

Response to the comments of Anonymous Referee #1

General comments:

Zhang et al used an oxidation flow reactor (OFR) to produce SOA at different relative humidities, and attempt to explain the changes in SOA loadings caused by the changing RH. Unfortunately, I do not find their explanations and conclusions to be based on solid evidence or argumentation. I also cannot envision that further analysis of their data would allow new insights into the topic of how humidity influences SOA formation. Therefore, I cannot suggest this manuscript for publication in ACP. I outline some of the main shortcomings below.

Response: We thank the Referee for their feedback on our manuscript. We have carefully considered the comments and have made significant revisions to address the concerns that the Referee raised.

In our revised version, we have conducted exocyclic double bond limonene ozonolysis experiments to verify the RH effects on multi-generation reactions of limonene. We have also added low concentration limonene experiments to provide further insights at low SOA loadings. In addition, we have revised the proposed mechanism and analysis for limonene and Δ^3 -carene ozonolysis. The responses are listed below in blue color text and the associated revisions to the manuscript are shown in red color text.

Major comments

1. The experiments were conducted at loadings far from atmospheric concentrations, yet this aspect, and how it might impact the relevance of the study to the atmosphere, was not discussed at all. The gas phase oxidation chemistry of monoterpenes is a complicated process, largely due to competing fates of RO₂ radicals under different conditions, and further reactions are very likely to take place after condensation into SOA (e.g. Pospisilova et al., 2020; Kalberer et al., 2004). As such, using offline SOA composition data to infer something about the reactions taking place in the first milliseconds after oxidant attack is extremely challenging and would, at the very least, require detailed analyses to exclude that any of the other stages of potential reactions are negligible. This was not done, and I cannot see that it would be possible with the data available in this study. In fact, the offline MS data is even said to stay relatively unchanged with the change in RH (line 194), so the chemical insights will be very limited, and really the only data used to draw conclusions on is the SMPS data. This is simply not enough for reaching any conclusive chemical understanding of the processes.

Response: To get a better understanding of the multi-generation reactions of limonene under low loadings, we have conducted a low-concentration limonene ozonolysis experiment (Fig. S6). The results of this experiment also revealed an enhancement effect of RH on limonene SOA similar to that observed at high loadings. This has been updated in the revised manuscript at Page 18, Line 339-348: **“To investigate the multi-**

generation reactions of limonene under low-concentration conditions, we conducted low-concentration limonene ozonolysis experiments, and the results are shown in Fig. S6. In these experiments, the limonene and O₃ concentrations were 20.5 ppb and 5.7 ppm, respectively. According to the experimental results, the number concentration of SOA formed from limonene ozonolysis increased by approximately 1.4 times under high RH, which is similar to the increase observed under high-loading conditions. The mass concentration increased by approximately 1.3 times at a precursor concentration of 20.5 ppb. The relatively small increase in mass concentration compared to the high-concentration conditions may be attributed to the less pronounced distribution of SVOCs at low mass concentrations. This result indicates that the enhancement effect on limonene SOA by high RH is still valid for low precursor concentrations.”

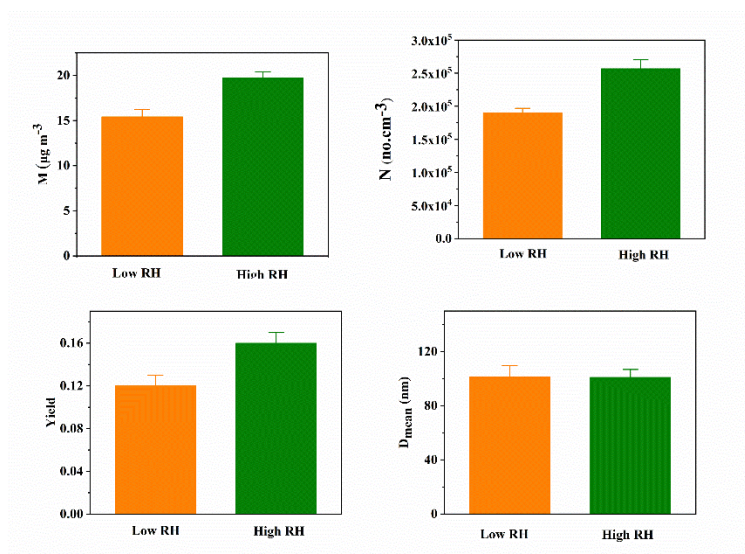


Figure S6. The SOA formation of low-concentration limonene under low and high RH (a) mass concentration (b) number concentration (c) SOA yield (d) mean diameter.

