

Experimental Considerations for Per- and Poly-fluorinated Alkyl Substances (PFAS) in Test Solutions for Toxicological Exposures

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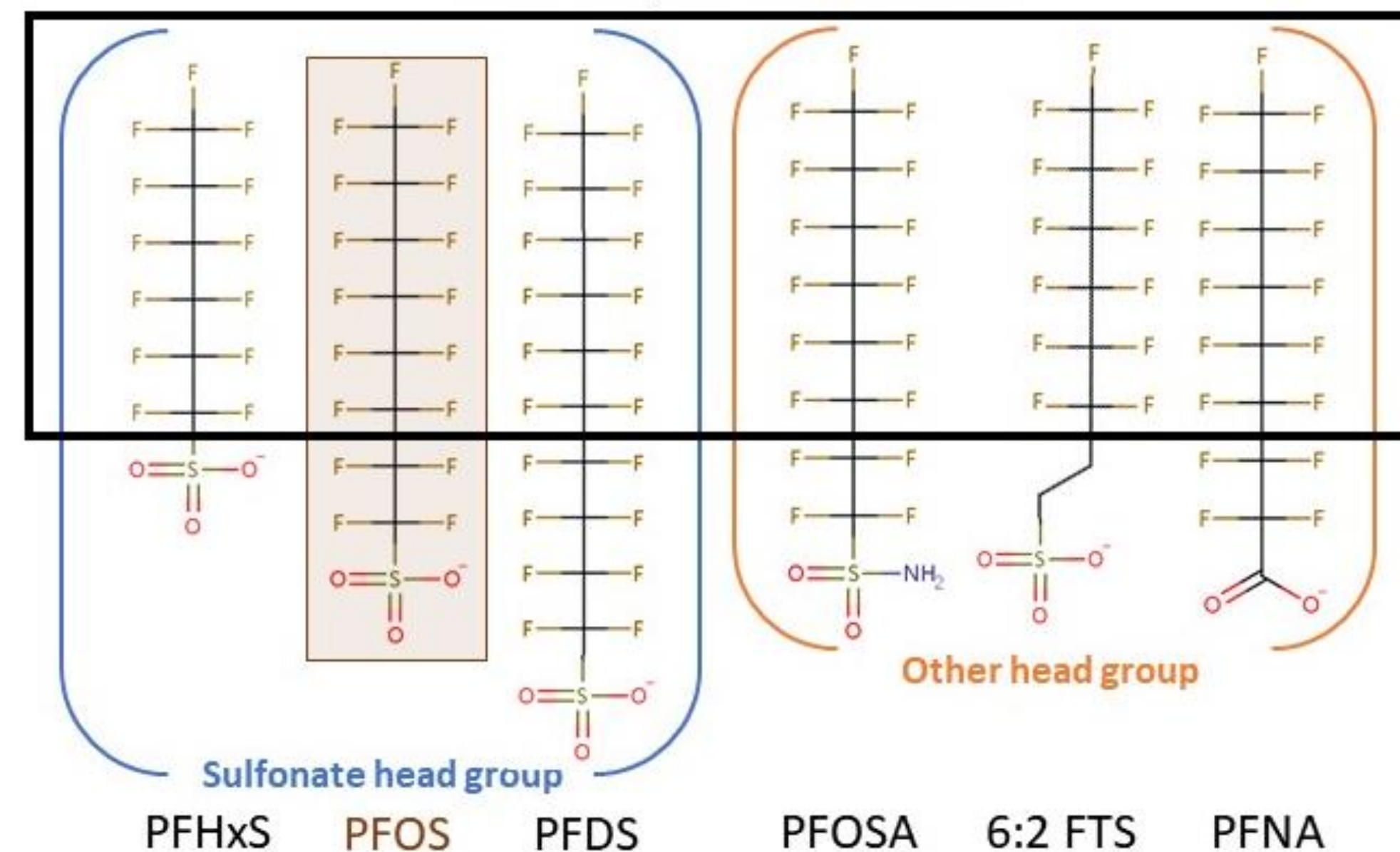
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Introduction

The unique chemical properties of certain PFAS can cause discrepancies between measured and targeted water concentrations during aquatic toxicity exposures. These same properties also increase the difficulty of accurate and precise analytical measurements of exposure waters. To address these concerns, behaviors of model PFAS under laboratory conditions in common aquatic toxicity assays are examined. Specifically, chemical purity, sorption to exposure chambers, stratification, and stability of exposure concentrations over time. Prior work by Rewerts et al. (2021) examined similar phenomena in PFAS. This work expands on those findings with additional experiments, including lower test concentrations and a broader array of chemicals. Results are presented with implications to ensure test design and measurement of PFAS concentrations accommodate the unique obstacles that arise with this class of chemical.

