A letter to reply the comments

We thank the reviewers for their thoughtful comments. We have carefully revised the manuscript accordingly. Our point-to-point responses to the reviewers' comments, which are repeated in italic, are given below.

ACPD manuscript: **10.5194/acpd-15-29673-2015**
Authors: **L. Yu, J. Smith, A. Laskin, K. M. George, C. Anastasio, J. Laskin, A. M. Dillner, and Q. Zhang**

To Reviewer #1

This study investigates the chemical evolution of SOA formed from reactions of phenolic compounds in the aqueous phase. The dataset that combines the AMS and DESI-MS measurements, in principal, provides important observations into the interplay of key processes including functionalization, fragmentation, and oligomerization in the SOA cloud processing. The role of aqueous photochemical aging in the phenolic SOA evolution is demonstrated in terms of transformation of elemental/molecular composition and volatility distribution. In view of this, I would recommend publication on ACP with some issues that require clarification.

Reviewer's comment:
1. The importance of aqueous phase photochemistry in the SOA aging relies on the availability of precursors. While results presented in the current study indicate that the aqueous photooxidation of phenolic compounds significantly alter the aqSOA signature, the abundance of phenolic compounds in the aqueous phase could substantially limit the power of this reaction scheme in the entire lifetime of phenol derived SOA. The authors are suggested to provide:
1) The average level of phenolic compounds in ambient aerosols or clouds;

Authors' reply #1: According to previous studies, the concentrations of phenols and substituted phenols measured in cloud and fog waters were typically in the range of 0.1-30 µM (*Anastasio et al., 1997;Sagebiel and Seiber, 1993*). In addition, concentrations exceeding 100 µM were predicted to present in fog waters in areas impacted by wood burning (e.g., in Northern California during wintertime) based on measured concentrations of these compounds in aerosol particles (*Anastasio et al., 1997*). This information is now provided in Sect. 2.1.

Reviewer's comment:
2) Compare the lifetimes of phenols in terms of further oxidized by OH radicals in the gas phase, vs. transported and dissolved into liquid particles;

Authors' reply #2: The lifetimes of phenols with respect to $^3C^*$ and •OH reactions in atmospheric fog and cloud water are on the order of minutes to hours during daytime (*Smith et al., 2014*). The lifetimes of phenols in the gas phase with respect to •OH reaction are of the same order as in the aqueous phase (i.e., hours) (*Feigenbrugel et al., 2004*). This information is now provided in Sect.1.

Reviewer's comment:
3) Discuss the relative importance of different reaction pathways of phenolic compounds in the atmosphere, mostly, OH-initiated oxidation in the gas phase, vs. aqueous phase.

Authors' reply #3: Our study indicates that during •OH-initiated oxidation of phenols in aqueous phase, oligomer formation was an important process initially and functionalization and fragmentation became increasingly more important as the reaction proceeds. Yee et al. [2013] investigated the gas phase photooxidation of phenols under low-NO_x condition using hydrogen peroxide as a source of •OH and observed no oligomer formation. They reported that demethoxylation and ring opening process were the major reaction pathways for gas phase oxidation of phenols. We have added this information in Sect. 3.4.

Reviewer's comment:
2. While the findings of this study are interesting, fundamental aspects in the methodology employed, i.e., the extent of agreement in the AMS and DESI-MS measurements, need to be addressed. I realize that additional experimental validation might be necessary:
1) Have the authors ever tried to atomize a solution composed of, say 1:1 v/v ammonium sulfate and organic standards (e.g., succinic acid found in the aqSOA samples), into AMS and checked the measured O:C or H:C ratios? This would be a good validation on capability of the atomizing procedure to generate equal amount of solutes in complex mixture.

Authors' reply #4: The AMS and the nano-DESI MS analyze organic aerosols using very different sampling and ionization techniques. Therefore, a direct comparison of their measurement results is not feasible and large differences may be observed. For example, Yu et al. (*2014*) reported that the average O/C ratios of phenolic aqSOA determined by AMS are systematically higher than the signal-weighted average O/C ratios determined based on nano-DESI mass spectra, for which one possible reason is that highly oxidized molecules with molecular weight less than 100 Da are not sampled by nano-DESI MS. Assuming similar ionization efficiency for all molecules identified in the nano-DESI mass spectra can potentially introduce large errors in calculation of average elemental ratios as well.

