**Atmospheric
Chemistry
and Physics
Discussions**

Interactive comment on "Insights into secondary organic aerosol formed via aqueous-phase reactions of phenolic compounds based on high resolution mass spectrometry" *by* Y. Sun et al.

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Response to the comments of referees

We thank two anonymous reviewers and Dr. Barbara Turpin for their thoughtful comments. We have revised the manuscript in response to the comments. Listed below are our itemized responses to each reviewer's comments, which are repeated in italic.

Response to Referee 1
This study by Sun et al. presents results of laboratory experiments showing the abil-

ity of phenolic compounds (phenol, guaiacol, syringol) to form high molecular-weight compounds and/or highly oxidized products. The results suggest that these species can act as precursors of secondary organic aerosol (SOA) under atmospheric conditions which is nicely supported by the comparison of mass spectra from the laboratory studies and a fog sample. Since to date the formation processes of SOA and especially the formation of highly oxidized products as identified in the current study are not well understood yet, this study represents an important new piece in our current understanding of SOA formation. The paper is well written and methodology and results are presented clearly. I have a few comments that should be considered before the publication of the manuscript.
General comments
- How well does the suggested reaction scheme agrees with prior studies that have analyzed the first-generation (reactive) intermediates of phenol oxidation into eh aqueous phase? While the authors cite a few studies, a more thorough discussion of prior studies might be useful (e.g., (Land and Ebert, 1967; Chun et al., 2000; Barzaghi and Herrmann, 2002).

The reaction mechanisms suggested in this study agree well with those reported in previous studies and the observations of the reaction products are consistent too. For example, in our phenol (C_6H_6O) experiment, we observed AMS evidence indicating the formation of catechol (1, 2-dihydroxybenzene), which was previously shown to be a product of OH radical addition to the phenol ring (Land and Ebert, 1967; Atkinson et al., 1992; Barzaghi and Herrmann, 2002; Olariu et al., 2002). We also observed guaiacol and syringol dimers, which were proposed as the products of coupling of phenoxy radicals (Kobayashi and Higashimura, 2003; Chang and Thompson, 2010).

In response to the reviewer's comments, in the revised manuscript we have expanded the discussions on mechanisms based on previous studies. In addition, Fig. 5 now shows the proposed formation mechanism of hydroxylated products, in addition to dimer and oligomer formation.

- An estimate of the time scales and efficiency of aqueous phase SOA formation from phenols in the atmosphere might be useful. This could be done by comparing the time scales of phenol losses by gas phase reactions vs SOA formation with the phenol uptake and subsequent SOA formation in the aqueous phase. (Such a comparison might have been done already in the companion paper Sun and Anastasio, in prep, 2010; if so, it should be referred to in the present paper).

We had briefly mentioned the time scale of aqueous-phase SOA formation from phenols in the first paragraph of section 3.1 in the original manuscript, but we have made this more extensive now. In the revised manuscript, background information on SOA yield and phenol reaction kinetics is given. The text reads:

"As described in Anastasio and Sun (2010), the lifetimes of phenols in atmospheric fog and cloud water drops with respect to OH radical reaction are on the order of a few hours during daytime; similarly, the illumination times required to produce the SOA products studied here in the presence of OH radical were approximately an hour (syringol), a few hours (guaiacol), and 20 hours (phenol). While all three compounds have similar rate constants with OH radical (NDRL data base), the exposure times for the three phenols vary because of large differences in the rates of direct photoreactions: while phenol has no direct photoreaction, this pathway is relatively rapid for syringol, and is intermediate for guaiacol (Anastasio and Sun, 2010). Because the lifetimes of gas-phase phenols with respect to OH radical reaction are of the same order as the aqueous phenols (i.e., hours) (Feigenbrugel et al., 2004), the relative importance of the aqueous- and gas-phase reaction pathways depends strongly on the gas-aqueous partitioning of the phenols. Based on Henry's law constants for the phenols (Sagebiel et al., 1992; Sagebiel and Seiber, 1993; Guo and Brimblecombe, 2007), under typical cold cloud conditions (5 $°C$, 0.3 g-H_2O m^{-3}), approximately 3% (guaiacol), 4% (phenol), and 15% (syringol) of these phenols will be in the aqueous phase; however, field measurements have shown that aqueous concentrations of

methoxyphenols are often a factor of three or four higher than predicted from Henry's law (Sagebiel and Seiber, 1993)."

