

EGU2020-11630

<https://doi.org/10.5194/egusphere-egu2020-11630>

EGU General Assembly 2020

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A novel method to quantify exchangeable hydrogen fraction in organic matter

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Stable isotope measurements of nonexchangeable hydrogen ($\delta^2\text{H}_n$) of bulk organic matter has emerged as a tool, with a wide range of applications in biology, biogeochemistry and forensics. However, reproducible and precise measurements of $\delta^2\text{H}_n$ between laboratories and methods are still challenging. One of the largest impediments to obtain accurate isotope ratios is to use reference materials of similar exchangeable hydrogen fraction (f_x) to the matrix of interest. The organic matter has typically three pools of hydrogen (H): (i) the adsorbed water, which can be minimized by extensive drying, (ii) the carbon bound H (the fraction of interest), which is non-exchangeable and cannot be removed and (iii) the non-carbon bound H, (i.e. N-, COO-, O-, and S-bound H) that cannot be removed but can be readily exchanged with the environmental moisture. Quantification of f_x based on dual water vapor isotope exchange and Isotope Ratio Mass Spectrometry (IRMS) have shown large variability in f_x between studies for the same organic matter type such as keratin. High variability in f_x between samples and standards can translate into a large impact on the measured isotopic values. Here we used a novel approach to independently quantify f_x in 21 natural organic material sources with minimal sample manipulation based on ^1H - ^2H exchange experiments and quantified through proton based liquid-state nuclear magnetic resonance (^1H -NMR) spectroscopy. The experiments were carried out at room temperature by immersing separate solid powdered samples in deuterated dimethylsulfoxide (background) and deuterium oxide (^2H source) followed by the quantification of the water generated in the supernatant fraction through ^1H -NMR using glucose as reference. At the same time, samples were analyzed through the most recent procedure of dual water vapor isotope equilibration method using online drying and equilibration in a UniPrep carousel. We discuss these findings and suggest that the proposed ^1H -NMR method of quantifying f_x is an independent and novel approach that can contribute to a better understanding of H exchangeability in a wider range of organic materials, critical for accurate measurement of the $\delta^2\text{H}_n$.