Author Comments in Response to Referee Comments 3

RC3: 'Comment on egusphere-2026-116', Anonymous Referee #3, 06 Apr 2026

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This paper presents the development and use of a GC/EI-MS mass spectral library for organic aerosol component and source identification. It is an extension of a previous version of the library published in Jen et al., 2019, extending the number of mass spectra from ~4800 to ~27,000. This is an impressive piece of work with a huge amount of work included to label up the mass spectra with an extensive set of metadata. A jupyter notebook has been developed to aid the user interface, providing series of useful tools for analysis. The authors indicate that other labs will be able to add new mass spectra to the library in the future providing an important and novel resource for the atmospheric science community. The paper is very well written and easy to follow. I suggest publication after addressing a small number of minor comments.

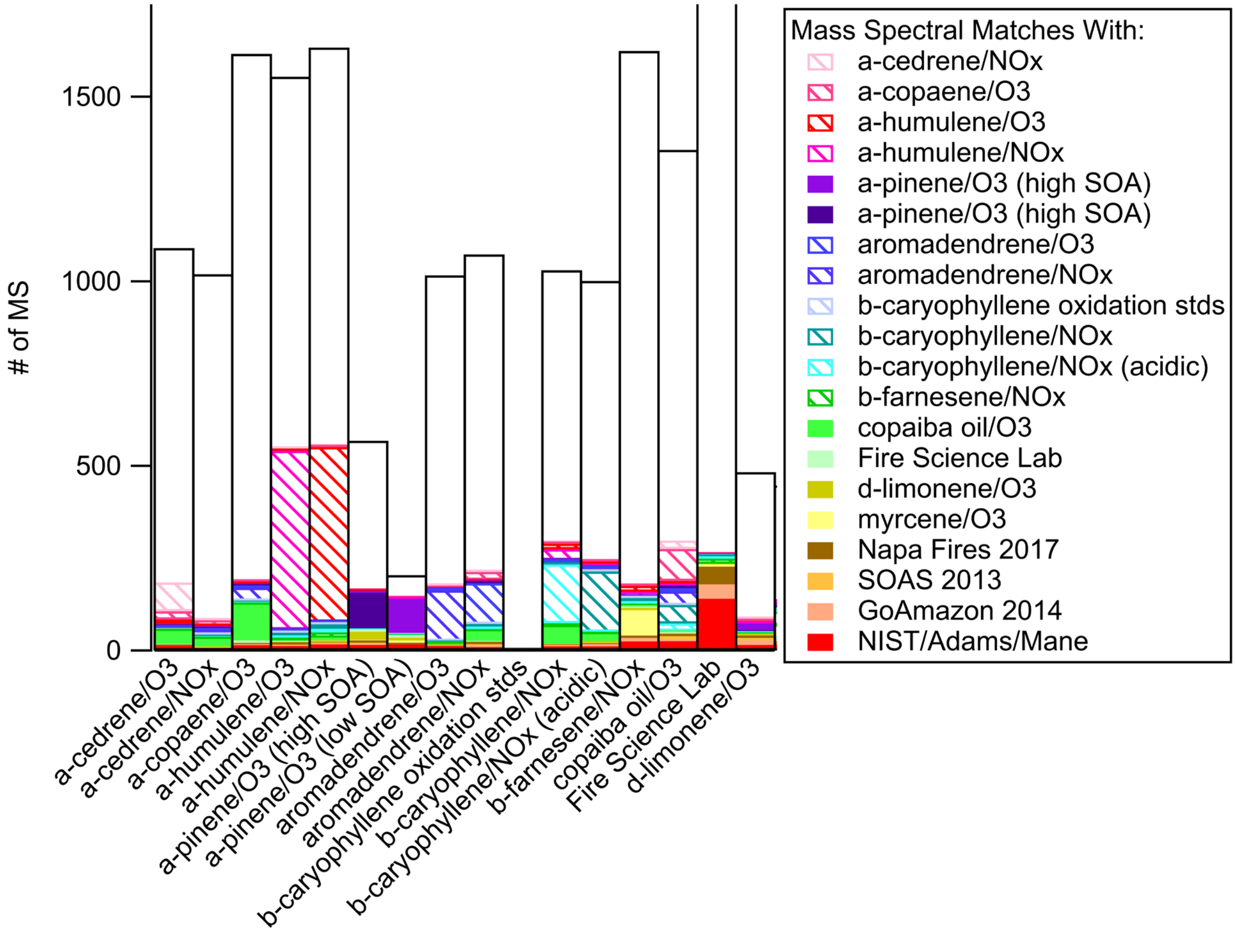
We appreciate Anonymous Referee #3's comments and notes on how to improve the quality of this manuscript. We have addressed all of Referee #3's comments inline below with author responses in italics and blue-colored font. Any line references are those in the original preprint.