Specific comments
p. 2917, l. 1/ 2: I suggest to reword 'hygroscopicity and radiative impacts' and state more clearly that it is rather 'aerosol mass and optical properties (absorption)' that will be affected.

We have replaced "hygroscopicity and radiative impacts" with "chemical and optical properties". In addition to affecting light absorption properties, the adding of the phenolic SOA mass may change the sizes of the particles and therefore their light scattering properties.

p. 2917, l. 22: Lim et al., and Altieri et al. did not investigate aqueous phase reactions of isoprene but reactions of their oxidation products (which are already covered by the list above).

The reviewer is right. We have revised the manuscript accordingly.

p. 2921, l. 18: Specify if 'molar yields' or 'mass yields' are meant. The latter should be even greater than unity and it is the value that is usually used in SOA model studies.

It is the "mass yields" that we reported. This point has now been clarified in the revised manuscript.

p. 2922/23: The finding that the mechanism (product distribution) is similar with and without OH radical is interesting. Is there any prior study (with similar/same species) that could suggest a possible mechanism? A reaction scheme (in more detail than in Figure 5) would be highly useful that elucidates OH and/or hv attack at the phenols.

Following the reviewer's comment, Fig. 5 has been expanded to show both mechanisms leading to phenol hydroxylation and oligomization. In addition, the discussions on mechanisms based on previous studies have been expanded in the revised manuscript. Note that the product distributions for reactions with and without OH radical only look similar on a big scale. There are important differences in carbon oxidation states as the SOA produced under light plus OH radical conditions are more oxidized than those produced under light only (Table 1), probably due to enhanced hydroxylation of the aromatic ring as well as to increased yields of carboxylic acids (e.g., oxalate) in OH radical-initiated reactions. In addition, no SOA was formed from an illuminated phenol (C_6H_6O) solution in the absence of H_2O_2 (i.e., light only). We have clarified these points in the revised manuscript.

p. 2923, l. 6 (and Figure 3, Figure S5b): How can the pH dependency of the products and yields be explained? This should be discussed in the context of these two figures.

The differences we see in AMS signal for the two different pH conditions with syringol (both in the presence of OH radical) are subtle (Fig. 3 and Fig. S5b). Coupled with the fact that we only have this pair of samples where we examined the effect of pH, we are not prepared at this point to speculate about how pH affects the chemical composition of the phenolic SOA, even though it's an interesting question. It's possible that the pH dependence of the products is related to the pH-dependent conversion of the dihydroxy aromatic radical to a phenoxyl radical (see the new version of Fig. 5), but more work would be needed to examine this.

p. 2924, l. 14: Previous studies of oxalate formation in the aqueous phase have shown that glyoxylate is its direct precursor. Could this be one of the unidentified peaks??

We did not analyze glyoxylate in this study but we suspect that it could be one of the

C2302

unidentified peaks judging from their retention times in the IC (Fig. S3). For example, according to Table S2 in Tan et al. (2009), glyoxylic acid (retention time, Rt = 9.7 min) is eluted right after the formic acid (Rt = 6.8). We see three unidentified peaks right after the formate peak in Fig. S3. One of them could be glyoxylic acid, although the IC column used in this study is different from that by Tan et al. (2009).

We mention this possibility in the revised paper: "Previous studies have shown that glyoxylate is a direct precursor of oxalate formation in the aqueous phase (Ervens et al., 2004; Lim et al., 2005). Although this compound was not analyzed in this study, it could be one of the unidentified peaks judging from their retention times in the IC (Fig. S3)."

p. 2928, l. 5/6: In previous SOA studies, there has been considerable discussion of the possibility of the reversibility of oligomers formation. Is there any hint in your experiments that the observed high SOA yields only occur at this relatively high water contents or that the products either decay back to their reactants and/or get further oxidized? In other words, should the observed high yields be regarded as maximum yields that decreases upon fog water evaporation

Our high SOA yields are measured on samples that have been blown down to dryness (see section 2.1 for the blow-down procedure) and so these yields are applicable to the case of evaporated aqueous drops. Thus the SOA products that we are measuring are stable and not subject to decomposition during the loss of water; this is consistent with the oligomers and organic acids that we have identified in the SOA. We have added a sentence at the end of the first paragraph in section 3.1 to clarify this.

