

Reviewer #1

Review of 'Phase partitioning and volatility of secondary organic aerosol components formed from α -pinene ozonolysis and OH oxidation: the importance of accretion products and other low volatility compounds'

This study utilized the Filter Inlet for Gases and AEROSols (FIGAERO), coupled with the high resolution time-of-flight chemical ionization mass spectrometer (HR-ToFCIMS), to explore the chemical composition and volatility of SOA generated from the α -pinene ozonolysis and OH oxidation in a glass chamber. This novel particle sampling and analysis technique showed its strong power in the molecular characterization of particle phase organic components: in addition to the previously identified products i.e., small carboxylic acids mostly, a spectrum of high molecular weight products, with assigned molecular formulas, is identified as well via a thermal desorption program. Overall, this study is a great complement to previous publications that employ LC/MS as a common analytical technique, and together, sheds light on the question of the SOA composition. Regardless of the fact that chemical structures of identified species are still unknown, the current observations are of significant value. A concern, however, is that the authors' primary conclusions regarding the mass fraction of acyl-containing compounds in the particle phase and the agreement between measured and modeled gas-particle equilibrium partitioning depends, at least to a certain degree, on a variety of factors in the experimental conditions, data processing protocols, and model parameterization. Uncertainties arising from these processes can potentially mask the current conclusions. To verify the reliability and generalizability of these results, systematic sensitivity tests in both experiments and simulations could be carried out, which might be beyond the scope of the present study. Nevertheless, I would suggest the authors soften the conclusions by considering and discussing the factors that might bias their conclusions.

Major comments:

1. One of the conclusions is that the particulate organic compounds detected by FIGAERO HR-ToF-CIMS can explain 20-50% of organic aerosol mass measured by AMS. This conclusion is drawn by 1) applying an instrument sensitivity of formic acid uniformly to the entire spectrum to obtain the mass concentration of each ion in the particle phase, and 2) measuring the overall organic mass by AMS. Uncertainties need to be given to constrain the calculated fraction, 20-50%, of identified products in α -pinene derived SOA. Specifically, what is the collection efficiency of the ammonium sulfate seed particles parameterized in AMS? Is it the default value 0.5 that is used for field environments? As the authors note in the experimental methods section, effloresced AS particles were used in the chamber experiments at RH varying from 35% to 65%. Could dry particles exhibit different bounce behavior than hydrated particles when impacting the heater? Can recent studies that focus on AMS collection efficiencies in chamber and field measurements help constrain the CE value used here (Matthew et al., AST, 2008; Middlebrook et al., AST, 2012)? Another way is to derive the total organic mass concentration from the SPMS measured total particle volume, together with the AMS measured org/sulfate ratio. Would that give a confirmatory result?

The composition and abundance of particles in JPAC were determined on-line using an aerosol mass spectrometer in conjunction with a SMPS. As the reviewer points out, the efficiency at which the AMS collects and vaporizes pure ammonium sulfate particles can differ from that for particles coated with organic. Therefore we used the SMPS data in addition to the chemical speciation from the AMS data to acquire time series for SOA and ammonium sulfate. The AMS organic and ammonium sulfate signals were used to calculate a particle density, with which we converted the total particle volume measured by the SMPS to total particle mass (in an identical fashion to (Ehn et al., 2014)).

In the manuscript, we have added the following text to further clarify that the SMPS and AMS were used together to constrain the SOA and sulfate mass loadings:

"We used the composition measured from the AMS and total particle volume from a SMPS to calculate particle mass of sulfate and SOA present in the chamber."

