Responses to Reviewer

Reviewer #3:

This paper presents the design and characterization of a new thermal desorption / NO_2 CEAS based instrument for the measurement of gas and particle-phase peroxy and alkyl nitrates, with calibration of PN and AN detection and correction for NOx interferences. The paper presents instrument laboratory characterization and some limited ambient testing. The work is well-motivated and the manuscript is clearly written; I have some questions and suggestions how the characterization and presentation could be improved which are detailed below. I recommend publication after revisions.

Major comments:

1. As this is an instrument description paper, I would recommend including the instrument schematic (currently Figure S1) in the main body of the paper, perhaps moving Table 1 to the SI (less important). I also recommend adding some key details to the instrument figure: (a) for the described pure N_2 sampling at the end of each measurement cycle, where is this N_2 introduced? (b) how does the flow through the "Multi inlet unit" work? Does the 1 lpm sample flow go through all channels continuously and either divert to the CEAS or exhaust, or does only one inlet at a time flow? If the latter, nice to also add to the SI a figure of the NO_2 signal during this switching and indicate what period of the measurements you use (there must be a switching time where the signal is stabilizing). Indicate lengths / dimensions on the figure – how long are the ovens vs. the "cooling region" after them? How large is the overall inlet box? Perhaps include a photograph to give the reader a better sense of how this looks deployed. When testing on ambient air, you mention the $PM_{2.5}$ inlet. How long is the inlet line ahead of this cyclone? What is the material? A photo of the sampling head and map of ambient sampling location would also be helpful.

Response: We thank the reviewer for this constructive suggestion. We agree that the reviewers' suggestions mentioned above will greatly improve the readability of the manuscript.

(1) We agree to incorporate Figure S1 into the main text and rename it as Figure 1, to move "Table 1" to the supplementary materials as "Table S1," and to update the figure and table numbering accordingly in both the revised manuscript and the supporting information. Additionally, we have added details regarding the introduction of high-purity N_2 to the revised instrument schematic (new Figure 1), provided an explanation in the revised manuscript, and labeled the length/dimensions of the heating region (20 cm) and cooling region (35 cm) in the figure 1:

"*The ultra-high-purity N_2 is supplied from cylinders and fed directly into the CEAS zero-air inlet, the N_2 flow rate matches the sampling.*" (lines 232-233)

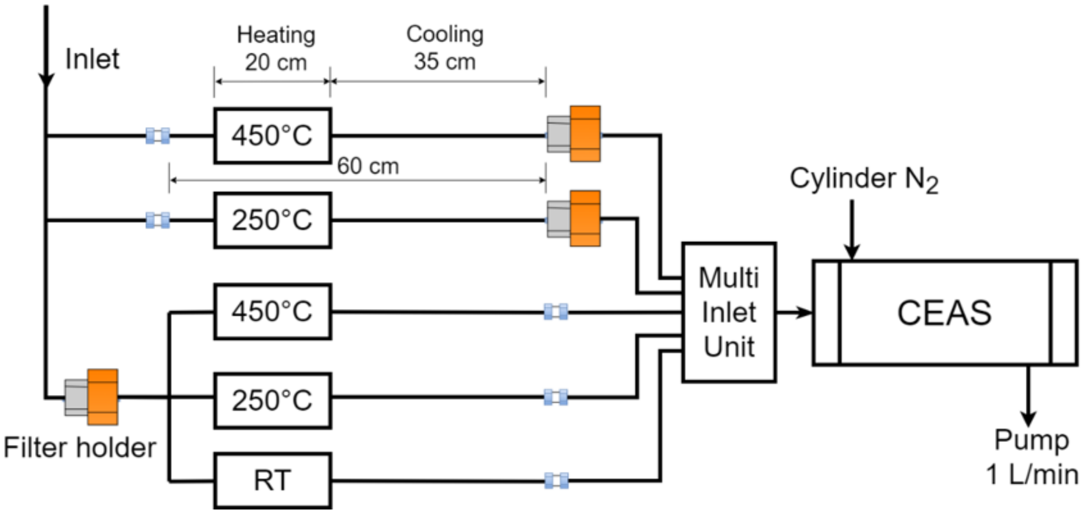

Figure 1 Schematic diagram of the five-channel TD-CEAS instrument.

