Response to anonymous referee #3

In their manuscript, "Measurements of higher alkanes using NO^+ PTR-ToF-MS: significant contributions of higher alkanes to secondary organic aerosols in China," the authors tackle an important but often overlooked issue: the contribution of high-molecularweight gas-phase alkanes to SOA formation. A relatively large body of work over the last decade has provide a fairly thorough examination of the composition of vehicle emissions and the importance of branched (and cyclic) alkanes; this manuscript uses this work as a starting place to explore their contribution to SOA in China. The work is made possible in large part by the recent development of the NO^+ PTR. Overall, I think this work addresses interesting questions, and makes real efforts to tackle the complexity of calibrating a new instrument for compound classes that may contain hundreds of isomers. However, there are a few scientific weak spots that need to be addressed before this work is ready for publication. These larger comments include some needs for clarification, and some more fundamental issues with how OH exposure and SOA potential are being calculated. I commend the authors for taking a step into some unknown territory, but some additional discussion (and possibly work) needs to be included.

Response: Thank you very much for your valuable comments and suggestions on our manuscript. These comments are all helpful for improving our article. All the authors have seriously discussed about all these comments. According to your comments, we have tried best to modify our manuscript to meet with the requirements for the publication in this journal. The details about the calculation of OH exposure was added in the supporting information of the revised manuscript. Two methods to estimate SOA formation from different VOCs classes were all presented in the manuscript and the supporting information. Point-by-point responses to the comments are listed below.

General Comments:

(1) There are some typo and English issues throughout. It is not sufficient to seriously detract from the science, but it is to the degree that the authors should have a native English speaker review and edit this manuscript.

Response: Thanks for your nice suggestion.

(2) Considering the focus of this work is on large alkanes, and the semi-volatile nature of these components, particularly at the lower temperatures of some of these measurements, this manuscript really does not provide enough detail on the sample line and inlet. How long is this

sample line? Is it heated all the way to the sample inlet, or just in the room? If it is not heated all the way to the inlet, I would have some misgivings about alkanes about C14 or so, there could be substantial losses or time lags for larger alkanes. Is there any evidence (observations or models) that suggest line losses and delays can be ignored? If the authors want to focus on gas-phase measurements of S/IVOC alkanes, more detail needs to be provided for the sampling.

Response: We included more information about the sampling in the revised manuscript. The schematic drawing of the inlet system for the measurement during campaigns is shown in the attached figure (Fig S2, Wu et al., 2020). The sampling line is ~8 m and ~3 m during PRD campaign and NCP campaign respectively. Ambient air was continuously introduced into ToF-MS through a Teflon tubing (1/4") with an external pump at 5.0 L/min. The calculated residence time for the inlet was ~3 s for PRD campaign and ~1 s for NCP campaign, respectively. The sampling line was heated all the way to the sampling inlet by an insulating tube with a self-controlled heater wire (40 ℃) wrapping outside. We conducted tubing loss experiments in the laboratory by introducing standards (2 ppb/1 ppb/0.5 ppb) of higher alkanes (*n*-C8-C15), monoaromatics (benzene, toluene, o-xylene, 1,2,4-trimethylbenzene), isoprenoids (isoprene, α-pinene) and naphthalene into PTR-ToF-MS through a 8 m Teflon tubing (1/4") at room temperature with an external pump at 5.0 L/min (Figure S11). The tubing loss of these standard compounds is found to be <5% except *n*-C15 of ~8% and naphthalene of ~10%. Given the fact that the sampling line was heated at 40 ℃ all the way to the inlet during the campaigns, we think the tubing loss would be ignored.

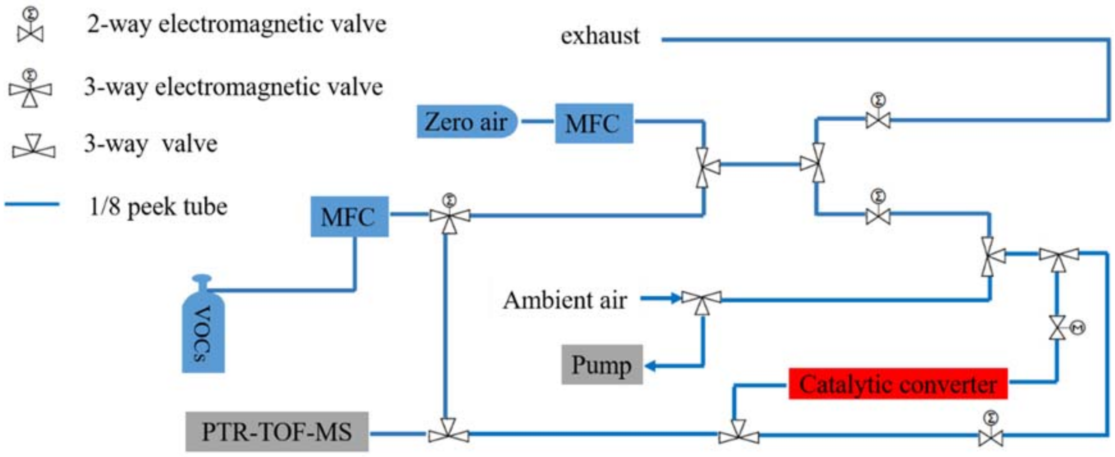

Figure S2. Schematic drawing of the inlet system for PTR-ToF-MS during the campaign. (Wu et al., 2020)

