RESTEK Pure Chromatography

Mitigating Matrix Issues via Filter Aid to Improve Solid-Phase Extraction Methods

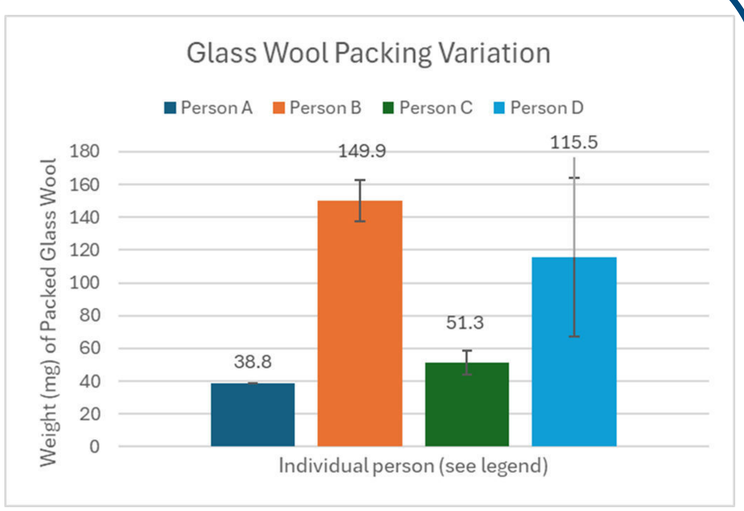
Sebastian Prisacariu, Jason Hoisington, Alexis Shelow, Restek Corporation, Bellefonte, PA, USA

Abstract

Solid phase extraction (SPE) stands as a valuable sample preparation technique for water samples, utilized in the extraction of substances such as PFAS and semi-volatiles from drinking water. Despite its efficacy, SPE encounters challenges in non-drinking water matrices where solid particles can obstruct SPE cartridges, prolonging extraction or necessitating multiple cartridges per sample. EPA Method 1633 recently introduced glass wool to address this issue; however, manually adding wool can be laborious and inconsistent. This presentation introduces an integrated filter aid for SPE cartridges, enhancing consistency compared to glass wool. Incorporating the filter aid mitigates matrix-related clogging, leading to expedited extraction, reduced cartridge consumption, and more reliable analytical outcomes.

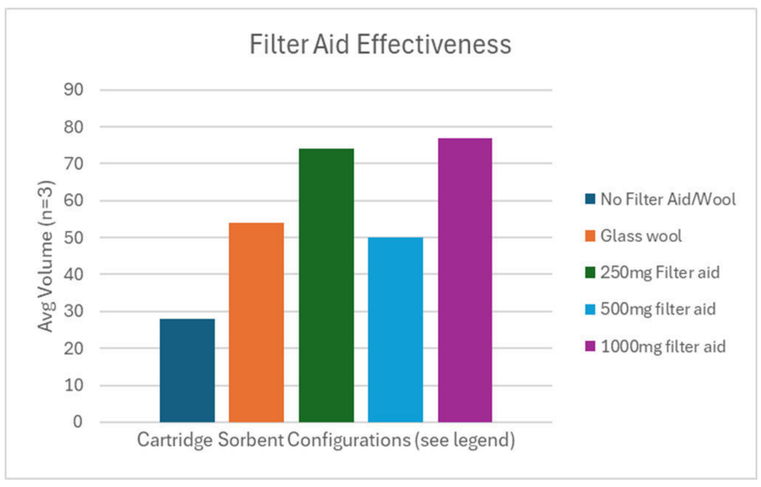
Improved Filtration Performance

Disadvantages of Glass Wool Packing



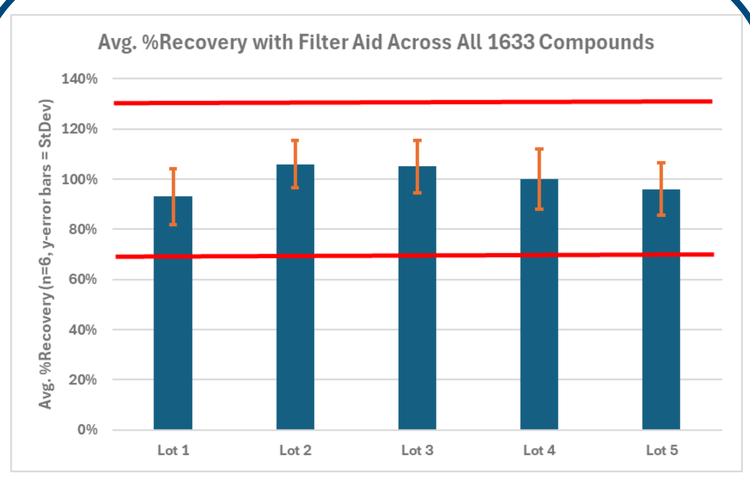
EPA method 1633 calls for the addition of silanized glass wool to be added at half the height of the WAX SPE cartridge barrel. With no specific weight given for the silanized glass wool, this can create inconsistencies in the amount packed in cartridge from cartridge-to-cartridge and technician-to-technician. The graph above demonstrates the variability in silanized glass wool being packed with 4 individuals packing 5 SPE cartridges per EPA guidelines.

Filter Type vs Sample Volume Pass-Through



ASTM Substitute Wastewater Matrix (D5905) was made to evaluate the effectiveness of each filter type outlined above. The substitute wastewater consists of flour, ocean salts, kaolin, Triton x100, light beer, and water. The limit of suspended solids of this matrix is 2-3x the amount that EPA 1633 calls for. The substitute wastewater was passed through each cartridge type until the cartridge became clogged and sample flow stopped. The graph above shows the volume (mL) of substitute wastewater that each cartridge was able to process.

1633 Analyte Recoveries



Analyte recoveries for the 1633 compound class was evaluated to ensure that the filter aid did not suppress or enhance the recoveries outside of the permissible ±30% range. Spiked recoveries were < ±10% for each compound. The graph above shows the average % recoveries for all compounds in 5 different lots of cartridges.

Conclusions

Laboratories conducting EPA 1633 method testing have reported issues of handling samples with high quantities of suspended solids. While the current recommendation of packing silanized glass wool into SPE cartridges can be used, there are notable challenges to this technique which stems from the variability from one technician across multiple SPE cartridges and technician-to-technician variability. Shown here is a much more effective solution, where an optimized filter aid has been added to a WAX/Carbon Dual Bed for improved sample extraction and cleanup. This solution eliminates variability and clogging, while maintaining excellent method performance, meeting the method requirements of ±30% recoveries for all analytes.

References

1. **U.S. Environmental Protection Agency.** Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. *EPA.* [Online] January 2024.
2. **ASTM International.** ASTM D5905-98(2018) Standard Practice for the Preparation of Substitute Wastewater. *ASTM.* [Online] December 24, 2018.