In terms of the reviewer's question about whether we did AMS analysis of a solution of organic and ammonium sulfate mixtures, the answer is yes. We atomized a solution composed of equal mass of ammonium sulfate and sucrose ($C_{12}H_{22}O_{11}$) and analyzed the resulting aerosol using AMS. The measured O/C and H/C ratios of organics (i.e., sucrose) were 0.95 and 1.98, respectively, which agree well the theoretical values of 0.92 and 1.83. In addition, we found that the mass ratio of organic to sulfate observed in the aerosol by AMS was similar to that originally in the solution. This indicates that the solute composition is preserved during the atomization procedure and drying. This information is now added in Sect. S2 in the supplementary information.

Reviewer's comment:
2) This question might be out of the scope of the current study, but, have the authors ever used the positive mode in the nano-DESI-MS technique on the phenolic aqSOA? If so, did the positive mode measurements give the similar molecular formulas for the dominant compounds as detected by the negative mode measurements?

Authors' reply #5: Yes, we performed nano-DESI MS analyses under both positive and negative modes. For each aqSOA sample, a large number of common molecules were identified by both modes. However, their relative abundances in the mass spectra showed differences between the two modes, likely due to the ionization efficiency differences. For example, ring opening species tended to be more abundant in the (-) MS compared to the (+) MS. A possible reason is that some ring opening species, such as organic acids, are more prone to deprotonation, forming negative ions, than protonation, which forms positive ions. We have added this discussion in Sect 3.2 and listed the top 10 most abundant products detected in the positive ion mode in Table S6.

Reviewer's comment:
3) Oligomerization could potentially occur during the electrospray ionization process (e.g., formation of non-covalent dimer of terpenylic acid in the alphapinene SOA sample). How could the authors distinguish the oligomers produced in the actual aqueous photochemistry from the artifacts generated in the ESI process?

Authors' reply #6: We tested this potential artifact by analyzing dark control samples containing phenolic precursors from each precursor. The solutions were analyzed using ESI-MS through direct infusion and no dimer or oligomer ions were detected. In addition, AMS analyses found ions representative of phenolic dimer and high oligomers only in the illuminated samples, not in the dark controls (*Yu et al., 2014*). These results indicate that oligomers were generated from aqueous photochemical reactions, rather than being an artifact from the ESI process. We have added this discussion in Sect. 3.2.

Reviewer's comment:
3. I consider the observation of ELOVCs formation during the early stage of aqueous photochemistry is one of the highlights of the present study. Could the authors comment on their formation mechanisms by comparing the differences in the chemical structures of the proposed dimers produced from OH- vs. ^{3}C-mediated pathways?*

Authors' reply #7: We characterized the molecular compositions of phenolic aqSOA formed at reaction half-lives and compared them between the •OH- and ^{3}C*-mediated pathways. Overall, aqSOA compositions were similar for the same precursor although the ^{3}C*-mediated reactions tended to be faster and produce more oligomers and hydroxylated species than the •OH-mediated reactions. The compositional difference between the two pathways was relatively small for SYR aqSOA but larger for PhOH aqSOA. In addition, the aqSOA from PhOH + •OH reaction at half-life was mostly composed of substituted monomers and ring opening species, whereas the aqSOA from the PhOH + ^{3}C* reactions contained a larger fraction of oligomers and their functionalized derivatives. More detailed discussions are given in Yu et al. (*2014*).

Reviewer's comment:
4. Page 29695, Table 1: The Henry's Law constants for SYR, GUA, and PhOH need to be given here.

Authors' reply #8: The Henry's Law constant for SYR, GUA, and PhOH is 2.5×10^4, 5.0×10^3, 1.5×10^4 M atm^{-1} respectively. This information is now provided in Table 1.

Reviewer's comment:
5. Page 29696, Table 2: Confidence intervals for the best-fit parameters need to be given.

Authors' reply #9: The 95% confidence intervals for the best-fit parameters are now given in Table 2.

To Reviewer #2

The authors present a detailed chemical characterization of aqueous SOA formation from phenol, syringol, and guaiacol oxidation with OH radical (from HOOH) and $^3C^*$ (from 3,4-dimethoxybenzaldehyde, or 3,4-DMB). They utilize AMS and nano-DESI MS to provide elemental ratios information and average molecular weights of the compounds formed during the reaction progression, finding that functionalized and high MW products (including oligomers) dominate the aqSOA at the beginning of reaction followed by increasing importance of fragmentation. The manuscript is overall well-written, though there are some major questions about the methodology used that need additional clarification, in particular the potential role of 3,4-DMB as both an oxidant source and precursor in aqSOA formation. The selection of experimental variables as compared to the real atmosphere should also be expanded, as it is harder to gauge if current interpretations of the data may be as atmospherically relevant. Further, the manuscript often refers to molecular level characterization with proposed molecular structures and the goal of mechanism development, but there are no attempts to propose mechanisms to support the structures or functionalization assumed. Still, this is a fundamental laboratory study with results that would be of interest to the community and appropriate for ACP, provided major changes are made as suggested below.