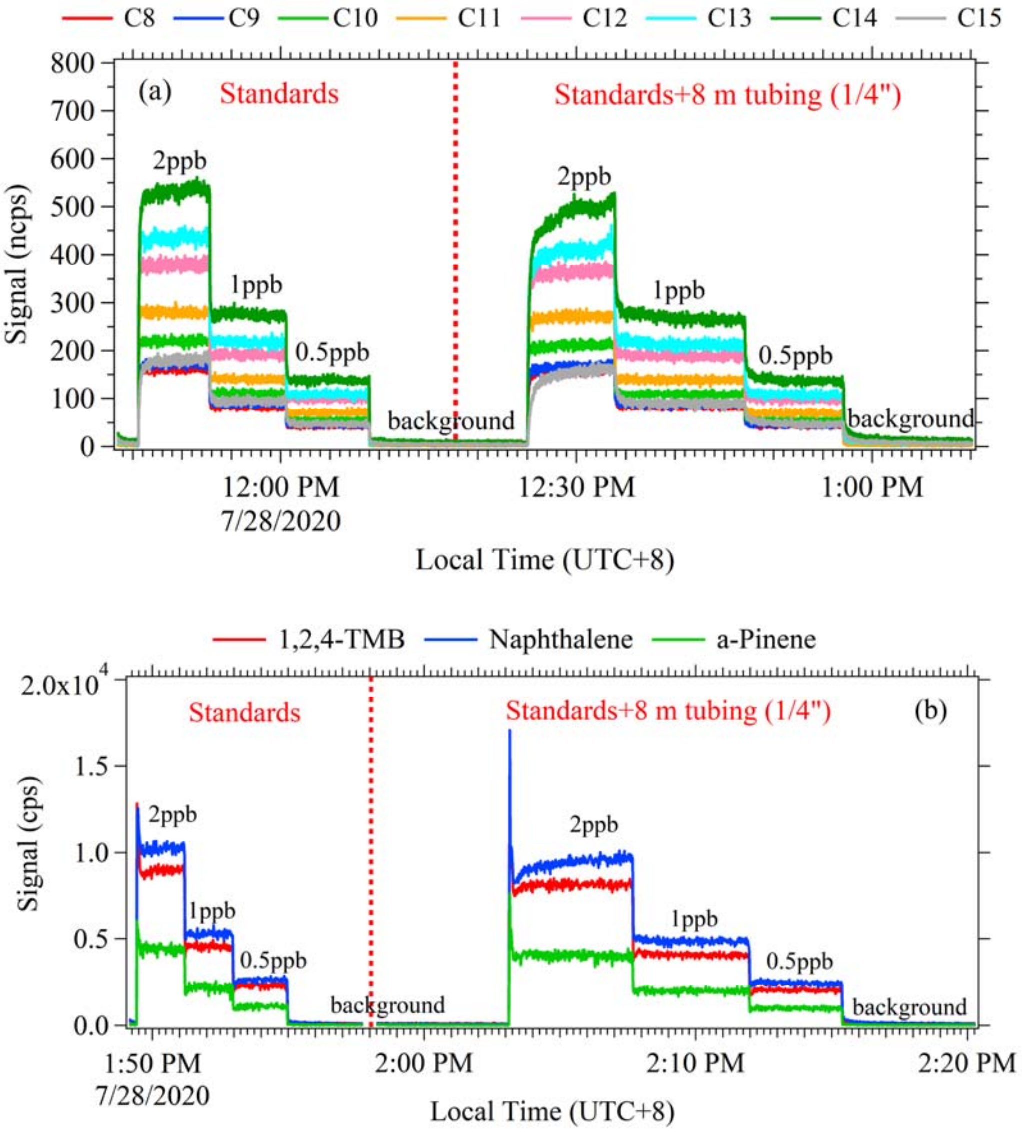

Figure S11. The tubing loss experiments of higher alkanes (*n*-C8-C15), 1,2,4-trimethylbenzene,α-pinene and naphthalene at room temperature using PTR-ToF-MS with an external pump at 5.0 L/min.

The sentences on line 205-216 of page 9-10 are modified as follows:

“Ambient air was continuously introduced into PTR-ToF-MS through a Teflon tubing (1/4”) with an external pump at 5.0 L/min, with tubing length of ~8 m and ~3 m during the PRD and the NCP campaign, respectively. The inlet tubing was heated all the way to the sampling inlet to avoid water vapour condensation by an insulating tube with a self-controlled heater wire (40 ℃) wrapping outside. The calculated residence time for the inlet was ~3 s for PRD campaign and ~1 s for NCP campaign, respectively. The tubing loss experiments were conducted in the laboratory by introducing standards of higher

alkanes (*n*-C8-C15), monoaromatics (benzene, toluene, o-xylene, 1,2,4-trimethylbenzene), isoprenoids (isoprene, α-pinene) and naphthalene into PTR-ToF-MS through a 8 m Teflon tubing (1/4”) at room temperature with an external pump at 5.0 L/min (Figure S11). The tubing loss of these compounds is found to be <5% except *n*-C15 (~8%) and naphthalene (~10%).”

3) In the discussion around "sensitivity", is that the response per mass of the m-1 ion, or the sum of all ions? If the former, it should be discussed in some of the relationships shown, as discussed in comments below. If the latter, how are those ions summed and attributed in the complex real-world data? Understanding of this terminology is critical for interpreting some of the figures.

Response: The “sensitivity” is that the response per mass of the m-1 ion with a unit of ncps/ppb in this study. We measured the fragmentation patterns of selected higher alkanes with NO^+ PTR-ToF-MS by introducing commercially acquired pure chemicals (Figure S8). We found that higher alkanes are mainly reacted through hydride abstraction by NO^+ forming mass (m-1) ions (m is the molecular mass). The fractions of m-1 ions account for more than 60% of total ion signals for >C12 *n*-alkanes (Figure 4 (a)). Thus, we use the response per mass of the m-1 ion for each higher alkane when performing sensitivity experiments using a gas standard with a series of *n*-alkanes. We added a sentence on line 147-150 of page 7 in the revised manuscript to explain the “sensitivity” where this word was first mentioned.

