A comparison of sorbent materials for offline TD-GC-MS breath analysis

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Introduction

- Offline analysis of breath samples by TD-GC-MS remains the gold standard for the detection of novel potential biomarkers in breath.
- The sensitivity of breath analysis is affected by a wide range of confounding factors including the sampling device and the sorbent materials used to trap VOCs.
- Due to the high relative humidity of breath there is a trade off between the ability of sorptive materials to retain the VOCs of interest and the amount of water captured on the tube [1,2].
- **Here we show that recovery of VOCs on sorbent tubes is impaired when loaded in a humid gas stream and that polar compounds are more effected.**

Table 1 – Summary of commonly used sorbent materials in breath research

Sorbent type	Material	Captive range	Surface area (m^2/g)	Hydrophobicity
Porus polymers	TenaxTA	C7 - 26	35	Very Good
	TenaxGR	C7 - 30	35	Very Good
Graphitized carbons	Carbograph 1TD	C5 – C14	100	Good
	Carbograph 2TD	C8 - 20	12	Good
	Carbograph 5TD	C5 - 8	560	Poor
Carbon molecular sieves	Carboxen 1000	C2 - 3	1200	Very poor
	Carbosieve SIII	Ethane - C5	800	Very poor

Methods

- Three sorbents were selected for comparison based on the most commonly used mixes in breath VOC analysis.
- Included one single bed tube, TenaxGR, and two dual bed tubes, TenaxTA/Carbograph1TD (TA1TD) and TenaxTA/Carbograph5TD (Biomonitoring).
- The average weight of water loaded onto sampling tubes when using the ReCIVA was calculated and a water bubbler was setup to humidify N_2. Appropriate dry purge times at 50 mL / min were obtained for each sorbent at loading volumes of 0.5 and 1 L.

