Review of Li et al. "Sensitivity of a Q-ACSM to chamber generated SOA with different oxidation states"

Description:
This manuscript describes measurements of the sensitivity of an ACSM to OA for three oxidant/VOC SOA systems produced in a mid-sized Teflon chamber using an ACSM, SMPS, DMA-APMA. Relationships of sensitivity and effective density to f44 are explored. Experiments were conducted in attempt to separate effects on sensitivity due to collection efficiency (CE) and relative ionization efficiency (RIE) of OA. Conclusions are made on trends of CE, RIE, phase state, HOA vs OOA, effective density, and oxidation state.

The manuscript is generally poorly written and makes a range of conclusions using flawed logic and speculation based on incomplete experimental evidence and lack of context from the existing literature. Due to some critical flaws (discussed below), I do not believe the manuscript is publishable in its present form. If it was published, it would "pollute" the literature with trends that are not well supported and may be due to experimental factors completely unrelated to those that are discussed.

With substantial additional work (including additional experimental evidence, analysis, and calibrations) and documentation, greatly scaled back conclusions, more thorough context, and, the dataset may eventually be publishable. Even in that case, the authors would need to present a convincing case of what parameters they are truly quantifying, including quantifying their uncertainties, and why the results are useful and better inform the analytical study of OA in the laboratory. In its current form, the manuscript is misleading and does not advance findings that are adequately constrained, new, or useful to the literature. Therefore, I strongly recommend that the paper is rejected, and the authors are encouraged to do the additional work and resubmit a much improved paper at a later time. See details below.

Critical Flaws

The paper really only quantifies an empirically-defined response factor, which is the ratio of the mass concentrations reported by the ACSM and calculated from the SMPS volume and the measured particle density. As thoroughly documented in the AMS literature, that response factor can have trends due to several effects:

$$RF = (RIE_{real} / RIE_{assumed}) * (E_{b,real} / E_{b,assumed}) * (E_{L,real} / E_{L,assumed}) * (E_{S,real} / E_{S,assumed}) * (SMPS_{calc} * SMPSvol_{real}) * (\rho_{real} / \rho_{assumed})$$

where $CE = E_b * E_L * E_S$ shows the 3 components of CE, due to particle bounce in the vaporizer, incomplete transmission by the aerodynamic lens, and non-spherical particle effects, respectively as defined by Huffman et al. (2005). It is also clear that errors in the estimation of particle volume from the SMPS number distribution and/or the density will also appear in RF.

When one is trying to characterize a widely used instrument and publishing the observed results as if they are representative of all instruments of the same type, great care needs to be taken to examine each of these terms and their uncertainties. The present paper has some speculative interpretations of RF based on RIE and E_b, even though it completely ignores all the other effects! My best guess is that E_L explains a substantial fraction, and potentially most of the trend in RF, as when the particles grow in the SOA experiments, the upper end of the distribution may fall outside of the ACSM lens transmission, thus creating an experiment and time-dependent trend on RF. It is possible that some of the other factors that are ignored in this paper may also contribute to the observed trend. For this reason the paper is not publishable.

The authors need to go back to the lab and present multiple additional pieces of information, including:

1) A detailed characterization of E_L vs. particle size for their own instrument. It is well-know that E_L varies for different instruments (and sometimes in time for a given instrument), especially at the upper end of the particle size. However, Figure 2 suggests that losses at the small end may also be a problem. These are challenging experiments, but they are doable. If the authors cannot accomplish this, they should not resubmit this paper elsewhere, as the results would be ambiguous, and it would be very misleading to report them as trends in E_b and RIE_{org} while ignoring E_L. Using the transmission curves from the literature is not acceptable, given substantial variability in E_L across instruments. See below for literature references.

2) Using the measured SMPS volume distributions, the measured density, and the measured ACSM transmission vs particle size (in d_{va} space, see DeCarlo et al., 2004), the authors can "trim" the SMPS volume to correspond to the volume actually sampled by the ACSM.