“Sensitivities of *n*-alkanes (C8-C15) standards were obtained during the campaign (Figure S6), which is defined as the normalized signal of hydride abstraction ions for each higher alkane at 1 ppbv with a unit of ncps/ppb.”

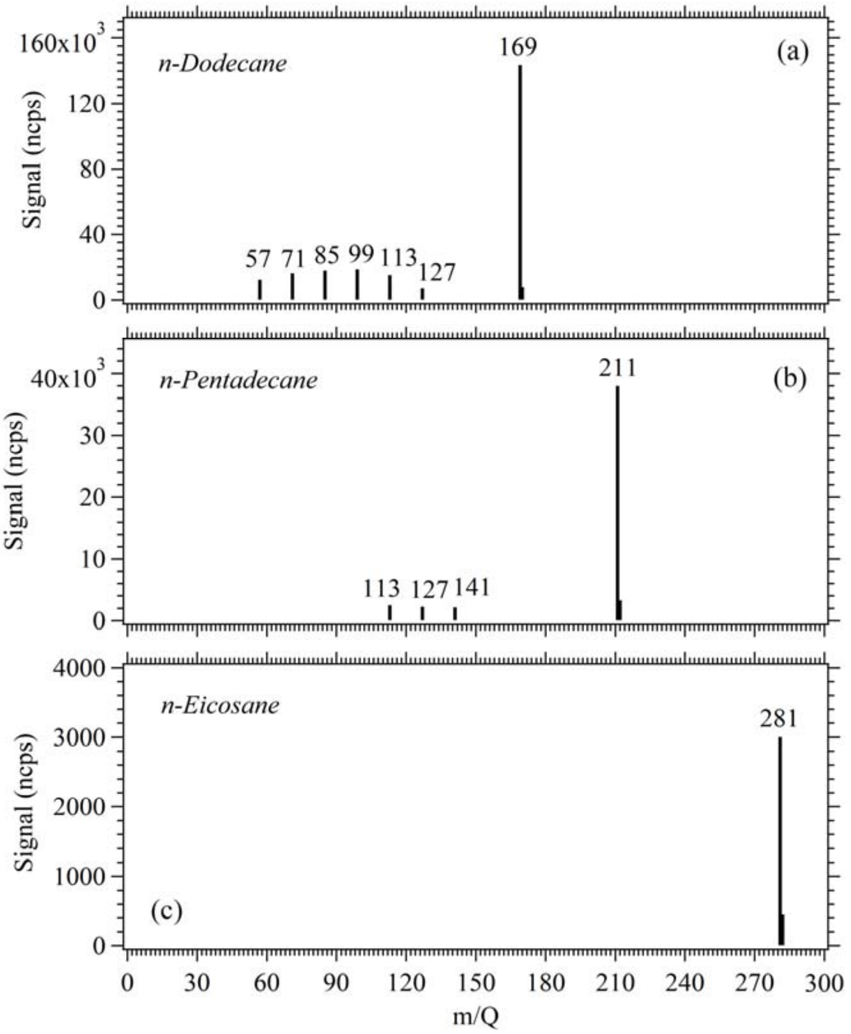

Figure S8. Mass spectra of the distributions of product ions from *n*-Dodecane **(a)**, *n*-Pentadecane **(b)** and *n*-Eicosane **(c)** with NO^+ PTR-ToF-MS. The signals of masses shown in the graph are the results after subtracting the isotopic signals during the high resolution peak fitting of the mass spectra.

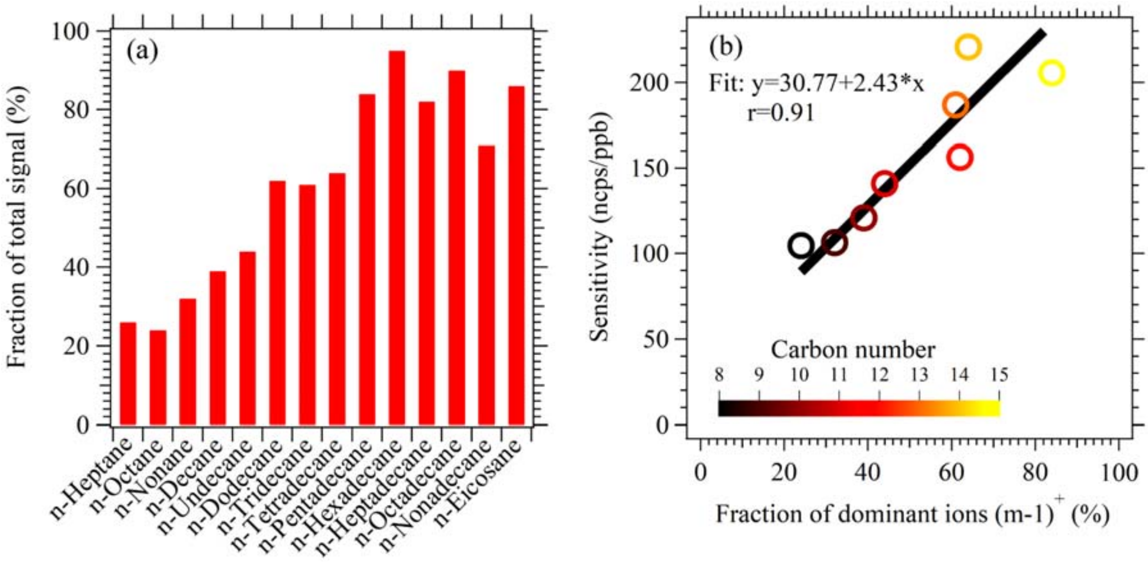

Figure 4. (a) The fractions of product ions (m-1) from hydride abstraction of C8-C20 *n*-alkanes in NO^+ PTR-ToF-MS. (b) Scatterplot of sensitivities under dry conditions versus the fractions of hydride abstraction ions for C8-C15 *n*-alkanes.