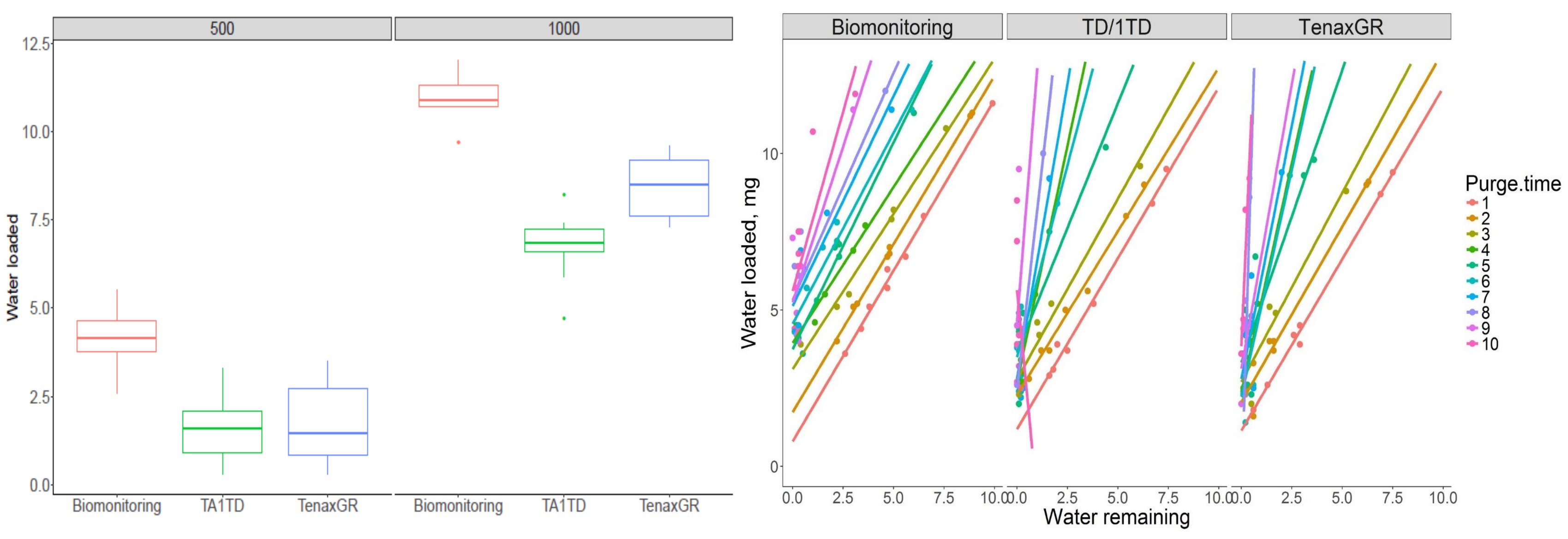

Figure 1 – Weights of water loaded onto the different sorbents using the loading rig (A). Water remaining after different purge times.

- Initial tests were performed using breath and a standards mix to investigate the effects of loading VOCs in humid gas. Subsequently breath samples were collected and analysed to compare the different sorbents.