3) It does not seem acceptable to present density measurements at only one size, especially if it is the peak of the number distribution. A few points across the distribution, and importantly the peak of the volume distribution, should be included for each system.

4) Evidence of good alignment of the aerodynamic lens in the ACSM should be presented. The particle beam width depends on particle size and shape, and if the lens is not well-aligned, then there can be particle losses that are strongly size-dependent, giving rise to another source of variation of E_L that would show up in RF.

5) Evidence of calibration of the SMPS size (using PSLs) and especially, volume concentration, needs to be presented. It is typical in field studies when multiple SMPSs are present (all operated by reputable groups) that the reported volume concentrations range a factor of 4-5. That's the typical accuracy that can be expected for an SMPS that is running on a lab and that has not been thoroughly calibrated and quality-checked. Even after careful work, discrepancies often remain, see for example Figure 8 in Wiedensohler et al. (2012). There are a number of papers that specify the checks that one has to do in order to ensure quantitative SMPS measurements, and it was especially glaring that this topic was not even mentioned in the present paper. See for example Wiedensohler et al. (2018) and Stolzenburg and McMurry (2018). An intercomparison to other instruments such as additional SMPSs and OPCs (for example UHSAS) would be useful to gain confidence in the measurements. Evidence of quantitative response of the CPC, ideally by comparison to an electrometer, but otherwise by comparison to several other CPCs (across the size range) is also needed.

6) Evidence of accurate density measurements using known materials, and across the particle size range contributing to the measurements shown, needs to be presented.

7) The uncertainty in each term of the equation for RF, as well as in 1-6 above, needs to be quantified, and the uncertainty needs to be propagated. Only then we can know if any of the reported trends for (for example) E_b have any meaning.

Also it should be noted that for internally mixed particles such as those generated here E_b will be a property of the particle that is influenced by its sulfate and OA content. So it is non-sensical to write $E_{b,SO4}$ and $E_{b,OA}$ (as the authors do in their notation) as if those were different quantities in the seeded experiments, or as if they were a property of the species and not the mixture.

Moreover, it is not standard practice to use ACSM or AMS measurements for quantitative yield experiments for chamber experiments in the first place, due to the complexities outlined above. Rather, researchers typically have used SMPS volume together with density estimates (or comparisons with filters as well as other methods), and OA/SO_4 ratios, to quantify yields. Jimenez et al. 2016 (which is referenced in this manuscript) states "Finally, we reiterate the need for direct calibration in laboratory experiments utilizing specific organic compounds or mixtures." So, this issue is not new and nor do the authors offer an advance in addressing the issue.

As discussed on that reference, the trends of AMS response observed for field and laboratory experiments are often different, due to the wider range of particle materials that can be made in the lab. Lab experiments typically have trouble replicating the OA composition observed in the atmosphere. Therefore, no implication should be made that the results from a lab study apply to field measurements. Any statements about the performance of the AMS / ACSM in field

measurements should be derived from the analysis of field measurements, of which there is an extensive record in the published literature. Comparisons of AMS with SMPS in field studies are not consistent with the trends shown here, see for example Jimenez et al. (2016) and references therein.

Other Important Comments:

As was extensively detailed by Reviewer 2, the manuscript is full of grammatical errors, confusing text, logical flaws and overstatements of conclusions. I agree with the comments of Rev. 2, and will thus focus on the most important additional points, and a few similar points with additional context or emphasis. However, the list is not comprehensive as it would be too time consuming to point out all the issues and any effort to revise and resubmit the paper should go beyond just the issues detailed by the reviewers. A detailed review of the Abstract is below, followed by other issues organized by topic.

Abstract:
L21: "comprehensively" is an extreme overstatement.

L25/L31/L32: "oxidation states" were not measured in this study.

L27: "exact mass" doesn't make sense.

L35: The cause of decreasing RF was not "pinpointed" or even significantly constrained.

L36: The CE was not determined quantitatively.