Table 1: It would be useful to add the OM/OC ratios to the precursor properties.

The OM/OC ratios of the precursor compounds are included in Table 1 in the revised manuscript.

C2303

Technical comment
p. 2924, l. 17: 'Ervens' misspelled

The typo was corrected

Figure 1: Add (f) in caption

"(f)" was added in the caption of Fig. 1.

Response to Referee 2

This manuscript is a valuable contribution to our understanding of multiphase reaction pathways of abundant precursors in the atmosphere. It is mainly based on laboratory work which provides a good insight into what might happen in the atmosphere. However, in spite of some efforts by the authors, the atmospheric implications of the results remain rather vague.
1) The authors do not elaborate on the gas-to-particle distribution of their precursors. All the compounds studied are rather volatile and has small Henry-constants (see e.g. Feigenbrugel et al., Atmos. Environ. 2004). Therefore they mostly remain in the gas phase even inside clouds, and their supply from the gas phase would remain rather limited (see eg. Gelencser and Varga, ACP 2005). Bulk laboratory experiments differ from multiphase reactions in that precursors are readily available for reactions and need not be replenished from the gas phase. With this in mind, the question arises how relevant is the initial concentrations of phenolic compounds in the solutions from atmospheric standpoint?

We are including a discussion of the gas-aqueous distribution (and reactions) of the phenols in the Anastasio and Sun (2010) manuscript and so have not gone into these details in the current paper, although we have added quite a bit of text to section 3.1 to address the reviewer's questions. The Henry's law constants for the phenols we

studied range from 4.8×10^3 M atm^{-1} for guaiacol to 2.6×10^4 M atm^{-1} for syringol at 5 °C (Sagebiel and Seiber, 1993). For a cloud with a liquid water content of 0.3 g-H$_2$O m^{-3}, the percent of these phenols that are in the aqueous phase are approximately 3% and 15%, respectively, at Henry's law equilibrium; however, field studies have shown enhancements in the aqueous concentration that are often a factor of 3 or 4 higher than predicted from Henry's law (Sagebiel and Seiber, 1993). We have added this information to the paper. The replenishment of the aqueous-phase phenols by partitioning from the gas phase is not a limiting factor since this mass transport is very fast (time scale of minutes) compared to aqueous reaction (time scale of hours) (Feigenbrugel et al., 2004).

As for the final question, regarding the relevance of the initial concentration of phenols we used (100 μM), our value is probably at the upper end of fog or cloud water values; we picked 100 μM in order to be able to easily measure the SOA products gravimetrically and by HR-AMS. During wintertime in air masses influenced by wood combustion, measured fog drop concentrations of syringol and guaiacol (and a few of their derivatives) in northern California range up to approximately 30 μM (Sagebiel and Seiber, 1993). However, this study underreports total phenol concentrations because the authors didn't measure a number of the phenols emitted from wood combustion, including phenol itself (C$_6$H$_6$O) and the benzenediols (i.e., dihydroxybenzenes). We have modified the manuscript (section 2.1) to discuss these issues and to indicate that our concentration is likely an upper bound for ambient aqueous phenols.

2) Aside from the above constraint, there is a significant element in the experimental protocol that could severely distort the characteristics of the resulting SOA: bringing the reaction mixture into dryness could result in substantial losses and semi- and even less volatile SOA products that can still be relevant under atmospheric conditions.

Yes, we agree that drying the reaction mixture will lead to an underestimate of the SOA

mass. However, based on our carbon balance calculation (see next response), the loss of semivolatile material is, on average, approximately 20% of the measured SOA mass.

