SHIMADZU

WP 134

One System, Multiple Solutions: Analysis of PFAS & Cyanotoxins in Water Adhering to EPA 537.1, 544, and 545

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1. Introduction

- PFAS are persistent synthetic chemicals found in consumer and industrial products, while cyanotoxins are harmful substances produced by cyanobacteria in water bodies.
- EPA Methods 537.1 and 533 are approved by EPA for PFAS analysis in drinking water; Methods 544 and 545 target cyanotoxins like microcystins, nodularin, cylindrospermopsin, and anatoxin-a.
- A single Shimadzu LCMS-8060RX system with automatic method switching enables efficient, accurate analysis of PFAS and cyanotoxins, reducing equipment needs and turnaround time.

2. Methods

Figure 1 illustrates the LC-MS system's method-switching capability, enabling seamless transitions between EPA Methods 537.1, 544, and 545 on a single platform. For EPA Method 537.1 (PFAS analysis), the flow path includes the delay column to remove background PFAS. In contrast, for EPA Methods 544 and 545 (cyanotoxin analysis), the delay column is bypassed to prevent unnecessary contaminations, ensuring accurate results. This automation enhances workflow efficiency and analytical flexibility.

To prevent mobile phase contamination when switching between PFAS and cyanotoxin analysis, a simple five-minute rinse (the flow path indicated by the red lines in Figure 2) with the appropriate mobile phase is shown to be sufficient.

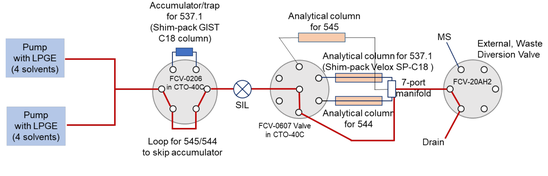

Figure 1: Flow diagram of the integrated LCMS-8060RX for multiple applications (red lines indicate the flow in the rinse step).

Table 2. LC and MS method conditions of the three EPA methods

Parameter	EPA 537.1	EPA 545	EPA 544
Analytical Column	Shim-pack Velox SP-C18 column, 2.7 µm, 2.1 x 50 mm	Shim-pack GIST C18 column, 2.0 µm, 2.1 x 100 mm	Shim-pack Velox SP-C18 column, 2.7 µm, 2.1 x 100 mm
Delay Column	Shim-pack GIST C18 5 µm, 3.0 x 50 mm	Not applicable	Not applicable
Injection Volume	2 µL	20 µL	10 µL
Column Oven Temp.	45 °C	40 °C	40 °C
Flow Rate	0.25 mL/min	0.3 mL/min	0.3 mL/min
Run Time	18 minutes	8 minutes	8 minutes
MS Interface	ESI Negative	ESI Positive	ESI Positive
Nebulizing Gas Flow	3.0 L/min	3.0 L/min	3.0 L/min
Heating Gas Flow	15.0 L/min	10.0 L/min	10.0 L/min
Drying Gas Flow	5.0 L/min	10.0 L/min	10.0 L/min
Interface Temp.	100 °C	300 °C	300 °C
DL Temp.	150 °C	250 °C	250 °C
Heat Block Temp.	250 °C	400 °C	400 °C

Figure 2 provides a detailed overview of the batch structure designed to evaluate the system's robustness and reliability during method switching. In total, the batch included 294 injections, covering null, solvent blank, rinse, and standard injections, with 54 hours of continuous operation.

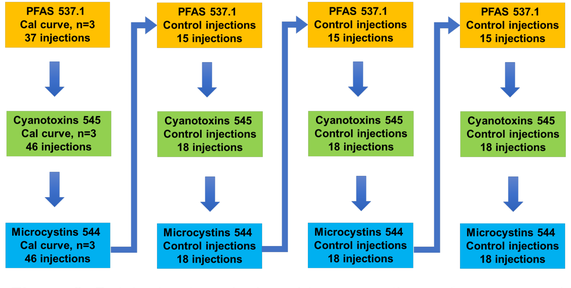

Figure 2: Batch structure designed to assess the performance and reliability of running three analytical methods on a single system.

3. Results

Calibration curves were successfully established for all the analytes in accordance with EPA Method 537.1, 545 and 544. All analytes demonstrated R^2 values exceeding 0.99, confirming strong linearity across the calibration range. The accuracy of all injections remained within 80% – 120%, and %RSD values for all calibration levels were below 15%.

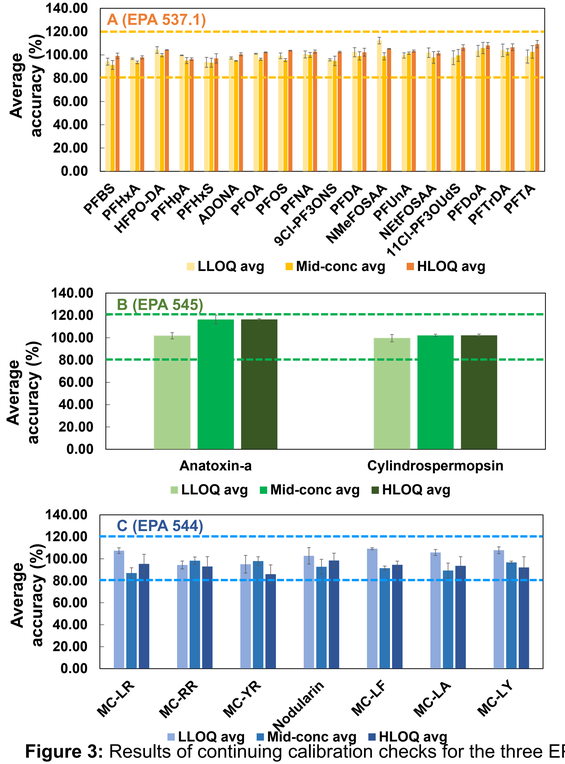

Figure 3: Results of continuing calibration checks for the three EPA methods.

Continuing calibration checks were performed after triplicate injections for each method—starting with 537.1, then 545 and 544—repeated three times to evaluate rinse effectiveness and system consistency. **Figure 3** shows accuracy at the LLOQ, mid, and HLOQ levels, with average accuracy on the y-axis and %RSD as error bars. All analytes maintained 80–120% accuracy and %RSD below 15%, confirming reliable performance and effective rinsing in preventing mobile phase contamination.

Figure 4 displays chromatograms of PFBS (negative ion, Method 537.1) and cylindrospermopsin (positive ion, Method 545) at the LOQ before and after method switching. Chromatogram No. 1 is from the initial calibration, while Nos. 2–4 are subsequent checks. Results confirm stable LOQ accuracy and consistent system performance despite method switching.

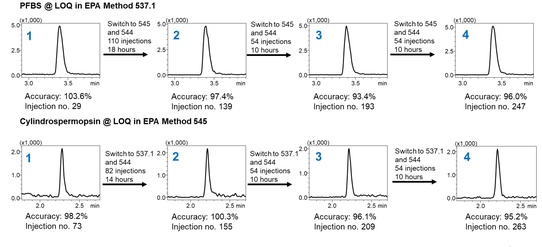

Figure 4: Mass chromatogram and quantitative accuracy of representative compounds before and after switching methods.

4. Conclusion

This study shows that PFAS and cyanotoxins can be accurately measured using one triple quadrupole mass spectrometer with automatic method switching. A quick five-minute rinse ensures clean transitions between methods, minimizing downtime and manual work. This streamlined setup reduces equipment costs, boosts efficiency, and supports high-throughput environmental testing, including rapid response to events like harmful algal blooms.

The authors are affiliated and funded by Shimadzu Corporation.