(2) We employed a separate TD inlet box design to prevent thermal interference during heating. Each inlet box measures 55 cm in length, 6 cm in width, and 9 cm in height. As suggested by the reviewers, we have included a photograph of the TD chamber in the supporting information as new Figure S1:

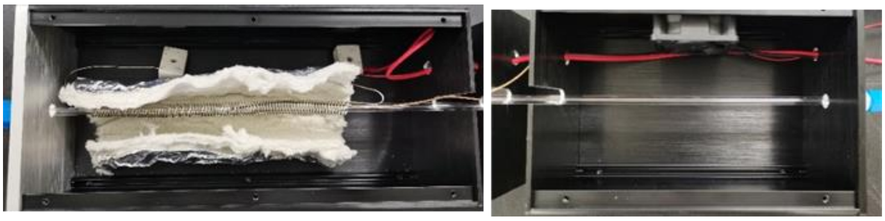

Figure S1 Photograph of TD inlet box.

(3) We have included a plot of the NO_2 signal during channel switching in the supporting information (Figure S2) and describe the operating procedure of the multi-inlet unit in detail in the manuscript, noting the data processing method used during the transition period (lines 226-230):

"*When the multi-channel inlet unit is in operation, only the sample flow from the currently selected channel is allowed to enter the analyzer. Since there is a transition period during which the NO_2 signal stabilizes after switching channels, the data from the first 15 seconds following the channel switch should be discarded, as shown in Figure S2.*"

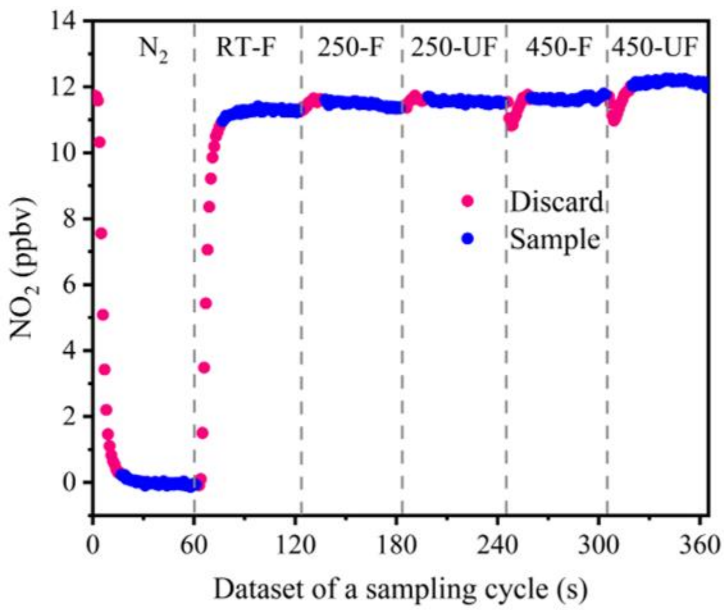

Figure S2 An example of a dataset during a sampling cycle. The red points represent data from the transition period following a channel switch, which is discarded during calculation. The blue points represent sample data.

(4) To aid the understanding of readers and reviewers, we have included a photograph of the sampling head and a schematic diagram of the sampling location in the supporting information (new Figure S6), and added an explanation in the revised Section 4.1 on lines 609-612:

"*When measuring ambient air, the air passes through a $PM_{2.5}$ cyclone separator into a 1/2-inch stainless steel main sampling tube with a high-flow pump used to draw air at the end (Fig. S6). The TD-CEAS system collects samples via a bypass line (PTFE, ~2 m) connected to the main sampling tube.*"

Figure S6 $PM_{2.5}$ sample inlet and sampling site location.

2. Your choice of $NaNO_3$ for particulate inorganic nitrate interference might minimize the effects you see; I think NH_4NO_3 is much more prevalent in the ambient atmosphere also in China, and it is much more semi-volatile and thus likely to cause interferences.

It would be preferable to test with this inorganic nitrate. If this is not possible, you should at least discuss and point to literature on this.

Response: We thank the reviewer for this important comment. We agree that NH_4NO_3 is the more atmospherically relevant interference species and that using $NaNO_3$ alone was not sufficient. Because we were not able to complete a direct NH_4NO_3 experiment before resubmission, we took two steps in the revision.