3) This point is manifested in the SOA carbon yield that can be deduced from the published data (from data reported in Table 1), though the authors themselves do not provide a carbon balance. For phenol, a carbon yield of about 25 % can be estimated with the notion that the initial compound cannot be detected. This raises the question that what happened to 75 % of the carbon atoms initially present in the solution: most of them had been lost as carbon-dioxide or highly volatile species already from the reaction solution; or they had been blown off upon evaporation from the Al cup? A TOC analysis of the liquid phase prior to the drying step could have helped.

We thank the reviewer for this helpful suggestion. We calculated the SOA carbon yields for our experiments and estimated that the carbon loss is on average 20%, not 75% as the reviewer suspected. Here we show one sample calculations:

a) The SOA mass yield of one syringol experiment (pH = 7, H_2O_2 added) is 108%
b) The OM/OC ratios for syringol and this non-volatile SOA are 1.61 and 2.21, respectively (see Table 1).
c) The carbon recovery in the non-volatile SOA can then be calculated as "108%/(2.21/1.61) = 79%". This means that 21% of the reacted precursor mass was lost, possibly as volatile and semivolatile species (e.g., CO, CO_2 and small organic species) during the processes of reaction and/or drying of the solution.

Detailed discussions on the SOA mass yields for different reaction systems will be given in Anastasio and Sun (2010). We have now added the following sentence in the revised manuscript: "However, based on the SOA carbon yields, we estimate that volatile and semi-volatile materials (e.g., CO, CO_2, and small organic compounds) lost during reaction and/or drying on average accounted for only 20% of the reacted phenol mass."

We also plan to analyze OC in the solution before the drying step in future experiments.

Minor comments:
1) The good match between the OM/OC data provided by the AMS and the gravimetry/TOC measurements is more than surprising given that AMS measures compounds in individual particles (with a varying degree of ionization efficiency) whereas gravimetry is carried out from blown-off bulk samples (with significant losses of semi-volatile species).

First, we need to reiterate that the AMS and gravimetric analysis were both performed on the blown-down bulk samples, which were reconstituted in purified water prior to AMS analysis (see the second paragraph of section 2.2).

Second, it is true that the SOA materials analyzed in this study are probably mixtures of many compounds with different ionization efficiencies (IE, which is defined as the number of ions detected per molecule of the parent species) in the AMS. But the OM/OC ratio of a mixture is dependent on the IE/MW ratios of individual molecules, not on their IE values; MW means molecular weight.

The IE values of organic molecules generally correlate well with the molecular weights (MW) of the molecules (Jimenez et al., 2003). This is especially true for molecules of same type, which applies to this study since the majority of the phenolic SOA compounds are likely oxygenated species. In other words, the IE/MW ratios for all the phenolic SOA species are approximately constant. Therefore, by counting AMS speciated ion signals, we are able to determine the elemental ratios and OM/OC for the SOA materials studied in this study with relatively good accuracy.

2) It is not surprising at all (despite statement in Page 2924 line 2) that SOA from phenol is more oxidized (have higher O/C ratios) yet it produces less high MW and high O/C species: the lower is the carbon number of the compounds containing oxygen, the higher will be the O/C ratio.

The O/C ratio depends on both carbon and oxygen numbers; for example, although phenol (C_6H_6O) contains the lowest carbon number in comparison to guaiacol ($C_7H_8O_2$) and syringol ($C_8H_{10}O_3$), its O/C ratio of 0.17 is the lowest too compared to 0.29 of guaiacol and 0.38 of syringol. Our conclusion is that the SOA product of phenol is overall more oxidized (i.e., higher O/C ratio) than those of guaiacol and syringol though the O/C ratio of the precursor, i.e., phenol (C_6H_6O), is lower than those of guaiacol and syringol.

Response to B. Turpin

This paper describes the results of a laboratory study of aqueous-phase photooxidation of phenolic compounds. Formation of lower volatility products, such as oligomers and oxalic acid, was observed in illuminated solutions. Reaction products exhibited similar characteristics as field aerosol samples. This paper is the first laboratory investigation of SOA formation from aromatic compounds through cloud processing. Though the paper is thorough and the results are presented clearly, we have a few comments for the authors to consider before publication.

