Responses to Referee # 3

We thank the reviewer for the careful review of our manuscript. The comments and suggestions are greatly appreciated. All the comments have been addressed. In the following, please find our responses to the comments one by one and the corresponding revisions made to the manuscript. The original comments are shown in italics. The revised parts of the manuscript are highlighted.

Anonymous Referee #3

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General Comments

This study identifies important HOMs (highly oxygenated organic molecules) from isoprene + NO3 reaction through chamber experiments. The identification of HOMs from NO3 oxidation have been less studied than those from OH or O3 oxidation, so this study fills an important gap in atmospheric chemistry. This study uniquely and in great detail connects many measured compounds to possible mechanistic formation pathways.

I suggest this paper be published with some minor revisions as specified below.

These minor revisions include some improvements to the mechanistic understanding and providing more information on how to interpret these laboratory results within the context of how SOA forms from isoprene + NO3 in the ambient atmosphere.

Specific Comments:

Page 5, 149. From the measurements of RO2, HO2, and NO3, can you approximate the fate of the RO2 radical in your experiment? Were conditions such that the RO2 predominantly reacted with another RO2, NO3, or HO2? Do you have an estimate of the lifetime of the RO2 radical in your experiments and how this compares to the RO2 lifetime in the ambient atmosphere. RO2 radical lifetime is often longer in the atmosphere compared to experiments. Would this possibly enhance the SOA yield for ambient conditions for HOMs?

Response:

In our experiments we measured radical concentrations directly. The reaction of RO_2 with NO_3 was determined to dominate over the reaction RO_2 with RO_2 and with HO_2 and HO_2 concentration, although the measured RO_2 and HO_2 are subject to uncertainties due to interference from NO_3. This is consistent with our understanding of the reaction system as there is no extra HO_2 source (Vereecken et al., 2021). However, a recent study found that a large portion of RO_2 is not measured by LIF and thus RO_2 was underestimated (Vereecken et al. 2021). Therefore, we expect the reaction of RO_2+RO_2 to be also important. Overall, we estimate that the RO_2 fate is dominated the reaction RO_2+NO_3 with significant contribution of RO_2+RO_2.

We also estimated RO_2 lifetime using the measured RO_2, NO_3, and HO_2 concentrations. The RO_2 lifetime is approximately 20-50 s in our experiments, which is generally comparable or shorter than the lifetime of RO_2 in the ambient atmosphere at night, varying from several 10 s to several 100 s (Fry et al., 2018), depending on the NO_3, HO_2, and RO_2 concentrations as well as specific RO_2. Assuming a HO_2, RO_2, and NO_3 concentration of 5 ppt, 5 ppt (Tan et al., 2019), and 300 ppt (Brown and Stutz, 2012) respectively, the RO_2 lifetime in our study is representative of nighttime RO_2 lifetime of urban atmosphere.

If the RO_2 lifetime increased, the autoxidation of RO_2 would be more important relative to its bimolecular reactions. This may enhance HOM yield and thus enhance SOA yield. However, on the other hand, reduced rate of RO_2+RO_2 at longer RO_2 lifetime, producing low-volatility dimers, can reduce the SOA yield via reducing dimer yield (McFiggans et al., 2019; Pullinen et al., 2020).

In the revised manuscript, we have added discussion on the fate and lifetime of RO_2.

"In these experiments, RO_2 fate is estimated to be dominated by its reaction with NO_3 according to the measured NO_3, RO_2, and HO_2 concentration and their rate constants for the reactions with RO_2 (MCM v3.2(Jenkin et al., 1997; Jenkin et al., 2003; Saunders et al., 2003; Jenkin et al., 2015), via website: http://mcm.leeds.ac.uk/MCM) despite uncertainties of the measured RO_2 and HO_2 concentration due to interference from NO_3. As a large portion of RO_2 is not measured by LIF (Vereecken et al. 2021) and thus RO_2 is underestimated, we expected the reaction of RO_2+RO_2 to be also important. Overall, we estimate that the RO_2 fate is dominated the reaction RO_2+NO_3 with significant contribution of RO_2+RO_2."

"The RO_2 fate is dominated the reaction RO_2+NO_3 with significant contribution of RO_2+RO_2, which can also represent the RO_2 fate in the urban areas and areas influenced by urban plume. Yet, it cannot represent the chemistry in HO_2-dominated regions such as clean forest environment (Schwantes et al., 2015)."