L37-39: "Our experiment results along with previous literature reports strongly implied that as the SOA oxidation state increases, SOA will transform gradually from a liquid state (CE » 1) into a solid (or glassy) state with a CE of 0.2~0.5." This is a gross overstatement of what the evidence presented in this manuscript supports. The trends observed may have been any combination of lens transmission effects, E_b effects of mixed dry ammonium sulfate / OA particles, OA RIE effects, OA CE effects, or variable errors on the SMPS or CPC used for comparison. Neither CE effects nor phase state effects were isolated in this study.

L39-41: "Meanwhile, the RIE of OA decreased substantially when SOA transformed from hydrocarbon-like OA (HOA) into more oxygenated OA (OOA) and may further decrease as O/C continued to increase." Like with E_b, the effects of RIE of OA were NOT isolated in this study. Moreover, the results presented here have no bearing on HOA since only chamber SOA was studied. Also, no data on O/C was presented here (inferring O/C from f44 for chamber SOA is not justified – see details below)

L41-44: "Our results indicated that the current Q-ACSM calibration procedure using a constant RIE may lead to somewhat underestimation of more oxidized OOA but overestimation of less oxidized HOA, i.e., a variable RIE shall be applied, most likely as a function of the SOA oxidation state." As stated above (and further explained below), RIE effects were not isolated.

More Detail on Lens Transmission Effects:
The ACSM lens transmission declines to zero for both small and large particles. In particular the upper size cut depends on the specific lens being used (Hu et al., 2017), and can even change in time after an instrument is moved to a different location. The transmission also depends strongly on the pressure inside the AMS aerodynamic lens, which can decline dramatically if the pressure in the lens is reduced (e.g. Bahreini et al., 2003) , for example due to a partially clogged critical orifice, or to just being set at the wrong pressure by using an improperly sized critical orifice. This value should be documented here for all experiments, and care should be taken to ensure that all SOA experiments are done with the same lens pressure used in characterizing E_l vs. size. The trends of the response factor shown here may be significantly influenced, or may even be dominated, by differences in the particle size ranges analyzed by the ACSM and SMPS. It is not acceptable to publish a paper that attempts to quantify CE and RIE and the ACSM without a detailed and careful quantitative calibration of the transmission of the ACSM vs size, and applying that transmission curve to all SMPS measurements. Using transmission curves from the literature is not acceptable, as there is considerable variation in those, and it is not known which literature curve (if any) may apply to the ACSM used here. I would expect to see several figures and pages of text devoted to calibrating the AMS size transmission before any conclusions about CE or RIE could be credible.

No volume size distributions are shown here for the different experiments, so it is impossible for the reader to even estimate the extent to which differences in particle size transmission could be a problem here.

As Reviewer 2 points out, the decreasing trend in RF vs f44 (Fig. 5a) could be simply due to an artifact of increasing particle size extending beyond the upper size limit of 100% aerodynamic lens transmission efficiency. While a well-performing lens can transmit particles with 100% efficiency up to ~550 nm vacuum aerodynamic diameter (~400-450 nm mobility diameter; Knote et al., 2011; Hu et al., 2017), lenses with reduced transmission at larger sizes is not uncommon (Liu et al., 2007; Takegawa et al., 2009; Hu et al., 2017; Campuzano-Jost et al. 2017), resulting in large losses for sizes as small as 450 nm vacuum aerodynamic diameter (~300-350 nm mobility diameter). Such effect may also play a role in the results presented in Fig. 6 and Fig. 7, as suggested by Rev. 2. Only then should the trends in RF can be considered reliable.