Offline ESI mass spectrometry analysis of particulate matter is an effective technique that provides valuable information about the SOA formed from the oxidation of VOCs. This technique is powerful to provide the molecular composition of SOA and enable the determination of the formation mechanisms of the oxidation products, including gas-phase reaction products. Indeed, many previous studies have applied offline ESI-MS for the identification of SOA components and their formation mechanism. For instance, Zhao et al. (2022) proposed the gas-phase formation pathways of dimer esters in SOA arising from the ozonolysis of α -pinene using the offline analysis with UPLC-QTOF-MS. Furthermore, employing offline HPLC/ESI-TOF-MS analysis, Inuma et al. (2007) proposed a detailed formation mechanism for two organosulfates associated with SOA formation. Additionally, Li et al. (2020) proposed gas-phase OH oxidation mechanism of long-chain alkanes using offline ESI-TOF-MS. Similarly, through offline UPLC/ESI-TOF-MS analysis, Thomsen et al. (2021) inferred that the α -pinene-derived analogue, cis-pinic acid, tends to stay in the gas phase and undergo further reactions before condensing.

In order to minimize the potential impact of post-collection reactions on our

experimental results, we employed a consistent treatment across all experiments, where the collected particles are immediately dissolved to avoid any potential influence of particle-phase reactions under different experimental conditions.

We did not observe significant changes in the type of mass spectrum peaks between low and high RH. However, we did observe variations in the peak intensities of certain specific products, which can be attributed to the influence of RH. These effects promote specific reaction pathways, thereby facilitating the formation of corresponding products.

In addition, to further verify the hypothesis regarding the influence of water on multi-generation reactions of exocyclic double bonds, we have conducted the endocyclic double bonds limonene ozonolysis under low O₃ concentration (67 ppb) and high precursor concentration (450 ppb). Under this condition, the ozonolysis mostly happened for the endocyclic double bond in limonene, leaving the exocyclic double bond almost unreacted, since the reaction of O₃ with endocyclic double bond is ~30 times faster than the reaction of O₃ with exocyclic double bond (Shu and Atkinson, 1994). As expected, when limonene was oxidized at only the endocyclic double bond, we observed a slight decrease in both the number and mass concentrations as the RH increased (Fig. S7). This is similar to the results obtained for Δ^3 -carene, which contains only an endocyclic double bond. The results of these control experiments provide more evidence that the multi-generation reactions play important roles in the limonene SOA enhancement by high RH. This was revised it in Page18, Line 349-356: “To further confirm the assumption that water-influenced multi-generation reactions of the exocyclic double bond enhance the SOA formation, we conducted two comparative analyses: firstly, we examined the ozonolysis of the endocyclic double bond in limonene, leaving the exocyclic double bond unreacted. This was done by applying a low O₃ concentration (~67 ppb), since the reaction of O₃ with endocyclic double bond is ~30 times faster than the reaction of O₃ with exocyclic double bond (Shu and Atkinson, 1994). Interestingly, when limonene was oxidized at only the endocyclic double bond, we observed a slight decrease in both the number and mass concentrations as the RH increased (Fig. S7). This is similar to the results obtained for Δ^3 -carene, which contains only one endocyclic double bond.”

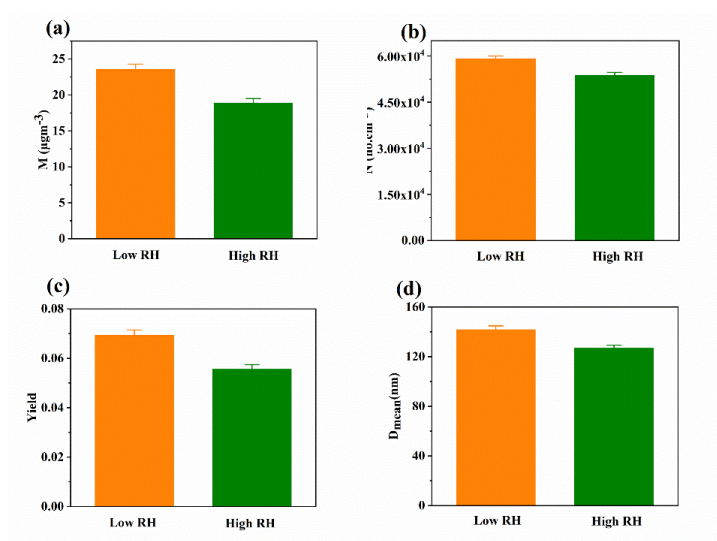


Figure S7. The SOA formation from endocyclic ozonolysis of limonene under low and high RH (a) mass concentration (b) number concentration (c) SOA yield (d) mean diameter. The initial concentration of limonene is 450 ppb and the concentration of O₃ is 67 ppb. Limonene ozonolysis primarily took place on endo-double bonds, with a rate constant of $2.01 \times 10^{-16} \text{ cm}^3 \text{ molec}^{-1} \text{ s}^{-1}$ (Shu and Atkinson, 1994). Based on this rate constant, it can be estimated that approximately 10% of the limonene was consumed by O₃ upon exiting the reactor.