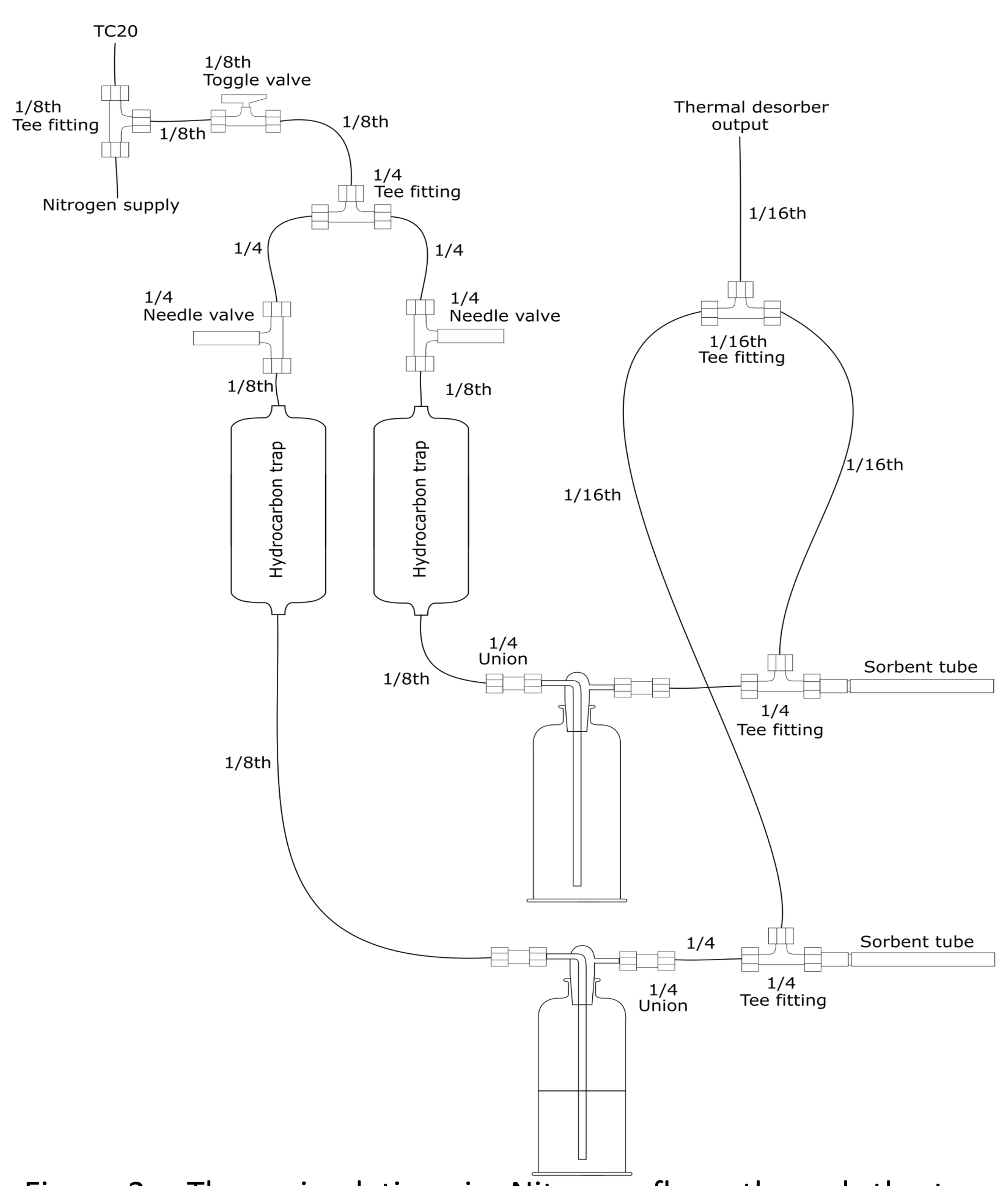

Figure 2 – The recirculation rig. Nitrogen flows through the traps before passing through dreschel flasks, one of which contains water. Helium carrying VOCs from the TD is tee'd into the flow just before the target sorbent tubes.

- The standards mix contained 30 VOCs, including a range of commonly found breath compounds and an alkane ladder.
- Lower airway breath samples were collected using a ReCIVA at the 2 sampling volumes and a flow rate of 200 mL / min.
- VOCs were loaded onto the sorbent tubes for comparison using the recirculation rig (figure 2). This allowed the standards mix or breath samples to be thermally desorped into flows of humid or dry N_2.

Results

- TenaxGR and TA1TD tubes required 4 min of purging for 0.5 L of breath sampled and 8 min for 1 L. Biomonitoring tubes required 8 and 16 respectively.
- All compounds in the standards mix and sampled breath showed reduced recovery in wet gas compared to dry gas when they were desorbed into the recirculation rig (figure 3).

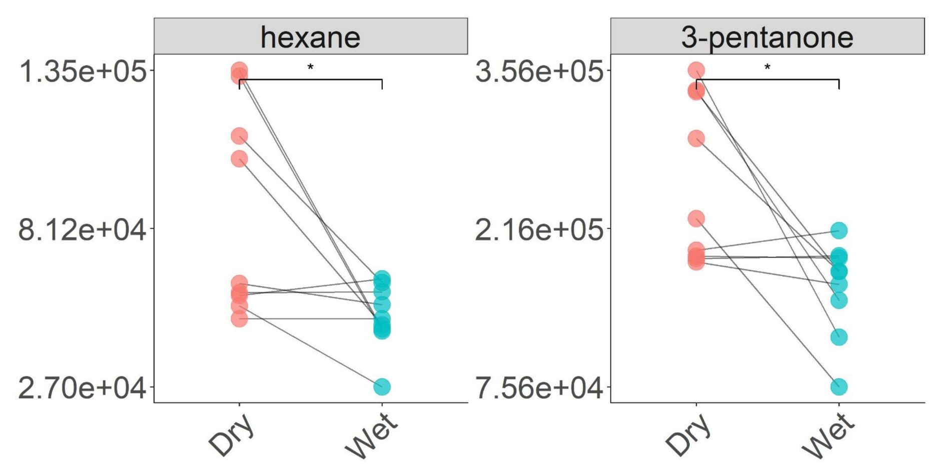

Figure 3 – Example compounds from the standards mix when desorbed into wet and dry gas.

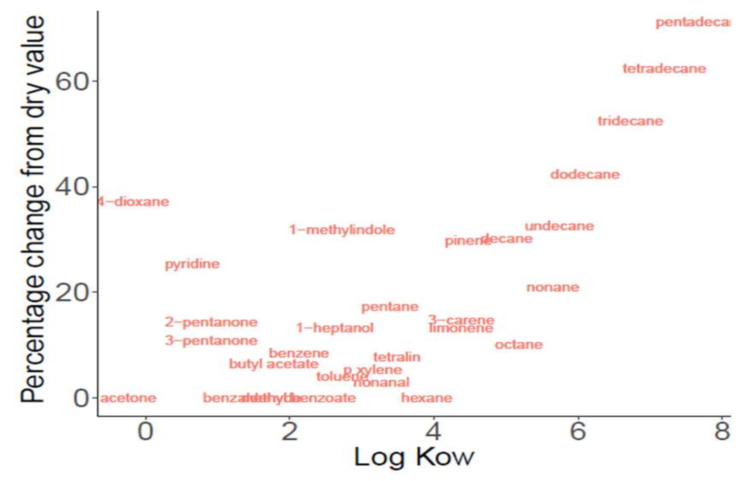

Figure 4 – The effect of chemical properties on the recovery of VOCs in wet and dry gas.