Citation: Rewerts et al. *Environmental Toxicology and Chemistry*. Vol. 30, pp. 667-688. 2021.

Common perfluorinated tail

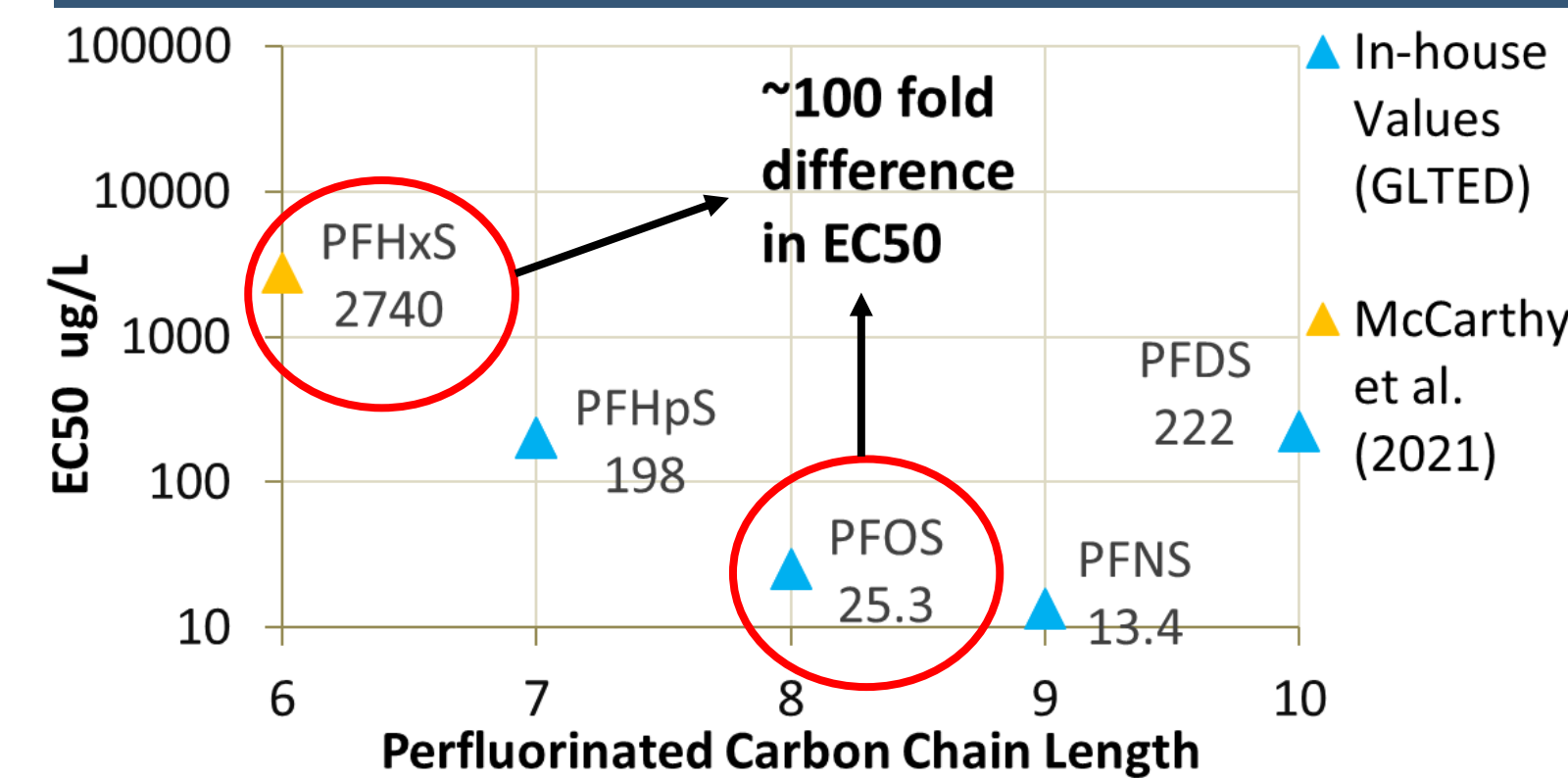


Above: The chemical structure of the six PFAS used in experiments measuring adsorptive loss to glass and plastic test vessels. Effects from variable carbon chain length are determined via the first three PFAS, followed by alterations to the sulfonate head group in the remaining three.