In summary, by keeping the same protocols in sample processing and analysis, the errors introduced from off-line analysis were minimized. The differences in specific products in the MS indicate the RH effects on the subsequent reactions after the ozonolysis of the exocyclic double bond. This mechanism was further verified by the control experiments at low ozone concentration. Therefore, we believe that the results here can improve our understanding of the multi-generation chemical processes of limonene.

2. There are several studies concluding that RH does not have a noticeable impact on the formation of the most oxidized products that are expected to contribute most to SOA (e.g. Surdu et al., 2023; Li et al., 2019). Surdu et al. also analyze the potential reasons for the RH-driven changes in relation to particle phase reactions and changes in partitioning.

Response: We acknowledge that relative humidity (RH) was reported not to have a noticeable impact on the formation of the most oxidized products such as in Surdu et al. (2023). However, it is important to note that this study was conducted under photooxidation conditions whereas our experiment was conducted under dark conditions. Furthermore, their investigation of SOA formation was carried out to achieve a steady-state particle growth, with RH gradually increasing while maintaining all other experimental conditions constant. In contrast, our experiment focused on exploring the influence of water throughout the entire oxidation process of SOA by using different initial degrees of humidity.

With regard to Li et al. (2019), although both their study and ours investigate ozonolysis, the experimental conditions are quite different. The ozone concentration and residence time of the reactor are 900 ppb and 60 s in Li et al. (2019), while they are ~6 ppm and 167 s in this study. This leads to a ~18 times higher ozone exposure in this study. In addition, the limonene concentrations are 1085 ppb and 321 ppb in Li et al, 2019 and in this study, respectively. All these differences lead to an easier ozone reaction of the exocyclic double bond in our study. In addition, the high ozone/limonene ratio in this study is believed to be more similar to the real atmospheric conditions. We have revised this in the revised manuscript in Page 17, Line 322-328: **“In contrast, Li et al. (2019) found negligible change in dimers and HOMs in limonene-O₃ system when changing RH from 0 to 60%. The discrepancy is mainly attributed to the different experimental conditions. The ozone exposure in this study is ~18 times higher than in Li et al. (2019), while the limonene concentration in this study is only ~30% of that in their study. These two conditions both favor the multi-generation reactions occurred at**

the exocyclic double bond of limonene and its products. Thus, we believe this leads to the different results regarding the formation of HOMs and dimers.”

3. In addition to their speculative nature, the chemical mechanisms drawn up and discussed are wrong/misleading concerning the sCI. Only a small part of the formed CI will stabilize (and thus be impacted by RH), as most of them will simply decompose through the typical vinyl hydroperoxide channel. Reaction with water vapor is normally only relevant for the stabilized CI. In this manuscript, it is proposed that ozonolysis produces sCI at a 100% yield (e.g. lines 206-208, Fig 3-4). This raises further questions concerning how well the authors have understood the reactions that they are using to explain their observations.

Response: We have redrawn Figures 3, 4 and S5 and revised the corresponding mechanisms. In the revised figures, the POZ formed from the reaction of the endocyclic double bond generates eCI₁ and eCI₂ with branching ratios of 0.35 and 0.65, respectively. The reaction pathways associated with eCIs are complex. However, the generation of sCIs and the OH pathway are believed to be dominant (Nguyen et al., 2016). It is inferred that about 46% of eCIs formed from α -pinene ozonolysis would stabilize (Tillmann et al., 2010). Furthermore, it has been observed that the presence of OH plays a significant role in promoting the formation of HOMs (Crouse et al., 2013). Therefore, in this study, we focus our discussion on the sCI and OH pathways which are illustrated in the proposed mechanism depicted in Figures 3 and 4.

These details have been updated in the revised manuscript at Page 12 as follows:

Line 218-221:

“In the context of eCI₁, several complex reactions occur, with the most dominant reaction being the generation of hydroxyl radicals (OH) and a reaction pathway known as sCI₁. The sCI₁ pathway can proceed through three distinct reactions, as depicted in Fig. 3.”

Line 224-226:

“The second and third pathways involve reactions of sCI₁ with carboxylic acids and carbonyls, respectively, leading to the formation of anhydrides and secondary ozonides. Additionally, the generated OH radicals can react with limonene, giving rise to another alkyl radical, C₁₀H₁₇O·.”

Line 236-238:

“In addition to the eCI₁ route, the eCI₂ pathway is also responsible for the generation of various products (Fig. 4). Since the reaction of the hydroxyl radical (OH) attacking limonene is already depicted in Fig. 3, our main emphasis in Fig. 4 is on the pathways involved in the generation of sCI.”