4) More detail is necessary on how they calculate OH exposure. I'm not familiar with the isoprene chemistry method - please include a description. And for the ratio of aromatics method, don't you need to start by assuming some ratio at the time of emissions? What is assumed here? Why does the OH exposure diurnal (Figure S9) look very different for the biogenics and the anthropogenics? Notably, in the NCP campaign this is also a large missing daytime source, could this be related to a "mistiming" of SOA caused by a bias in the OH exposure calculation? Or what might be the missing source?

Response: We included more detail about the calculations of OH exposure in the revised supporting information. **The observed ratios between m+p-xylene and ethylbenzene were used to estimate the OH exposure ($[OH] \times \Delta t$) by Roberts et al. (1984):**

$$[\boldsymbol{OH}] \times \Delta \boldsymbol{t} = \frac{\mathbf{1}}{\boldsymbol{k}_{\mathbf{m+p-xylene}} - \boldsymbol{k}_{\boldsymbol{ethylbenzene}}} \times [\boldsymbol{ln}(\frac{\mathbf{m+p-xylene}}{\boldsymbol{ethylbenzene}})_{\boldsymbol{t=0}} - \boldsymbol{ln}(\frac{\mathbf{m+p-xylene}}{\boldsymbol{ethylbenzene}})_{\boldsymbol{t}}]$$

Where the initial emission ratios of m+p-xylene/ethylbenzene were estimated according to the correlation of m+p-xylene with ethylbenzene during campaigns. The ratio of 4 and 1.5 were used in the PRD campaign and the NCP campaign, respectively (Figure S29).

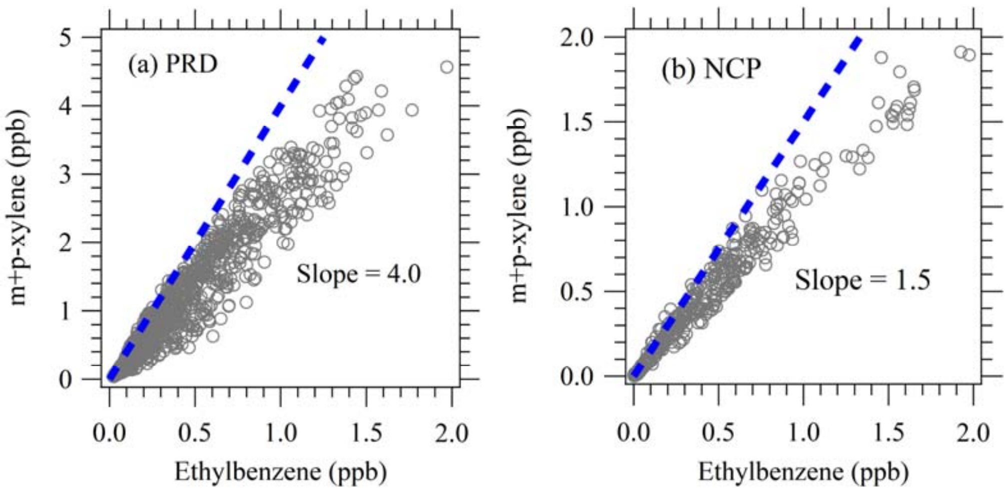

Figure S29. Correlation of m+p-xylene with ethylbenzene in PRD (a) and NCP (b). The dashed lines in both graphs indicate the estimated initial mission ratio of m+p-xylene/ethylbenzene.

Isoprene are mainly photo-oxidized through the reactions with OH radical in the atmosphere and its primary first-generation reaction products are formaldehyde, MVK and MACR (Apel et al., 2002) . The reaction processes of isoprene oxidized by OH radical are mainly as follows:

$$Isoprene + OH \rightarrow 0.63HCHO + 0.32MVK + 0.23MACR \quad k_1=1.0\times10^{-10}\ cm^{-3}\ s^{-1}\ (Eq.\ S1)$$

$$MVK + OH \rightarrow Products \quad k_2=1.9\times10^{-11}\ cm^{-3}\ s^{-1}\ (Eq.\ S2)$$

$$MACR + OH \rightarrow Products \quad k_3=3.3\times10^{-11}\ cm^{-3}\ s^{-1}\ (Eq.\ S3)$$

where k_1, k_2, k_3 are the rate constants of the reactions. According to above reactions, the relationship between MVK/Isoprene, MACR/Isoprene, (MVK+MACR)/Isoprene and the reaction time Δt can be decribed as follows (Apel et al., 2002):

$$\frac{MVK}{Isoprene} = \frac{0.32k_1}{k_2-k_1}\left(1-\exp((k_1-k_2)\,[OH]\Delta t)\right) \qquad (Eq.\ S4)$$

$$\frac{MACR}{Isoprene} = \frac{0.23k_1}{k_3-k_1}\left(1-\exp((k_1-k_3)\,[OH]\Delta t)\right) \qquad (Eq.\ S5)$$

$$\frac{MVK+MACR}{Isoprene} = \frac{0.32k_1}{k_2-k_1}\left(1-\exp((k_1-k_2)\,[OH]\Delta t)\right) + \frac{0.23k_1}{k_3-k_1}\left(1-\exp((k_1-k_3)\,[OH]\Delta t)\right) \quad (Eq.\ S6)$$

where $\frac{MVK+MACR}{Isoprene}$ can be derived from the measurements by PTR-ToF-MS. Then the OH exposure ($[OH]\Delta t$) of isoprenoids can be obtaind from Eq. S6.