2. The authors suggest that equilibrium gas-particle partitioning is applicable to chamber conditions by showing in Figure 4 decent agreement in the particle-phase fractions of pinic acid, pinonic acid, and norpinic acid between measurements and predictions. There are too many uncertainties in both modeling and measurements to draw this conclusion very firmly. From the modeling perspective, the prediction of vapor pressure from different estimation methods varies by orders of magnitude (see comparison in Compernelle et al., ACP, 2011). This introduces large uncertainties in the predicted particle-phase fraction of compounds (F_p). Next, what measurement is used here to yield COA? Do the AMS measurements agree with those from SMPS? These issues need to be clarified. For the experimental measurements, my impression is that the compound particle-phase fraction (F_p) is calculated directly from the gas/particle-phase measurements. How do particle and vapor wall losses influence the calculated F_p ? The turbulence mixing status is influential in determining the wall loss rate for both particles and vapors. As in Ehn et al. Nature, (2014), the wall loss rate of ELVOC is predicted as $\sim 0.011 \text{ s}^{-1}$ in an actively mixed reactor, for which that the lifetime of ELVOC with respect to wall loss is only 1.5 min, much less than the residence time of 50 min in the glass chamber.

We have attempted to clarify this section. We use a combination of the SMPS and AMS to determine the OA in the particles (see above response). Our goal was not to test the accuracy of literature vapor pressure measurements or parameterizations. Rather, we wished to test whether there was a linear relationship expected between measured and predicted F_p in response to OA as would be predicted from equilibrium partitioning theory. Given that vapor pressures for many atmospherically relevant compounds are unknown, it is useful to “test” a combination of the group contribution approach for estimating vapor pressures and partitioning based on equilibrium partitioning theory. If the model and observations are linearly related for changes only in OA, but with a constant bias, that result would be consistent with equilibrium partitioning and imply that a single adjustment of vapor pressure (or activity coefficient) is required.

However, if, as for some compounds studied, there is significant scatter in the modeled vs measured F_p , and that scatter correlates with chemical conditions in the chamber, that result implies either that equilibrium partitioning is not valid or that the measurements used to test partitioning are flawed. We show that by using the measured thermogram, which implies thermal decomposition at higher temperatures influences the detection of more volatile compounds used in the F_p tests (Fig 4). That is, after correcting for the multi-modal desorption profiles to account for the role of thermal decomposition, then measured and modeled F_p are more linearly related (regardless of what vapor pressure is used) therefore suggesting that partitioning theory describes the loss of vapor in the gas phase and increasing particle phase fraction during changes in the chamber.

The absolute agreement (how near to the 1:1 line the data fall) is well within the uncertainty in vapor pressure estimation, as the reviewer notes, but our measurements, which are independent constraints of the gas to particle partitioning, actually constrain the effective (activity coefficient adjusted) vapor pressures of these compounds.

No doubt, wall loss is a significant sink of organics from both the gas and the particle phase in this chamber (as we noted in Ehn, et al 2014). However, the impacts of wall loss on the above analysis is limited, and would be most important if we attempted to extract the absolute saturation vapor pressure (which is not of interest in this paper) of the compounds. Running the chamber in continuous flow mode and using only data when reaction conditions were constant (only seed concentrations were changed), we measured the combined equilibrium of gases between particles and walls. The relative fraction of material in the gas phase that is lost likely depends on the wall conditions, and the relative C^ of each compound but almost certainly biases gas phase concentrations of ELVOC like compounds low in the gas phase.*

Minor comments:

1. Page 4472, Line 10-15: As shown in the calibration experiments, benzoyl peroxide was detected as the benzoate anion, and less efficiently, benzoic acid. A recent study also found the transformation of isoprene-derived peroxides (ISOPOOH) to carbonyls (MACR and MVK) in GC and PTR-MS (Rivera-Rios et al., GRL, 2014). If the decomposition of O-O bounds occurs during the thermal desorption process, how would the authors estimate the fraction of peroxide functionality that is originally in the particles? Another related

question, for each ion detected in the particle phase, is there a corresponding ion with the same molecular assignment detected in the gas phase?

We do not attempt to estimate the fraction of peroxide functionality in the particles, in part because of thermal decomposition of O-O bonds, and also because as we illustrate, acetate ionization appears to detect peroxy acids and carboxylic acids with similar sensitivity. The only way that peroxide functionality is quantified in this approach, is if the decomposition (or direct desorption) yields a peroxy acid or carboxylic acid. Acetate ionization is relatively insensitive to non acyl keto groups (ketones, aldehydes, esters) and non-acyl peroxides.

