Identification and Quantification of Gaseous Organic Compounds Emitted from Biomass Burning using Two-Dimensional Gas Chromatography/Time-of-Flight Mass Spectrometry

Hatch et al.

We thank the referees for their comments, which have helped to improve our manuscript. Our responses are provided below.

Referee #1

The authors present results from an experimental study exploring the identification and quantification of non-methane organic compounds (NMOCs) emitted from 6 fuels. In this study 722 compounds were either positively or tentatively identified and for the first time sesquiterpenes were identified in gas-phase BB emissions. The study and the manuscript are well organized and documented. Nevertheless I have several comments before this manuscript can be accepted for publication in Atmospheric Chemistry and Physics.

Questions: Page 23241, Line 24: How were the samples brought back to the lab? Were plants uprooted or just their leaves/pine needles used as samples? Can you describe potential differences in gas-phase products that one might expect between these two types of samples? How long were samples kept prior to experiments? I see that it is listed in section 3.35 but it needs to be listed in the experimental.

An overview of the fuels and their treatment during FLAME-4 has been provided by *Stockwell et al*. (2014). For coniferous fuels, fresh, green boughs (branches with needles attached) were cut and burned. For grasses/straw, stems were cut; they were not uprooted. Except ponderosa pine, which was cut locally, all fuel samples were shipped to the fire lab. Samples were stored for a few days to a few months prior to burning with the longer-term storage occurring in a humidified refrigerator. Storing cut plants likely reduces fuel moisture and possibly the emissions of biogenic compounds, as discussed in Section 3.5 for monoterpenes. *Stockwell et al*. (2014) also compared the FLAME-4 results to field measurements of fire emissions.

We have added the following text to section 2.1.1:

"The fuel treatment during FLAME-4 has been described by *Stockwell et al.* (2014). In general, fuel samples were shipped to the FSL and stored from a few days to a few months with longer-term storage occurring in a humidified refrigerator; instances identified where storage time may have influenced emissions are noted in Section 3.3."

Page 23243, Line 6: What percentage of semi-volatile compound loss is expected from the tubing? This can be tested by taking a semi-volatile compound and flowing through your system measuring the concentration before and after to determine loss. This should also be tested for SQT.

Loss of semi-volatile compounds during sampling is a well-known problem that requires systematic characterization, which is beyond the scope of this manuscript. All sampling methods have artifacts and we have done our best to minimize these in our sampling setup and analysis. In particular, Teflon tubing is often used for NMOC analysis; for example, minimal sampling artifacts (<10%) were observed for a standard mix of NMOCs following 200 m of Teflon tubing (*Schnitzhofer et al*., 2009), which is more than 20x longer than the tubing used in this study. As a specific example, sesquiterpenes are among the least volatile compounds detected in this work. *Helmig et al*. (2004) found ~90% recovery for sesquiterpenes after passing through nearly 4 m of PTFE tubing. Further, *Pollmann et al*. (2005) found nearly complete recovery of sesquiterpenes after passing through a sodium thiosulfate impregnated filter at low ozone concentrations. Therefore, we expect—for the compounds reported in this work—that losses to surfaces were minimal and within the reported the uncertainties. Although sampling losses likely influenced the range of compounds that could be detected.

In section 3.3.5, we have added the following text:

"Further, *Helmig et al*. (2004) found high (~90%) recoveries for sesquiterpenes following nearly 4 m of Teflon tubing."

Page 23243, Line 20: Typically glass-fiber filters are baked at ~ 600 C to remove adventitious carbon. How do you know that these filters have been baked at a high enough sample to remove impurities?

We recognize that filters are typically baked at much higher temperatures and acknowledge that baking at ~130 C likely did not completely remove all impurities, particularly those with lower volatility. The room temperature desorption method used to extract the analytes targets much higher volatility compounds than the solvent extraction methods that are often used with filter samples. Therefore, pre-baking to 600 C was not necessary in this case. Further, the compounds presented in Table 2 were not observed in the blank and background filters that were treated in the same manner. Thus, although other impurities may be present, it does not negate that the reported compounds can be attributed to biomass burning smoke.

Page 23244, Line 1: What were the typical masses of the background and after PM collection?

The PM collected on the background filter was negligible, compared to a net gain of up to ~3 mg for the smoke samples.