The different diurnal patterns for the biogenics and the anthropogenics are mainly due to their different emission patterns. Much more fresh emissions for biogenic compounds during the daytime than nighttime, leading to the low OH exposure during the daytime for these compounds. The photochemical age of the air mass after mixing is the nonlinear addition of the photochemical age before mixing. Generally, the photochemical age of the mixed air mass is closer to that of the fresh air mass (Parrish et al., 1992). The large missing daytime source in the NCP might be some other SOA precursors or formation pathways (e.g. aqueous reactions) (Kuang et al., 2020)

The math on Eq. 1 makes sense, but I'm not sure the assumptions do. This assumes that the air behaves like a plug flow reactor from some emissions source, and then just oxidizes until the measurement site. Wouldn't local emissions (that perhaps have a different oxidation history) change things dramatically? Given that benzene and TMB aren't present in diesel or motor oil (which is the source of higher alkanes), this might be an issue. Though they are both from vehicle emissions, so maybe not. This might all be more compelling with more detail on how OH exposure is calculated.

Response: When calculating OH exposure for anthropogenic compounds, we use the ratio between m+p-xylene and ethylbenzene. Currently, given the fact that we don't have sufficient data to evaluate the specific contributions from different emission sources for all these anthropogenic compounds, we assume that m+p-xylene, ethylbenzene and higher alkanes have the same dominant emission sources during campaigns. In the near future, we will conduct VOCs measurements from typical emission sources in China.

Specific comments:

Line 135. The relationship between humidity and sensitivity seems fairly robust, so correctable, but I'm not sure a would classify a 50% drop in sensitivity as a "slight" decrease. If the "sensitivity" is to m-1, is the drop due to a change in fragmentation, or a drop in all ions? As it also due to a shift of NO^+ to H_3O^+ as water content increases, or does increasing humidity not increase H_3O^+?

Response: We modified the description about the relationship between humidity and sensitivity. We did several times humidity experiments in the laboratory by diluting higher alkanes standard into humidified air (relative humidity of 0-95% at 25℃) to reach approximately 1 ppb mixing ratio. The Figure 3 (b, c) summarize all the experiments data at different times to derive the relationships for C12 and C15 alkanes. The “sensitivity” is to m-1 ion. As shown in the attached Figure (a), the normalized signals of all product ions and the fragment ions are decreasing with the increase of humidity. These decreasing patterns are probably due to the decreasing reagent ions (NO^+ and O_2^+) as the humidity increases. Thus, the humidity correction should be applied for the quantitation of higher alkanes using NO^+ PTR-ToF-MS.

We modified and added sentences on line 163-168 of page 7-8 in the revised manuscript as follows:

“As shown in Figure 3(b, c) and Figure S7 (a), the normalized signal of all product ions (m-1) and the fragment ions of *n*-alkanes (C8-C15) standards are decreasing with the increase of humidity. These decreasing patterns are probably due to the decreasing primary reagent ions (NO^+ and O_2^+) as the humidity increases Figure S7(b). Thus, the humidity correction should be applied for the quantitation of higher alkanes using NO^+ PTR-ToF-MS.”

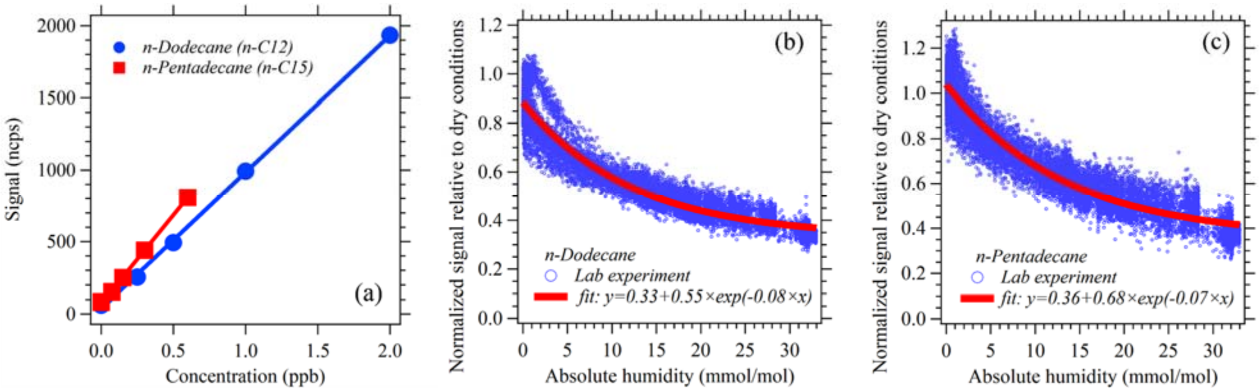

Figure 3. (a) Calibrations of *n*-Dodecane and *n*-Pentadecane under dry conditions; (b) Humidity dependence of *n*-Dodecane. (c) Humidity dependence of *n*-Pentadecane.