Collection Efficiency and Relative Ionization Efficiency Sections:
The sections on CE (sect. 3.3) and RIE (sect. 3.4) are extremely under-supported, misleading, and highly speculative. Given the experiments conducted and methods used, separation of the effects of CE and RIE does not seem possible. This applies to both relative trends and absolute values. Those sections should be completely eliminated unless unambiguous new evidence can be provided. The RIE section provides no experimental evidence and only an incomplete discussion of the published literature on this topic. The CE section discusses experiments where SOA was coated on top of dry ammonium sulfate seed and the "Response Factor (RF)" was calculated. However interpretation of the results are flawed. The RIE of sulfate was not calibrated, even though it may vary substantially (e.g., Zhang et al., 2017). Despite the lack of sulfate calibration, the authors assume an RIE of sulfate and calculate a CE of ammonium sulfate that depends proportionally on that value. While it is stated that the absolute value is not important but rather just trends matter, in the same paragraph the argument that when a lot of SOA is added to the ammonium sulfate seed, a CE of unity is observed which supports that the aerosol is now in a liquid state. Not only does that line of logic require that ammonium sulfate is calibrated, it also requires that the RIE of the OA is known. So, generally the main figure of that section (Fig. 7) is very problematic since the RIE of SO4 and OA are not known. Also, note that calculation of a CE for dry ammonium sulfate of 0.28 in this work does not narrow the range of uncertainty from lack of calibration for ammonium sulfate RIE since a range of a factor of 2 (0.2-0.4) has been observed for dry ammonium sulfate (Hu et al., 2017 and references therein). Moreover, equating the trends in the RF to changes in the RIE of the SOA is not necessarily justified since the effects may be the "results of a combination of a AS core and organic shell" as the authors state in Line 305. Thus, any trends in Fig. 7 may be dominated by the SOA coating thicknesses. However, changes in CE and RIE of the SOA may also be factors. Consequently, any quantitative conclusions that can be drawn about CE of OA is extremely under-constrained here. In order to separate RIE and CE effects, a direct, unambiguous measurement of CE needs to be made (e.g., see Xu et al., 2018).

Incomplete Literature Context / Major Omissions:
Several instances stand out as having a major lack of literature review/support or simply omitting fundamental references on topics discussed. For example the Kuwata et al. (AS&T 2012) paper which formulates the relationship between SOA density and O/C and H/C is never referenced or discussed (or any other paper on such relationships). A reader might get the impression that this manuscript was the first to show a correlation between SOA density and oxidation. Also, the paper Xu et al. (AS&T 2018), which extensively investigates RIE of OA for a large range of compounds and SOA (unambiguously separating CE and RIE), is completely missing. Another example is the section "Effects of f44" (3.2), where only a few peripheral references are provided in discussion of these results; whereas, there is a large body of work discussing trends in f's and elemental ratios for SOA chamber studies. Even the papers introducing the f44 vs f43

diagram (Ng et al., 2010; 2011) are not referenced! Without such context, it is impossible to determine if anything new was learned from the present studies.

<u>f44, O/C, Oxidation State:</u>
The use of f44 is used throughout the manuscript to be equivalent to O/C and "oxidation state," which is an unjustified step for chamber data. The authors state in Lines 200-203: "Since *m/z* 44 signal basically reflected the oxygen content in OA, the O/C ratio can be deduced directly from f44, both of which have been widely used to represent the oxidation state of OA (Canagaratna et al., 2015)." While this may be true for ambient OA, this does not necessarily apply to chamber data. Canagaratna et al. (2015) (Fig. 8a) shows that relationship for ambient, standards, and laboratory produced SOA (a-pinene, toluene, isoprene, sesquiterpenes). Ambient OA and some standards follow a well-established relationship. However, taken together the chamber SOA shows no correlation, which is not surprising since the presence of acids (which produce CO2+ in the AMS/ACSM) and other oxidized functional groups would not necessarily be expected to track for specific oxidant/VOC systems. Only sesquiterpenes and a-pinene fall on the ambient parameterization line and are clustered within a narrow region.

Moreover, if the RIEs of OA indeed vary a lot for the systems studied then it would seem very possible that changes in f44 could be controlled as much by changes in the overall OA sensitivity (the denominator of f44 = *m/z* 44 / OA) as by the actual relative abundance of *m/z* 44 in the SOA. CO_2^+ in the AMS is expected to be largely formed from the decarboxylation of organic acid groups on the vaporizer, forming CO_2 gas, which would have a constant RIE, and thus would not track changes in overall OA RIE.