Methods

Quantitation: Compounds were separated by HPLC and detected with a Thermo ISQ mass spectrometer. Quantification was performed by isotope dilution. Further details provided in figure captions.

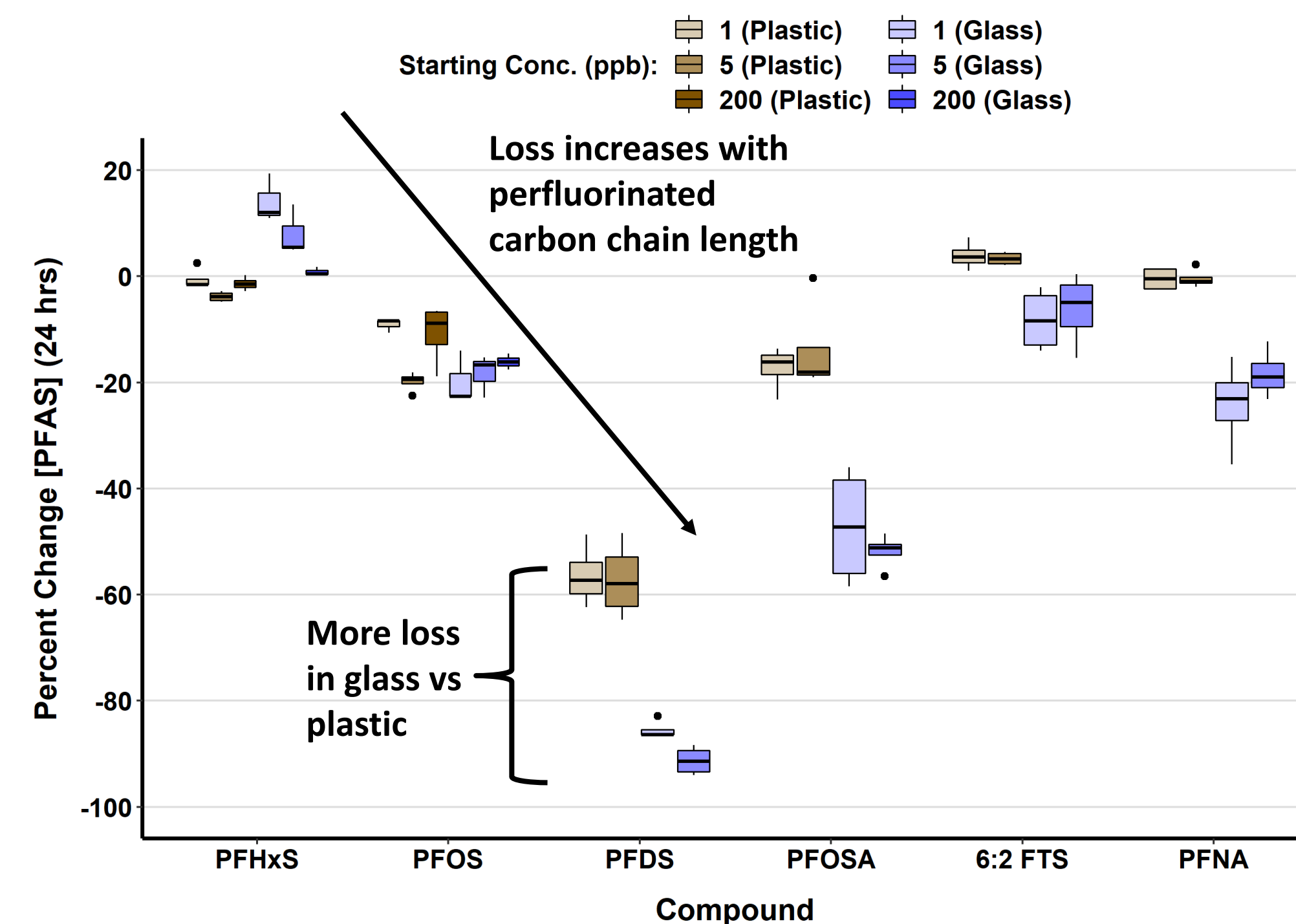
Purity Analysis



Vendor For PFHxS	A	B	C
% Contamination	0.46	3.83	6.19
% PFOS Contamination	ND	2.17	4.84
PFOS Contamination at PFHxS EC50 (ug/L)	ND	59.4	132.7

Citation: McCarthy et al. *Environmental Toxicology and Chemistry*. Vol. 40, pp. 2319-2333. 2021.