Page 23247, Line 20: What oxygenated compounds displayed high breakthrough? These should be asterisked in tables and charts.

As indicated in the caption of Table A1, all compounds that we believe may be underestimated are listed in italics. We have now mentioned this in the text, as well, towards the end of section 3.1.

Page 23251, Line 20: Need to direct reader to Table A1 for abundance of benzene and toluene. How much are your measurements under predicting these values? Why are you under predicting?

We have added a reference to Table A1. The underestimation of benzene/toluene (due to MS detector saturation) is discussed in section 2.3; we have added a reference to this section. A comprehensive comparison of FLAME-4 NMOC measurements using four different techniques will be presented in a separate study.

Page 23257, Line 24: Why could the relative mass of needle vs. wood not be determined? What are you visually observing?

The relative mass could not be determined because the branches were burned intact and not as separated piles of twigs and needles. There is no way to measure the pre-fire mass of the needles and wood separately without destroying the natural fuel geometry. Thus it is difficult to reasonably estimate the relative masses of needles and twigs. However, after the fires most of the needles were gone, but much of the wood was only charred.

Page 23260, Line 1: Is there information regarding the lignin content of the plant fuels used in this study?

We assume the referee is referring to page 23261, lines 1-2 where we discuss potential differences in lignin content between the two grass species measured in this study. Unfortunately, we could not find published literature stating the lignin content of these grasses, nor was it measured in this study. We stated as such on page 23261, line 3. To clear up the confusion we have revised the sentence to "Although the biomass composition of these grasses have not been **reported.**"

Minor Questions: Page 23241, Line 16: What differences can potentially occur if the fuel sample is not standing upright?

The fuel arrangement can influence the combustion conditions. Piles of grasses will likely burn under relatively more smoldering conditions than grasses standing upright.

Page 23246, Line 25: What vapor pressure ranges are not characterized by this approach?

In Section 3.1, we have added the vapor pressures for the compounds roughly bounding this GCxGC measurement range (3-methyl 1-butene and sesquiterpenes).

Page 23247, Line 1: What is the polarity range that can be used for GC?

The accessible polarity range depends on the sampling method and GC columns used for analysis, as well as the thermal stability of the analyte compounds. Further, molecular polarity is not a well-defined property (*Pankow and Barsanti*, 2009), and thus it is not straightforward to define the measureable polarity range. As a full description of GC methods is outside the scope of this manuscript, we provided examples of compounds that did not elute under the GCxGC conditions of this study (page 23247, lines 2-3).

Page 23254, Line 2: How much higher are 1-alkenes relative to C12. How much higher are terminal alkene SOA yields relative to internal alkenes?

For the first question, we assume the referee is asking how much higher the SOA yields are for 1-alkenes relative to alkanes (up to C12). Per the data presented in *Ziemann* (2011), there is not a consistent difference in the SOA yields between the 1-alkenes and linear alkanes, with the alkene SOA yields ranging from ~17-117% higher. Similarly, SOA yields for the terminal alkenes range from ~20-380% higher than internal alkenes. We have added these estimates to the text.

Page 23259, Line 6: How much error do you expect?

We have updated the text to: **"Considering the wide range of reported SOA yields among the MT isomers (<10-60% (*Griffin et al.*, 1999; *Lee et al.*, 2006)),** prediction errors may be significant considering the large and variable contribution of these species in the smoke of coniferous fuels (Figures 1 and 2)."

Page 23259, Line 16: What are their volatilities and atmospheric lifetimes?

We have revised the sentence to:

"SQTs have historically been difficult to measure (*Bouvier-Brown et al.*, 2009; *Pollmann et al.*, 2005) due to their relatively low volatilities **(P_{vap}, ~1×10^{-3} kPa at 25 °C (*Helmig et al.*, 2003)**) and high reactivities **(atmospheric lifetimes on the order of minutes to hours** (*Atkinson and Arey*, 2003))."

Page 23259, Line 22: What are negligible ozone concentrations?

We have changed 'negligible' to '**near-zero**'.

Page 23261, Line 6: What were the levoglucosan concentrations in WG compared to the other fuels?

Because only the fraction of the levoglucosan marker (via AMS measurements) was reported in the cited study, as opposed to levoglucosan concentrations, we have decided to eliminate this comparison.