Comments

Page 7: For the sesquiterpenes, there are over a 1000 MS each. What is the S/N ratio used for peak identification? It would be useful to know how much overlap there is between the MS for these experiments.

The ratio of each chromatographic peak value to signal noise is >10 for these samples, though additional constraints of minimum peak area, peak height, and peak area were additionally applied typically limiting this signal:noise > 20. While exact overlap of the MS for these sesquiterpene experiments is a current topic for our next analyses of UCB-GLOBES data, we know based on Figure 5 that on average ~ 30% max of MS are matched. Thus, 30% is the highest possible overlap with other sesquiterpene systems, if all matched MS were matched with MS in other sesquiterpene systems. The breakdown by MSM (while preliminary here) can be seen in AC Figure 1 below. The greatest overlap in MSM for the sesquiterpene systems are those in which the same precursor was utilized, e.g. alpha-

humulene/O3 and alpha/humulene/NO_x. While there are other overlaps across these sesquiterpene systems, the majority of MS remain unmatched to any UCB-GLOBES entry or any external known MS from NIST MS, Adams Essential Oil, or MANE Fragrance databases. There is a greater extent of the MSM being found with copaiba oil/O_3 system though, indicative of similar oxidation products as expected from a complex mixture of terpenes.



AC Figure 1: Mass spectral matches by UCB-GLOBES dataset for laboratory oxidation datasets. Hashed bars are indicative of MS found in sesquiterpene systems.

Line 115: I think the bracket at the end of this line should be a comma or else a second close bracket is needed.

Thank you for noticing this. A comma has been added in place of the open parentheses, "…without context of additional data such as Kováts chromatographic retention index, a proxy for elution time of compounds relative to a standard series such as n-alkanes that can be compared across instruments (Kováts, 1958)."

Line 144: Most of these lab SOA samples are without seed aerosol, this should be stated here.

Thank you for noticing this. In fact, all laboratory oxidation samples were conducted with seed aerosol, except for the monoterpene experiments performed in a flow tube by Zhang et al., (2018), *so we have corrected the experimental details in Table 1 accordingly. We have also modified these lines in the manuscript, now reading, "Additionally, aerosols were also collected from seeded laboratory oxidation experiments of single hydrocarbon precursors including several sesquiterpenes (α-cedrene, α-copaene, β-caryophyllene, α-humulene and α-farnesene) and an essential oil rich in terpenes (copaiba essential oil, Amazon origin) under varied oxidant conditions (H_2O_2 as OH precursor with added NO_x or ozonolysis) in the U.S. Environmental Protection Agency National Exposure Research Laboratory as described in previous work (Jaoui et al., 2013; Yee et al., 2018; Zhang et al., 2018). Ozonolysis of monoterpene species such as α-pinene, d-limonene, and myrcene in the absence of seed aerosol were also tested in an oxidation flow reactor at UC Berkeley (Zhang et al., 2018), while reaction of α-pinene with nitrate radical ($NO_3^\bullet$) in the presence of seed aerosol was performed in the Georgia Tech Environmental Chamber (GTEC) facility (Walters et al., 2025). Oxidation of furan biomass burning intermediates, 3-methylfuran with OH and furfural with OH (HONO as OH precursor in both cases) were also conducted in GTEC with seed aerosol (Joo et al., 2024). Lastly, isoprene oxidation via reaction with OH radical (H_2O_2 as precursor, in the absence of added NO_x) was conducted with seed aerosol in the Pacific Northwest National Laboratory chamber facility according to previous methods (Chen et al., 2023)."*

Line 214: Has accessing the library been tested by another lab or have the mass spectra been compared to those generated on another instrument?