Adsorptive Loss to Plastic and Glass Vessels

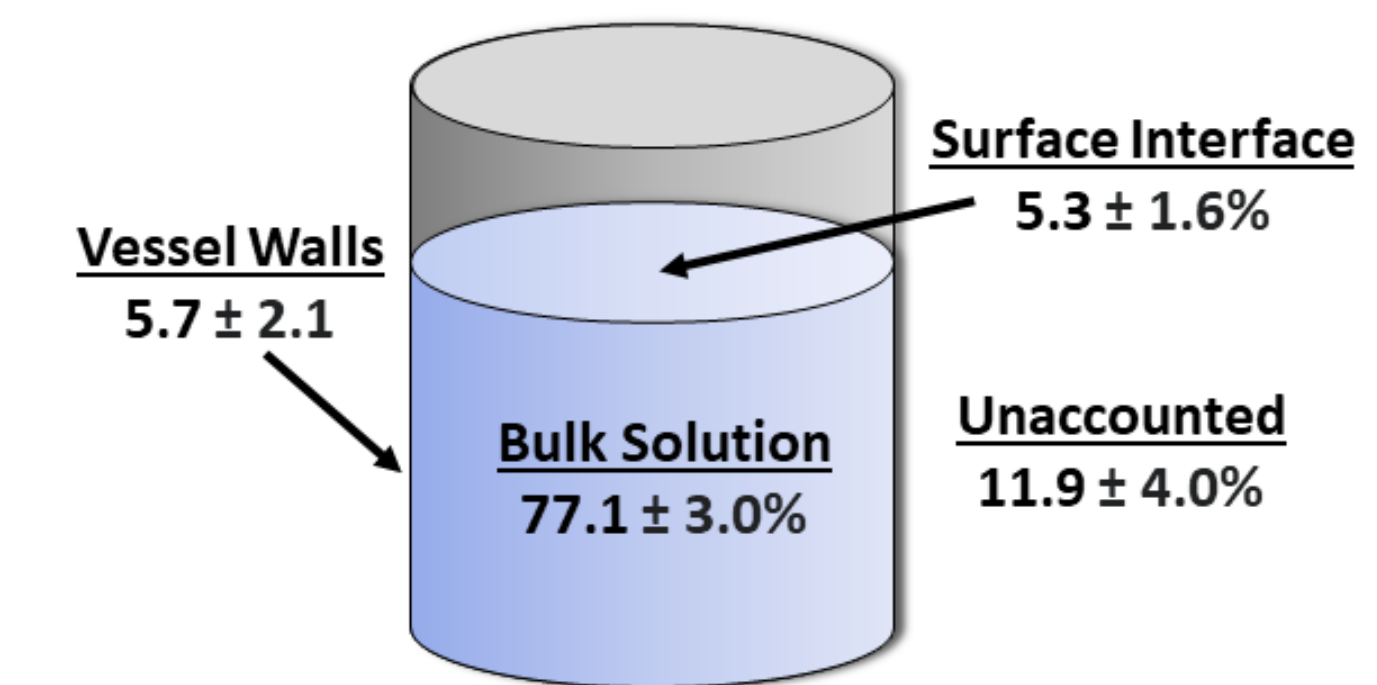


Above: 24-hour adsorptive losses of PFAS at 1, 5, and 200 parts per billion (ppb) in 100 mL glass beakers and 30 mL plastic (Rigid Plastic #6 – polystyrene) cups. Starting concentrations were within +/- 25% of given nominal. Solutions were agitated gently on an elliptical shaker for the 24-hour test duration to ensure mixing.