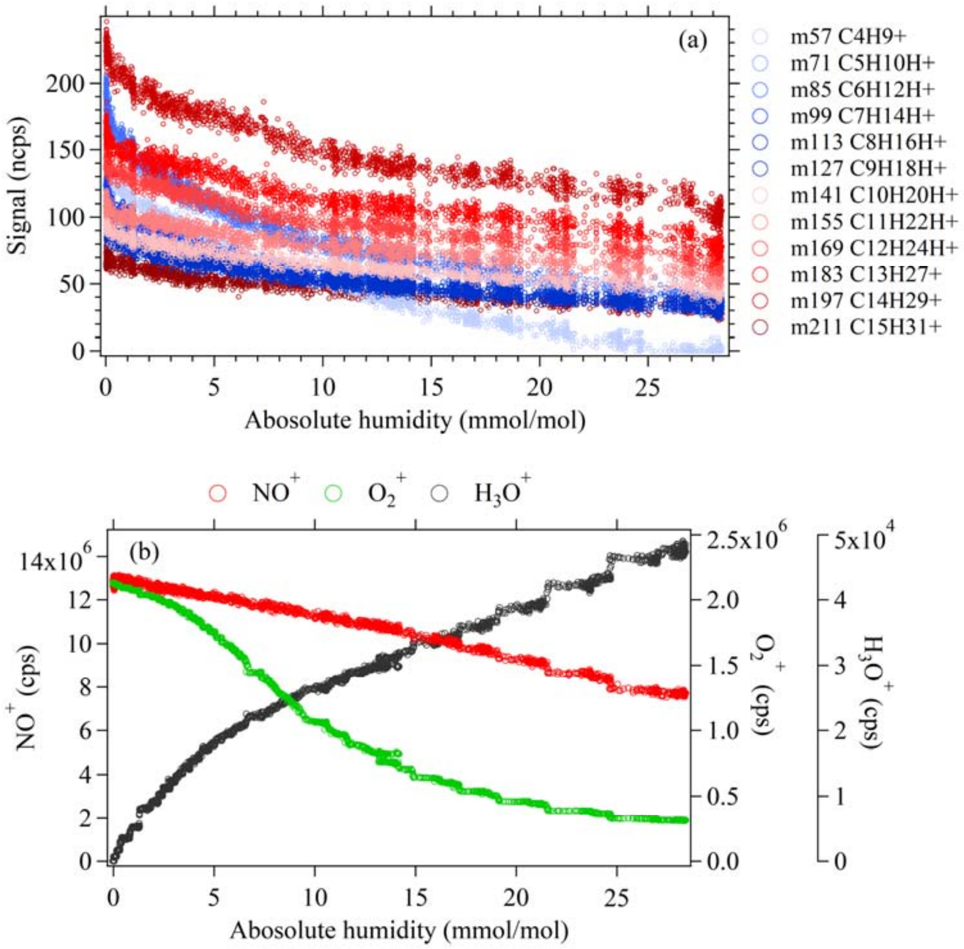

Figure S7. Humidity dependence of all product ions and the fragment ions for *n*-alkanes (C8-C15) (a), and primary ions (NO^+, O_2^+, H_3O^+) (b).

Line 144. Is this slope between fraction of m-1 and sensitivity just due to the decrease m-1? In other words, is the total ion count produced the same, and just the fragmentation different, or is the total ion count lower for lower alkanes?

Response: We used a mixed standards of *n*-alkanes (C8-C15) to do the sensitivity experiments with NO^+ PTR-ToF-MS. Since almost all the higher alkanes produce the fragmentation ions of m/z 57, m/z 71, m/z 85, and m/z 99, we are not able to calculate the total ion count for each higher alkane with our experiments in this study. But the reason for this slope might be explored in the future when we have sufficient experimental conditions.

Line 144-145. Are calibrations of C16-C21 not available directly just because it is hard to get them into the gas phase, or was there some other reason?

Response: The commercial mixed standards of *n*-alkanes (C8-C15) we used in this study are all at 100 ppb except *n*-C15 at 30 ppb, because the lower vapour pressure of *n*-C15 make it difficult to obtain higher concentration of 100 ppb in the same cylinder. The vapour pressures of *n*-C16-*n*-C21 are even lower than that of *n*-C15. It is not possible to prepare *n*-C16-*n*-C21standards with concentrations of > 30 ppb into the mixed standards of *n*-alkanes (C8-C15) in the same cylinder. Moreover, the very low vapour pressure compounds are not stable in the cylinder.

Line 160. Should be "except" instead of "expect Line 162. How was this switching achieved? Was it just a change in voltages? Are there residual effects observed, and/or do some transitional data need to be removed?

Response: We replaced the “expect” with “except” in the revised manuscript. More details about the automatic switches between H_3O^+ chemistry and NO^+ chemistry are also included on line 199-205 of page 9 in the revised paper.

“Switching between H_3O^+ and NO^+ ion mode are provided by the PTR-MS Manager (v3.5) software developed by the Ionicon Anlytik (Table S1). The pressures of drift chamber are held constant at 3.8 mbar in both modes during the campaigns (Figure S10(a)). It usually takes <10 s for H_3O^+ ions and ~60 s for NO^+ ions to re-stabilize after automatically switching between the two measurement modes (Figure S10(b)). The ambient measurement data during the transition period (~1 min) was discarded.”

Table S1. The settings of the voltages of ion source voltages (Us, Uso), drift tube (Udrift, Udx) and pressure of drift tube (pDrift) during automatical switching between NO^+ mode and H_3O^+ mode, respectively.

Setting	NO^+ mode	H_3O^+ mode
Us	40 V	150 V
Uso	100 V	80 V
Udrift	470 V	920 V
Udx	23.5 V	46 V
pDrift	3.8 mbar	3.8 mbar