Page 23262, Line 14: This sentence should be more quantitative.

We have updated the sentence to:

"However, due to the small molecular sizes **(<C_7)** and relatively low concentrations of the observed compounds **(~2-11% of the total EF),** they are not likely to contribute significantly to BB SOA."

Table 3: The estimated SOA mass could be better constrained by using average OH, O3, and NO3 atmospheric steady state concentrations, typical gas-phase bimolecular rate constants for the category of interest and SOA mass yields. Assuming that each

compound reacts completely is not a fair estimate of SOA mass since some compounds react very slow.

We have updated Table 3 with SOA estimates that account for differences in OH-reactivity among the compound classes.

Figures 1-6: Having the GC spectra is not necessary in each figure and should be put in supplemental information. Axis and labels are very small they font should be increased.

The authors collectively and respectfully disagree that the GCxGC chromatograms are not necessary. We think that they provide a strong visual representation of the complexity of biomass burning NMOC emissions. Further, as this is the first reported application of GCxGC for the analysis of gas-phase BB emissions and 2D-chromatograms are not widespread in the related literature, we think it is important to retain the chromatograms in the manuscript.

We have adjusted the colorscale to better highlight the minor peaks. The font size has also been increased.

Minor Comments: Several sentence need references, some examples below: Page 23238: Line 23, and Line 25; Page 23239: Line 3; Page 23252: Line 26; Page 23255, Line 1; Page 23261, Line 1.

We have added references where needed.

Page 23238, Line 24: Sentence is awkward, revise.

We have revised the sentence to "Wildfires and prescribed burns occur globally with highly variable fuel types and burning conditions."

Page 23251, Line 23: Sentence is awkward, revise.

The sentence has been revised to: "Further, higher molecular weight aromatic HCs were detected than are typically reported elsewhere (e.g. $\leq C_9$, (*Akagi et al.*, 2011; *Andreae and Merlet*, 2001); more recently, unspeciated C_{11} alkyl aromatics (*Yokelson et al.*, 2013)).

Page 23251, Line 25: Compilations is an odd word to use here.

We have changed to 'BB reviews'.

Page 23251, Line 3: Extra period after fuels

The error has been corrected.

Page 23253, Line 2: Awkward sentence, revise.

We have revised the sentence to:

"Guaiacols are commonly measured in smoke from coniferous fuels (*Jiang et al.*, 2010;

Saiz-Jimenez and De Leeuw, 1986), as these softwoods contain lignins composed primarily of guaiacyl units (*Shafizadeh*, 1982)."

Page 23253, Line 11: There is an additional reference from this group that also shows aqueous-phase reactions with OH and phenols have 100% SOA mass yields.(Sun et al., 2010)

We have included the suggested reference.

References Sun, Y.L., Zhang, Q., Anastasio, C., Sun, J., 2010. Insights into secondary organic aerosol formed via aqueous-phase reactions of phenolic compounds based on high resolution mass spectrometry. Atmos. Chem. Phys. 10, 4809-4822.

Referee #2:

This paper presents results of highly detailed analyses of sorbent tube samples collected during laboratory test burns of 6 biomass fuels during the FLAME-IV study. Thermal-Desorption 2D-GC-TOF-MS analysis was conducted on gaseous non- methane organic compounds (NMOC; ranging from 'volatile' to 'intermediate volatility') extracted from Tenax/Carbon sorbent tubes collected from well mixed smoke collected during the FLAME studies. A large number of compounds were identified and quantified based on instrument response to either authentic standards of identified com- pounds or chemically similar compounds. The analysis described in the paper appears to be carefully done, with sources of uncertainty identified and the results placed in context with prior investigation in this area. The data and analysis will be of great interest to a wide swath of researchers investigating atmospheric impacts of biomass burning (BB) emissions. These results and discussion will be of particular interest atmospheric modelers and those studying the potential precursors for the secondary organic aerosol (SOA) production observed in biomass burning emissions (which are, as noted, substantially under-predicted by current models and a large source of uncertainty in models). Therefore, the paper is highly suitable for publication in ACP. I concur with most of the questions raised by the other referee, so look forward to these points being addressed. Here I highlight some (mostly) minor points, mostly with the aim of improving the clarity of the manuscript.

P23239, L8-9: This example is not the best way to demonstrate the health and environmental impacts of long range transport of BB emissions – not so many folks in the Arctic.