"The RO_2 lifetime is approximately 20-50 s in our experiments, which is generally comparable or shorter than the lifetime of RO_2 in the ambient atmosphere at night, varying from several 10 s to several 100 s (Fry et al., 2018), depending on the NO_3, HO_2, and RO_2 concentrations. Assuming a HO_2, RO_2, and NO_3 concentration of 5 ppt, 5 ppt (Tan et al., 2019), and 300 ppt (Brown and Stutz, 2012) respectively, the RO_2 lifetime in our study is comparable to the nighttime RO_2 lifetime (50 s) found in urban locations and areas influenced by urban plume. In areas with longer RO_2 lifetime such as remote areas, the autoxidation of RO_2 is expected to be more important relative to its bimolecular reactions. This may enhance HOM yield and thus enhance SOA yield. However, on the other hand, at lower RO_2 concentration and thus longer RO_2 lifetime, reduced rates of RO_2+RO_2 reactions producing low-volatility dimers can reduce the SOA yield via reducing dimer yield (McFiggans et al., 2019; Pullinen et al., 2020)."

Page 5, line 160. Please provide more detail here on using the H2SO4 sensitivity for the HOMs. Are there certain HOMs this assumption would apply more too? For example, does this assumption apply more to HOMs that are more oxygenated or have a higher C? Please specify the overall uncertainty in HOMs in the main text (It looks like you calculate this in the supplement). Is there need to add uncertainty here for using the H2SO4 sensitivity directly for the HOM sensitivity?*

Response:

Accepted.

In the revised manuscript, we have added more details on the H_2SO_4 sensitivity. Also we have added the overall uncertainty in HOM yield, which was shown in the supplement.

Since HOM contain more than six oxygen atoms and their cluster with nitrate ions are quite stable (Ehn et al., 2014), the charge efficiency of HOM is thus assumed to be equal to that of H_2SO_4, which is close to the collision limit (Viggiano et al., 1997). We do not expect this sensitivity applies more to certain HOM than other HOM since HOM are all highly oxygenated with multiple functional groups. If HOM do not charge with nitrate ions at their collision limit or the cluster formed break during the short residence time in the charger, its concentration would be underestimated as pointed by Ehn et al. (2014). Thus, our assumption provides a lower limit of the HOM concentration. We have further discussed the uncertainty of using the sensitivity of H_2SO_4 for HOM as follows.

"Since HOM contain more than six oxygen atoms and their clusters with nitrate ions are quite stable (Ehn et al., 2014), the charge efficiency of HOM is thus assumed to be equal to that of H_2SO_4, which is close to the collision limit (Viggiano et al., 1997). If HOM do not charge with nitrate ions at their collision limit or the clusters formed break during the short residence time in the charger, its concentration would be underestimated as pointed by Ehn et al. (2014). Thus, our assumption provides a lower limit of the HOM concentration."

Figure1. Please add the names for the top m/z on panel b like done for panel a. It looks like many of the top m/z's are the same, but maybe some are unique. It's hard to compare by eye because the m/z lines are so small. Coloring the m/z label by their type listed in the pie chart would also be useful for the reader.

Response:

Accepted.

Page 11 line 292: Because you can measure OH and NO3, can you approximate how much isoprene and the first-generation NO3 nitrates react with OH versus NO3 in your experiments? This may lend insight into the products you are detecting. For example, the C5H8O2 compounds mentioned above seems more likely to form from OH oxidation than the H-shift in scheme S1a and S1b (Kwan 2012 Fig 5)? The reaction rate constant for the first-generation nitrates reaction with NO3 is low compared to OH rate constant (Wennberg 2018). From this information, can you connect how your laboratory results should be interpreted to the ambient atmosphere? For example, how long lived are NO3 derived first-generation nitrates in the ambient atmosphere are they likely to react again with NO3 or with OH at dawn?

Response:

In our experiments, the reaction of OH with isoprene contributed less to the isoprene consumption than the reaction of O_3 with isoprene as the OH is mainly formed by isoprene+O_3 and the OH yield is less than one. Therefore, the reaction of OH with isoprene is negligible (<3%) for isoprene loss. This is consistent with the contribution determined using measured OH concentration, despite some uncertainty of measured OH concentration due to the interference by NO_3. In light of the negligible role of OH in isoprene consumption, we think that $C_5H_8O_2$ is more likely formed by the reaction with NO_3 and subsequent H-shift.

In the revised manuscript, we have discussed the role of OH in isoprene consumption.

"The contribution of the reaction of isoprene with trace amount of OH, mainly produced in the reaction of isoprene+O_3 via Criegee intermediates (Nguyen et al., 2016), is negligible as the OH yield is less than one (Malkin et al., 2010) and thus its contribution is less than that of isoprene+O_3. This is consistent with the contribution determined using measured OH concentration, despite some uncertainty in measured OH concentration due to the interference from NO_3."