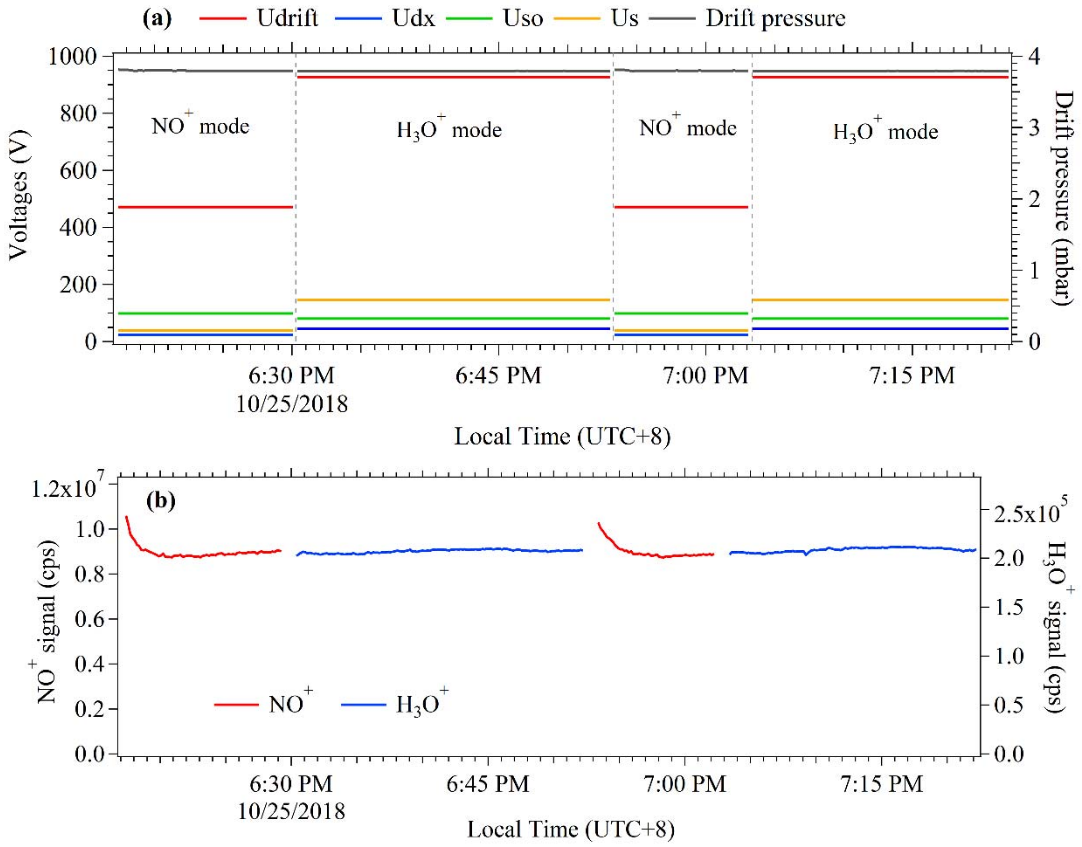

Figure S10. An example of the voltages of ion source voltages (Us, Uso), drift tube (Udrift, Udx) and pressure of drift tube (a), and the signal changes of primary ions (b) during automatical switching between NO$^+$ mode and H_3O^+ mode, respectively.

Line 167. I'm not quite sure what the "insulated tube" is - do they mean heater rope or something?

Response: We revised the sentence. "**The inlet tubing was heated all the way to the sampling inlet to avoid water vapour condensation by an insulating tube with a self-controlled heater wire (40 ℃) wrapping outside.**"

Line 185. Do the authors mean there are limitations to its application? Or just that it hasn't been applied very often?

Response: We revised the sentence. "**Although NO$^+$ chemistry has been shown to be valuable in measuring many organic species, the applications in real atmosphere of different environments are still rare**".

Line 187-190. The comparison between H3O+ and NO+ and GC-MS/FID is very encouraging. There are some significantly non-unity slopes though (0.47 for benzene) -do the authors have an explanation for this?

Response: Yes, the benzene of H_3O^+ measurement is not well consistent with that of NO^+ measurement in the formal period of PRD campaigns. We don’t know the exact reason for this. Considering the good consistency of benzene between NO^+ measurement and GC-MS measurement, we used the benzene from NO^+ measurement in this study.

Line 276. A Table of SOA yields used would be helpful for the SI

Response: We included a table of SOA yields in the revised supporting information (Table S3).

Table S3. The calculated average SOA yields of higher alkanes in PRD and NCP.

Compounds	**Formula**	**Average SOA**	**Average SOA**
Octane	C_8H_{18}	0.003±0.002	0.006±0.004
Nonane	C_9H_{20}	0.010±0.005	0.017±0.010
Decane	$C_{10}H_{22}$	0.026±0.012	0.040±0.021
Undecane	$C_{11}H_{24}$	0.058±0.020	0.080±0.036
Dodecane	$C_{12}H_{26}$	0.106±0.032	0.142±0.059
Tridecane	$C_{13}H_{28}$	0.249±0.061	0.305±0.103
Tetradecane	$C_{14}H_{30}$	0.329±0.070	0.388±0.118
Pentadecane	$C_{15}H_{32}$	0.386±0.081	0.450±0.135
Hexadecane	$C_{16}H_{34}$	0.428±0.086	0.492±0.141
Heptadecane	$C_{17}H_{36}$	0.488±0.096	0.556±0.156
Octadecane	$C_{18}H_{38}$	0.664±0.079	0.704±0.139
Nonadecane	$C_{19}H_{40}$	0.773±0.056	0.792±0.105
Eicosane	$C_{20}H_{42}$	0.860±0.025	0.863±0.054
Heneicosane	$C_{21}H_{44}$	0.877±0.025	0.870±0.046

Line 301-304. Are there emissions sources for only alkanes? I would guess not, in which case these alkanes must be getting emitted alongside cyclic saturated hydrocarbons (e.g., cycloalkanes), which actually contribute similar or more to petroleum fuels (e.g., Gentner et al., 2012, Table S5). These compounds are expected to have broadly similar (or slightly higher) SOA yields. This would have you substantially overestimating SOA in NCP at night - the authors should comment on possible explanations or biases.

Response: In this study, the higher alkanes represent only the total concentration of linear and branched isomers, without considering the cyclic alkanes. Yes, some studies have indicated that cyclic alkanes also contribute importantly to SOA formation (Tkacik et al., 2012; Gentner et al., 2012). In the near future, we will also try the possibility to measure these cyclic alkanes. Including these cyclic alkanes would enhance the estimation of SOA production shown in this study.

Figure 5. I'm not quite sure what data is being shown here. Gentner provides diesel fuel data in table S5 of that publication (this is used to approximate exhaust in that manuscript). That data doesn't agree with what is shown here though - for C8 it is 37%, and all the values are near or above 21%. The Isaacman paper is actually just a single fuel sample that is included in the Gentner data set, so could probable be excluded. That paper also provides gasoline data, which is not shown. In Figure 9 of the Chan paper, the branched-to-normal ratios are given for vehicle exhaust, which convert to between 13 and 41% normal for C24 through C21, which are fairly different than those shown. Similarly, in Figure 5, the branched-to-normal ratios for LA are shown at multiple oxidation times, and reach levels of 20 to 50% for those same carbon numbers - this is simlar to the values shown, but I think not quite right. I think the numbers for Bakersfield (based on Figure 4 from that paper) should be much lower, 5-10%, for all carbon numbers. Values should also be included for direct vehicle emissions from Worton et al., 2014, dx.doi.org/10.1021/es405375j. Basically, some explanation for how these numbers were all compiled is necessary (even if it's just in the SI), because they don't look quite right to me.

