What's in your water!

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Overview

Monitoring and safeguarding water quality are crucial for ensuring the well-being of both human populations and the environment. Examples of chemical contamination in water bodies includes organic contaminants such as polyfluoroalkyl substances (PFAS), pharmaceuticals, pesticides, and chemical warfare agents. Most of the the traditional water assurance methods require grabbing water samples from the source and performing GC-MS/LC-MS analysis in the lab for trace-level organics detection. This approach, while accepted as the gold standard, comes with a prohibitive cost and logistical burden. To alleviate this burden, Detect-ION has adapted its fieldable gas sensor for in-situ water analysis, thereby obviating water storage and transport while delivering sensitivity as low as 1 µg/L, a threshold recommended per EPA guidance for water potability.

Materials and Methods

We adapted our compact 10-L Preconcentrator-Thermal Desorber-Gas Chromatograph-Mass Spectrometer (TD-GC-MS) system, originally designed for trace gas sensing and demonstrated extraction, identification, and quantification of volatile and semi-volatile organic contaminants (VOCs/SVOCs) from water matrices. To extract VOCs from the water medium, we bubbled ultra-pure N_2 gas through the water beaker at 3 LPM and subsequently swept the N_2 stream through the VOC preconcentrator sorbent. Between the bubbling setup and the preconcentrator, we used a Nafion-based dehumidification stage to prevent the preconcentrator sorbent from getting exposed to excessive water vapor. After the sampling step, we performed a 10-s thermal desorption of the VOCs collected on the sorbent followed by a subsequent 3-min GC-MS analysis. Additionally, we performed multiple analysis steps sequenced with increasing water sample temperature to bubble and collect SVOCs, such as methyl salicylate, with much lower vapor pressure.

Water Sensing Testbed

Detect-ION leveraged the recently developed miniature and fieldable gas chromatograph-mass spectrometer (GC-MS) system called "ACHILLES" and augmented the sampling design to extract volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) from the water matrix to perform a comprehensive chemical analysis of its constituents. To do this, we built a VOC extraction stage that integrates with the ACHILLES hardware upstream of its embedded VOC preconcentrator stage. **Figure 1** shows the block diagram of the water sensor testbed using ACHILLES.

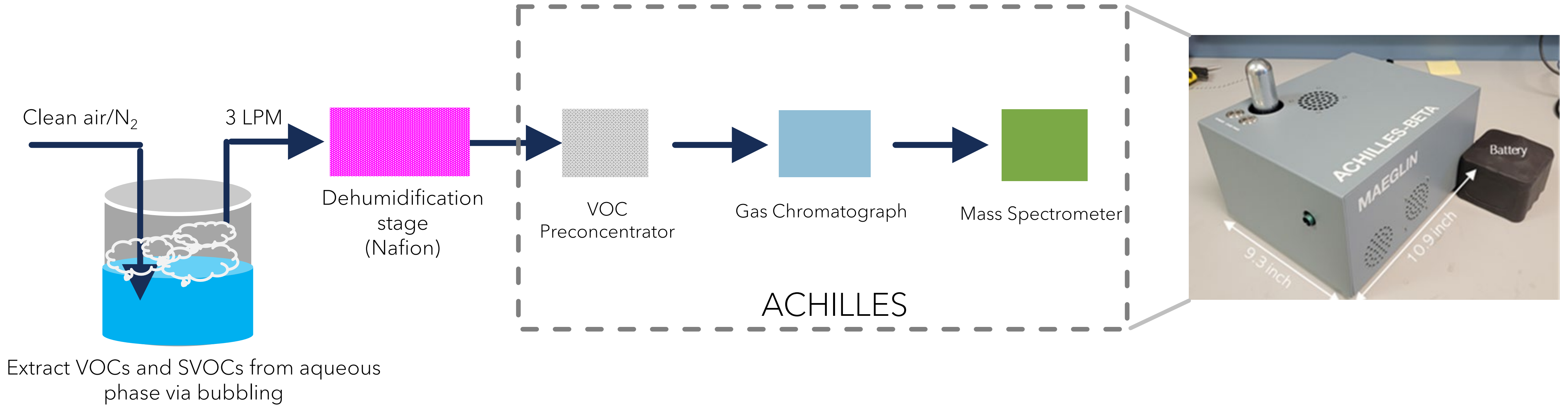

Figure 1. Block diagram shows the adaptation of the ACHILLES system as a water contamination sensor.