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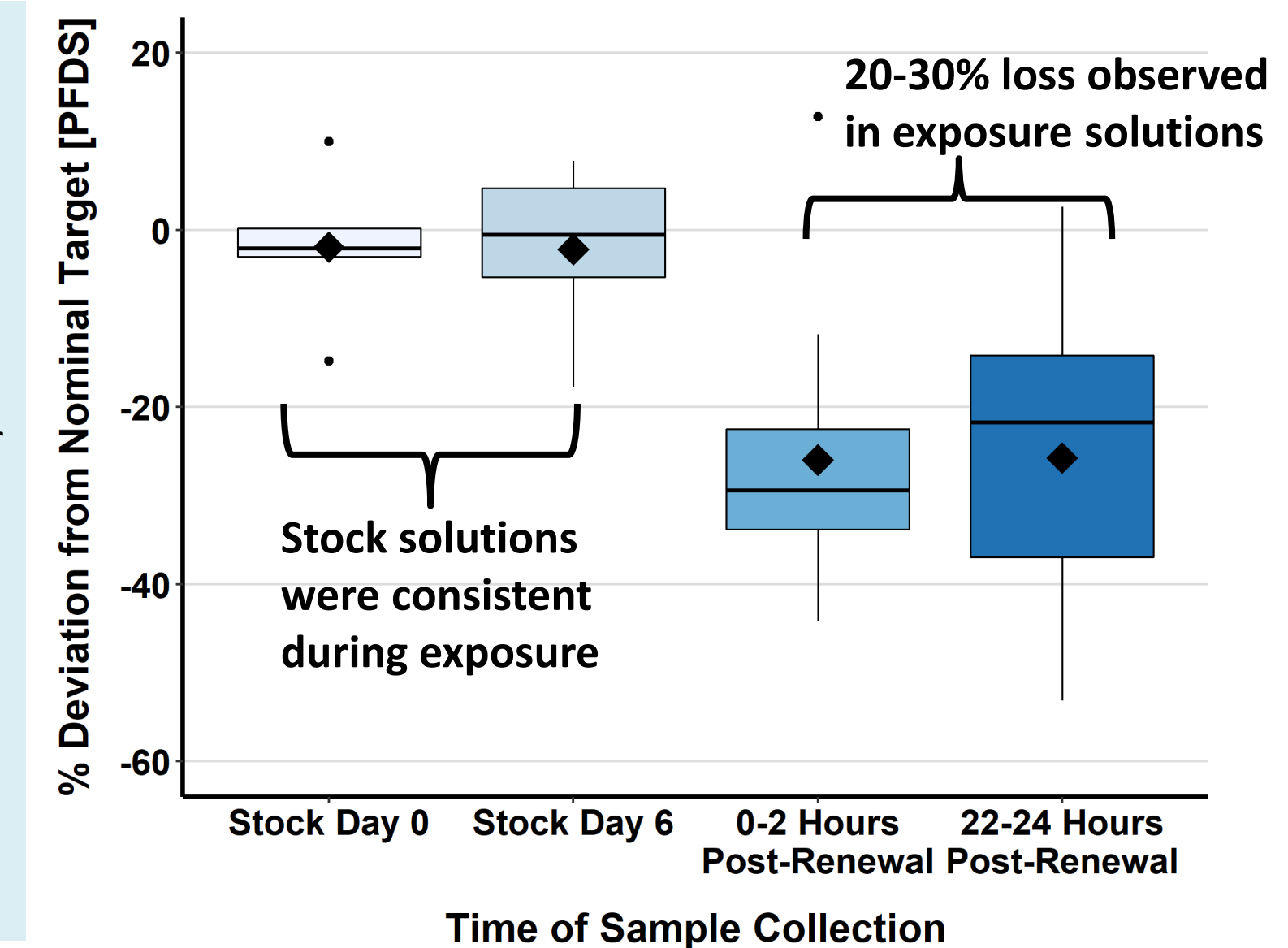
Further Loss Studies

24-Hour Partitioning of PFOS in Glass Beakers



At Left: The partitioning of PFOS at 6 ppb after 24 hours in 100 mL glass beakers. Interface measurements were made via capillary absorption into circular paper towel cutouts followed by methanol extraction. Chemical adsorbed to vessel walls was extracted with methanol after test vessels were emptied.

At Right: Percent deviations from nominal PFDS concentrations in a 7-day static renewal *C. dilutus* exposure with 24-hour renewal. Stocks were stable for the duration of the exposure. Loss was apparent in exposure solutions and occurred within 0-2 hours of renewal. Triangles indicate mean values.



Key Considerations for Exposures

- ❖ Purity of PFAS materials varies greatly across vendor and must be analyzed before exposure to ensure contamination will not skew toxicological results.
- ❖ Adsorptive loss increases with increasing perfluorinated carbon chain length and is greater in glass test vessels than plastic test vessels.
- ❖ Alterations to the sulfonate head group change adsorptive loss, which becomes more severe in sulfonamides and less pronounced in fluorotelomer sulfonates.
- ❖ Loss to container walls and enrichment of the surface layer both contribute to loss of chemical from the bulk solution.
- ❖ Stable concentrations can be obtained in stock solutions if time is allowed for loss to reach a stable equilibrium prior to the exposure.
- ❖ Loss appears to occur quickly, within the first 0-2 hours after renewal for PFDS.

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