For the first-generation NO_3 nitrates, their reaction rates with OH and with NO_3 calculated using the reaction constants (Wennberg et al., 2018) and OH and NO_3 concentrations are comparable, with both contributing significantly to the loss of first-generation NO_3 nitrates as the rate constant with OH is much higher than that with NO_3.

Regarding the lifetime of first-generation nitrates in the ambient atmosphere, according their rate constants with OH and NO_3 (Wennberg et al., 2018), their lifetime are 5 h and 1.3-4 h, respectively, with respect to the reaction with OH and NO_3 assuming a typical OH concentration of 2×10^6 molecules cm^{-3} (Lu et al., 2014; Tan et al., 2019) and NO_3 concentration of 100-300 ppt in urban areas. Therefore, they likely react further with OH and NO_3 at dawn. Therefore, our results are relevant to the ambient urban atmosphere and areas influenced by urban plumes.

In the revised manuscript, we have added discussion on the relevance to ambient atmosphere as follows.

"We observed the second-generation products formed by the reaction of first-generation products. The lifetime of first-generation nitrates in the ambient atmosphere, according their rate constants with OH and NO_3 (Wennberg et al., 2018), are ~5 h and ~1.3-4 h, respectively, with respect to the reaction with OH and NO_3 assuming a typical OH concentration of 2×10^6 molecules cm^{-3} (Lu et al., 2014; Tan et al., 2019) and NO_3 concentration of 100-300 ppt in urban areas (Brown and Stutz, 2012). Therefore, they have the chance to react further with OH and NO_3 at dawn. In our experiments, the lifetimes of these first-generation nitrates with respect to OH and NO_3 are comparable to the aforementioned lifetime due to comparable OH and NO_3 concentrations with these ambient conditions. Therefore, our findings on the second-generation products are relevant to the ambient urban atmosphere and areas influenced by urban plumes. Some of these products such as $C_5H_{810}N_2O_8$ and multi-generation nitrooxyorganosulfates have been observed in recent field studies in polluted megacities in east China (Hamilton et al., 2021; Xu et al., 2021)."

"3N dimer such as $C_5H_9N_3O_{10}$ as well as 2N-monomers such as $C_5H_8N_2O_8$ and $C_5H_8N_2O_{10}$ have been observed in a recent field study in polluted cities in east China (Xu et al., 2021)."

Adding pictures of the molecules to schemes S1-S4 would be very beneficial for the reader.

Response:

Accepted. We would also like to note that these schemes and other schemes in this study only show example isomers and pathways to form these molecules. It is likely that many of the reactions occurring are not the dominant channels as otherwise there would be much higher HOM yield. We have added these notes in the revised manuscript.

"We would like to note that the scheme and other schemes in this study only show example isomers and pathways to form these molecules. It is likely that many of the reactions occurring are not the dominant channels as otherwise there would be much higher HOM yield as discussed below."

Page 13 line 350. Can you explain how this statement connects with these schemes more. I do not follow as both scheme 2 and scheme 3 have an example of a nitroxyhydroperoxide and a hydroxy nitrate? Also the likelihood of each pathway being relevant in your experiments seems more related to the RO2 fate (i.e., reaction with another RO2 *or HO2) than with the loss rate of nitrooxy hydroperoxides and hydroxy nitrates in Ng et al., 2008. Can you include this into your explanation too?*

Response:

We apologize that there is a typo here. We meant that the reactions with hydroxy nitrate is more likely. In the revised manuscript, we have modified this sentence. As the reviewer pointed out, RO_2 fate is also an important factor, which affects the relative concentration of nitroxyhydroperoxide and hydroxy nitrate. In our experiments, RO_2 fate is dominated by the reaction with NO_3 and RO_2. Therefore, hydroxyl nitrate is expected to be higher than nitroxyhydroperoxide. Overall, we have revised the discussion as follows:

"Additionally, C5-hydroxynitrate concentration is expected to be higher than that of nitrooxyhydroperoxides because RO_2+RO_2 forming alcohol is likely more important than RO_2+HO_2 forming hydroperoxide in this study. Therefore, it is likely that $C5H_9N_2O_n$• M2a series was mainly formed from $C_5H_9NO_4$ instead of $C_5H_9NO_5$, while $C_5H_9N_2O_n$• M2b were formed from $C_5H_9NO_4$ followed by an alkoxy-peroxy step. That is, Scheme 2a and 3b were more likely."