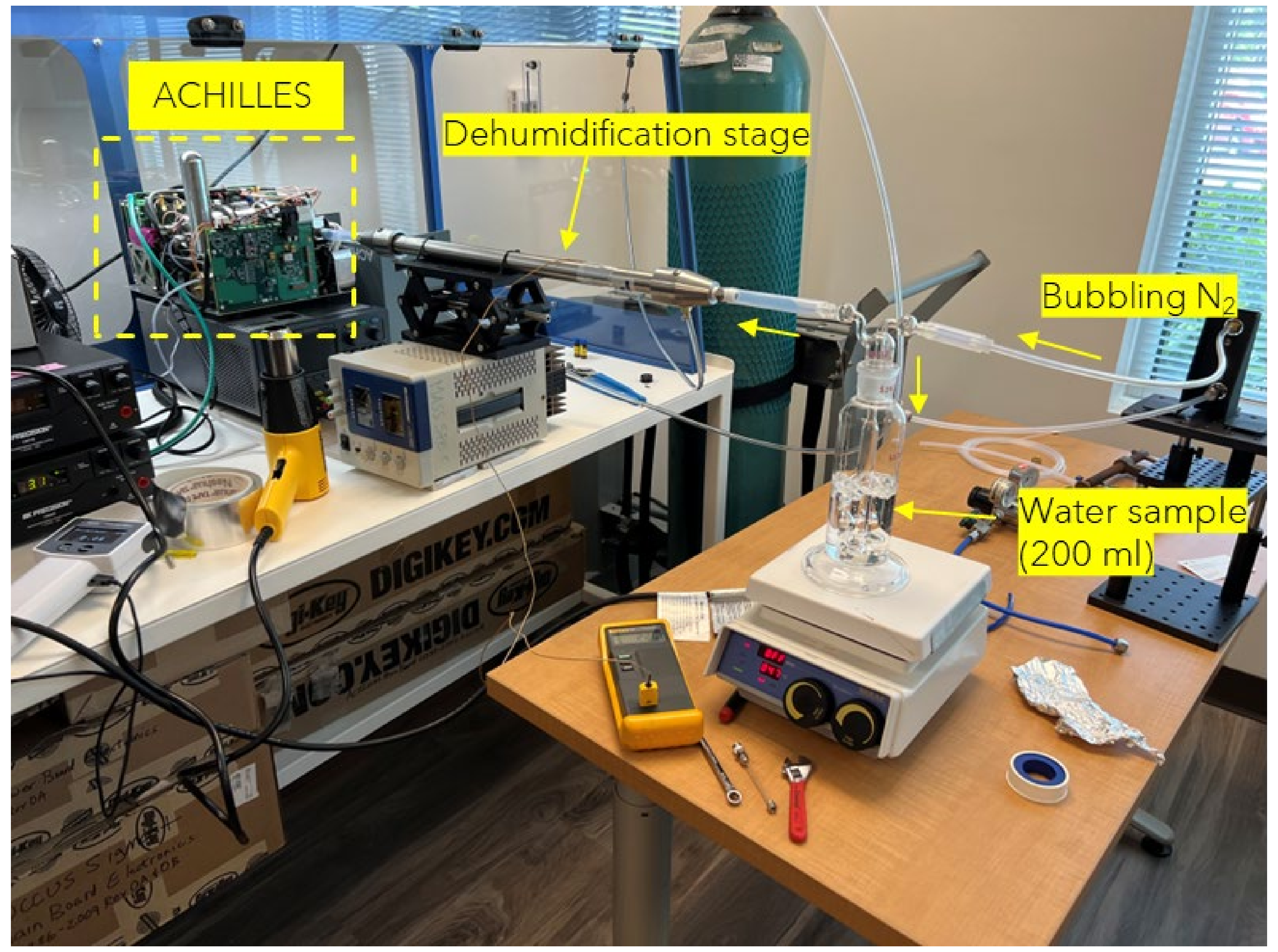

Figure 2. Testbed developed to the demonstrate the feasibility of ACHILLES as a water sensor.

Detect-ION collected early feasibility data using a setup with a provision to 1) bubble high purity N_2 gas at 3 LPM through the water sample; 2) control the temperature of the water sample; and 3) dehumidify the sample using a Nafion-based stage between the water sample and ACHILLES's preconcentration stage to avoid the sorbent from being exposed to excessively high humidity levels. **Figure 2** shows a testbed photo illustrating the three main subsystems, namely the ACHILLES sensor, dehumidification stage and the bubbling setup.

