PFAS analysis on the SCIEX 7500 system: 15 months of robustness data

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This technical note demonstrates the robustness of the SCIEX 7500 system over 15 months of routine PFAS analysis with various water samples. Quality control (QC) samples, spiked at 10 ng/L with L-PFHxS, L-PFOS, L-PFOA and L-PFNA, showed accuracies generally within ±1 standard deviation of the mean and all QC samples were with 30% of the mean values (Figure 1). Only 1 preventative maintenance (PM) service was performed during the 15-month timeframe. During the analysis, approximately 100–200 injections were performed per week and other non-PFAS applications were also run. These results highlight the strong robustness of both the analysis method and the SCIEX 7500 system.

A robust analysis is paramount to the long-term viability of routine PFAS analysis. In this technical note, the developed method was validated for 26 PFAS compounds of interest in LC-MS-grade, drinking, ground and surface water samples.[1] Modifications were made to the LC system to reduce background contamination, including replacing or bypassing any components of the system that contribute to PFAS contamination. Specifically, system components containing fluorinated ethylene propylene (FEP) and Teflon were bypassed or replaced with polyether ether ketone (PEEK), when possible.

Key benefits of long-term PFAS analysis using the SCIEX 7500 system

- **Long-term stability of QC samples with no loss in sensitivity:** Robustness demonstrated by all QC samples within ±30% of the mean over 15 months
- **Minimal maintenance:** Preventive maintenance was only performed once during the 15-month period. Prior to the preventive maintenance, the calibration specifications were met with only front-end cleaning required.
- **Diversity of water samples analyzed:** Several different water matrices were analyzed, including ground, surface and drinking water