First, we rewrote the manuscript to state this limitation explicitly, rather than implying that the $NaNO_3$ test ruled out inorganic nitrate interference in general. Second, we added an order-of-magnitude upper-bound estimate based on literature values: using a typical Pearl River Delta NH_4NO_3 loading below 10 μg m^{-3} and a conservative 50% conversion efficiency at 450 °C, the potential interference is estimated to be on the order of 1.4 ppbv NO_2 equivalent. We now state clearly that this estimate is only an upper bound and that the exact magnitude for our inlet remains unquantified until direct NH_4NO_3 experiments are performed.

we have added discission in the revised manuscript (lines 567-579):

"*It should be noted that the thermal tests described above were conducted using* $NaNO_3$ *as a proxy for particulate inorganic nitrate. While* $NaNO_3$ *is thermally refractory,* NH_4NO_3 *is likely to dominate much of the urban inorganic nitrate burden in China and is substantially more semivolatile. Literature data indicate that* NH_4NO_3 *begins to decompose at temperatures as low as ~400 °C, and that its conversion to* NO_2 *at 450°C is less than 50% (Garner, et al., 2020). Because this temperature range overlaps with the ΣANs setpoint used here, ambient* NH_4NO_3 *could contribute a positive artifact to measured ΣANs. Using a typical Pearl River Delta* NH_4NO_3 *mass concentrations of <10 μg* m^{-3} *(Liu et al., 2026) and a conservative 50% conversion efficiency, we obtain an order-of-magnitude upper bound of ~1.4 ppbv* NO_2 *equivalent. The exact magnitude for our inlet remains unquantified because* NH_4NO_3 *was not tested directly in the laboratory. This is an important limitation of the current study, especially for cool or heavily polluted conditions.*"

3. Section 4.3: ambient timeseries and evaluation against PAN. First paragraph of this section discusses briefly the TD-CEAS data shown in Figure 6, focusing on average and standard deviations and day/night differences. To better highlight the diel structures you discuss, I recommend adding a diurnal average plot that shows the average daily cycle of the relevant species, ideally with a second panel showing temperature, because this will help interpret the diel behavior. Then add this to the discussion: is the day /night different consistent with the temperature pattern? To me the relative concentrations of particle-phase ANs measurements compared to gas-phase look very high. Was it quite cold? DO you expect lots of high MW heavy nitrates? The particulate PNs also seem very high, if they are indeed dominated by PAN … is the gas to particle ratio you see here consistent with the known volatility of PAN and the ambient temperature? Also it would be nice to show your ambient temperature NO_2 channel here compared to the Thermo 42i NO_2 measurement. Is it possible to add more ambient data to better capture the representative diurnal cycles?

Response: We thank the reviewer for these very helpful suggestions. We agree that the original field discussion interpreted the diel behavior too aggressively. In the revision we did two things at the same time: we added the diurnal-average figure requested by the reviewer, and we softened the interpretation of the particle-phase signals, especially pΣPNs.

(1) Daily average plot

As suggested, we have added a diurnal average plot (new Figure. 9) in the revised manuscript. The data were taken from the entire campaign period, Specifically, the temperature and relative humidity data were obtained from the Wushan monitoring station of Guangdong Ecological Environmental Monitoring Center, located ~2 km northwest of GIG.

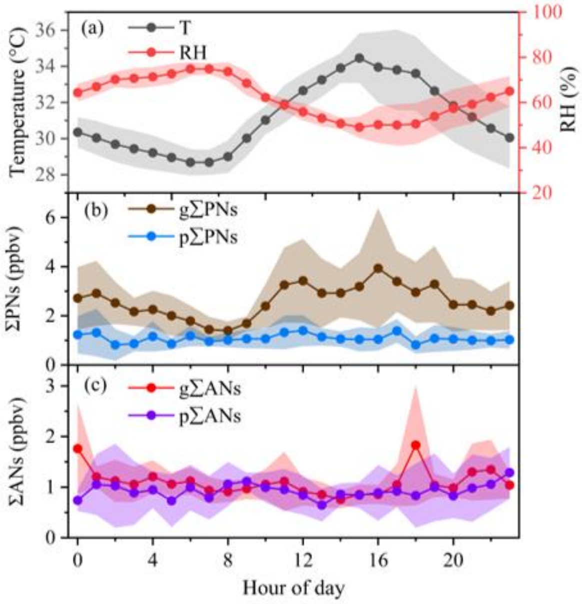

Figure 9. Daily average of (a) temperature and relative humidity, (b) gas- and particle-phase ΣPNs, and (c) gas- and particle-phase ΣANs. Shading indicating the error of ±1 σ.