Our high-resolution fitting is applied to data from both phases (gas and particle). In general, a signal is measured for the vast majority of identified compounds in both phases, but the extent varies greatly. During a gas-phase measurement, the signal for many high-mass compounds is low and not statistically above the background. For low-mass compounds, these are either only in the gas-phase measurement or appear at the highest desorption temperatures (from decomposition presumably). There are then many in between which have detectable presence in both phases see (Lopez-Hilfiker et al., 2014) for details.

2. Page 4473, Section 3.1: The initial concentrations of α -pinene and O₃, and the amount of α -pinene reacting with O₃ and OH radical at the end of the experiment need to be given. Also, could the authors estimate the overall OH exposure in the chamber? If the reaction timescale is the same as the residence time, 50 min, the existence of a substantial amount of high molecular weight organic products suggests an intense reaction pathway either in the gas phase or the particle phase.

α -Pinene concentrations going into the chamber were measured by a quadrupole PTR-MS to be 27 ppb and at the exit of the chamber the α -pinene concentration under ozonolysis was 10 ppb (17 ppb reacted) and 5 ppb (22 ppb reacted) during OH oxidation.

3. Page 4482, Line 6-7: Recent evidence has shown that α -pinene-SOA might be semisolid. Might be helpful to cite here (e.g., Renbaum-Wolff et al., PNAS, 2013).

We have added this citation to the manuscript in the discussion section.

Reviewer #2

General Comments: This paper describes a set of experiments that investigated the gas-particle partitioning and volatility of SOA formed from the reactions of α -pinene with O₃ and OH radicals. The experiments were conducted in glass and Teflon reaction vessels and the gas and particle composition and SOA volatility were analyzed using a Filter Inlet for Gases and Aerosol coupled to a high resolution chemical ionization mass spectrometer (FIGAERO HR-ToF-CIMS) and also an Aerosol Mass Spectrometer (AMS). Acetate ions were used for ionization, which is fairly specific for carboxylic acids and similar compounds. The results indicate that a large fraction of the SOA is composed of carboxylic acids and similar compounds, and that the SOA contains both monomers and oligomers, with the latter decomposing at high temperatures to form the detected monomers. When effects of oligomers on observed gas-particle partitioning are accounted for in a gas-particle partitioning model the agreement between the measurements and model are significantly improved. The results provide important new insights into SOA composition, particle phase reactions, SOA volatility, and gas-particle partitioning. The paper should certainly be published in ACP, although I have a few comments and suggestions the authors should first consider.

Specific Comments:

1. Page 4470, lines 19–24: Do the authors have any idea about wall losses of gaseous organic compounds in the stainless steel line or Teflon line?"

Wall losses of gases during transit from the chamber to the instrument are likely significant problem for many compounds. As noted above however, while the absolute abundance was likely perturbed by chamber and sampling surfaces (as in any chamber measurement), most of our conclusions do not depend strongly upon absolute abundance.

We chose to use a metal inlet tube for the particle phase inlet to ensure that small particles were not lost due to surface charging which can vary in an unpredictable way when using non-conductive tubing like Teflon. Semi-volatile compounds present in the gas and particle phases are potentially lost on the stainless walls during transit to the filter which would likely favor evaporation from the particles in an effort to re-achieve equilibrium with the gas phase, and would bias our particle phase semi-volatile compounds (ex. pinic, pinonic, nor-pinic) low. However, as shown in the manuscript, we measure more of these compounds in the particle phase than expected based on literature estimates of equilibrium vapor pressures. Keeping the inlet short (20 cm), by placing our instrument directly under the chamber (inside the temperature controlled chamber housing) helped to lower the effect of sampling biases – though the absolute wall loss as a function of volatility was not measured.

We should note however, that while it could be that semi-volatile compounds are stripped from the particle phase by stainless steel tubing, this would also be the case for the AMS instrument and the SMPS, both of which sample through small diameter stainless steel or copper tubing. That is, such sampling biases are a general problem, not one unique to our instrument. We have also sampled the UW 0.7 m³ Teflon bag containing α -pinene ozonolysis SOA through a Teflon tube, and did not find dramatically different volatility distributions using the FIGAERO. Thus, we think this effect for our sampling conditions was minor, but could be significant with very long, unconditioned sampling lines.