1. Field studies suggest that the concentration of phenol in cloud/fog/rain samples is usually less than 1 micromolar (Harrison et al., 2005). Formation of oligomers is less favored at lower precursor concentration (Tan et al., 2009). The authors should justify the high precursor concentrations they used in experiments, or they should address other possible conditions in the atmosphere (i.e. aerosol water).

While the concentration of individual phenols in ambient drops is typically below 1 μM, in areas where there is significant wood combustion the total aqueous concentration of phenols can reach up to several tens of micromolar. We have added a few sentences to section 2.1 to discuss this. The text reads: "The initial solutions were composed of air-saturated Milli-Q water containing 100 μM of a single phenol, sulfuric acid or sodium borate to adjust the pH; in experiments with OH radical we also added 100 μM

H_2O_2 (Table 1). We used 100 μM of initial phenol in order to be able to readily measure the SOA products. While this concentration is much higher than typical ambient levels of individual phenols (approximately 0.1 - 1 μM; (Anastasio et al., 1997 and references therein)), it is not much higher than the upper range of total concentrations of methoxyphenols observed in winter fog drops in northern California, where measured values range up to approximately 30 μM (Sagebiel and Seiber, 1993)." In addition, the Sagebiel and Sieber study likely underreports total phenol concentrations because the authors didn't measure a number of the phenols emitted from wood combustion, including phenol itself (C_6H_6O) and the benzenediols (i.e., dihydroxybenzenes).

In our ongoing work we are examining how SOA yields and phenol loss kinetics depend upon phenol concentration.

2. Was the solution saturated with air? Oxygen is an important radical scavenger. In the proposed mechanism (Figure 5), the authors should consider the competition between radical-radical reactions and radical - O2 reactions.

Yes, all solutions were air saturated. We have added this fact to the revised manuscript (section 2.1).

3. The authors indicate that the phenol + simulated sunlight did not produce SOA in the caption of Table 1. In contrast, SOA material was observed in the phenol + OH radical experiment. However, the authors conclude that "The mass spectral patterns and ion compositions of SOA formed from the same precursor are similar for both of the experimental conditions (i.e., light only as well as light plus OH)" at pg. 2922 line 29.

We thank Dr. Turpin for pointing this out. The term "phenols" is used to refer to phenolic compounds in broad sense. It could be confused with "phenol" - the compound. To avoid this confusion, we now use "phenol (C_6H_6O)" in the caption of Table 1 to be explicit. We also revised the sentence originally on page 2922 line 29 to be clear that

we are only referring to guaiacol and syringol: "For guaiacol and syringol, the mass spectral patterns and ion compositions of SOA formed from the same precursor are similar for both of the experimental conditions (i.e., light only as well as light plus OH radical)."

As written, the importance of OH radical in the SOA formation from phenol is not clear. In general, are the yields higher in the presence of OH? Is formation faster in the presence of OH?

We are including these details in the Anastasio and Sun (2010) manuscript, so we didn't describe them in this paper. The biggest impact of OH radical is on the kinetics: the rate of phenol degradation is much faster in the presence of OH radical compared to the case of illumination only. In terms of SOA yields, compared to exposures with light and OH radical, SOA yields are approximately the same in the absence of OH radical (with the exception of phenol, which does not form SOA during illumination only). In addition, the addition of H_2O_2 as an OH radical produced more oxidized SOA, probably due to enhanced hydroxylation of the aromatic ring as well as to increased yields of carboxylic acids (e.g., oxalate) in OH radical-initiated reactions. This point is stated in the manuscript.

4. This work uses the photolysis of 100 micromolar H_2O_2 by simulated sunlight to produce OH radical. Is it possible for the authors to estimate the OH radical concentration in reactions? 100 micromolar H2O2 is indeed atmospheric relevant. However, this H_2O_2 concentration could lead to lower OH radical concentration than atmospheric conditions. Gas-to-droplet partitioning, nitrate photolysis, and Fenton chemistry are important OH radical sources in cloud droplets, while the direct photolysis of H_2O_2 could be a minor source. If the OH radical concentration is lower than cloud physics model predicted (Jacob, 1986), it could explain modest differences between experiments with and without OH radical. As OH radical can promote the formation of oxalate, it is also possible oxalate could be a more important product

under atmospheric relevant conditions.

