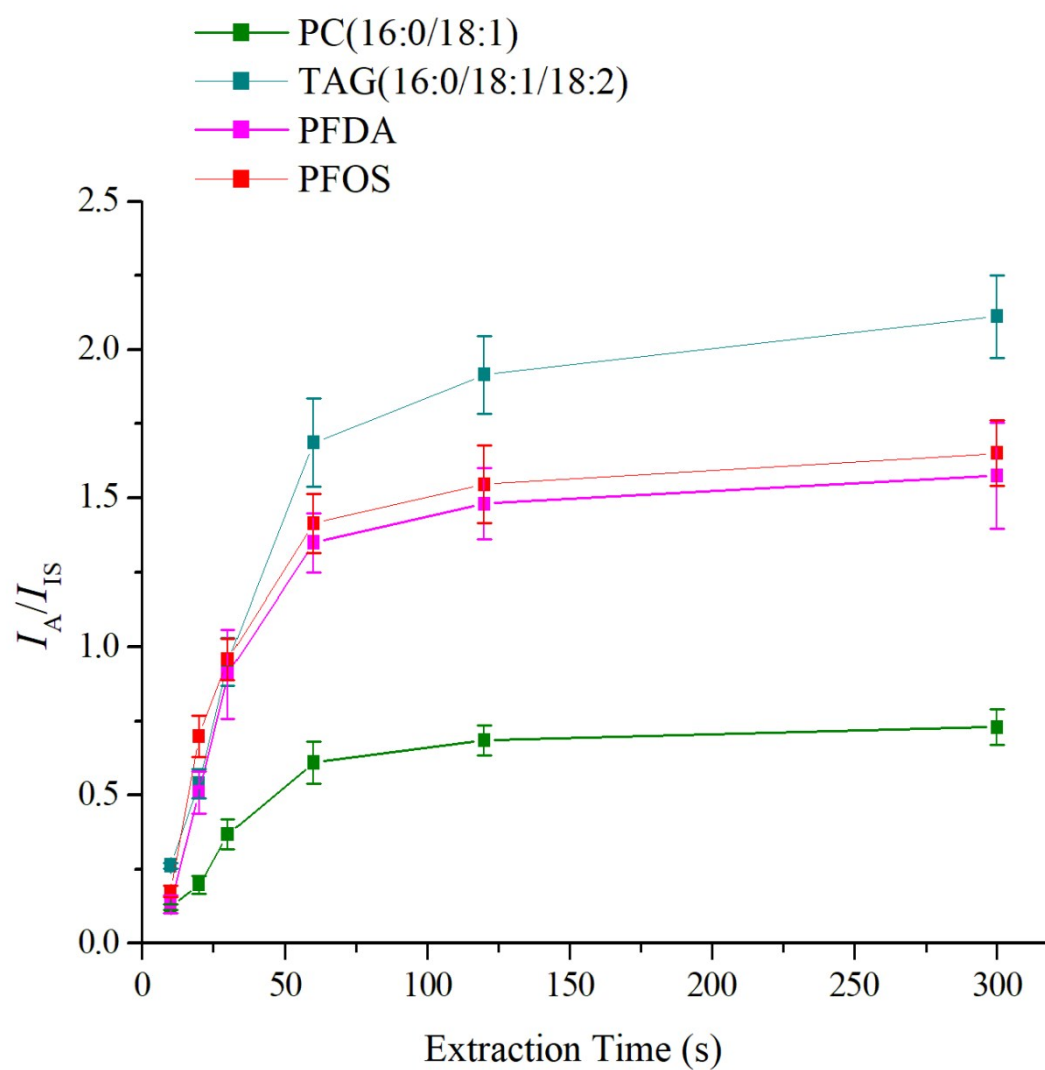
E-mail: jwdeng@126.com



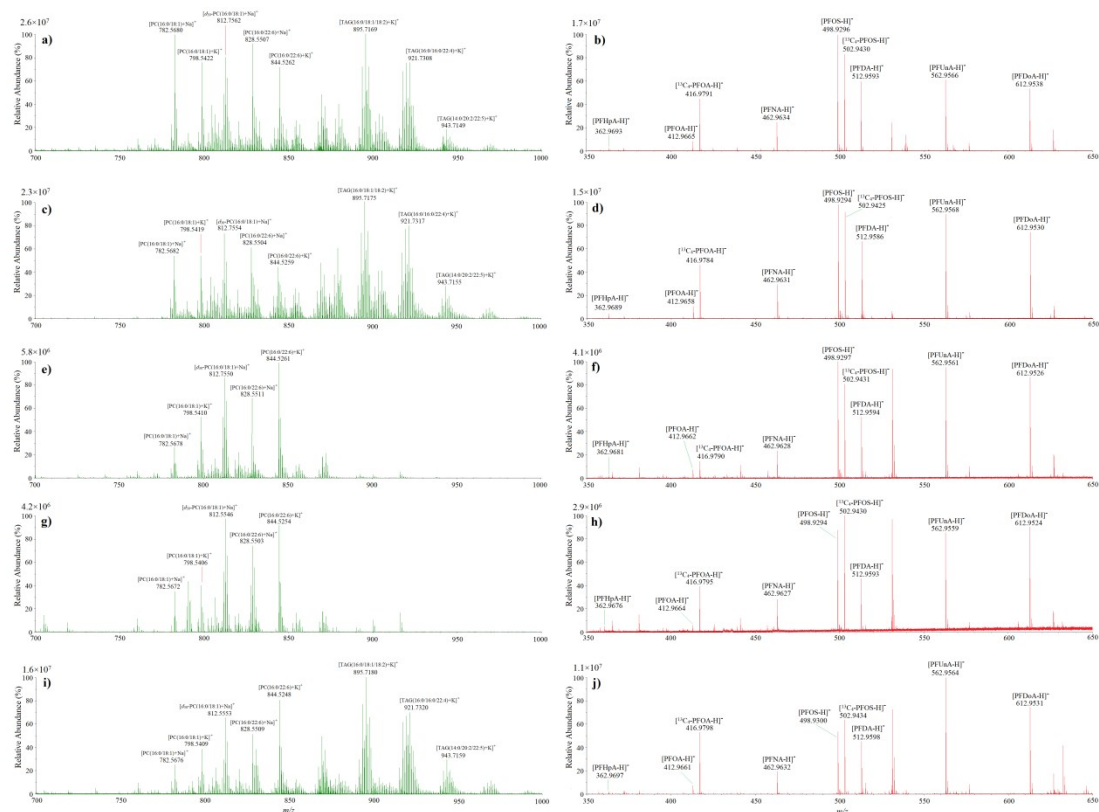
**Fig. S1** Effects of high voltage on signal intensities.



