

We sincerely thank the Referee for the valuable comments. Our manuscript has been revised according to the comments from the Referee and our responses to the comments are as follows. For clarity, the comments are reproduced in blue, authors' responses are in black and changes in the manuscript are in red.

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## Responses to Anonymous Referee #2

### General comments

The authors have studied SOA formation from photo-oxidation of 1,2,4-trimethylbenzene under different concentrations of NO<sub>x</sub>, SO<sub>2</sub> and NH<sub>3</sub> in an indoor smog chamber. The reactants and the particle generation and growth were monitored using a series of standard instrumentations. The chemical functional groups were characterized by ATR-FTIR in each experiment and inorganic constituents were analyzed using ion chromatography. The molecular level information is provided using UPLC-HRMS and dd-MS<sup>2</sup> scans. Ten new organosulfates are identified in the presence of SO<sub>2</sub>, including 3 in which the origin was previously unknown and some previously reported to be originated from biogenic precursors. Formation mechanisms for 8 of the newly identified organosulfates are proposed based on previous literature. Their results indicate that SO<sub>2</sub> is a key parameter for ultrafine particle formation. A synergistic effect of NH<sub>3</sub> and SO<sub>2</sub> in particle formation is also shown, indicating the importance of reducing both SO<sub>2</sub> and NH<sub>3</sub> emissions to improve lowering PM. Their results also suggest that ammonium sulfate form by the reaction of NH<sub>3</sub> with H<sub>2</sub>SO<sub>4</sub> facilitate aerosol formation and growth through condensation of organic vapors.

This article advances the current knowledge of aerosol formation from photo-oxidation of a typical aromatic hydrocarbon in the presence of NO<sub>x</sub>, SO<sub>2</sub> and NH<sub>3</sub>, therefore is of interest to the scientific community of ACP. The manuscript is well written and organized. The experiments are well executed, methods are explained adequately, results are discussed thoroughly, and conclusions are well supported. I have some minor comments to improve the quality of this manuscript that are listed below. I recommend accepting this manuscript for publication in ACP with minor revisions.

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## Specific comments

Line 13 Indicate the major emission sources of TMB.

### Author Reply:

Aromatic hydrocarbons can dominate the VOC budget in certain urban areas with  
35 TMB being one of the most significant species (Ran et al., 2009). TMB is mostly  
emitted from anthropogenic sources such as solvent use (Mo et al., 2021). Recent  
studies have recognized TMB as an important species in SOA formation in the  
atmosphere (Wang et al., 2020; Zaytsev et al., 2019; Mehra et al., 2020). Hence, the  
following text has been added in the abstract.

40 Aromatic hydrocarbons can dominate the volatile organic compounds budget in the  
urban atmosphere. Among them, 1,2,4-trimethylbenzene (TMB), mainly emitted from  
solvent use, is one of the most important secondary organic aerosols (SOA) precursors.

Line 45 Define 'certain regions.

### 45 Author Reply:

The original text has been supplemented as follows: High levels of SO<sub>2</sub>, NH<sub>3</sub>, and  
VOCs have been reported in certain regions such as Guangzhou (Zou et al., 2015),  
Beijing (Meng et al., 2020), Handan (Li et al., 2017) in China. During haze pollution  
episodes, Li et al. (2017) observed that SO<sub>2</sub> levels can be up to 200 ppb in Handan,  
50 China. A recent study also showed significant increasing NH<sub>3</sub> levels in the atmosphere  
over the United States and the European Union (Warner et al., 2017).

Line 54-56 Add reference/s.

