Waters™

Enhancing Environmental Testing: Comprehensive Guide to PFAS Analysis Using EPA Method 1633 and LC-MS/MS

EXECUTIVE SUMMARY

Per- and polyfluoroalkyl substances (PFAS) are a group of human-made chemicals that have become pervasive in the environment due to their extensive use and persistence. Concerns about their potential impact on human health and ecosystems have prompted stringent regulatory measures for their monitoring. The United States Environmental Protection Agency (EPA) Method 1633 provides a comprehensive approach to analyzing PFAS in various environmental matrices, ensuring compliance with the Clean Water Act and other regulatory requirements.

This white paper explores the critical aspects of PFAS analysis using EPA Method 1633, focusing on the role of advanced Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS) technology. We will outline best practices for sample preparation, the importance of robust training and support, and future innovations to enhance the detection and analysis of PFAS. Whether you're establishing new testing capabilities or enhancing existing ones, this guide offers essential insights and guidance.

INTRODUCTION TO PFAS AND REGULATORY CONTEXT

PFAS are a large group of chemically related compounds that have been used since the 1940s in various industrial applications and consumer products, such as non-stick cookware, water-repellent clothing, and firefighting foams. PFAS are known for their resistance to degradation, leading to widespread environmental contamination and bioaccumulation in living organisms.

The potential health risks associated with PFAS exposure, including cancer, thyroid disease, and immune system dysfunction, have led to increased regulatory scrutiny. In response, the EPA has developed Method 1633, a rigorous analytical procedure designed to detect and quantify 40 different PFAS compounds across various environmental matrices, including wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue. Method 1633 is a cornerstone for environmental compliance programs under the Clean Water Act, Superfund sites, and remediation efforts at Department of Defense (DoD) installations.

UNDERSTANDING EPA METHOD 1633

EPA Method 1633 is a comprehensive analytical method designed to identify and quantify PFAS in both liquid and solid environmental samples. Finalized in January 2024, this method was developed in collaboration with the US Department of Defense's Strategic Environmental Research and Development Program (SERDP). It is validated through a multi-laboratory study, ensuring reliability and accuracy in diverse matrices.

Key applications of EPA Method 1633 include:

- **Clean Water Act Compliance:** Ensuring that wastewater discharge meets environmental safety standards.
- **Superfund Sites:** Monitoring and mitigating PFAS contamination at hazardous waste sites.
- **Remediation and Investigation Programs:** Supporting environmental clean-up efforts, including those at military installations.

ROLE OF LC-MS/MS IN PFAS ANALYSIS

Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS) has become the gold standard for PFAS analysis, owing to its high sensitivity, selectivity, and accuracy. This technique effectively separates and identifies PFAS compounds even at trace levels (parts per trillion), making it indispensable for environmental testing.

KEY ADVANTAGES OF LC-MS/MS TECHNOLOGY:

- **High Sensitivity:** Capable of detecting PFAS at very low concentrations, ensuring compliance with stringent regulatory limits.
- **Selectivity:** Differentiates between PFAS compounds and potential interferences, reducing false positives and enhancing data reliability.
- **Quantitative Accuracy:** Provides precise quantitation, essential for environmental monitoring and risk assessment.
- **Reproducibility and Robustness:** Ensures consistent results across different samples and laboratories, which is crucial for regulatory compliance.

CASE STUDY: LC-MS/MS IN ACTION

A recent validation study using certified reference materials (CRM) demonstrated the effectiveness of LC-MS/MS in PFAS analysis. For wastewater samples, the method showed a mean trueness of 92%, while soil samples demonstrated a mean trueness of 97%, well within the acceptable ranges. These results highlight LC-MS/MS's ability to provide reliable, high-quality data, supporting environmental safety and regulatory adherence.

SAMPLE PREPARATION: CRITICAL STEP FOR ACCURATE ANALYSIS

Sample preparation is a vital component of PFAS analysis, directly impacting the accuracy and reliability of results. EPA Method 1633 emphasizes using Solid-Phase Extraction (SPE) techniques to handle complex environmental matrices.

SOLID-PHASE EXTRACTION (SPE) TECHNIQUES:

1. **Weak Anion Exchange (WAX) Cartridges:** These are used for their selective retention of PFAS, particularly acidic species, through ion exchange mechanisms. By adjusting the pH during extraction, WAX cartridges can effectively isolate PFAS from complex matrices.
2. **Graphitized Carbon Black (GCB):** In combination with WAX, GCB is used to remove additional matrix interferences, enhancing the clarity and accuracy of results. This dual approach is critical when analyzing samples with high organic content or other potential interferences.

BEST PRACTICES IN SAMPLE PREPARATION:

- **Matrix-Specific Preparation:** Different sample types (*e.g.*, wastewater vs. soil) require tailored preparation steps. For instance, aqueous samples undergo WAX cleanup followed by GCB, whereas solid samples use the reverse order.
- **Performance-Based Flexibility:** EPA Method 1633 allows for method modifications as long as the performance criteria (*e.g.*, recovery rates, relative standard deviations) are met. This flexibility enables laboratories to optimize their workflows and improve efficiency.
- **Automation and Efficiency:** Utilizing automated systems, such as the PromoChrom™ SPE-03, can streamline the sample preparation process, reduce manual handling, and increase throughput, thereby improving lab productivity.

TRAINING AND SUPPORT: ENSURING SUCCESS IN PFAS ANALYSIS

Implementing EPA Method 1633 and LC-MS/MS technology requires comprehensive training and ongoing support to ensure successful adoption and consistent performance. Waters Corporation offers a range of training solutions designed to meet the needs of environmental professionals.