In the Teflon gas phase inlet 12cm compounds like formic and pinonic acids, which are higher volatility, are passed efficiently based on laboratory calibrations and response tests, however larger molecular weight compounds that are more likely to be irreversibly lost to inlet surfaces are likely underestimated.

2. Page 4472, lines 15–20: I'm a little confused by this discussion about detection of compounds containing acyl groups. Are the authors saying that all compounds with an acyl group: carboxylic acids, peroxyacids, diacylperoxides, esters, aldehydes, ketones, acylperoxynitrates, etc., have detection sensitivities approximately similar to formic acid? This could be stated more clearly by saying more specifically which compounds they are talking about. Many readers may not know that acyl group-containing compounds is a very large class of compounds.

We will clarify in the text, we've yet to find a single functional group name to classify the group of compounds acetate detects as carboxylate anions. To date we have calibrated the acetate ionization approach to a suite of carboxylic acids, peroxy-acids, and diacylperoxides, all of which were detected at corresponding carboxylate anions with similar high sensitivity. Acetate ionization appears to be rather insensitive, at least in terms of forming a deprotonated anion, to a suite of alcohols, hydroxy ketones, aldehydes, and esters. We have not tested acyl peroxy nitrates. We therefore wanted to be conservative and make it clear that we could not conclude all signals were carboxylic acids given the current interest in carboxylic acids as a component of SOA (Heald et al., 2010).

3. General comment beginning with Section 3.2: I encourage the authors to read the paper by Docherty et al., ES&T,39, 4049–4059 (2005). They conducted a very similar study on SOA formed from monoterpene ozonolysis using temperature-programmed thermal desorption with electron ionization (TPTD-EI) and reached many of the same conclusions as this study regarding monomers and oligomers in SOA and the volatility distribution, and also showed that much of the SOA was organic peroxides.

We have added this citation to section 3.2:

“This idea is consistent with previous work using a TPTD which also investigated the multimodal nature of thermally desorbed SOA from the reaction of α -pinene and ozone (Docherty et al., 2005) which concluded a large fraction of the SOA formed consisted of organic peroxides.”

4. General comment beginning with Section 3.2: The discussion regarding oligomer decomposition to form monomers could probably be explained using a plausible mechanism based on what is known about the

chemistry of these compounds. To my knowledge, the only reversible bimolecular reactions that are likely to form carboxylic acids involve the decomposition of esters to a carboxylic acid + alcohol + water. Although carboxylic acids that contain an aldehyde group can cyclize to form a hydroxyfuranone, which can then ring-open to form the carboxylic acid-aldehyde, the corresponding bimolecular reactions are unlikely due to entropy effects. Considering, however, that ester decomposition involves acid catalyzed hydration, and that no strong acid is present in the system and water is probably lost from the particles during sampling and in the early stages of heating, it seems unlikely that the carboxylic acids observed at high desorption temperatures have come from ester decomposition. A more likely scenario is that the SOA contains peroxy-pinonic acid, peroxy-norpinonic acid, peroxy-pinonic acid, and peroxy-norpinonic acid, and that these peroxide compounds react in the particles with pinonaldehyde (a major reaction product) to form the corresponding low volatility acylperoxyhemiacetals, which thermally decompose upon heating to pinic acid, norpinonic acid, pinonic acid, norpinonic acid, and pinonic acid. See general mechanism in Ziemann and Atkinson (2012) in reference list and Docherty et al. (2005) referenced above, which draws from the alpha-pinene ozonolysis mechanism in Jenkin, ACP, 4, 1741-1757 (2004). The authors may also want to look at the paper by Strollo and Ziemann, Atmos. Env., 77, 534-543 (2013), which uses TPTD-EI to demonstrate the effects of reversible and irreversible oligomerization reactions on SOA formation and volatility for a different system.

We agree with the reviewer that this is a plausible explanation for our observations, and we already do mention this mechanism in Section 4 as a possible source of low volatility compounds. However based on gas-phase models of α -pinene ozonolysis under the conditions of the JPAC experiment, very little peroxy acid is predicted to be formed, given the expected $RO_2/HO_2 > 1000$. Nor do we have definitive observational evidence with this data set that peroxy acids are actually present. Therefore, while we have added additional references to the above mentioned papers, we refrain from drawing a conclusion as to the relative importance of acyl-peroxy-hemi-acetals in the SOA formed.