Response: Linear alkanes and branched alkanes follow the same molecular formula: C_nH_{2n+2}. When using NO^+ ToF-MS, we measure the total concentrations of linear alkanes and branched alkanes with the same formulas since this technique doesn't differentiate isomers. We collected data from literatures which are derived from GC-based techniques to calculate the mass fractions of *n*-alkanes in higher alkanes with the same formula from various ambient and emission studies, with the purpose of emphasizing the importance of contribution of branched isomers to higher alkanes concentrations at each carbon number. The fraction of *n*-alkane at each carbon number is calculated as follows:

$$Fraction_{linear} = \frac{Concentration_{linear}}{Concentration_{linear} + Concentration_{branched}} \times 100\%$$

As shown in the figure, *n*-alkanes contribute ~5-60% to higher alkanes concentrations from C8 to C25, indicating that branched isomers contribute up to ~40-95%. This indicates the measurement of branched isomers is also important. We checked the data collected from references and modified the figure (Figure 6) as suggested. A table of data used in this figure was also included in the revised supporting information (Table S2). The data from Worton et al., 2014 was not included because those high alkanes data are particle phase. In this study, we only focus on the gas phase alkanes and their importance to SOA.

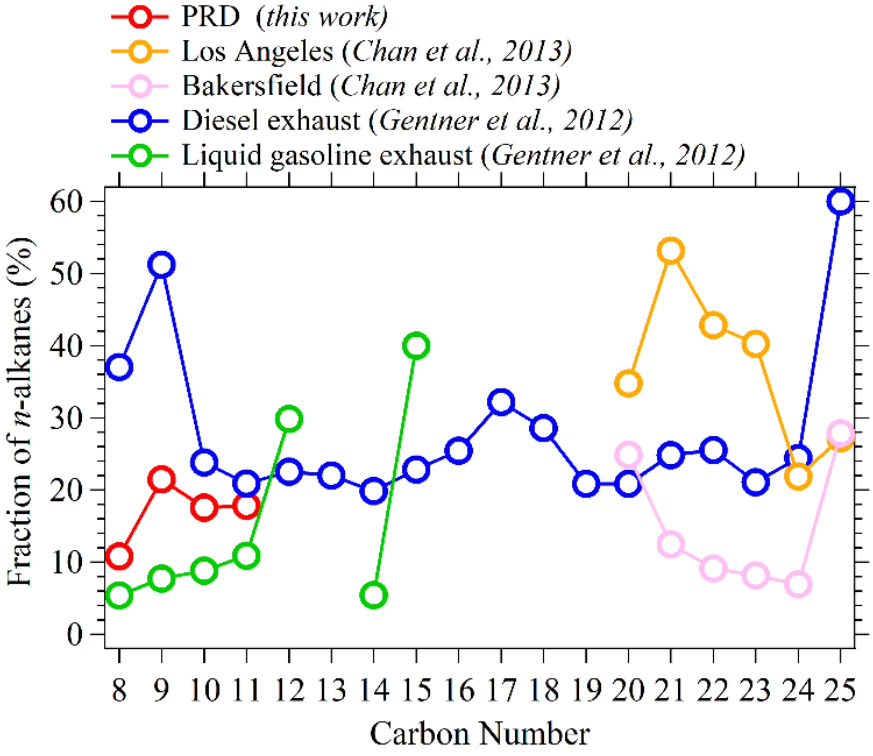

Figure 6. Fractions of *n*-alkanes in higher alkanes with same formulas derived from this study, ambient air in Los Angeles, Bakersfield and in vehicle exhausts.

Table S2. Fractions of *n*-alkanes in higher alkanes with same formulas derived from this study, ambient air in Los Angeles, Bakersfield and in vehicle exhausts.

Carbon Number	Fraction of *n*-alkanes in higher alkanes with same formulas				
	PRD[a]	Los Angeles[b]	Bakersfield[b]	Diese exhaust[c]	Liquid gasoline exhaust[c]
8	10.82	/	/	37.04	5.39
9	21.48	/	/	51.22	7.71
10	17.56	/	/	23.81	8.81
11	17.81	/	/	20.91	10.88
12	/	/	/	22.54	29.82
13	/	/	/	21.98	/
14	/	/	/	19.84	5.41
15	/	/	/	22.86	40
16	/	/	/	25.44	/
17	/	/	/	32.16	/
18	/	/	/	28.57	/
19	/	/	/	20.83	/
20	/	34.78	24.85	20.87	/
21	/	53.16	12.47	24.82	/
22	/	42.85	9.11	25.51	/
23	/	40.24	8.14	21.05	/
24	/	21.85	6.92	24.44	/
25	/	27.17	27.87	60	/

[a]:This work; [b]: Chan et al., 2013; [c]: Gentner et al., 2012

Reference:

Apel, E. C., Riemer, D. D., Hills, A., Baugh, W., Orlando, J., Faloona, I., Tan, D., Brune, W., Lamb, B., Westberg, H., Carroll, M. A., Thornberry, T., and Geron, C. D.: Measurement and interpretation of isoprene fluxes and isoprene, methacrolein, and methyl vinyl ketone mixing ratios at the PROPHET site during the 1998 Intensive, Journal of Geophysical Research: Atmospheres, 107, ACH 7-1-ACH 7-15, 10.1029/2000JD000225, 2002.

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