Authors' reply #1: Our responses to the reviewer's comments on selection of experimental variables and mechanistic discussions of aging processes are provided below where more detailed comments are given.

Reviewer's comment:
1. Pg. 29675: It would be more beneficial if the authors could do a calculation to report the fraction of the initial carbon that is estimated to end up down the pathways of fragmentation, functionalization, and oligomerization pathways.

Authors' reply #2: The composition of phenolic aqSOA continuously evolved during each reaction, with oligomerization, functionalization, and fragmentation pathways occurring in parallel. Therefore, the aqSOA samples analyzed in this study were always a mixture of different products. Since the AMS uses high energy electrons for ionization that tends to cause extensive fragmentation of molecules, the AMS spectra provide limited information on individual molecules. On the other hand, due to matrix effects and significant variation in ionization efficiencies of different classes of compounds (*Kebarle and Verkerk*, *2010*), the molecular information determined by nano-DESI MS is generally not quantitative. We are therefore unable to calculate the fraction of the initial carbon that ended up down an individual pathway.

Reviewer's comment:
2. Pg. 29676 lines 12-19: The sequence of these two sentences seem contradictory. In the first, the statement that oxidative fragmentation becomes more important on the timescale of atmospheric aging seems to be in conflict with the second stating that SV-OOA eventually turns into LV-OOA with atmospheric processing and transport. Do these processes really happen on different timescales? Definition of "atmospheric aging" timescale versus "atmospheric processing and transport" timescales seem warranted.

Authors' reply #3: AMS only measures particulate organics, thus the observation of less oxidized SV-OOA being converted into more oxidized LV-OOA during atmospheric aging does not conflict with what's said in the previous sentence "*… oxidative fragmentation usually becomes more important over the course of atmospheric aging, leading to the formation of increasingly more oxidized organic aerosol as well as volatile molecules that are lost from the particles*." In addition, here we talk about "atmospheric aging" and "atmospheric processing and transport" in a general sense and mean to refer to processes with a broad range of time scales. To avoid confusion, we have revised the sentence and it now reads "*Analyses of ambient aerosol datasets acquired worldwide with aerosol mass spectrometers (AMS) have indeed shown that less oxidized, semi-volatile oxygenated organic aerosol (SV-OOA) generally evolves into highly oxidized, low-volatility oxygenated organic aerosol (LV-OOA) due to atmospheric aging [Jimenez et al., 2009; Morgan et al., 2010; Ng et al., 2010].*"

Reviewer's comment:
3. Pg. 29678-29679 2.1: There is no explicit description of how the phenols are actually measured and calibrated for. Please clarify the instrumentation and methodology.

Authors' reply #4: The concentrations of phenol and 3,4-DMB are measured by a high-performance liquid chromatograph (HPLC) with a UV-visible detector. Details of the instrumentation and methodology are reported in Smith et al. [2014]. We provided this information in Sect. 2.1.

Reviewer's comment:
4. Pg.29678, line 21: Can the authors describe in more detail the selection of 3,4-DMB as a source of $^3C^*$ as oxidant source and provide a mechanistic schematic of this oxidation scheme? Considering the dimethoxy features that are similar to that of the methoxyphenol precursors used, what bearing does this have on the data interpretation? How do the authors separate oligomerization, for example between cross reactions of 3,4-DMB and the oxygenated products formed during reaction? This also seems to lead to interferences as stated in p. 29682, lines 27-29.

Authors' reply #5: 3,4-dimethoxybenzaldehyde (3,4-DMB) was selected as a source of $^3C^*$ in this study because 1) they represent non-phenolic aromatic carbonyls which are emitted in large quantities from wood combustion (*Hawthorne et al., 1992;Simoneit et al., 1999;Schauer et al., 2001*) and 2) they are readily dissolved in water, absorb light to form $^3C^*$, and catalyze the oxidation of phenols with little or no loss of the aromatic carbonyl (*Anastasio et al., 1997;Smith et al., 2014*). The mechanistic schematics for $^3C^*$ formation from 3,4-DMB are reported in Anastasio et al. (*1997*) and Smith et al. (*2014*).

The concentration of 3,4-DMB (5µM) used in this study was much lower than the initial phenol concentration (100 µM). In addition, 3,4-DMB acted mostly as a catalyzer for the oxidation of phenols and its loss rate was much slower than the oxidation of the precursors during aqueous reactions of phenols (*Smith et al., 2014*). These results indicate that 3,4-DMB was unlikely a significant source of aqSOA and that the cross-reactions between 3,4-DMB and the oxygenated products were unlikely important during this study either. Related discussions are now included in Sect 2.1.