### Author Reply:

55 The following studies have been added to the references list.

1. Estillore, A. D., Hettiyadura, A. P. S., Qin, Z., Leckrone, E., Wombacher, B.,  
Humphry, T., Stone, E. A., and Grassian, V. H.: Water Uptake and Hygroscopic Growth  
of Organosulfate Aerosol, Environ. Sci. Technol., 50, 4259-4268,  
10.1021/acs.est.5b05014, 2016.
- 60 2. Fleming, L. T., Ali, N. N., Blair, S. L., Roveretto, M., George, C., and Nizkorodov,

S. A.: Formation of Light-Absorbing Organosulfates during Evaporation of Secondary Organic Material Extracts in the Presence of Sulfuric Acid, *ACS Earth Space Chem.*, 3, 947-957, 10.1021/acsearthspacechem.9b00036, 2019

3. Hansen, A. M. K., Hong, J., Raatikainen, T., Kristensen, K., Ylisirniö, A., Virtanen, A., Petäjä, T., Glasius, M., and Prisle, N. L.: Hygroscopic properties and cloud condensation nuclei activation of limonene-derived organosulfates and their mixtures with ammonium sulfate, *Atmos. Chem. Phys.*, 15, 14071-14089, 10.5194/acp-15-14071-2015, 2015.

4. Riva, M., Chen, Y., Zhang, Y., Lei, Z., Olson, N. E., Boyer, H. C., Narayan, S., Yee, L. D., Green, H. S., Cui, T., Zhang, Z., Baumann, K., Fort, M., Edgerton, E., Budisulistiorini, S. H., Rose, C. A., Ribeiro, I. O., RL, E. O., Dos Santos, E. O., Machado, C. M. D., Szopa, S., Zhao, Y., Alves, E. G., de Sa, S. S., Hu, W., Knipping, E. M., Shaw, S. L., Duvoisin Junior, S., de Souza, R. A. F., Palm, B. B., Jimenez, J. L., Glasius, M., Goldstein, A. H., Pye, H. O. T., Gold, A., Turpin, B. J., Vizuete, W., Martin, S. T., Thornton, J. A., Dutcher, C. S., Ault, A. P., and Surratt, J. D.: Increasing Isoprene Epoxydiol-to-Inorganic Sulfate Aerosol Ratio Results in Extensive Conversion of Inorganic Sulfate to Organosulfur Forms: Implications for Aerosol Physicochemical Properties, *Environ. Sci. Technol.*, 53, 8682-8694, 10.1021/acs.est.9b01019, 2019.

5. Zhang, Y., Chen, Y., Lei, Z., Olson, N. E., Riva, M., Koss, A. R., Zhang, Z., Gold, A., Jayne, J. T., Worsnop, D. R., Onasch, T. B., Kroll, J. H., Turpin, B. J., Ault, A. P., and Surratt, J. D.: Joint Impacts of Acidity and Viscosity on the Formation of Secondary Organic Aerosol from Isoprene Epoxydiols (IEPOX) in Phase Separated Particles, *ACS Earth Space Chem.*, 3, 2646-2658, 10.1021/acsearthspacechem.9b00209, 2019.

85 They are cited in the revised manuscript as follows:

The presence of OSs could alter aerosol morphology (Riva et al., 2019), viscosity (Riva et al., 2019; Zhang et al., 2019), particle acidity (Riva et al., 2019), phase state (Zhang et al., 2019), hygroscopicity (Estillore et al., 2016; Hansen et al., 2015), and optical properties (Fleming et al., 2019), thereby resulting in large climate effects.

Line 98-99 Briefly indicate what MCM is and add a link to the version.

**Author Reply:**

The Master Chemical Mechanism (MCM, <http://mcm.leeds.ac.uk/MCMv3.2/>, last access: 23 February 2021) is a near-explicit chemical mechanism that can describe, in detail, the tropospheric degradation of numerous VOCs. The oxidation processes of VOCs are highly complex. Explicit chemical mechanisms might cause missing oxidation pathways and products, thus leading to large gaps between the modeled and measured SOA mass. To the best of our knowledge, the oxidation mechanism of TMB has not been updated in the MCM since 2005. Wang et al. (2020) suggested that recently identified autoxidation pathways for OH oxidation of TMB were not included in the current MCM and the detected products are more diverse than the products shown in MCM. The updates for the OH-initiated oxidation mechanism of TMB can be achieved only when the rate, branching ratios and product distributions can be explicitly obtained. Therefore, more investigations are needed to further explore the detailed chemical processes for OH oxidation of TMB. According to the suggestion of the Referee, the following text was added in the revised manuscript.