Mass spectra from UCB-GLOBES have been used informally by other labs conducting GC-MS measurements to aid in their identifications/source classification of observed organic aerosol species. We have also compared mass spectra to some of those from our in-house GC-MS instruments noting that MS variations can result from the fact that 1-D GC separation MS can have additional spurious ions from closely eluting peaks that are not present in the GCxGC-MS in UCB-GLOBES for some compounds. McGlynn et al., (2025) *performed an extensive search of the ~4800 FSL_FIREX2016 mass spectra against that in the NIST MS Database 2024 (pre-release), for which additional identifications were made, though still only ~10% of the compounds were identified. This demonstrates that for at least ~480 mass spectra from this dataset within UCB-GLOBES, sufficient MS matching is achieved when comparing the MS to NIST MS (which contains many records of mass* *spectra acquired with other instruments). For those ~ 640 compounds' mass spectra among ambient samples that have been positively identified (as shown in Figure 5), this also results primarily from MS matching with MS in databases of known compounds acquired on other instruments (i.e., NIST MS Database, Adams Essential Oil, Mane Fragrance Company).*

Table 2: The description under the "name" "Compound name or given name as an unknown compound…." doesn't really make sense. Also, are the metrics OSc, O:C etc calculated minus the silyl groups? This should be clarified.

Thank you for pointing this out. We have updated the description to, "Compound name including trimethylsilyl groups if applicable (e.g., levoglucosan, 3TMS); otherwise a placeholder name given by an analyst as an unknown compound (e.g. UNK_AHG_HZ_SOAS_394, following the format:

UNKnown_PrincipalInvestigatorInitials_AnalystInitials_Dataset_PeakNumber)" to clarify the formats encountered under the "Name" metadata field.

The predicted chemical properties (e.g., OSc, O:C, Nc) are predicted for the underivatized compound (i.e., minus the silyl groups) as the reviewer points out. We have updated the descriptions for these metadata in Table 2 by adding "…of underivatized analyte" to clarify this.

Line 260: Were the molecular formulas predicted based on the isotope ratios of the molecular ions where applicable.

Thank you for this question. For this prediction method, we did not utilize isotope ratios here as we were not using the high-resolution MS data in this analysis. However, for some datasets within UCB-GLOBES (i.e, NAPA_2017_YL, Soasox, and FSL_FIREX2016_v2.MSP), isotope ratios and/or complementary vacuum ultraviolet ionization data (a softer ionization method which can preserve the molecular ion) were utilized by analysts to determine the MW and thereby noted under the "MW" metadata field. We have expanded the discussion here on Line 259 to add, "…the "MW_Prediction" metadata field was set as "0" or empty. In contrast, the "MW" metadata field was populated under the following cases: 1) Positive MS identification resulting in known chemical structure, 2) analyst determined MW from examination of isotopic ratios and the molecular ion, and 3) analyst determined molecular ion from complementary analysis of samples using vacuum ultraviolet ionization (also reported under "VUV_Exact_Mass" and applicable to only a few datasets in UCB-GLOBES). Custom code was also written to report the base peak (m/z of the highest intensity peak) and the five highest intensity ions."

Line 281 – What does the LDY mean? It isn't explained previously.

The "LDY" is part of an alphanumeric formatted Universal ID (UID), serving as a unique identifier for an MS entry within UCB-GLOBES. By default, it takes the form of Curator'sInitials-####, so we have added this description under Table 2 for the metadata description of UID as, "Universal ID, a unique identifier for the MS within UCB-GLOBES (e.g., LDY-22110, following a format of CuratorInitials-MSNumber)."

Line 300- Why did you pick ~800 compounds for the template? Why did you not do a full non-target analysis and then align the peaks?