OH radical steady-state concentrations in our SOA experiments were on the order of 10^{-15} M, which is in the range measured (in the lab) for cloud and fog drops during day time. Although we did not include all ambient sources of OH radical in our experiments, this was partially offset by the fact that we also did not include all of the sinks that are present in ambient drops.

5. Pg. 2919 line 2, the authors took the sample when half of the precursor re-acted. It would be useful if the authors can provide the approximate duration for each experiment. I guess the duration should vary for different precursors under different conditions. For example, the degradation of precursors could be faster when OH presents. This duration could also provide some insights into reaction kinetics.

The kinetic data is treated in more detail in our forthcoming manuscript (Anastasio and Sun, 2010). But in the revised manuscript, we have added a section (3.1) on background information on SOA yields and phenol reaction kinetics. The text reads:
"As described in Anastasio and Sun (2010), the lifetimes of phenols in atmospheric fog and cloud water drops with respect to OH radical reaction are on the order of a few hours during daytime; similarly, the illumination times required to produce the SOA products studied here in the presence of OH radical were approximately an hour (sy-ringol), a few hours (guaiacol), and 20 hours (phenol). While all three compounds have similar rate constants with OH radical (NDRL data base), the exposure times for the three phenols varied because of large differences in the rates of direct photoreactions: while phenol has no direct photoreaction, this pathway is relatively rapid for syringol, and is intermediate for guaiacol (Anastasio and Sun, 2010). Because the lifetimes of gas-phase phenols with respect to OH radical reaction are of the same order as the aqueous phenols (i.e., hours), the relative importance of the aqueous- and gas-phase reaction pathways depends strongly on the gas-aqueous partitioning of the phenols. Based on Henry's law constants for the phenols (Sagebiel et al., 1992; Sagebiel and

Seiber, 1993; Guo and Brimblecombe, 2007), under typical cold cloud conditions (5 °C, 0.3 g-H_2O m^{-3}), approximately 3% (guaiacol), 4% (phenol), and 15% (syringol) of these phenols will be in the aqueous phase; however, field measurements have shown that aqueous concentrations of methoxyphenols are often a factor of three or four higher than predicted from Henry's law (Sagebiel and Seiber, 1993)"

6. Pg. 2927 line 15, this conclusion might be too broad and strong. Biomass burning could produce VOCs besides phenolic compounds. This work did not investigate the aqueous-phase chemistry of other biomass burning VOCs. The analysis only suggests that cloud processing of phenolic compounds could explain the observed SOA components very well, as indicated in the next sentence.

We agree and have revised the sentence as "Overall, these results suggest that aqueous-phase processing of phenolic compounds (e.g., from biomass burning emissions) may be an important pathway for SOA formation in atmosphere."

7. How water soluble are these precursor compounds? What are their Henry's law constants and how do they compare with other SOA precursors?

The water solubilities (20 °C for phenol; 25 °C for guaiacol and syringol) are: 880 mM for phenol (http://en.wikipedia.org/wiki/Phenol), 194 mM for guaiacol (Sagebiel and Seiber, 1993), and 128 mM for syringol (Sagebiel and Seiber, 1993).

The Henry's law constants at 5 °C are: 5.4 $\times10^3$ M atm^{-1} for phenol (Guo and Brimblecombe, 2007), 4.8 $\times10^3$ M atm^{-1} for guaiacol (Sagebiel and Seiber, 1993), and 2.6 $\times$ 10^4 M atm^{-1} for syringol (Sagebiel et al., 1992). We have added the calculated aqueous fraction of each species for a typical cold cloud or fog to section 3.1.

8. Drying – first paragraph of results – was this also done with the dark control? What was found?

C2312

Yes, we dried the dark controls in the same way that we dried the illuminated solutions; we found negligible formation of SOA in the dried dark samples. These results are described in the last sentence of section 2.1.

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C2314

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