(2) Day/night pattern with temperature

We agree with the reviewer that temperature is a key organizing variable. In the revised manuscript we now interpret the increasing daytime gΣPN fraction as qualitatively consistent with warmer conditions favoring the gas phase, while explicitly noting that the absolute magnitude of pΣPN remains uncertain because pΣPN is obtained by channel subtraction and has not been directly validated with a particulate PN standard. For ΣANs, we retained the more modest interpretation that lower nighttime temperature is consistent with a larger particle-phase fraction of lower-volatility organic nitrates. We have added discussion on this point in Lines 670-685:

“*The trends in daytime gΣPNs and temperature are positively correlated, as shown in Figure 9. The fraction of gΣPNs, suggesting a gas-to-particle transformation process of ΣPNs. The proportion of gΣPNs (gΣPNs/(gΣPNs +pΣPNs)) increases from less than 60 % in the morning (08:00, ~29 °C) to nearly 80 % in the afternoon (16:00, ~34 °C), which is qualitatively consistent with warmer conditions favoring the gas phase. Relative humidity also affects ambient ΣPNs concentrations. We therefore regard the* *daytime evolution of gΣPNs as robust in a relative sense. By contrast, the absolute magnitude of pΣPNs should be interpreted more cautiously because pΣPN is obtained by channel subtraction and has not been directly validated with a particulate PN standard. The residual particulate signal may reflect a combination of less volatile PN components, temperature-dependent partitioning, and subtraction uncertainty. Relative humidity may also influence ΣPNs through PAN hydrolysis and heterogeneous uptake on particles (Salas et al., 2020; Sun et al., 2022). The nighttime increase in gΣPNs is not interpreted quantitatively because $ClNO_2$, N_2O_5, and HONO can contribute to gas-phase channels after dark. For ΣANs, the particle-phase fraction increases as temperature decreases, which is consistent with partitioning of lower-volatility organic nitrates towards the condensed phase during nighttime conditions.*"

(3) Comparison of ambient temperature NO_2 channel and Thermo 42i NO_2 measurement
We thank the reviewer for the suggestion. We did not add a direct comparison to the Thermo 42i because the two instruments do not measure NO_2 with equivalent selectivity, and without a dedicated side-by-side characterization the comparison could be misleading rather than helpful. We now state that limitation more clearly in the response and in the manuscript.

4. Second paragraph of this section discusses the gPNs comparison to PAN, and refers to Figure S5. This figure raises another major question for me – gPNs looks closer to PAN validation method than corrected gPNs. Is the correction biased by having been developed using PAN, so that in ambient air when sumPNs are not all PAN it will skew the data? This should be addressed in the discussion. If you check the uncorrected PNs diurnal cycle, does it make more sense in terms of expected PN volatility? Is it possible that this correction introduces spuriously high PNs signal (and then possibly in both particle and gas phase)?
Response: We thank the reviewer for this important observation. We agree that the correction cannot be presented as composition-agnostic, because it was developed using PAN. We therefore revised the manuscript so that the corrected daytime gΣPNs is described explicitly as a PAN-anchored operational estimate.
At the same time, we retained the correction because the raw gΣPNs data occasionally fell below independently measured PAN, which is physically implausible and indicates a negative inlet bias in the uncorrected channel. The revision now states both points together: correction is necessary to remove an obvious artifact, but the corrected values should still not be interpreted as composition-independent absolute ΣPN totals when the ambient mixture departs from PAN. We have added the following statements in the revised manuscript (Lines 694-701):
"*Based on the comparison of raw ΣPNs and PAN (where gΣPNs are, at certain times, unreasonably lower than PAN), it is necessary to correct the raw data because the uncorrected values occasionally imply a physically implausible negative inlet bias. At the same time, the correction remains PAN-anchored. In the real atmosphere, ΣPNs comprise a mixture of peroxy nitrates, whose thermal dissociation products may not*

behave identically to the PA radical. The corrected daytime gΣPNs values should therefore be interpreted as operational ΣPN estimates constrained by PAN, rather than as composition-independent absolute totals."

