

**Analytical and Bioanalytical Chemistry**

**Electronic Supplementary Material**

**Analysis of perfluorinated alkyl substances in Spanish sewage sludge by liquid chromatography-tandem mass spectrometry**

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## Results and Discussion

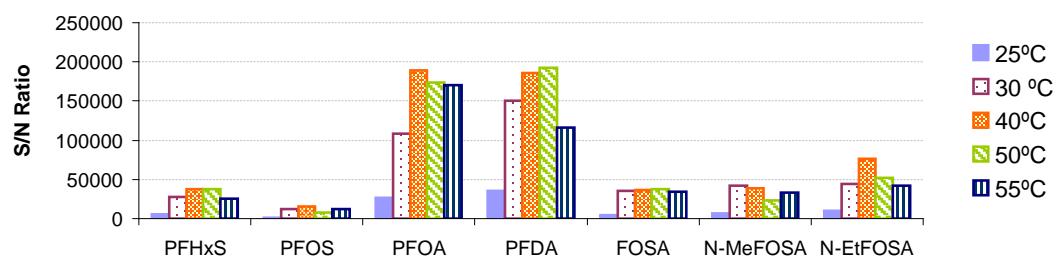
### HPLC-MS/MS conditions

Some parameters were optimized to improve the separation of the target compounds, to maximize the ion formation and facilitate their introduction to the mass spectrometer.

The phase mobile chosen to reach an adequate chromatographic separation of the target compounds was 2mM ammonium acetate in Milli-Q water (A) and methanol (B). Once phase mobile was chosen, the oven column temperature was studied in a range from 25 to 55°C. At 25°C and 30°C the S/N ratio was too low, the maximum S/N ratio was achieved at 40°C. At 50°C and 55°C values were decreasing again (see Fig. S1)

Fig. S1

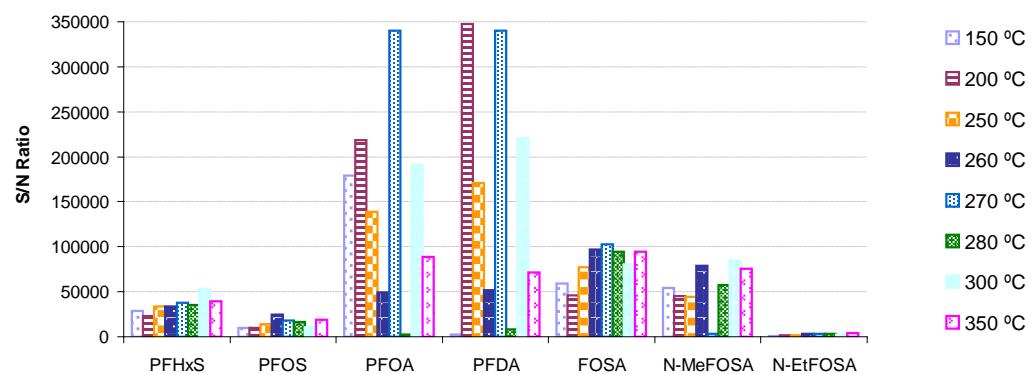
## Evaluation of the effect of the oven column temperature in the analysis of PFAS.



Several electrospray ionization chamber parameters were optimized to maximize ion formation and transmission and minimize noise and source contamination. The drying gas temperature was studied, for the mobile phase selected, in the range from 150 to 350°C (see Fig. S2). Most of compounds had a good S/N ratio at 270°C, except N-MeFOSA which showed a low S/N ratio when this temperature was used. In the case of N-MeFOSA, the best S/N ratios were reached at 260 and 350°C, but these temperatures could not be used for the rest of the compounds because of their S/N ratios decrease. The drying gas pressure adequate at 270°C was 25psi.

Fig. S2

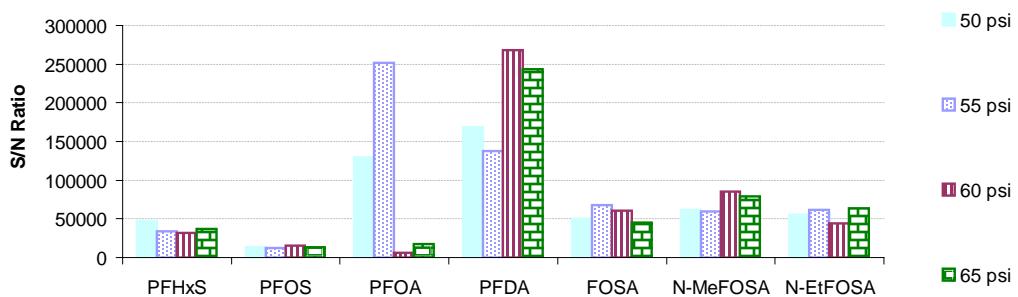
## Evaluation of the N<sub>2</sub> drying gas temperature in the analysis of PFAS.



The nebulizing gas pressure was evaluated in the range of 50-65psi, being 55psi the pressure providing the best results for most of the compounds studied. In the case of 60psi and 65psi the signal of PFOA decreased although the values for the rest of the compounds were acceptable (Fig. S3).

**Fig. S3**

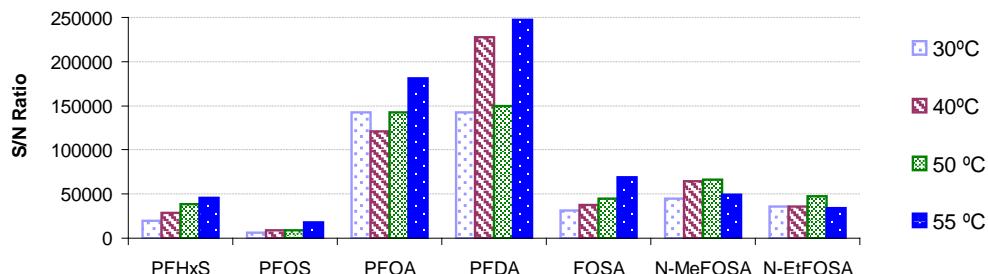
Evaluation of the N<sub>2</sub> nebulizing gas pressure in the analysis of PFAS.



Spray chamber temperature was tested between 30°C and 60°C. In this case, 55°C provided the best S/N ratio for most of the compounds; higher temperatures did not allow a good ionization (Fig. S4).

**Fig. S4**

Study of the effect of the spray chamber temperature in the analysis of PFAS.



Spray chamber temperature was evaluated in a range from 30 to 60°C.  
At 60°C, operational problems appeared, indicating that this temperature was not adequate.

The spray shield voltage facilitates the introduction of the ions formed in the spray chamber toward the mass spectrometer. This parameter was ranged from -300 to -600v, and the value selected was -450v. The X-Y position and voltage of the spray needle was necessary to maximize the nebulization efficiency and optimize the sensitivity and maximum robustness. The needle voltage was ranged from -1000 to -4000v; in this case, -2000v was considered as optimal value.

Some MS/MS parameters were optimized to achieve the suitable collision induced dissociation (CID) of the parent ions for formation of the product ions. The collision gas pressure was tested in the range of 1.6 and 2mTorr; 1.8mTorr provided good results for most of the analytes (Fig. S5).

**Fig. S5**

Study of the effect of the collision gas pressure in the analysis of PFAS.