Technical Comments:

1. Page 4465, line 16: Probably should be “high molecular weight organics” or “large organic molecules”.

Changed accordingly.

Reviewer #3

General comments: This manuscript discusses and interprets measurements from a Filter Inlet for Gases and AEROSols (FIGAERO) coupled to a High Resolution Time-of-Flight Chemical Ionization Mass Spectrometer (HR-ToF-CIMS). Measurements were taken of gas and particle-phase species formed during ozonolysis and OH oxidation of alpha-pinene in a glass chamber and a Teflon chamber. The FIGAERO HR-ToF-CIMS is capable of measuring gas and particle-phase species – gas species are measured when bypassing the FIGAERO, and particle-phase species are measured by first collecting particles on a filter and then slowly heating the filter and measuring the vaporized species. The acetate ion was used for chemical ionization which is primarily sensitive to organic acids and similar species. Particulate matter concentrations were also measured using an aerosol mass spectrometer, and total organic aerosol mass loadings were compared to the measurements from the FIGAERO HR-ToF-CIMS. Desorption profiles (signal versus filter temperature) were used to estimate species’ vapor pressure, and the authors conclude that 50% or more of the organic aerosol has saturation mass concentrations below 10^{-5} micrograms/m³, implying that fraction should be treated as effectively nonvolatile. The FIGAERO HR-ToF-CIMS allows for semi-continuous analysis of gas and particle phase species and as such presents a significant asset to the research community’s measurement capabilities. This is, to the best of my knowledge, the first manuscript quantifying organic particulate matter volatility from FIGAERO HR-ToF-CIMS data, and it is hence of great interest to the research community. The manuscript is well written and should be published in ACP after all reviewers’ comments have been addressed. I have a few concerns about quantification of organic species as detailed below.

Specific comments:

1. Considering that the authors compare particle-phase measurements from the FIGAERO HR-ToF-CIMS to organic aerosol measurements from the AMS, the AMS data collection and analysis should be described

in more detail. AMS measurements and data analysis are subject to significant uncertainty, for example due to particle bounce at the vaporizer resulting in a non-unity collection efficiency, which could even change over the course of this experiment as the ratio of inorganic seed aerosol to organic aerosol changes.

This has been addressed in responses to reviewer #1 above.

2. In order to quantify FIGAERO HR-ToF-CIMS species concentrations the author use the instrument's sensitivity to formic acid and apply that sensitivity factor to all measured species (hundreds of species). They argue that "this assumption is reasonable within a factor of 1.5" based on calibration data shown in Fig. 1. I am not convinced for several reasons: a) The sensitivity factors in Fig. 1 seem to span 2 orders of magnitude, so it is not clear to me why this assumption is thought to be reasonable within a factor of 1.5 b) The sensitivity factors in Fig. 1 are for carboxylic acids only. As the authors note the FIGAERO HR-ToF-CIMS with the acetate reagent ion is sensitive to all "acyl compounds" which includes but is not limited to carboxylic acids. Thus, it seems appropriate to also measure sensitivity of the instrument to non-acid compounds and compare to the sensitivity of the acids before determining / estimating a general sensitivity factor.

We have calibrated to a series of compounds with different functionalities (as discussed in the manuscript and shown in Figure 1 – not just acids). We state that acetate ionization does not only detect acids, but also other functional groups, so as to not mislead the reader to think that all detected compounds have been unambiguously speciated as acids.

However, there are two more important points regarding calibration and our conclusions that should be stressed. First, after calibrating to dozens of compounds spanning a large molecular weight range, we find that the sensitivity to multi-functional carboxylic acids is no higher than that to formic acid. That such compounds converge to a similar sensitivity is not surprising. Differences are likely related to mass-dependent ion transmission and ToF duty cycle, which are almost certainly captured by the factor of 1.5 uncertainty.

