Review for Measuring molecular singlet oxygen ($^1O_2$*) from atmospheric photosensitizers: Intercomparison of techniques, irradiation setups, data analysis and protocol recommendations

Gemmell et al. compared singlet oxygen ($^1O_2$*) measurements from four photosensitizers across four photoreactor setups at three institutions. Using their results, they determined the factors influencing the rates of light absorbance, $^1O_2$* steady-state concentrations, and quantum yields. Consequently, they made five recommendations to improve the accuracy and reproducibility of $^1O_2$* measurements, which include considering wavelength-dependent quantum yields, avoiding suppression of $^1O_2$*, controlling and reporting photoreactor temperature, considering light scattering from nanoparticles, and conducting control experiments. These recommendations will help standardize $^1O_2$* measurements across different laboratories in studying photochemical processing of atmospheric aerosols and droplets. In general, this is a thorough and valuable intercomparison study. The authors have conducted rigorous control experiments and thoughtfully addressed potential artifacts. This manuscript makes a valuable contribution to standardizing $^1O_2$* measurements. The recommendations are practical and well-supported by the intercomparison data. However, there are some points that need clarification before acceptance for publication:

Major Comments

- Scattering artifacts: The supplement's Section S8 and Figures S15-S17 address a previously underappreciated artifact. The sensitivity analysis showing up to 48.8% error in Rabs for lignin is compelling. However, I notice that the lignin absorbance before and after 0.22 μm filtration (Figure S15) shows negligible difference, yet the Mie scattering modeling (Figure S16) suggests significant scattering contributions. This apparent contradiction needs explanation in the main text. I suggest that the authors clarify that scattering can occur from particles smaller than the filter pore size, and that the absence of filtration effect does not rule out scattering artifacts. This nuance is important for readers who might incorrectly conclude that filtration alone solves the problem.
- UCD photon flux calibration (Section S11): This section reveals a significant methodological challenge that deserves more attention in the main text: The observation that "the ratio of the peak in photon flux at 548 nm to the peak at 347 nm varies from 2.0 to 5.2" across 60 measurements, and that they had to use perinaphthenone to constrain the long-wavelength flux. I suggest adding a brief discussion in Section 3.2.2 or 3.2.3 about the importance of validating spectrophotometer measurements with chemical actinometry or reference sensitizers, especially when internal reflections or positioning variability may affect spectral shape. This is a practical recommendation that would be useful for home-built photoreactors.
- Temperature dependence: Figure S19 is particularly striking: the 30°C vs. 22°C experiments show dramatically different [$^1O_2$*]ss even after applying temperature-corrected rate constants. This suggests that temperature affects not just the probe kinetics, but also potentially the sensitizer photophysics and/or $^1O_2$* production efficiency. The recommendation to control temperature within 20 to 25°C seems sound, but the manuscript should acknowledge that the underlying causes of this temperature sensitivity are still not fully understood and warrant further investigation. Also, the recommendation to control "within a minimum range of 20 to 25°C" is vague. What is the acceptable variability within an experiment? Across experiments? I suggest the authors consider providing quantitative guidance (e.g., ±1 °C, ±2 °C).
- Line 580: "[FFA]0 < 145 μM": I understand that this threshold comes from Ossola et al., but is it universally applicable? I think the appropriate concentration may depend on the

photosensitizer's $^1O_2$* production rate and the light source intensity? I suggest the authors consider adding guidance on how to verify that probe scavenging is negligible for a given system.

- Line 225: The statement that "the triplet state of perinaphthenone does not react with furfuryl alcohol" is supported by Schmidt et al., but is this universally true across all conditions (pH, concentration ranges)? I suggest the authors consider adding a brief note about the conditions under which these holds.
- Relative vs. Absolute quantum yield methods: This paper presents both methods but it could more clearly guide readers on when to use each approach. The statement that values were "consistently 15% within each other" (line 458) is helpful, but what is the threshold for "acceptable" agreement? Providing more specific guidance will be useful.

Specific Technical Comments

- Units' consistency: Table S1 reports Rabs for perinaphthenone in units of $\times 10^{-6}$ mol_{photon} L^{-1} s^{-1}, which is consistent with the main text Figure 4a. However, equation 1 in the main text gives units of $mol_{photons}$ cm^{-2} s^{-1}. This discrepancy should be resolved. There should be consistency between the equations, text, and figures.
- Blank controls (Section S1, Figure S1): All three labs show negligible FFA decay in blank controls, which is reassuring. However, the Ircelyon blank appears to show a slight downward trend. Is this within experimental uncertainty? Also, to avoid any confusion, I suggest renaming "Lyon Blank" in the figure's legend to "Ircelyon Blank".

Minor Comments

- Figure 5 caption: "Normalized to peak (i.e., peak value = 1)": The authors should consider adding "for each photoreactor setup individually" to clarify that normalization is per setup, not global.
- Atmospheric implications section: This section is somewhat general. The authors should consider adding a more concrete example of how the recommendations would improve model parameterization.
- Building a new photoreactor Section: This is interesting but feels disconnected from the rest of the main text. The authors should consider integrating it into the recommendations or moving to SI.
- Recommendation 5 (Control experiments): The authors should consider stating that results from deoxygenated experiments should be interpreted cautiously since removing O_2 changes the system fundamentally (e.g., ^3C* lifetime increases, other pathways may emerge). The absence of FFA decay in N_2-purged samples confirms no ^3C* + FFA reaction, but I don't think it proves that $^3C^* + O_2 \rightarrow {}^1O_2^*$ is the only pathway in oxygenated conditions.
- Section S8.2: The sentence "Using a particle concentration of 1.18×10^{13} m^{-3} and a mean particle diameter of 142 nm (values scaled from Bieber et al. (2024))". The term "scaled" implies modification. Please clarify whether these are directly from Bieber et al. or adjusted for this study. If adjusted, the authors should explain the scaling rationale.
- Section S11, line 86: "… perinaphthenone 1O2* quantum yield" There is a typo here (missing superscripts and subscripts). Should be "$^1O_2$*".