To test the system for a range of sampling times, we made a 250 µg/L dilution of EPA-624 using reagent water that was obtained from a deionized and purified water source. The EPA-624 was procured from Sigma Aldrich (Supelco 46966) with a concentration of 2000 µg/ml each of 26 VOC components in methanol. We used 25 µL of the EPA-624 in 200 mL of reagent water to make the 250 µg/L analyte solution. To ensure our test setup was clean enough, we ran a test using pure reagent water. Any contaminants detected in this test would represent contributions from the reagent water and/or the plumbing/surfaces between the water bubbler system and the dehumidification stage.

Preliminary Results

TIC A of **Figure 3** shows the pure reagent water total ion chromatograph (TIC) and shows that no signal is detected above the instrument noise. Subsequently, we sampled the EPA-624 standard for increasingly longer times and obtained TIC B, C and D. Between each experiment set, we performed internal blanks to ensure the preconcentrator sorbent was clean for the subsequent experiments. The MS sensitivity is fixed but the overall system sensitivity depends on the time for which the gas stream was sampled through the preconcentrator.

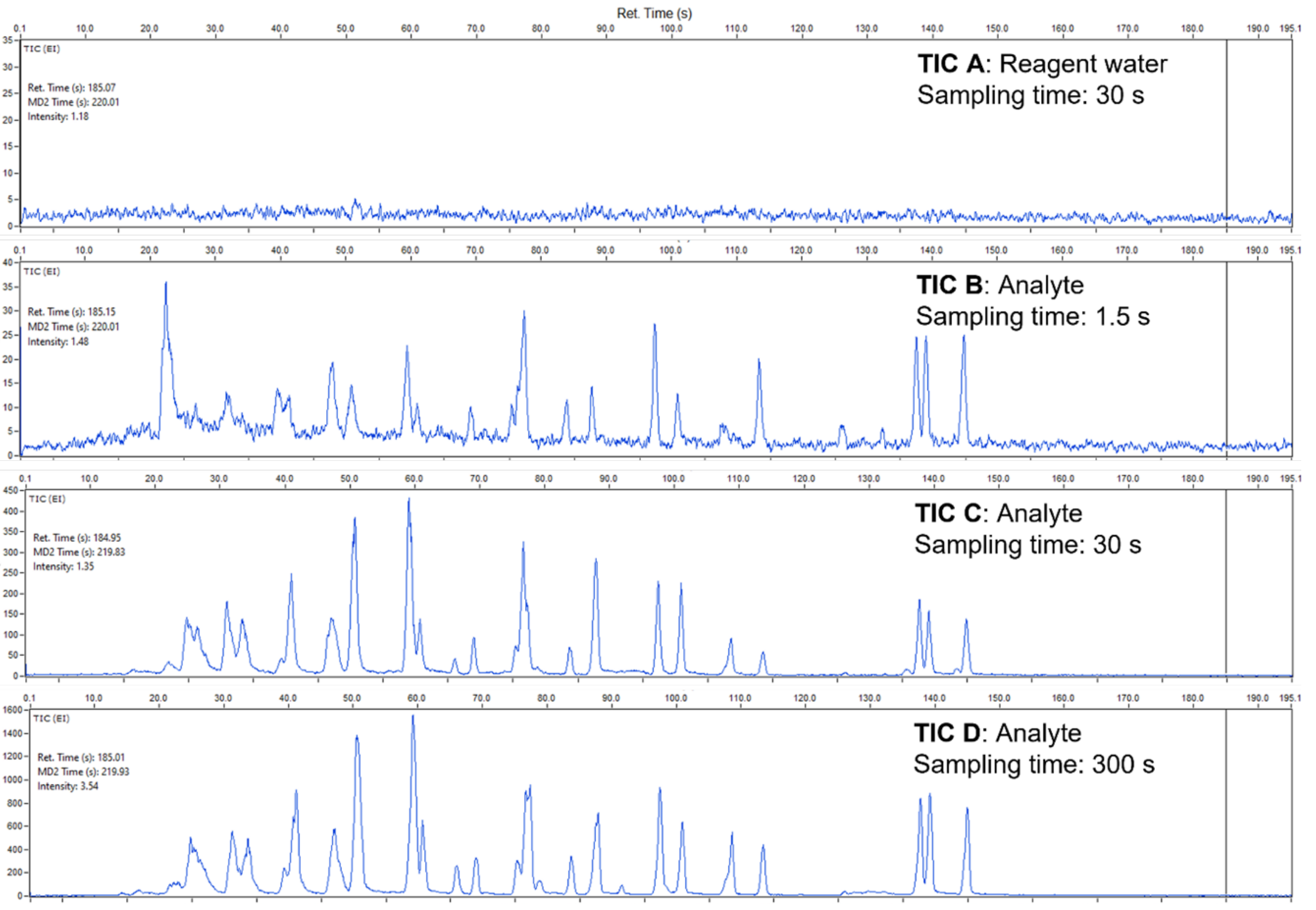

Figure 3. TIC plots generated by the ACHILLES system using the ad hoc bubbling system to detect VOC contaminants from a water sample (250 µg/L at 22° C) for a range of sampling time. Note that TIC A shows that prior to adding the EPA-624 standard, the system response from bubbling through a pure reagent water sample was below ACHILLES noise.

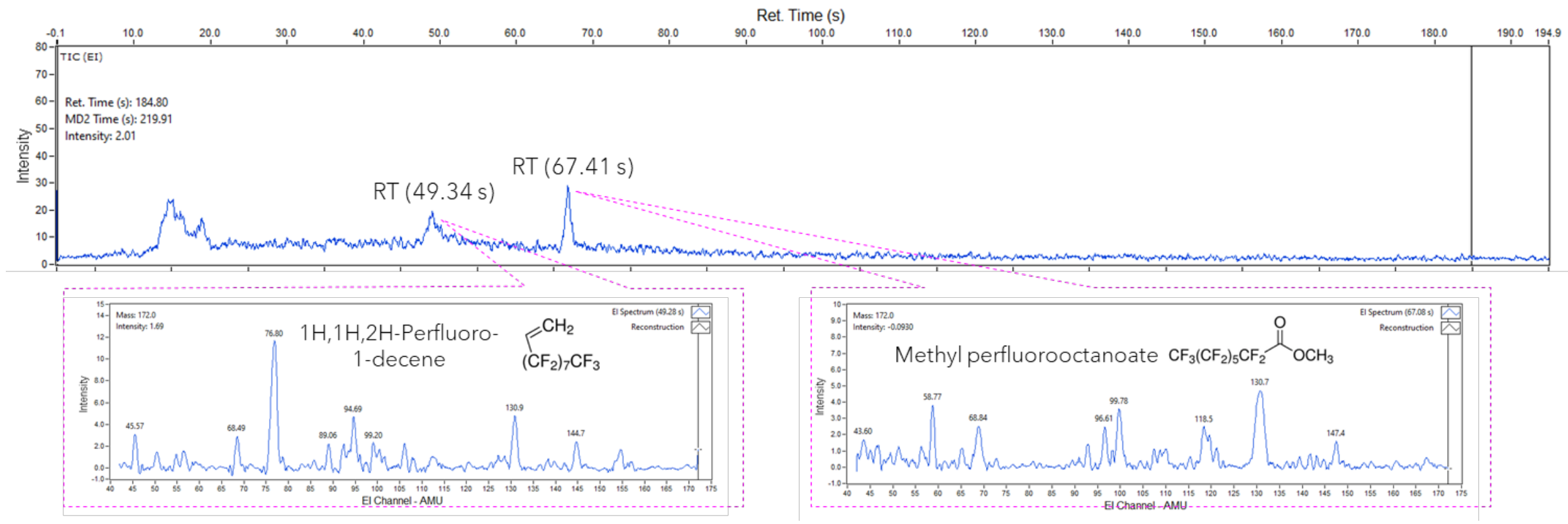

Figure 4. We also generated TIC plots by using the ACHILLES and ad hoc bubbling system to detect PFAS in a water sample (10 µg/L at 36° C) for 30-s sampling time.

Summary

- Our engineering design review indicates that ACHILLES can be easily modified to incorporate a compact water sampling stage, upstream of the VOC preconcentrator.
- The sorbent can be protected from high humidity levels during sampling via a Nafion or molecular sieve-based desiccant stage.
- An elevated temperature range of the water sample enables detection of SVOCs.

Acknowledgments

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