

Title: Litter decomposition enhances volatile organic compound emission from a freshwater wetland: insights from year-round in situ field experiments

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The authors report VOC fluxes from the decaying leaf litter of a freshwater wetland plant, *Phragmites australis*. The reported fluxes include an impressive list of 62 VOCs: 5 aromatics, 15 OVOCs, 30 NMHCs, 9 HVOCs, 3 VOSCs. Many of these compounds are difficult to quantify well, thus requiring proof of careful methodology. The proof, unfortunately, is not provided, leading to doubts about data quality. The problem is compounded by the use of incomplete references. The methods section cites two papers (Wang et al, 2023 and Liu et al. 2021) to broadly explain 'analysis steps'. The cited studies employ slightly different methods and themselves cite a range of earlier papers for broad methodological statements. It becomes a nearly impossible task to follow the actual methods of VOC quantification. Specific papers should be cited for specific methods, and only if they truly align. For example, Wang et al., 2023, uses a different instrument altogether (GC-MSD-FID from Agilent vs. a GC-MSD from Shimadzu), so it should be made evident why this paper is cited.

Below, I compile a checklist for methodology that should be included for field measurements of VOC fluxes. While several items are addressed in this study, other critical aspects are missing.

<b>SAMPLE STORAGE</b>	
Canister blank	Yes. Filled with N2, evacuated, tested for contamination. Described in S1
Canister stability	Stability of compounds was not tested for the compounds of interest, even though they have different degrees of stability in canisters. Compounds with poor stability or even production within canisters from other compounds (e.g., VOSCs) need to be checked.
<b>FIELD SAMPLING</b>	
Flux chamber blank (sampling equipment only)	Chamber blank/control experiments using empty chambers were not conducted, which is concerning because of the use of foam board to make the chamber float. Separate issue: chambers are described as 1.1m x 1.1m x 1.1m (Line124), but it appears that another smaller chamber (35x35x15cm) is then placed inside the larger box (Line 140), is that correct?
Leakage / sample replacement	A correction is needed to account for the removal of 3.2 L samples taken from an 18 L chamber. This is not a large correction, given that the first sample is taken at the beginning when outside air = inside air, but it should be considered (if using 3 points instead of 2). Leakage/sample replacement may be responsible for the non-linearity of concentration change that was observed.
Replication	Yes. 3 times per treatment of leaf litter (Line 126-128).

<b>INSTRUMENT</b>	
Instrument blanks	Yes. N2-filled canisters analyzed. Described in S1
Hardware and chromatography details	Yes. Entech pre-concentrator with Shimadzu GC-MSD. Temperature programming. Note that running in SCAN mode is less precise than SIM (Lines 156-170)
Method of Peak ID	Yes. By retention time and m/z matching standards.
Instrument precision	Not reported. Precisions are needed for each compound.
Duplicate runs	Not reported.
Detection limit	Not reported.
Use of Gas standards	Diluted PAMS and TO-15 standard mixtures are cited. DMS and DMDS standards were used but not described. For VOCs without standards, they were “semi-quantified based on the species whose RTs were close to them or based on the species which have a similar chemical structure to them.” This is not an appropriate method, as there is no reason to believe the instrument sensitivity or ion fragmentation will be the same. These compounds must be removed from any quantitative analysis.
Gas standard traceability	Missing. Need to add source of standard and standard traceability (if available)
Calibration curves	Calibration curves were run at: 0.5, 1, 5, 15, 30 ppb for PAMS and TO-15. 0.1, 0.5, 1, 2, 4 ppb for sulfur.
Cal Curve over range of concentrations	May not be valid, as VOC concentrations / ranges were not reported. Calibration curves might not be appropriate for the range of concentrations observed. If concentrations <0.5 ppb (or <100 ppt for VOSCs), then the calibration curve is a one point calibration.
<b>FLUX CALCULATIONS</b>	
Curve fitting procedures	“VOC concentrations in chamber headspace had already leveled off at 30 in. Thus, here the first two points, which could capture the initial fluxes, were used to calculate VOC fluxes (Zhang et al., 2021).” What is meant by ‘leveled off’, and do all chambers show this trend? Zhang et al. only used 2 point flux calculations for one compound and only in the <10% of cases when $r^2 < 0.75$ . Here, the 2-point method appears to be used for all VOCs. Is the system leaky or is there a dilution effect from withdrawing large samples (see above)?
Flux detection limits	Given the precision of each compound, what are their flux detection limits? When is a flux determined to be not significantly different from zero? How are these observations treated?

In summary, numerous revisions are needed to address the methodological omissions and to improve the quality of flux data. Measuring 62 VOC fluxes is impressive, but how many of these fluxes are real? I suspect several of the measurements have larger uncertainties than presented and/or are below detection levels. Also, aggregating fluxes by VOC type can summarize a large set of results, but this process obscures what is happening with specific

compounds. By aggregating by mass, heavier compounds can dominate the overall picture. Please provide more information about the measurements that are defensible, add caveats where needed, and exclude results that are unreliable. Only then can the reader be fully able to assess the scientific contributions of the work.