Page 4, lines 124–134

In addition, the Master Chemical Mechanism (MCM, <http://mcm.leeds.ac.uk/MCMv3.2/>, last access: 23 February 2021) is a near-explicit chemical mechanism that can describe, in detail, the tropospheric degradation of numerous VOCs. A recent study reported that identified autoxidation pathways during OH oxidation of TMB were not included in the current MCM and the detected TMB products were more diverse than the products shown in MCM (Wang et al., 2020). TMB photooxidation is highly complex and sensitive to environmental conditions. The updates for the OH-initiated oxidation mechanism of TMB can be achieved only when the rate constants, branching ratios and product distributions can be explicitly obtained. It is necessary to further investigate the detailed chemical processes for TMB photooxidation.

Line 210 Indicate how the mass calibration was performed.

**Author Reply:**

We used standard calibration solutions provided by the manufacturer to calibrate the MS instrument every five days. The following text was added in the revised manuscript.

125 Page 9, lines 259–260

The MS instrument was calibrated every five days with standard calibration solutions provided by the manufacturer.

130 Line 200 Indicate the mass resolution in full MS, top N in dd-MS<sup>2</sup>, isolation width (mass window) etc

**Author Reply:**

In the revised manuscript, detailed parameters are indicated as follows:

Page 9, lines 253–259

135 MS spectra were recorded in the range of  $m/z$  50 to 750 in full MS scan with a mass resolving power of 70000 (FWHM at  $m/z$  200). The full MS scan was followed by data-dependent MS/MS (dd-MS<sup>2</sup>) scans using stepped collision energies of 20, 40, and 60 eV via high-energy collisional dissociation. The resolution was 17500 and an isolation width of 2  $m/z$  units was applied for the dd-MS<sup>2</sup> scan. The other parameters for MS<sup>2</sup> experiments were as follow: AGC target,  $2 \times 10^5$ ; maximum IT, 50 ms; loop count, 3;  
140 minimum AGC target,  $1 \times 10^5$ ; apex trigger, 2–6 s; dynamic exclusion, 6 s.

Line 277 Add initial growth rates to Table 1 or SI.

**Author Reply:**

145 The aim of depicting Fig. 2 in the original manuscript was to clearly reflect the changes of the particle nucleation time, initial growth rate, and mean diameter with the initial SO<sub>2</sub> concentration. The specific particle parameters including the particle initial growth rate have been summarized in the table below.

**Table R1.** Particle parameters for experiments 1–8.

Exp.	Nucleation time (min)	Particle mean diameter (nm)	Initial particle growth rate (nm h <sup>-1</sup> )
1	70	125.5	46.53
2	15	109.9	20.09
3	10	121.2	27.42
4	10	130.6	31.09
5	100	123.1	23.51
6	20	118.5	19.30
7	10	112.4	22.14
8	10	136.5	29.82

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Table R1 has been added in the revised supplement as Table S1 and the caption of Fig. 2 have been also updated as follows.

**Figure 2.** Particle nucleation time as a function of initial SO<sub>2</sub> concentration under low-NO<sub>x</sub> (open circles) and high-NO<sub>x</sub> (solid circles) conditions (Exps. 1–8). The symbol color indicates the particle mean diameter and symbol size represents the particle initial growth rate. Values of the particle parameters are listed in Table S1.

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Line 277-280 Can the authors elaborate the reasons for the observed non-linear response of the particle diameter with initial SO<sub>2</sub> concentrations?

160 **Author Reply:**

OH-initiated oxidation of VOCs can yield a number of products with different degrees of volatility (Xu et al., 2014). The growth of aerosol particles is related to the coagulation, the condensation of non-volatility oxidation products, and gas-particle partitioning of semi-volatility organic compounds (SVOCs). For SVOCs, the evaporation is important after partitioning to the particle phase. Therefore, the rate at which SVOCs participate in the particle growth is lower than their condensation rate. Interestingly, recent studies showed that the particle-phase chemistry such as heterogeneous reactions of SVOCs are substantially pronounced for particle growth (Shiraiwa et al., 2013; Paasonen et al., 2018; Apsokardu and Johnston, 2018). Apsokardu and Johnston (2018) explored the influences of particle-phase chemistry on the growth rate of aerosol particles with a kinetic growth model and found that some