Minor comments:

1. Consider adding a few additional recent organic nitrate references to your introduction, e.g.: https://amt.copernicus.org/articles/15/459/2022/amt-15-459-2022.pdf, https://doi.org/10.1002/2016GL069239, https://doi.org/10.5194/acp-18-15419-2018, https://doi.org/10.5194/acp-16-5969-2016

Response: We thank the reviewer for these excellent references. We have added all four recommended citations (Day et al., 2022) and (Fisher et al., 2016) on lines 56-57, (Kiendler-Scharr et al., 2016) on line 69, (Zare, et al., 2018) on line 51 to the Introduction section. These additions significantly strengthen the literature review.

2. For comparison of the PAN fraction of total sumPNs: https://acp.copernicus.org/articles/25/5893/2025/

Response: We thank the reviewer for this valuable suggestion. In the revised manuscript, we have added a comparison of the PAN fraction in total ΣPNs with the results reported by Andersen er al. (2025) on Lines 690-691:

"*This proportion ranges from 48% to 78% at the temperate forest site in Paris, France (Andersen et al., 2025).*"

3. There is another recent instrument paper in press you could check and think about how these ideas are relevant to your measurements: https://egusphere.copernicus.org/preprints/2026/egusphere-2026-157/egusphere-2026-157.pdf

Response: We thank the reviewer for bringing this very relevant study to our attention. We have carefully read the paper by Dewald et al. (2026). The study developed a denuder TD-CRDS system (D-TD-CRDS) for accurate measurement of particulate nitrate. The denuder selectively removes gas-phase NOy, allowing direct measurement of particulate nitrates. A Nafion dryer placed upstream of the denuder extends its lifetime and improves removal efficiency. The system was tested with both inorganic nitrate (NH_4NO_3) and organic nitrates (generated from NO_3-initiated oxidation of limonene), and the results were cross-validated using an aerosol mass spectrometer (AMS). This work offers valuable insights for our study in the following aspects:

(1) Particle transmission/removal efficiency: In our study, we use a filter to remove particles, which provides a high retention efficiency (≥ 99.5 % for 0.3 µm particles). However, we have not yet characterised particle transmission efficiency through the sampling lines. Although many studies report high transmission efficiencies in similar tubing setups, we recognize that experimental verification is needed in future work.

(2) Standards: The thermal dissociation behaviour may differ depending on the chemical form of nitrate. Therefore, calibration experiments should cover the main nitrate species expected in ambient air. This multi-species approach, as demonstrated in the reference study, is something we plan to adopt in future method development.

4. The last sentence of the introduction (lines 141-144) is repeated information from earlier in that paragraph, not needed.

Response: We thank the reviewer for this careful reading. We agree that the last sentence of the Introduction (lines 141–144 in the original manuscript) merely repeats information already stated earlier in the same paragraph. Therefore, we have deleted this redundant sentence "We provide systematic laboratory characterization, including dissociation behavior, calibration strategies for gaseous and particulate standards, and quantification of interferences relevant to high-NOx environments, and we demonstrate the instrument performance during an urban field deployment." from the revised manuscript.

5. Around lines 201-204: please add more detail on the filters. What material / pore size is it? How often you need to replace it? Did you check signals before and after filter replacement in ambient sampling?

Response: We thank the reviewer for this helpful comment. We realize that the description of the filters used in our sampling system was insufficient. We have now added the missing details in the revised manuscript (lines 239-242):

"*The filters are made of PTFE and has a diameter of 47 mm, with retention efficiency of 99.5% or higher for 0.3 μm particles. The filter is replaced once a week during field campaigns, or earlier if the analyzer's flow rate decreases and in the event of air pollution.*"

We acknowledge that we did not systematically record the TD-CEAS signal specifically before and after filter replacement during ambient sampling. The filter used in our system is 0.3 μm PTFE membrane filter. PTFE is chemically inert and has very low affinity for adsorption of ONs, and the pore size (0.3 μm) is chosen to remove particles with retention efficiency of 99.5%. Additionally, we regularly performed zero-air checks (every sampling cycle) and recorded baseline signals to ensure that there is no persistent contamination or signal drift before and after the filter replacement.