- Recovery of compounds with low octanol/water partition coefficients (Kow), such as acetone, was less affected by loading in wet gas than those with high Kow values such as pentadecane (figure 4).
- Repeat sampling on the ReCIVA allowed the sorbents to be separated by PC-DFA (figure 5).
- The first discriminant allowed separation of all 3 sorbents and was predominantly driven by higher recovery of light VOCs in Biomonitoring and TA1TD than TenaxGR

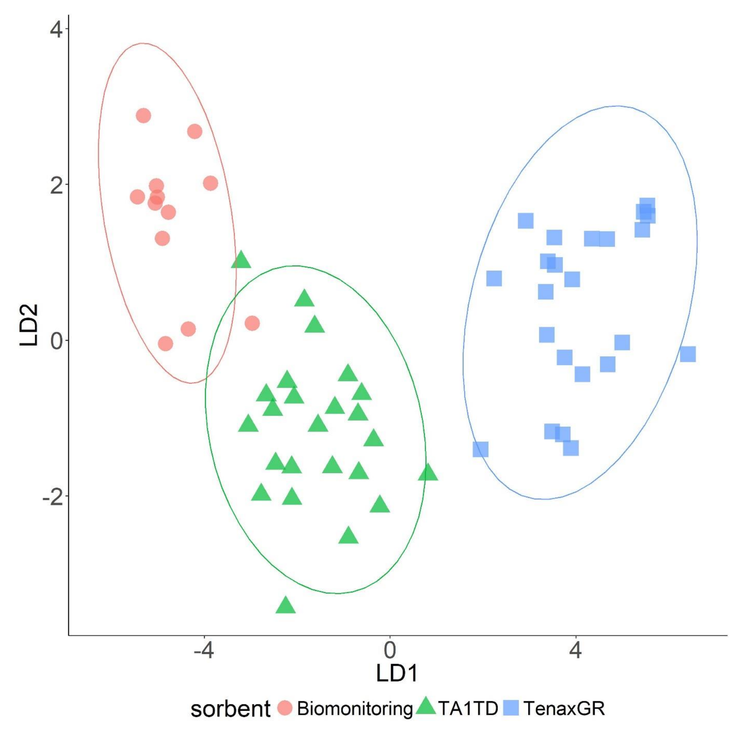

Figure 5 – A PC-DFA with 95% confidence intervals of breath VOCs trapped on the different sorbents using the ReCIVA.

Discussion

- The selection of sorbent tubes remains an important parameter in the design of breath VOC experiments and establishing appropriate dry purging times for the selected sorbents is required to ensure consistent results from TD-GC-MS.
- The recovery of VOCs is affected by the humidity of the carrier gas and is a significant issue for devices such as the ReCIVA where large amounts of water are loaded onto the tube.
- Due to the recovery of compounds being affected by their chemical properties care should be taken to try and control the humidity of the gas that is loaded onto sorbent tubes.
- Further work is required to determine if the hydrophobicity of the sorbent impacts the loss of signal.

References:

1. Gallego, E., Roca, F. J., Perales, J. F., & Guardino, X. (2011). Comparative study of the adsorption performance of an active multi-sorbent bed tube (Carbotrap, Carbopack X, Carboxen 569) and a Radiello®diffusive sampler for the analysis of VOCs. Talanta, 85(1), 662–672.
2. Ras, M. R., Borrull, F., & Marcé, R. M. (2009). Sampling and preconcentration techniques for determination of volatile organic compounds in air samples. TrAC - Trends in Analytical Chemistry, 28(3), 347–361.