In my opinion the submitted manuscript addresses an interesting scientific issue, i.e., the study on the volatility of HULIS aerosol compounds. The text is understandable for the reader. The objectives are clearly defined and raised conclusions are coherent. However, I would opt to likely consider moving the manuscript first to the ACPD full review process. This is because I have a few remarks as stated below:

A) Limited number of ambient samples

The authors made their research based only on 4 filter samples. No justification was provided as to the choice of such a limited number of samples. Certainly, during the full ACPD review phase the authors would be requested to build up their scientific story on the results obtained from the analysis of more ambient samples, say 10 or larger due to the increased complexity of HULIS (e.g., Environ. Sci. Technol., 2016, 50 (4), 1721).

B) Analytical procedure for the preparation of HULIS extracts

The authors blindly believe that a whole fraction of HULIS is greatly soluble in the aqueous phase. However, in the light of recent papers dealing with the chemical composition HULIS (e.g., Environ. Sci. Technol., 2016, 50 (4), 1721; Atmos. Chem., 2015, 72, 65), aerosol-derived HUmic LIke Substances represent a complex chemical mixtures, including high-molecular-weight aliphatics (primarily C_{27}−C_{32}) with small proportions of −CH_3, −OH, and C=O groups. These are poorly soluble in water, thus the extraction with pure water only may lead to the substantial loss of analyte to be further subjected to the H-TDMA analysis. I am highly surprised that the authors did not take it into account despite it is a basic approach in the analytical atmospheric chemistry: a sample preparation is the most crucial factor. I would suggest broadening a result discussion with providing additional data obtained for samples extracted with less polar solvents, say acetonitrile and water acetonitrile (50/50v). The same could be applied for another solvent couple: methanol and methanol-water (50/50), as recently have been suggested by Lin (Environ. Sci. Technol., 2014, 48(20), 12012). Moreover, I am a bit concern of the selection of SPE method for HULIS water extracts since it may result in a dramatic loss of highly oxidized and water soluble products, such as organosulfates (nitroxyorganosulfates).