6. Line 285-286: phrase "across trace to heavily polluted levels": meaning is unclear to me.

Response: We thank the reviewer for pointing out this ambiguity. We agree that the phrase "across trace to heavily polluted levels" is too vague. To avoid any confusion, we have revised the original sentence as follows (lines 358-361):

"*PAN derived from the TD−CEAS channel agrees closely with GC-ECD ($R^2 = 0.988$; slope = 0.987; Fig. 3) from clean background (e.g., PAN < 1 ppbv) to heavily polluted conditions (e.g., PAN > 10 ppbv).*"

7. Line 289-290: Switch from talking about gas-phse to particulate AN standards was confusing to me at first. I think I understand now that you only used the gas-phase AN standards for the themal curves, but could not produce a stable enough concentration to calibrate with them, therefore you only calibrate the ANs channel with 2-EHN. If I've understood that correctly, perhaps reword this first sentence of this section to clarify.

Response: We thank the reviewer for this careful reading and for pointing out the confusing transition from gas-phase to particulate ANs standard at the beginning of this section. To avoid confusion, we have corrected sentence "Stable gas-phase ΣAN standards at specified mixing ratios are difficult to generate reproducibly by vapor dilution" of this section to the following below in the revised manuscript (lines 367-369):

"*Because stable gas-phase AN standards at known mixing ratios were not available in our setup, we established an operational particulate ΣANs calibration using filter thermolysis of 2-EHN (Yu et al., 2021).*"

8. Also, around line 307: "calibration conditions" – did you run these EHN tests through the full inlet system with filter and all, or separately inject these samples directly into the CEAS? Good to clarify here whether this calibration is only of the CEAS instrument response to themalized EHN, or whether it also tests the filters / subtraction from the inlet system.

Response: We thank the reviewer for seeking this important clarification. When we performed the particulate phase channel calibration using a 2-EHN standard solution, the gas flow passed through the complete sample introduction system, including the heated quartz tube and filter membrane, and the blank was subtracted from the final results. We realized that our original description was ambiguous. To clarify, we have added the following sentence in the revised manuscript (lines 390-392):

"*It is important to note that we conducted the experiments using the complete sampling system and subtracted the blank from the results.*"

9. Line 316: simultaneously -> independently: I think this is what you mean, that both NO and NO2 can separately vary in different ways, that they don't necessarily always track together?

Response: We thank the reviewer for this careful linguistic suggestion. Our original wording "vary simultaneously" could be misinterpreted as implying that NO and NO_2 concentrations change in sync. What we intended to convey is that under high-NOx conditions, the competing pathways involving RO_2 radicals with both NO and NO_2 occur simultaneously (i.e., in parallel), not that the two gases vary together. To avoid confusion, we have revised the sentence "These competing pathways are expected to be especially important under high-NOx conditions, where NO and NO_2 coexist at elevated concentrations and vary simultaneously." to the following below on lines 399-401:

"*These competing pathways are expected to be especially important under high-NOx conditions, where NO and NO_2 coexist at elevated concentrations and the competing reactions take place in parallel.*"

10. Line 351-352: I disagree that Figure 4 shows good agreement at low concentrations -it looks pretty far off across the board.

Response: We thank the reviewer for this critical observation. We agree that the correction results of the lookup table do not show as ideal a fit at either low or high concentrations as we initially stated. We have revised the text "By comparison, the reproduced lookup-table approach performed well at low concentrations (R^2=0.975) but increasing overestimated PAN at high concentrations, particularly under high-NOx conditions." in the manuscript accordingly on lines 436-439:

"*By comparison, the reproduced lookup-table approach did not perform well across the entire concentration range, despite an overall correlation of 0.975 (R^2), the method increasingly overestimated PAN, especially under high-NOx conditions.*"

11. Line 359 & line 362: clarify the term "gradient": you mean different sequences of NO concentrations, I think?

Response: We thank the reviewer for this important comment. To avoid ambiguity, we have replaced "different NO gradients" with "a series of NO concentrations" on line 456 and deleted "gradient" on line 459 in the revised manuscript.

12. Line 375: "thermally dissociates at"

Response: We thank the reviewer for this grammatical correction. We agree that the verb should be in the third-person singular form to agree with the subject. We have corrected "thermally dissociate at" to "thermally dissociates at" on line 517.

13. Section heading 3.3.3 on line 386: Should also already mention HNO3, HONO, NH4NO3 in the section heading and discuss them in the text below.