Reviewer's comment:
5. Pg. 29680, line 18: Can the authors describe more why it is reasonable to assume peroxide groups are negligible in the given systems?

Authors' reply#6: Organic peroxides were not identified among the top 50 most abundant aqSOA products based on nano-DESI MS analysis. Thus the amount of peroxide groups are likely negligible in this study. We added this information in Sect. 2.3

Reviewer's comment:
6. Pg. 29682 line 27-pg. 29683, line 2: The extent by which 3,4-DMB is a participant in the formation of aqSOA production is very confusing. The authors describe here that ~ 70% of the original 3,4-DMB is reacted after 6 hrs and likely forms low volatility species. This means there is another reactant in this system and therefore it should be included as part of [ArOH] that is tracked over time and included in yield calculations as a precursor to aqSOA. This would likely lower the yields.

Authors' reply #7: It is true that losses of 3,4-DMB was observed, but this reaction was much slower than the oxidation of phenols. We now show the concentration profiles of 3,4-DMB as a function of illumination time for all three $^3C^*$ reactions in Fig. S1 in the supplementary information. The evolution profiles of phenol precursors are shown in Fig. 1a–1c. In addition, the initial concentration of 3,4-DMB was 20 times lower than those of the precursors. For these reasons, it is unlikely that 3,4-DMB was an important source of aqSOA in this study. Note that [ArOH] represents the concentration of the phenol; it does not include 3,4-DMB.

Reviewer's comment:
7. Pg. 29683, line 11: Considering there is no proposed "reaction mechanisms" to form phenolic aqSOA presented in the current manuscript, the authors should remove this phrasing or provide a proposed mechanism based on the reported molecular composition. In general, the

manuscript would be improved greatly if there were more mechanistic schemes presented to go along with the proposed routes of fragmentation, functionalization, and oligomerization.

Authors' reply #8: We presented phenolic aqSOA formation mechanisms in Yu et al. (*2014*) and the possible reaction pathways were indeed proposed based on the molecular information from nano-DESI analyses. In the present study, we examined the photochemical aging of aqSOA by comparing the molecular compositions of aqSOA in three filter samples collected over the course of each reaction. Since various oligomerization, functionalization, and fragmentation reactions appeared to occur in parallel, the coarse time resolution as well as the qualitative nature of the nano-DESI results only allowed us to draw relatively general conclusions about the aging pathways based on bulk changes in the relative abundances of oligomers, derivatives, and ring-opening species. In order to achieve more refined mechanistic understanding of the photochemical aging of phenolic aqSOA, we may need to conduct additional experiments and data analysis which are out of the scope of the present manuscript.

Reviewer's comment:
8. Pg. 29686, lines 6-8: Did authors do experiments with these systems stopping illumination mid-run to check if the "higher MW oligomeric compounds" fate is only photodegradation or photolysis? That is, these product signals should stabilize. Is additional oxidation with OH or $^3C^*$ *not a potential route?*

Authors' reply #9: In this study, we did not stop illumination during experiments to check the stability of the higher MW oligomeric compounds in solution. However, there are evidences that these compounds were chemically stable in absence of lights. For example, comparing aqSOA formed at reaction half-lives from online experiments and in N_2 blown-down samples, we found their AMS spectra were highly similar throughout the entire *m/z* regions. Since our N_2 blown-down procedure usually takes several hours to dry a sample, it appears that aqSOA composition, including high molecular compounds, was stable in solutions without sunlight exposure. In addition, our nano-DESI MS analyses were performed on Teflon filters collected over the courses of different reactions, suggesting that high molecular compounds are stable in dry particles as well.

Reviewer's comment:
9. Pg. 29687, lines 1-2: Authors should give more detail on the calculation of their illumination being equivalent to several days of tropospheric aging. How do authors derive an equivalent aqueous OH exposure or $^3C^*$ *exposure that is on the same basis as atmospheric aging/processing. This would best be explained in Section 2.1 as part of the methodology for choices in precursor and oxidant concentrations.*

Authors' reply #10: The comparison of the photochemical kinetics in the RPR-200 Photoreactor System with that in the ambient is discussed in George et al. [2015]. We calculated the steady-state concentrations of •OH and the rate constant for the formation of $^3C^*$ in the RPR-200 Photoreactor System. For $^3C^*$ exposure, the rate of light absorption was ~7 times faster in the RPR-200 Photoreactor System than that in the midday winter solstice sunlight in Davis. The steady-state concentration of •OH in the RPR-200 Photoreactor System under the reaction condition was ~6.5 times higher than the average fog water value normalized to Davis winter solstice sunlight. The lifetime of phenolic precursors was ~6.5 times longer in the ambient fog water than that in RPR-200 Photoreactor System. All the information mentioned above is our basis for estimating the ambient values. This information is now provided in Sect. 2.1.