Second, while we have determined that acetate ionization, which initially was thought to be mostly selective towards carboxylic acids, in fact is sensitive to a larger suite of compounds, its sensitivity to carboxylic acids is uniformly higher than to any other functionalities expected in this mixture and that we have tested, e.g. ketones, alcohols, esters, simple hydroperoxides. The exceptions, as noted in the paper are peroxy acids and benzoyl peroxide (a di-acyl peroxide) which are detected with similar sensitivities as a suite of mono, di, and multi-functional carboxylic acids. By applying a sensitivity that is far higher than the actual for many compounds which might have only the other functionalities, we therefore underestimate their potential contribution. The AMS and SMPS provide a strong constraint on the importance of other functionalities, which appear to have a limited impact on our main conclusions.

Thus, while the measured individual sensitivities can span a large range, by assuming the highest sensitivity measured for a multifunctional carboxylic acid (which are no more sensitively detected than formic acid), and assuming minimal losses during transmission between the FIGAERO and the ionization region, we conclude that the reported fraction of total OA measured by the FIGAERO is a lower limit.

3. Based on analysis of the desorption profiles the authors conclude that at least 50% of the SOA is due to essentially non-volatile species (very low C*). I am concerned about this quantitative measure considering that a single sensitivity factor was applied to all measured species. This result will be biased if species of different vapor pressure have different sensitivities in the FIGAERO HR-ToF-CIMS. Has this been investigated (sensitivity to a compound vs. the compound's C*)? The authors conclude that much of the low-volatility compounds can be attributed to accretion products (e.g. oligomers). Have there been measurements of the sensitivity of the FIGAERO HR-ToF-CIMS to an oligomer and its corresponding monomer? Would one expect the oligomer and monomer sensitivity to be similar? Finally, if after consideration of my comments the authors still conclude that their data suggest that over 50% of the SOA is essentially non-volatile, it would be appropriate to comment on the implication of such a result to the larger research community, e.g. how would it affect organic aerosol modeling efforts.

We have calibrated to large molecular weight mono-carboxylic acids (e.g. stearic, palmitic, etc). These are not detected with substantially different efficiency than other acids (e.g. formic acid), so we do not expect oligomers such as C₁₈ – C₃₀ compounds detected during desorptions to have dramatically different ionization efficiencies assuming they contain carboxylic acids. If they do not contain carboxylic acid groups, then it is possible these are underestimated (see previous responses). Higher m/Q ions are not transmitted as efficiently as lower m/Q ions in this instrument (given the instrument tuning), and as such that would bias the directly measured oligomer content low. However, in the range between formic acid (m/Q 45, sensitivity 20 cps/ppt) and steric acid we see little change in detection efficiency (steric acid: m/Q 284, sensitivity 17 cps/ppt. palmitic acid: m/Q 256, sensitivity 22 cps/ppt).

A more important aspect related to the abundance of monomers vs dimers, is that some fraction of the oligomers likely thermally decompose before desorbing, as demonstrated in the paper given that we observe monomers appearing at higher temperatures than expected during the desorption process. If we simply reported the total abundance of monomers measured during a thermogram, we would overestimate the contribution of monomers, and underestimate the contribution of oligomers substantially without taking into account the clear discrepancies in volatility.

We have clarified the conclusion the reviewer mentioned, that over 50% of SOA is effectively non-volatile. We conclude that at least 50% of the SOA measured by the acetate FIGAERO HR-ToF-CIMS is effectively non-volatile.

Based on the arguments presented above on sensitivity, it seems likely that this fraction is significant compared to the total SOA, but we can only give a range based on our sensitivity. While there could be different sensitivities for the monomers and dimers, it is more likely that the sensitivities are lower (and mass concentrations higher) for oligomers than we assume. It would be difficult to have a response function which dramatically changed the thermogram shape. Zero signal no matter the calibration coefficient is still undetectable mass. Thus, if there is mass with very high C, the acetate FIGAERO HR-ToF-CIMS is blind to it, and our conservative sensitivity limits suggest such a contribution must be <50 – 75% of the total SOA. The possible biases we can come up with are generally implying that we underestimate the contribution of oligomers, but that latter conclusion is not robust enough to apply a uniform correction. As such, we have added a cautious statement about the implication for models.*

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