Response: We thank the reviewer for this suggestion. We agree that HNO_3, HONO, and NH_4NO_3 are relevant to the thermal dissociation behavior of reactive nitrogen species and should be mentioned in the section heading and discussed in the text. Accordingly, we have revised the section heading (now Section 3.3.3 Other interferences (nitro-aromatics, $ClNO_2$, N_2O_5, HONO, HNO_3, NH_4NO_3)) and added a dedicated discussion in the manuscript on lines 555-566:

"*HONO undergoes thermal or photolytic decomposition to yield NO. Pérez et al. (2007) and Friedrich et al. (2020) reported initial decomposition temperatures of HONO at 450 °C and 400 °C, respectively, with complete thermal decomposition occurring only in the range of 650–700 °C. Consequently, in the ΣPNs channel (250 °C) of this study, HONO is not thermally decomposed, while in the ΣANs channel (450 °C), the decomposition efficiency does not exceed 10 %. Ambient HONO concentrations at urban sites are typically below 3 ppbv (Wang et al., 2025), resulting in NO production from thermal decomposition of less than 300 pptv. Moreover, HONO is rapidly photolyzed after sunrise, thus exerting minimal influence on daytime TD measurements. Overall, the thermal decomposition of HONO in the ANs channel produces only trace amounts of NO, which may nonetheless interfere with TD channels and systems that target NO_2 as the analyte.*"

The discussion regarding HNO_3 and NH_4NO_3 have already been addressed in the comments above on lines 567-579.

14. Lines 445-446: nighttime gas phase OH data vs. particle-phase ON retained: Both are shown in Figure 6, and it's not clear from that figure alone why you think nighttime

gANs is particularly suspect… should be discussed in more detail, see comments about Figure 6 & interpretation above.

Response: We thank the reviewer for this important comment. We agree that the original text did not explain clearly enough why nighttime gas-phase interpretation is more problematic than the corresponding particle-phase retrievals. We therefore revised the manuscript in two ways. First, we now state explicitly that nighttime gas-phase ON data are not used quantitatively because $ClNO_2$, N_2O_5, and HONO can interfere with the heated channels after dark. Second, we softened the wording for particle-phase data: rather than calling them simply "reliable", we now describe them as more robust than the nighttime gas-phase values, but still operational, because paired-channel differencing reduces but may not completely remove common-mode gas-phase interferences.

We have clarified this in the revised manuscript and added an explanation on lines 640-643:

"*This is because the presence of other reactive nitrogen oxides at night, such as* $ClNO_2$*,* N_2O_5*, and HONO, as described in Section 3.3.4 (which form at night and photolysis during the day), can significantly interfere with ON measurements.*"

Additionally, in accordance with the above reviewer comments, we have added a detailed discussion of diurnal trends in the manuscript on lines 670-685, please see the comments above for a detailed response.

15. Line 497: "inorganic nitrate" – hopefully you will also test NH_4NO_3 and then you can mention that here, but if not, please specific NaNO3 here, since I am not convinced you have generally ruled out any inorganic nitrate interferences.

Response: We thank the reviewer for this important clarification. We agree that the original term was too broad. In the revised manuscript we now specify "inorganic nitrate ($NaNO_3$)" where appropriate and state separately that NH_4NO_3 remains an unresolved interference source for our system, for which we provide only a literature-based upper-bound estimate rather than a direct validation.

16. Line 513-514: This statement seems in conflict with your previous mentions that you can't really trust nighttime gANs measurements.

Response: We thank the reviewer for pointing out this inconsistency. We have revised the manuscript so that the data-usage hierarchy is now stated consistently throughout: daytime gas-phase ONs are the most robust component of the field interpretation; nighttime gas-phase ONs are shown only qualitatively; and nighttime particle-phase ONs are retained as operational outputs because the differencing framework reduces, but does not guarantee complete removal of, common gas-phase interferences. The revised sentence in the Conclusions now avoids claiming blanket nighttime reliability and restricts the interpretation accordingly.

We have revised the sentence "The observed diel behavior, characterized by daytime enhancement of gas-phase ONs and nighttime enhancement of $p\Sigma$ANs, illustrates the utility of this approach for probing ON phase partitioning and reactive nitrogen processing in urban air." to

"*The observed diurnal variation of gas-phase ONs and the day-night pattern of particle-phase ONs provide a useful approach for investigating the daytime phase partitioning of ONs and the transformation processes of reactive nitrogen in urban air.*" (lines 734-737)

17. Figure 4b: Slope is misspelled in both annotations.

Response: We are very sorry for our incorrect writing. The typo "Slpoe" has been corrected to "Slope" in revised Figure 5b.

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