Therefore, the use of the terms O/C and oxidation state should be avoided beyond an explanation that it might be roughly indicative of oxygen content and possibly of acid content.

<u>Clarity in Scope:</u>
All conclusions and interpretation should be scaled back to clearly state to apply to only RF (CE x RIE), effective density, f44 (not O/C or oxidation state), three oxidant/VOC SOA systems, dry conditions, and laboratory SOA. No extrapolation to ambient OA should be given or implied, since no evidence is presented and current evidence in literature suggests that such an extrapolation is not warranted – see e.g., Xu et al., AS&T 2018, Jimenez et al., AS&T 2016.

<u>Figure 8:</u>
This figure should be removed: no axis are given, and the data for several of the trends have not been derived in this paper, so this is really a cartoon from the author's imagination, which has no place in a paper. If the authors do all the extra work to make this a serious study, then the figure should be included with quantitative axes and with error bars for both X and Y.

In addition, the trend in density is not new and was published at least half a decade ago (Kuwata et al., 2012). The trends in CE and RIE of OA are not demonstrated in this paper and are especially misleading. The trends in RF may be dominated by lens transmission effects, and may have a contribution from the instrument that is being compared with, and can only be supported with further evidence. HOA is not studied here at all.

Also note that a published study (Pajunoja et al., 2016) reported the opposite trend for bounce vs. O/C, in that study bounce *decreased* as O/C increased.

Other Miscellaneous Comments (substantial and minor):
- L75: Real time composition measurements is the key development that AMS made possible/routine (not sized-resolved).
- L93-103: In this paragraph where CE and other effects on AMS sensitivity are introduced, it would be useful to instead use the more precise terms of each of the components of CE: lens, shape-related, and bounce effects (CE = $E_L * E_s * E_b$; Huffman et al., AS&T 2005).
- L101-103: This statement about the high uncertainty in CE and IE is unclear whether it applies to ambient or laboratory data and should be clarified (since it is not true for ambient data, per the Jimenez et al. 2016 reference cited).
- L124-125. AMS light scattering does not employ laser-based vaporization.
- L126-130. This statement claiming that the increased fragmentation in the capture vaporizer will "highly complicate the AMS quantification process" does not reflect conclusions of the Hu et al., 2017 paper cited here, nor any of the several other papers investigating the capture vaporizer. For example Hu et al. (2018a) state that "tracer ion marker fractions, which are used to characterize the impact of different sources are still present and usable in the CV." and Hu et al. (2018b) state "Consistent time series of positive matrix factorization (PMF) factors and their fractions of total OA were found across the CV and SV in the three very different ambient data sets ranging from biogenic- to anthropogenic-dominated, indicating limited loss of source determination information despite the increased fragmentation." It appears that this statement was added to dismiss this method as a practical way to reduce CE uncertainties. In fact, in the recent paper by Xu et al., AS&T, 2018, it was shown that the capture vaporizer can be used as a method to isolate changes in OA RIE. Given the other limitations of the ACSM, the authors may want to consider performing a similar study using a CV, which would make E_b~1 and thus substantially simplify the interpretation of the trends on RF.
- L162: "self-synthesized" is confusing. Consider alternative phrasing such as "synthesized in house".
- L177: "*m/z* calibration", not "mass calibration"
- L195: add "a" before "strong"
- L235-238: "Especially, laboratory studies have shown that the ozonolysis of α-pinene can lead to considerably higher SOA yield than that from OH initiated reactions (Yao et al., 2014). Therefore, in this work we were focusing on the isoprene-OH and α-pinene-O3 reactions only".

This does not seem like a good reason to omit these data from the analysis. OH oxidation is an important loss for a-pinene in the atmosphere and makes substantial SOA. This data should be shown and discussed. Also, Yao et al. should not be the primary reference here.
- L243: grammar.
- L291: grammar
- L293: grammar

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