We have included a different example showing the influence of Siberian wildfires on ozone concentrations in North America.

P23243, L7-8: Considering the extensive testing conducted to look at sorbent break-through and volatilization from the filters, it is surprising that so little attention is paid here. Some effort to quantify the extent of this effect and identify compounds that might be under-estimated because of this potential source of compound loss would be very helpful.

Sesquiterpenes are among the least volatile compounds detected in this work. *Pollmann et al*. (2005) found nearly complete recovery of sesquiterpenes after passing through a sodium thiosulfate impregnated filter at low ozone concentrations. Further, *Helmig et al*. (2004) found ~90% recovery for sesquiterpenes after passing through PTFE tubing. Therefore, we expect—for the compounds reported in this work—that losses to surfaces were minimal and within the reported the uncertainties. Although sampling losses likely influenced the range of compounds that could be detected.

In section 3.3.5, we have added the following text:

"Further, *Helmig et al*. (2004) found high (~90%) recoveries for sesquiterpenes following nearly 4m of Teflon tubing."

P23244, L1-2: This sentence doesn't make sense to me. How would you know if mass was loss from the background filter during PM collection? Perhaps 'PM collection' should be 'filter desorption'? In any case, this description should be clarified.

To avoid confusion, we eliminated this problematic statement. The PM collected on the background filter was negligible, compared to a net gain of up to ~3 mg for the smoke samples.

P23245, L10: GC Column types are not listed, though are referred to later. It would be helpful to list them here.

We have added the GC column types to the text.

P23245, L21-22: I found this description of background correction in general unclear. What statistical method was applied to test whether peaks were 'statistically different from zero'?

We have made minor changes to the text to improve the clarity and modified 'statistically different from zero' to:

"Compounds were removed from consideration if their concentrations were **negative or not significantly different from zero following background subtraction**."

P23246, L1-7: How was the most chemically similar compound determined? Are there cases where this selection would have a large effect on the quantification of the compound, and if so is this accounted-for in the uncertainty calculation? Also, I found the distinction between the use of the calibration line and the response factor unclear.

We have modified the text to clarify the distinction between calibration curves and response factors. Regarding determination of the most chemically similar standard, we have added the following text:

"Tentatively identified analytes were quantified using the calibration curve of the most chemically similar standard compound, **as determined by comparing functional groups, carbon number, degree of unsaturation/conjugation, and aromaticity. Given the large number (~275) and wide range (Table A1) of standard compounds analyzed in this work, reasonable surrogate standard compounds were available for most of the compounds detected in the biomass burning samples**."

Differences in instrument sensitivity between the analytes and surrogate standards may have an influence on the quantification; therefore we have adjusted the minimum uncertainty to 50% of the reported EFs for these compounds (compared to 20% minimum for compounds with associated standard compounds).

P23246, L25: What is meant by 'lower volatility compounds' and 'not well characterized'? Some attempt at quantifying the range of volatility identified (e.g. vapor pressures, RT, C*), and the uncertainty in quantification should be included (also relates to discussion of sampling losses above).

We have eliminated the questionable sentence. In Section 3.1, we have added the vapor pressures for the compounds roughly bounding the GCxGC measurement range (3-methyl 1-butene and sesquiterpenes).

P23247, L4-7: What effect does this 'wrap around' have on the quantification of these and neighboring compounds? Can this be specified and is it reflected in uncertainties for these compounds?

Wrap around should not significantly affect the quantifications of neighboring compounds due to the high sampling rate of the time-of-flight mass spectrometer and mass spectral deconvolution capabilities of the analysis software. The quantification of compounds that wrapped around and have an associated standard (e.g., furfural, phenol) should also not be significantly impacted because the calibration curves were generated under the same instrument conditions (i.e., wraparound was the same). However, the wraparound effect may influence quantifications that were performed using surrogate standards. A compound that wrapped around but quantified with a standard that did not is likely underestimated and vice versa. We note that the minimum error bars were increased to 50% of the reported EF for the tentatively identified compounds to account for the possible differences in instrument sensitivity between the analytes and surrogate standards.

We have added the following text in section 3.1:

"Such wraparound effects should not influence the quantification of the positively identified compounds because these effects were captured in the calibration curves. For a limited number of compounds, wraparound may have influenced quantifications of tentatively identified compounds that wrapped around yet quantified with a surrogate standard compound that did not (or vice versa)."