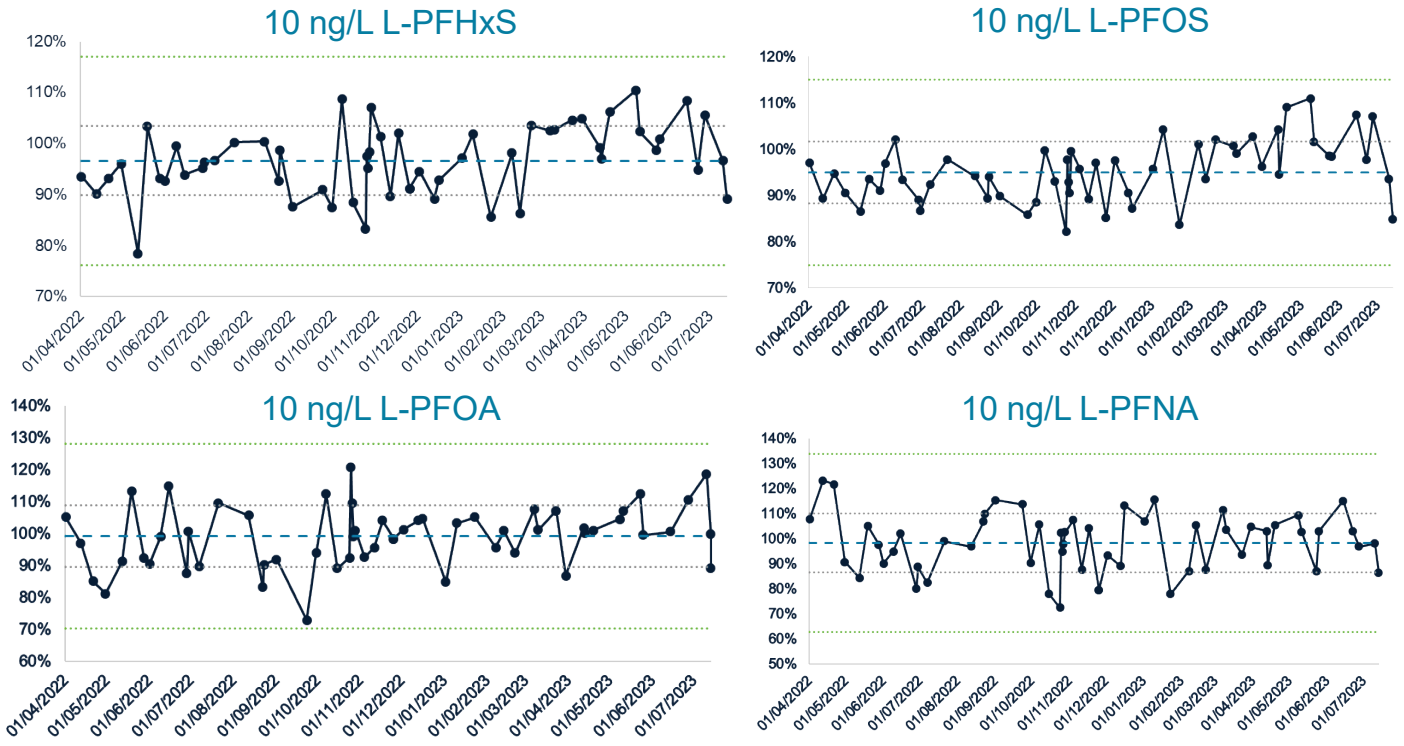

Figure 1. Percent accuracy plots of 4 regulated PFAS compounds at multiple timepoints. The plots highlight the robustness observed. The grey dotted lines show ±1 standard deviation and the green dotted lines show ±3 standard deviations. During this period, no value fell outside of ±30% of the mean. The QC samples were spiked into matrix and were representative of multiple different water samples.

Additional analysis details

To further clarify how the QC data were collected, the analysis batch is included below. Step 8 introduces the QC samples analyzed in matrix. Step 14 outlines the flushing procedure used following sample analysis.

1. 10 ng/L standard: Injected in triplicate to check the instrument performance
2. Diluent blank: Used as the 0 ng/L calibration curve standard
3. Calibration standards and sample diluent: A calibration curve was constructed across concentrations ranging from 0 to 50 ng/L in ultrapure water
4. Diluent blank
5. Procedure blank: Blank sample that underwent all sample preparation steps
6. QC sample in ultrapure water
7. Blank QC sample in matrix
8. QC sample at 10 ng/L in matrix
9. Diluent blank
10. 10 water samples (various sources)
11. QC in ultrapure water and sample diluent: Repeated every 10 samples to check for drift
12. Diluent blank: Repeated every 10 samples
13. Calibration curve
14. End of method: The column was flushed with 50:50 (v/v), mobile phase A/mobile phase B for 15 minutes. The column was flushed with 99% mobile phase B for 5 minutes. The flow stopped and the instrument was placed into standby.

Prior to sample analysis, the consumables used for sample preparation and storage were flushed with 50:50 (v/v), methanol/acetonitrile to ensure that background levels were minimized. These consumables included the sample centrifuge tubes with screw cap and the polyethylene (PE) syringe for filtration. Resistor capacitor (RC) filters were flushed and the first 2 mL of filtrate and pipette tips (both positive displacement and air displacement tips) were disposed of.

Modifications to the LC system

To achieve clean blanks, modifications were made to the LC system to mitigate PFAS contamination as much as possible. The LC system used here was a Shimadzu LC-40 system. The changes described here might vary between systems, depending on configuration and flow path.

The original R-0 to R-2 rinse selection block in the autosampler was bypassed. Only 1 rinse liquid was used, which was connected directly to the low pressure valve (LPV) using a PEEK tube. Subsequently, the original Teflon 1/16” tubing connecting the LPV to the bottom of the rinse port was replaced by 1/16” PEEK tubing.

The LC parts replaced with PEEK included:

- Eluent lines from bottles to the degasser, with filters removed
- Couplings on the degasser (nut and ferrule)
- Eluent lines from the degasser to the pump and autosampler
- T-piece inlets, including couplings in the pumps
- R-3 rinse line to the autosampler
- The rinse port and rinse pump tubing from the autosampler to the ports, including couplings
- Pump seals were replaced with PFAS-suitable seals
- The plunger seals of the measuring pump and pumps A and B were replaced

For complete method details and more information please refer to reference #1.

References

1. An ultra-high sensitivity analysis of PFAS compounds in multiple water sources, SCIEX technical note, RUO-MKT-02-15059-A.

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