TRAINING PROGRAMS AND RESOURCES:

- **Onsite and Virtual Training:** Customized training sessions provide hands-on experience with the instrumentation, methods, and software. Virtual options offer flexibility and convenience.
- **Application Success Guides:** Detailed reference materials guide users through every step of the PFAS analysis process, from sample preparation to data interpretation.
- **Expert Consultation:** Access to Waters Corporation's team of experts ensures that laboratories receive the technical support needed to overcome challenges and optimize performance.
- **Certification Programs:** Training programs culminate in certification, documenting proficiency in PFAS analysis methods, which can be beneficial for compliance and quality assurance purposes.

FUTURE INNOVATIONS IN PFAS TESTING

The field of PFAS analysis is continuously evolving, driven by the need for greater sensitivity, faster processing times, and reduced operational complexity. Waters™ Corporation is at the forefront of these innovations, offering solutions that enhance the efficiency and effectiveness of PFAS testing.

EMERGING TECHNOLOGIES AND METHODS:

- **Dual-Phase Cartridges:** Combining WAX and GCB into a single cartridge simplifies the extraction process, reducing preparation time and minimizing potential errors.
- **Automated Sample Preparation:** Systems like the PromoChrom SPE-03 automate the SPE process, enhancing reproducibility and throughput while freeing up laboratory personnel for other tasks.
- **Advanced LC-MS/MS Platforms:** The ACQUITY™ Premier UPLC™ coupled with the Xevo™ TQ Absolute mass spectrometer pushes the boundaries of sensitivity, enabling the detection of even lower PFAS concentrations.

EXPERT Q&A: ADDRESSING COMMON CONCERNS IN PFAS ANALYSIS

To further enhance the understanding of PFAS analysis and address real-world concerns, we have included insights from a recent webinar where experts answered frequently asked questions about PFAS analysis:

1. **What filter material is recommended to minimize PFAS contamination during sample pre-treatment?**
   - **Answer:** Acrodisc syringe filters are recommended to minimize PFAS contamination. It's also essential to ensure that filters and other consumables are PFAS-free and to pre-rinse with LC-MS-grade methanol to reduce potential contamination. Consistent monitoring of PFAS levels in the lab environment is key.
2. **What type of tubing is recommended for automated sampling in wastewater to avoid PFAS contamination?**
   - **Answer:** We recommend using tubing that does not contain Teflon or similar PFAS-related materials. Waters Corporation offers kits that replace such tubing in LC-MS systems. Additionally, using an isolator or delay column can help separate potential contaminants from the analytical peaks, ensuring accurate results.
3. **Is training and technical support included when leasing an LC-MS/MS instrument?**
   - **Answer:** Yes, Waters Corporation offers comprehensive training and technical support as part of the leasing agreement. This ensures that users are fully prepared to operate the equipment and implement PFAS analysis methods effectively.
4. **What measures can be taken to mitigate PFAS contamination in the laboratory environment?**
   - **Answer:** Various factors can contribute to PFAS contamination, including specific types of clothing, cosmetics, and cleaning products. Waters Corporation provides a guide on mitigating PFAS contamination, which includes best practices to maintain a PFAS-free environment in the laboratory.
5. **Can PFAS contamination be introduced from gases used in the analysis?**
   - **Answer:** While gases can be a source of PFAS contamination, using an isolator column helps delay and reduce this contamination. Ensuring that gases are free from PFAS is essential, and regular checks should be conducted to maintain low contamination levels.

6. **Are there methods for analyzing PFAS in air samples?**
   - **Answer:** Although Waters Corporation does not directly provide air sampling equipment, there are methods for PFAS analysis in air, typically involving sampling onto filters followed by analysis using compatible systems. These methods can be adapted for more volatile PFAS compounds.
7. **Which EPA methods are recommended for analyzing PFAS in drinking water?**
   - **Answer:** The primary EPA methods for drinking water analysis are EPA 537.1 and EPA 533. The choice depends on specific regulatory requirements. These methods are well-established for reliable PFAS detection in drinking water.
8. **Have plant samples been analyzed for PFAS content?**
   - **Answer:** Yes, PFAS analysis has been conducted on plant samples, including fruits, vegetables, and baby food, using methodologies similar to EPA 1633. Waters Corporation has detailed application notes on these procedures, providing a useful reference for laboratories.
9. **What is the impact of sodium hypochlorite on PFAS samples in wastewater?**
   - **Answer:** PFAS compounds are resistant to degradation, including treatment with sodium hypochlorite. Therefore, using sodium hypochlorite in wastewater disinfection should not affect the integrity of PFAS samples, which will remain detectable using standard analysis methods.

CONCLUSION

As environmental regulations become more stringent, the need for reliable and accurate PFAS analysis methods becomes increasingly critical. EPA Method 1633, coupled with advanced LC-MS/MS technology, provides a robust framework for detecting and quantifying PFAS in various environmental samples. By investing in comprehensive sample preparation techniques and leveraging expert training and support, laboratories can ensure compliance, safeguard public health, and contribute to environmental protection.

Waters Corporation is committed to supporting your PFAS analysis needs. Our innovative solutions, expert training programs, and ongoing support services are designed to help you achieve the highest standards of accuracy and reliability. Contact us today to learn more about how we can assist you in enhancing your lab's capabilities and meeting your PFAS testing goals.

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