Page 13 line 370: Is it also possible that instead of C5 nitrooxy carbonyls reacting more slowly with NO3 than C5 hydroxy nitrates that instead less HOMs are formed from C5 nitrooxy carbonyls because of the carbonyl group leading to more fragmentation (e.g., in MACR OH oxidation H-shifts lead to losing CO - Crounse 2012)? Have you considered this?

Response:

It is possible that more fragmentation in the further H-shift of peroxy radicals formed in the reaction of C5 nitrooxy carbonyls can also contribute to our observation of low abundance of $C_5H_7N_2O_n$. In the revised manuscript, we have added discussion on this point.

"This fact is consistent with the finding of Ng et al. (2008) that C5-nitrooxycarbonyls react slowly with NO_3. Additionally, the peroxy radical formed in the reaction of C5-nitrooxycarbonyls with NO_3 likely leads to more fragmentation in H-shift as found in the OH oxidation of methacrolein (Crounse et al., 2012), which may also contribute to the low abundance of $C_5H_7N_2O_n$."

Page 16 line 447: The rate constants for RO2 + RO2 reaction are heavily structure dependent, so this assumption does not really hold in atmospheric chemistry. This should be considered here. For example, in schemes 2 and 3, the dominant RO2 isomers of C5H9N2O9 and C5H9N2O10 will not be the one pictured. The one pictured will most likely lead to HOMs. The dominant one will be the peroxy radical in the tertiary position, which will likely lead to fragmentation and not HOMs. This tertiary peroxy radical will react with other RO2 much more slowly than secondary or primary peroxy radicals (Jenkin 1998, (Jenkin et al., 1998)), so you would not necessarily expect very much ROOR from these RO2 radicals even though they are dominantly detected. Have you considered this?

Response:

We agree with the reviewer that tertiary RO_2 formed may react slower than secondary and primary RO_2. $C_5H_9N_2O_9$• or $C_5H_9N_2O_{10}$• is likely a mixture of different isomers, including both the secondary and tertiary RO_2. And we have no evidence that $C_5H_9N_2O_9$• or $C_5H_9N_2O_{10}$• contains more fraction of tertiary RO_2 than primary and secondary RO_2. For example, the dominant precursor of $C_5H_9N_2O_9$•, isoprene hydroxyl nitrates, are likely (1-ONO_2, 2-OH) and (1-ONO_2, 4-OH) isoprene hydroxyl nitrate (IHN). The dominant RO_2 formed by the reaction of these IHN with NO_3 is likely secondary or tertiary RO_2. In the revised manuscript, we have added discussion on different reaction rate of different RO_2.

"Admittedly, the assumption of different RO_2 having similar rate constant in accretion reactions may not be valid. For example, self-reaction of tertiary RO_2 is slower than secondary and primary RO_2 (Jenkin et al., 1998; Finlayson-Pitts and Pitts, 2000). Different rate constant may also lead to the observation that the most abundant dimers could not be explained the most abundant RO_2."

Page 21 line 588: How was this HOM yield calculated? Is it from the first injection of isoprene or over the entire experiment?

Response:

HOM yield is calculated for the first isoprene addition period. In the revised manuscript, we have described this and used "primary HOM yield" in place of "HOM yield" to avoid ambiguity.

"The HOM yield in the oxidation of isoprene by NO_3 was estimated for using the sensitivity of H_2SO_4. It was derived for the first isoprene addition period to minimize the contribution of multi-generation products and to better compare with the data in literature, thus denoted as primary HOM yield (Pullinen et al., 2020) and was estimated to be $1.2\%^{+1.3\%}_{-0.7\%}$."

Conclusions: As related to the questions above, please include in more detail how to interpret these laboratory results within the context of how SOA forms from isoprene + NO3 in the ambient atmosphere. How do your laboratory conditions compare to the ambient atmosphere (e.g., RO2 fate (reaction with NO3, RO2, HO2, isomerize), RO2 lifetime, fate of the first-generation organic nitrates reaction with NO3 at night or OH at sunrise)?

Response:

Accepted. We have included more discussion on the ambient relevance of our laboratory study in the conclusion part as mentioned in the response to former comments.

Technical comments:

Scheme 2a: missing NO3 group on second molecule. In Scheme 2b, is the 3rd label really a H-shift? It looks like this should be reaction with RO2/NO3?

Response:

We thank the reviewer for pointing out our mis-labelings. In the revised manuscript, we have corrected them.

Scheme 3b: missing NO3 group on second molecule. And the last molecule OOH should be OH?

Response:

We thank the reviewer for pointing out our mis-labelings. In the revised manuscript, we have corrected them.

Page 9 line 235 there are two "in"

Response:

Corrected.

Figure S3, isoprene is spelled incorrectly.

Response:

Corrected.

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