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SVOCs can undergo accretion reactions in the particle-phase to further accelerate the particle growth. Paasonen et al. (2018) also showed that the increase in particle growth rate is related to particle-phase reactions. The study of Shiraiwa et al. (2013) highlighted the importance of particle-phase chemistry in the changes of SOA size distribution and suggested that particle-phase chemistry depends to some extent on particle acidity. Organosulfates can be produced by particle-phase reactions involving interactions between organics and inorganics. In this work, we measured organosulfates only in SO<sub>2</sub>-involved photooxidation, indicating that additional particle-phase reactions can occur under SO<sub>2</sub>-involved conditions. Increasing initial SO<sub>2</sub> levels could induce the formation of more sulfate (Fig. S4) and the enhancement in the particle acidity during photooxidation (Liu et al., 2016; Kroll et al., 2006). The elevated particle acidity can promote more SVOCs to transform into non-volatile products in the particle phase (Lin et al., 2014; Lal et al., 2012). Additional SVOCs could be moved from the gas phase to particle phase to increase the particle size and the evaporation of SVOCs can be inhibited, thereby promoting the particle growth.

It is somewhat interesting that the particle mean diameter in low-SO<sub>2</sub> ( $[\text{SO}_2]_0 < 100$  ppb) experiments is smaller than that in SO<sub>2</sub>-free experiments. Our result is in line with the study of Wyche et al. (2009), who attributed this phenomenon to the larger number of particles produced under SO<sub>2</sub>-involved condition. In this work, the presence of 59 ppb SO<sub>2</sub> caused the maximum number concentration of particles to increase by  $8.5 \times 10^4 \text{ cm}^{-3}$  under low-NO<sub>x</sub> condition. When the SO<sub>2</sub> level increased from 0 to 68 ppb in high-NO<sub>x</sub> experiments, the corresponding particle number concentration increased from  $2.9 \times 10^4$  to  $9.3 \times 10^4 \text{ cm}^{-3}$ . Therefore, the amounts of products that condensed onto each aerosol particles may significantly decrease in low-SO<sub>2</sub> experiments, which can result in the decrease in particle diameter (Liu et al., 2015). The promoting effect of particle-phase chemistry on the particle size growth may not offset the inhibiting effect of the emergence of large number of particles on the particle size growth, thereby leading to the low particle diameter in low-SO<sub>2</sub> experiments. We have added the following text to explain the reasons for the observed non-linear response of the particle diameter with initial SO<sub>2</sub> level.

Aerosol particles can grow in different ways such as gas-particle partitioning of semi-volatility organic compounds (SVOCs). Since the evaporation of SVOCs is important after partitioning to the particle phase, the rate at which SVOCs participate in the particle growth is lower than their condensation rate. However, recent advances give an insight that the particle-phase chemistry such as heterogeneous reactions of SVOCs are substantially pronounced for the particle growth (Shiraiwa et al., 2013; Paasonen et al., 2018; Apsokardu and Johnston, 2018). Organosulfates can be produced by particle-phase reactions involving interactions between organics and inorganics. In this work, organosulfates were only detected in SO<sub>2</sub>-involved photooxidation, indicating that additional particle-phase reactions can occur under SO<sub>2</sub>-involved conditions. Increasing the initial SO<sub>2</sub> level could induce the formation of more sulfate (Fig. S4) and the enhancement in the particle acidity during photooxidation (Liu et al., 2016; Kroll et al., 2006). The elevated particle acidity can promote more SVOCs to transform into non-volatile products such as oligomers and other high molecular mass compounds in the particle phase, thereby promoting the particle growth (Lin et al., 2014; Lal et al., 2012). Then, additional SVOCs could be transferred from the gas phase to the particle phase to increase the particle size. However, the particle mean diameter in low-SO<sub>2</sub> ([SO<sub>2</sub>]<sub>0</sub> < 100 ppb) experiments is smaller than that in SO<sub>2</sub>-free experiments. Our result is in line with the study of Wyche et al. (2009), who attributed this phenomenon to the larger number of particles produced under SO<sub>2</sub>-involved condition. The presence of 59 ppb SO<sub>2</sub> caused the maximum number concentration of particles to increase by  $8.5 \times 10^4 \text{ cm}^{-3}$  under low-NO<sub>x</sub> condition. When the SO<sub>2</sub> level increased from 0 to 68 ppb in high-NO<sub>x</sub> experiments, the corresponding particle number concentration increased from  $2.9 \times 10^4$  to  $9.3 \times 10^4 \text{ cm}^{-3}$ . Therefore, the amounts of products that condensed onto each aerosol particles significantly decreased in low-SO<sub>2</sub> experiments, which could result in the decrease in particle diameter (Liu et al., 2015). The promoting effect of particle-phase chemistry on the particle size growth may not offset the inhibiting effect of the emergence of large number of particles on the particle size growth, thereby leading to the low particle diameter in low-SO<sub>2</sub> experiments.