P23247, L24-27: Given that values with which to compare are available, this comparison should be made and the extent by which these values are 'lower bounds' reflected in the discussion and the table.

A full comparison of all NMOC measurements by four different techniques during FLAME-4 is underway and will be presented elsewhere. We have added a statement to that effect in the text. Values that we believe are underestimated are listed in italics in Table A1.

P23249, L23: Presumably 'compounds emitted during smoldering combustion'?

We have implemented the suggested wording.

P23250, L14: This would suggest that it cannot be at all explained by differences in MCE?

We have eliminated the word 'entirely'.

P23255, L27: Disproportionately relative to what?

We have eliminated the word 'disproportionately'.

P23256, L6-9: It is not clear why NO2/NO ratio is discussed here and whether high- or low-NOx yields should be applied? Should this be VOC/NOx?

The text correctly references NO2/NO ratios. As described in *Chan et al*. (2010), the SOA formation from unsaturated aldehydes through a PAN channel is dictated by the ratio of NO2/NO. Given the significant emissions of unsaturated aldehydes from biomass burning and the relatively high NO2/NO ratios in smoke plumes, we are postulating that such pathways occur in smoke plumes and may have a significant influence on the SOA yields from these light aldehydes. We refer the reader to the Chan et al (2010) reference for further information.

P23256, L23: 'Largest category' is ambiguous. Referring to the figure I presume it is largest mass emission factor, but this could be interpreted to mean the largest number of identified compounds. This kind of ambiguity is found in several other places and I suggest that the revised manuscript be edited with an eye towards removing such ambiguity.

The text stated "the largest category in BS smoke by EF". We have revised it to "the largest EF category in BS smoke…" and clarified other ambiguous references throughout the text.

P23257, L25: 'Visual observations' of the mass of fuel burned? This seems fairly untrustworthy? Can this be quantified in any way?

Unfortunately, this cannot be quantified any more reliably than indicated. There is no way to measure the pre-fire mass of the needles and wood separately without destroying the natural fuel geometry. However, after the fires most of the needles were gone, but much of the wood was only charred.

P23258, L14: No correlations are presented here.

We have changed ‘correlations’ to ‘similarities’.

P23259, L5-11: This is the first mention of PTRMS data in the manuscript, and there’s little reasoning behind this sudden comparison (nor is the acronym defined) or references to works that rely on these values, and what the issues with that might be. Some context should be provided. Further, while the suggestions in this paragraph are likely quite welcome, it would help if a bit further guidance is provided. For example, if ratios from essential oil samples are used, how might these be normalized for use in as emission factors for modeling purposes? This discussion might be transferred to the conclusions section.

We have eliminated the PTRMS comparison at this point in the manuscript. If unspeciated measurements of MTs are available, this could be distributed over the relative proportions of specific isomers reported for steam-distilled essential oils. We have added this suggestion to the text.

P23260, L28: Should be section 3.3.2

We thank the referee for catching this mistake. It has been corrected.

P23265, L8-10: The references listed include OA enhancement ratios for some of these same fuels under similar conditions. Why not do a direct comparison, where possible?

The primary goal of the SOA calculations was to demonstrate the relative importance of the major chemical classes and not necessarily to reproduce measured OA enhancements. These cited references typically report OA enhancement ratios for a single burn per fuel. Given the high variability of emissions from burn to burn and oxidation conditions among smog chamber experiments, direct comparisons to past experiments is difficult. Further, we do not expect that we are capturing all of the compounds contributing to SOA formation, nor the dynamics of the system (e.g., POA evaporation). Thus we prefer to place our estimates in the context of the range of reported OA enhancements.

Figures 1-6: The chromatograms don’t add much to the presentation, so can be moved to the supplement and the additional real-estate potentially used to selectively point out some noteworthy features of the bar charts.

The authors collectively and respectfully disagree that the GCxGC chromatograms are not necessary. We think that they provide a strong visual representation of the complexity of biomass burning NMOC emissions. Further, as this is the first reported application of GCxGC for the analysis of gas-phase BB emissions and 2D-chromatograms are not widespread in the related literature, we think it is important to retain the chromatograms in the manuscript.

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