Details of the analysis and approach for tracing ~800 compounds during the 2013 Southeast U.S. study (Soasox dataset) are described in (Zhang et al., 2018). Firstly, we note that this was one of the earliest datasets analyzed before we developed methodology for cataloguing full MS of peaks and creating custom MS libraries for automated peak matching through MS matching. Second, this was the largest dataset, comprising 254 filter samples vs. 29 samples in the case of FSL_FIREX2016, 186 samples in the case of GoAmazon, and 74 samples in the case of Napa, CA Fires 2017. The extent of effort to align all peaks in both GC dimensions was challenging with the analysis software at that time. Further, 2nd dimension shifts are much more variable with instrument condition and after breaks in analysis, and due to a modulation cycle of 2.3 sec with limited polarity range in internal standards, it is challenging to precisely configure a 2nd dimension LRI system to help with alignment and create a consensus template through 253 successive additions of any novel peaks after conducting a non-targeted chromatographic peak detection and MS search on each sample chromatogram. While ~800 compounds were detected in one particular sample of this study (chosen because of its rich diversity visible in the sample chromatogram), Zhang et al. did look at other sample chromatograms to check for other "missing" novel analytes amongst another approximately 10 samples and added them into the overall template ultimately used to track analytes across all 254 samples.

Line 356 – Can you explain why you think the BBOA/MT/SQT and BBOA/SQT can be interpreted as terpene derived primary biomass burning organic aerosols rather than small common oxidation products.

Thank you, this is a good point. After reexamination of the MS here, we reinterpret them to be as Referee 3 suggests, small common oxidation products, as the predicted chemical properties suggest Nc < 10 for each species with OSc ranging -0.8 to 0. We have revised this line to read, "The MS under BBOA/MT/SQT and BBOA/SQT categories could be further interpreted as shared common products derived from terpene oxidation and biomass burning based on Ch3MS-RF predicted carbon numbers <10 and average carbon oxidation *states ranging -0.8 to 0 (See Section 3.4 Improving chemical knowledge of unknown UCB-GLOBES compounds through chemical properties prediction)."*

Line 364: There is a large drop in compounds assigned as ASOA. There are no laboratory experiments for anthropogenic VOC precursors and so this is likely to be underestimated, especially where highly oxidised low C products are formed. Also, quite a few have been transferred to isoprene SOA. Could this be due to anthropogenic isoprene emissions?

While we assign these compounds to the isoprene oxidation category, ISOP, because there are MS matches between ASOA with ISOP datasets, we cannot be certain if they derive from anthropogenic isoprene emissions. As these shared ISOP oxidation products correlate in time with known ASOA tracers, we could consider these MS to be indicative of shared oxidation products between isoprene and anthropogenic emissions under these conditions and/or that these are isoprene oxidation products generated under the influence of anthropogenic emissions. Further insight could be gained in the future with laboratory experiments of anthropogenic VOC precursors and additional experiments simulating isoprene-derived SOA formation under anthropogenic influence.

Line 381: The number of hits is perhaps surprisingly low. Do you have any insights into the missing species based on the calculated metrics?

We agree with Referee #3 on this point. The GoAmazon MS dataset represents a highly unique, chemically diverse set of ambient compounds. In fact, the low number of hits with NIST MS Database became one of the main motivations for conducting laboratory oxidation studies of the sesquiterpene systems included in UCB-GLOBES, as > 20 sesquiterpene species were much more prevalent in the Central Amazon than in the Southeast U.S. (Yee et al., 2018). *In addition, GoAmazon includes several MS representing compounds from biomass burning influence that are not also observed in Napa, CA 2017 fire smoke as well as simulated burns of Western U.S. fuels in FSL_FIREX2016. We hypothesize that in part, the chemical diversity observed in the Central Amazon, results from the confluence of cross-reactions of many more available VOC precursor types and their intermediates (which has yet to be fully simulated in the laboratory) under varied natural, anthropogenic, and biomass burning conditions. In addition, the environment is also much more humid (average relative humidity > 80%) which is challenging to achieve in laboratory settings and warmer (average temperature > 26 °C). The extent of aqueous-phase chemistry both in the aerosol and/or through aerosol-cloud processing is also a potential mechanism by which these unique species are generated in the Central Amazon and have yet to be duplicated in the laboratory.*

Line 392: It would be useful to suggest the types of experiments that need to be done to extend the range of atmospheric conditions – biogenic + NOx, anthropogenic SOA etc.