Figure 4 – Indicate what error bars represent.

**Author Reply:**

235 The error bars represent uncertainties in the SOA yield results. The uncertainties were calculated from error propagation using the sum of the uncertainties in TMB data and the systematic error of SMPS. The following note was included in the caption of Fig. 4.

240 The error bars represent uncertainties in the SOA yield results and the uncertainties were calculated from error propagation using the sum of the uncertainties in TMB data and the systematic error of SMPS.

Figure 5 – Indicate the experiment numbers relevant to a) to f)

**Author Reply:**

245 The experimental numbers have been added in the caption of Fig. 5 as:

**Figure 5.** ATR-FTIR spectra of aerosol particles generated from TMB/NO<sub>x</sub> (a, Exp. 1; b, Exp. 5), TMB/NO<sub>x</sub>/SO<sub>2</sub> (c, Exp. 4; d, Exp. 8), TMB/NO<sub>x</sub>/NH<sub>3</sub>/SO<sub>2</sub> (e, Exp. 12), and TMB/NO<sub>x</sub>/NH<sub>3</sub> (f, Exp. 10) photooxidation.

250 Lines 346-348 – Add detailed information of the common products observed in SO<sub>2</sub> free and SO<sub>2</sub> involved experiments to the SI.

**Author Reply:**

The detailed information of representative products mentioned in the original manuscript were provided in Table S3 in the revised supplement.

255

Table S3 – Indicate clearly whether this table shows the compounds that are detected in both SO<sub>2</sub> free and SO<sub>2</sub> involved experiments with NH<sub>3</sub> or the products only formed from the experiments involved both NH<sub>3</sub> and SO<sub>2</sub>.

**Author Reply:**

260 The major products shown in Table S3 are the products detected in both SO<sub>2</sub>-free and SO<sub>2</sub>-involved experiments with NH<sub>3</sub> addition. The revised table in the supplement

is now labeled as Table S4 and the caption was revised as: **Observed products in both SO<sub>2</sub>-free and SO<sub>2</sub>-involved experiments with NH<sub>3</sub> addition.**

265 **Tables S2 and S3 – Add UPLC retention times.**

**Author Reply:**

The retention times have been added in the indicated tables, which are now labelled as Table S3 and S4.

270 **Technical corrections**

Line 105 – Add ‘in the atmosphere’ to the end of “Given the ubiquity of SO<sub>2</sub>, NH<sub>3</sub>, and TMB...

**Author Reply:**

275 According to the suggestion of the Referee, we have added “in the atmosphere” at the indicated place in the revised manuscript.

Line 200 – It is better to write it as data-dependent MS/MS (dd-MS<sub>2</sub>) scans

**Author Reply:**

We have modified it in the text.

280

Line 206 – Add B after 3%.

**Author Reply:**

We have added “B” in the revised manuscript.

285 **Figure 6 – Label the red structures as OS-226, OS-228...etc. (Authors may replace the chemical formula with their abbreviated names as the structures are shown.)**

**Author Reply:**

The red structures in Fig. 6 have been marked with OS-266, OS-228...etc.

290 **Figure 10 – Match the color of the TMB on the figure with carbon number (should be light blue as it has 9 carbons)**

## Author Reply:

We have corrected it in the revised manuscript.

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## References

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