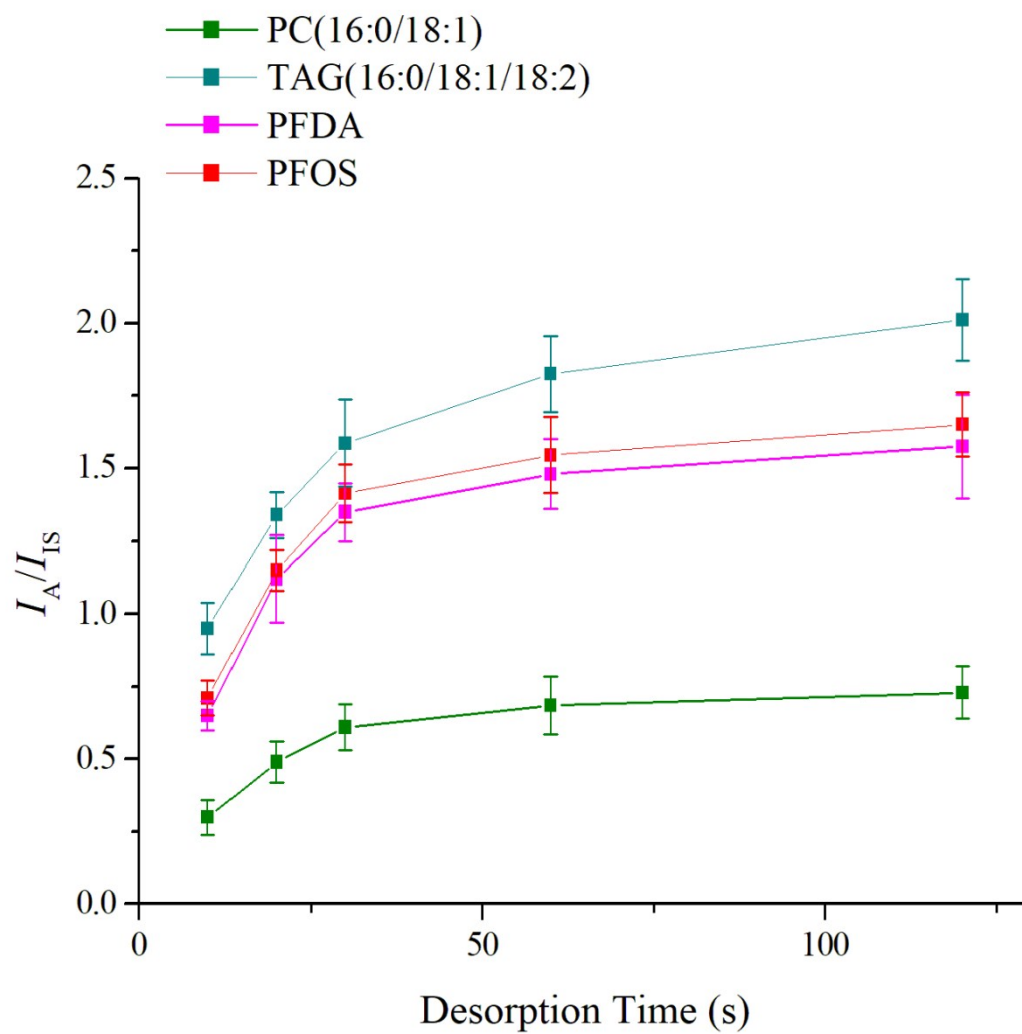
**Fig. S2** Effects of high voltage on signal duration time.



**Fig. S3** Effects of extraction time on signal intensities.



**Fig. S4** Mass spectra for microscale analysis of zebrafish muscle after PFASs exposure by SCP-nanoESI-MS using different desorption/spray solvents. a) positive and b) negative ion spectra with methanol as desorption/spray solvent, c) positive and d) negative ion spectra with acetonitrile as desorption/spray solvent, e) positive and f) negative ion spectra with methanol/water ( $v/v=1:1$ ) as desorption/spray solvent, g) positive and h) negative ion spectra with acetonitrile/water ( $v/v=1:1$ ) as desorption/spray solvent, and i) positive and j) negative ion spectra with methanol/chloroform ( $v/v=1:1$ ) as desorption/spray solvent.



**Fig. S5** Effects of desorption time on signal intensities.