Reviewer's comment:
10. Pg. 29688, lines 12-15: The authors should address the extent by which their findings that ELVOCs/oligomers formed in these experiments may be affected by methodology described in Section 2.1. For example, how do the solution concentrations of precursors chosen (are these actually reasonable for cloud droplets of biomass burning plumes?), the solution pH being adjusted to 5 (is that typical of cloud droplets), and full drying of the aerosol before sampling affect the product distributions observed?

Authors' reply #11: Previous studies indicate that the concentrations of phenols and substituted phenols measured in cloud and fog waters are typically in the range of 0.1-30 µM (*Anastasio et al., 1997;Sagebiel and Seiber, 1993*) and that in areas impacted by wood burning (e.g., in Northern California during wintertime), concentrations exceeding 100 µM is possible in fog waters (*Anastasio et al., 1997*). Thus, the concentrations of phenolic precursors used in this study are atmospherically relevant. The pH = 5 condition was also atmospheric relevant since it is within the typical pH range (3 – 5) of cloud water (*Bormann et al., 1989;Castillo et al., 1983*). This information is now provided in Sect. 2.1.

The aerosol generation process unlikely had a significant effect on the SOA product distributions either. For example, we compared the AMS spectra of flash-frozen and blown-down phenolic aqSOA samples and found that they were almost identical, indicating similar chemical compositions of the two sample types (*Yu et al., 2014*). The difference between the two sample types are that flash-frozen samples contain dissolved volatile species, low-volatility species, and unreacted precursors, while the blown-down samples are composed of only low-volatility species. Unreacted precursors and dissolved volatile species should all evaporate during the nebulization and drying process. By comparing total organic carbon concentrations observed in flash frozen and blown-down samples respectively, we estimated that dissolved volatile species accounted for only 2.7-6.6% of total carbon originally present in the reactants (*Yu et al., 2014*). These results indicate that the detection of ELVOCs/oligomers was not biased by the methodology used in this study.

Reviewer's comment:
11. Pg. 29681, line 14: Recommend changing "decomposition" to "reaction" or "decay", so as not to imply the

Authors' reply #12: We revised it accordingly.

Reviewer's comment:
12. Figure A1. Include a legend of the different experimental conditions represented by the data plotted as in Figure 6.

Authors' reply #13: Done as suggested.

Reviewer's comment:
13. Pg. 29682, line 17-18: Reword so that it does not sound like -H, -OH, -OCH3 add to a double bond (in the way that for example, OH radical, would add to a double bond). Authors probably mean that the double bonds become saturated with incorporation of these functional groups?

Authors' reply #14: We have revised the sentence to clarify this point. The sentence now reads "*The H/C values of aqSOA are also higher than those of the precursors (Fig. 1g-i), suggesting hydrogen incorporation mechanisms which include electrophilic addition of the hydroxyl radicals to the double bonds and electrophilic aromatic substitution reactions to attach more hydrogen-rich functional groups such as –OCH_3 or –$C(=O)CH_3$ to the benzene ring*".

Reviewer's comment:
14. Pg. 29682, line 22: Add in reference to Fig. 1d-f as well since refer to O/C figures here too.

Authors' reply #15: Done as suggested.

Reviewer's comment:
15. Table 3: "Top 10" should be changed to "Top 18".

Authors' reply #16: The 18 compounds listed in Table 3 represent the top 10 most abundant compounds identified in the aqSOA from each stage of photoreactions. To avoid confusion, we changed the title of Table 3 to "*Most abundant compounds identified in SYR aqSOA formed during different stages of the $^3C^*$-mediated reactions*". We also clarified in the table footnotes: "[a] *Molecular formulas and proposed structures of 18 compounds identified according to (-) nano-DESI spectra. They represent the top 10 most abundant aqSOA compounds observed at each reaction stage…*"

Reviewer's comment:
16. P. 29685, lines 15-17: Reword this sentence as it is confusing at the phrase, "…increase with aging time."

Authors' reply #17: This sentence has been revised and it now reads "*In addition, the average OS_C values of phenolic aqSOA all fall within the range observed for ambient SOA and increase with aging time, generally moving in the direction from SV-OOA toward LV-OOA.*"

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