Thank you for this suggestion. We have added the following recommendations for suggested types of experiments here and we also allude to this need in the updated conclusion section in address of Referee #3's comment on Line 516 recommending a more general Future Directions section. We add to Line 394, "Biogenic VOC oxidation was found to be particularly sensitive to anthropogenic emissions in this area (Franklin et al., 2023; de Sá et al., 2017, 2018, 2019; Shrivastava et al., 2019). New experiments focusing on Central Amazon conditions: biogenic VOC precursors (e.g., ~10s of ppt$_v$ *monoterpenes) with varying NO*$_y$ *[0-20 ppb*$_v$*] and O*$_3$ *[0-40 ppb*$_v$*] levels at high relative humidities (80-93 %) and temperatures (26-28 °C) in the absence and presence of anthropogenic VOC precursors and/or primary anthropogenic OA could provide new MS that help elucidate some of the mechanisms behind the compounds in the urban plume-influence category. However, such high relative humidities and low precursor and oxidant concentrations are typically challenging to achieve in a laboratory setting."*

Line 418: Does the statement about "29 % of MS are considered to have a MSM" relate to the ambient samples? Its not clear.

Thank you for pointing out this need for clarification. This line has been modified to reflect that we refer to 29% of all MS in UCB-GLOBES, "Despite generous thresholds for considering potential mass spectral matches (i.e. FMF ≥ 700, RMF ≥ 800), only 29% of all MS in UCB-GLOBES are considered to have a MSM, with the remaining 71% of MS unmatched to other MS within UCB-GLOBES, NIST 20, Adams Essential Oil MS Library, or the MANE Fragrance MS Library (Figure 5)."

We also revised the abstract lines starting with Line 53 to clarify this point, "While OA from laboratory oxidation experiments in UCB-GLOBES are highly diverse chemically, on average only 29% of UCB-GLOBES MS have a mass spectral match to another MS entry in UCB-GLOBES and/or in databases of known compounds (i.e. NIST MS Database, Adams Essential Oil, MANE Flavor and Fragrance Company). This indicates that roughly 70% of UCB-GLOBES MS are unique thus far, not observed more than once among the laboratory oxidation samples and ambient data in UCB-GLOBES MS. Further, only 18% can be positively identified using these databases or known authentic standards."

Line 420, The meaning of the sentence starting with "on average, 18 % of MSM…." Is not clear and should be rewritten.

This sentence has been modified accordingly, "On average, 18% of ambient data MS have MSM amongst an authentic standard or the aforementioned libraries of known compounds *leading to positive identification, emphasizing the need to better understand the sources and processing of most atmospheric species catalogued here."*

Figure 5: Do the hashed bars indicate positive identification using NIST or other known MS libraries?

Yes, that is correct. We have modified the last sentence of the caption to state this more explicitly as, "Hashed magenta bars indicate mass spectral match with a known compound in external libraries and/or with an authentic standard leading to positive identification (on average 18%)."

Line 446: The section on Ch3MS-RF is less convincing than the rest of the paper. The ML model was built on 130 standard compounds, but the library has 27,000 spectra. What is the cross over in terms of chemical functionalilty compared to the expected products. For instance, how does the model deal with the formation of nitrate groups in the OSc calculation for the a-pinene + NO3 experiment? In figure 6c, the nitrate chemistry looks very different and the abundance of dimers is much higher - perhaps the associated reference has some detailed analysis for comparison? More detail is needed here of the limitations of this approach.

We appreciate this comment and can expand in greater detail on the appropriateness and applicability of Ch3MS-RF for the MS in the library. As the reviewer points out, Ch3MS-RF was originally built on 130 standard compounds. These standard compounds are listed in Table A1. of Franklin et al. (2022) and were selected for their wide-varying volatility, functionality (e.g., alkane, acid, alcohol, aldehyde, ketone, polyaromatic hydrocarbons), and structure types (e.g., phenol, methoxyphenol, quinone, sugar, sterol, sesquiterpene, known terpene-derived oxidation products, isoprene-derived oxidation products) to be as representative of the chemical complexity of ambient organic aerosol species as possible with available standards. While nitrocatechol, diethyltoluamide, hexadecanamide, phthalimide, and quinoline, were the only N-containing standards feasible to acquire at the time and organonitrate compounds (as well as organosulfates) are typically not amenable to our GCxGC analyses, we do not expect Ch3MS-RF predictions for the a-pinene + NO3 experiment to predict chemical properties of organonitrate compounds, unless their MS features sufficiently overlap with the MS features used in the training set. As random forest modeling does not extrapolate and has a tendency to underpredict property extremes of Avg. OSc and Nc space (Franklin et al., 2022), we might expect a true greater spread of these properties compared to what is currently predicted for the a-pinene + NO3 case by Ch3MS-RF. That is, if the MS of the GC-amenable a-pinene + NO3 oxidation products are that far different from MS of the test case (~130 standard compounds) and that of the extrapolation case (71 identified GoAmazon *compounds) as used in Franklin et al., (2022), which demonstrated Ch3MS-RF to have mean absolute error accuracy of +/- 0.25 for Avg. OSc and +/- 1.8 for Nc, then, we would expect greater uncertainties for this case. In addition, position in GCxGC space (along with derivatization indicators) is a reasonable indicator of chemical properties (as compounds are chromatographically separated across volatility and polarity space). So, most compound types amenable to this analysis, we believe, have the potential to translate pretty well even for functional groups that Ch3MS-RF has not been explicitly trained in.*

We have summarized this briefly by revising the discussion in the manuscript as follows, "Briefly, Ch3MS-RF is a random forest model developed and trained on an authentic standard of ~130 compounds sampled with the same setup in this study. Mass spectral features and first-dimension retention index are utilized for chemical properties prediction of the underivatized analyte, the results of which are included as metadata entries accompanying each MS in UCB-GLOBES. The compounds used in training were selected to be as representative of the chemical complexity of ambient organic aerosol species as possible with available authentic standards. They span wide-varying volatility, functionality (e.g., alkane, alkene, carboxylic acid, alcohol, aldehyde, ketone, ester, amide), structure (e.g., polyaromatic hydrocarbons, phenol, methoxyphenol, quinone, sugar, sterol), and source types (e.g., known terpene- and isoprene-derived oxidation products). Note that Ch3MS-RF predictions here are inherently limited to compounds amenable to this GC x GC analysis (e.g., organosulfates and organonitrates would be excluded). Franklin et al., (2022) demonstrated Ch3MS-RF to have mean absolute error accuracy of ± 0.25 for Avg. OSc and ± 1.8 for N_c *when applied to the test case (~130 authentic standards) and the extrapolation case (71 identified GoAmazon compounds). As random forest modelling does not extrapolate and tends to underpredict property extremes of Avg.* OS_c *and* N_c *space (Franklin et al., 2022), we might expect a wider spread of the true chemical properties' values and higher uncertainty in predictions for any encountered MS with very different MS features from the training and extrapolation datasets."*

Line 516: The future directions are too specific to the authors group and existing samples. I would prefer something more generic that can be applied to the broader community.

Thank you for this suggestion. We have added an additional paragraph to cover broader future directions applicable to the broader community as follows:

From the present analyses of UCB-GLOBES, we recommend a few research priorities going forward to advance understanding of the chemistry and fate of organics in the environment. Firstly, the community would benefit from openly shared quality *annotated GC/EI-MS mass spectra of a wide range of environmental and laboratory sample types. Second, we would benefit from technical advancements to conduct and catalogue organic species in atmospheric laboratory simulations that are closer to atmospheric conditions with higher relative humidities (> 75 % RH), lower organic aerosol mass loadings, and lower concentrations of precursors and oxidants. We also recommend new datasets simulating oxidation of a mixture of VOC precursors and their oxidation intermediates, as cross-VOC system reactions as would happen in the atmosphere are not sufficiently represented in UCB-GLOBES yet. Inherent to these recommendations are challenges to technical feasibility, ensuring experiments are at sufficient concentrations for detection, and trade-offs of adding too many variables to the environmental matrix. However, achievements toward these recommendations could provide some of the keys to elucidating the true mechanisms and chemical complexity in ambient observations. Finally, we hope that UCB-GLOBES and other available environmental 70eV EI-MS data will be utilized for comparisons across environmental reservoirs (e.g., lithosphere, hydrosphere, biosphere, and atmosphere), so that the emission, transport, and fate of organic carbon across these sectors can be traced and better quantified through processes such as emission, reaction, and deposition.*

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