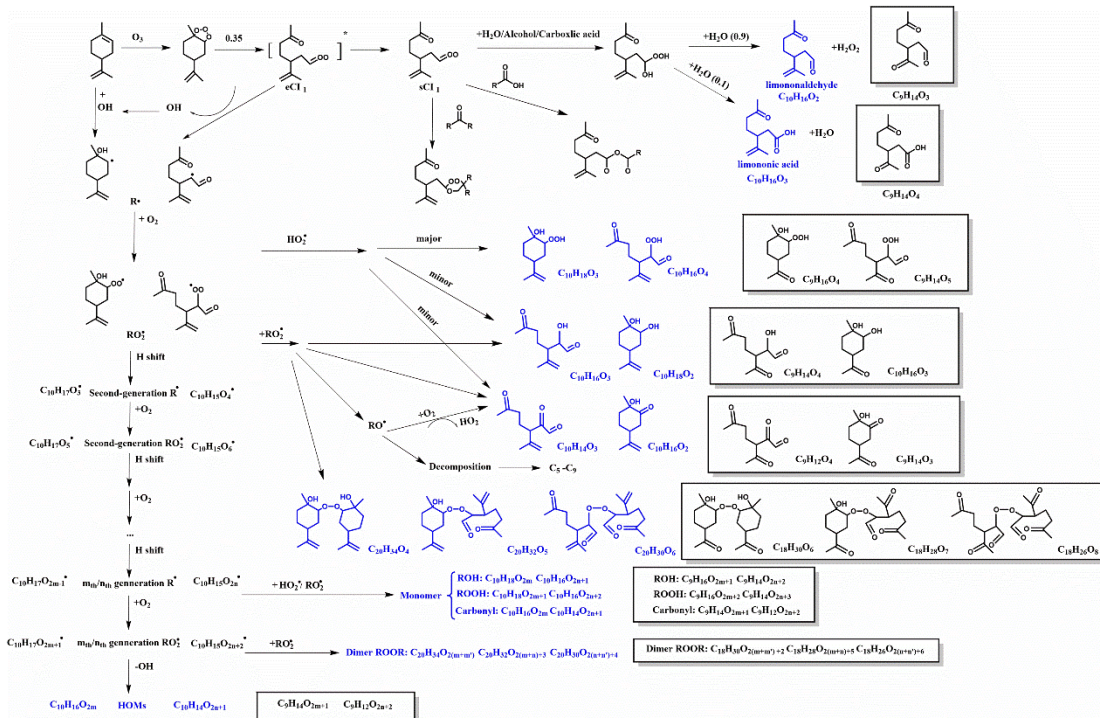


Figure 3. Proposed formation mechanism for SOA formation from eCI_1 oxidation under high RH. The compounds in blue and in boxes are identified using UPLC/ (-) ESI-Q-TOF-MS.

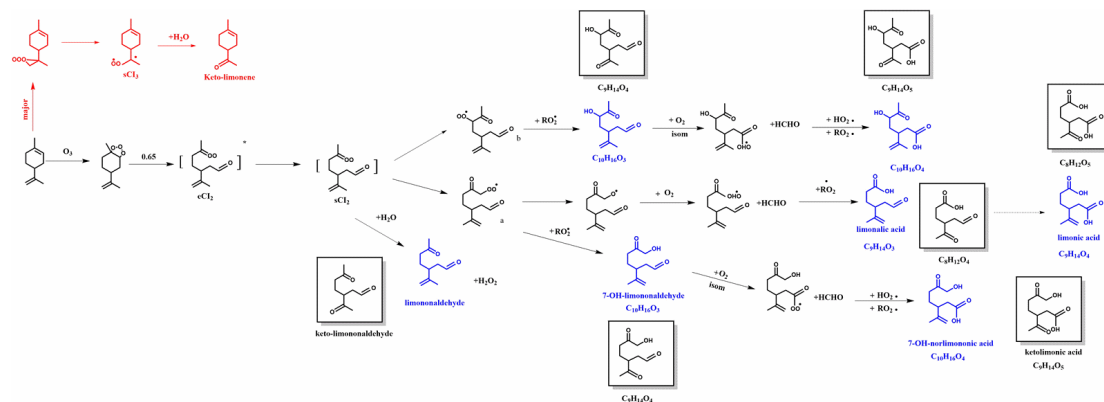


Figure 4. Proposed formation mechanisms for SOA formation from eCI_2 and exocyclic double bond oxidation under high RH. The compounds in blue and in boxes are identified using UPLC/ (-) ESI-Q-TOF-MS.

C ₁₆ H ₂₀ O ₆	7.24×10 ²	C ₁₀ H ₁₄ O ₁₁	3.90×10 ³
C ₁₃ H ₁₈ O ₉	3.36×10 ²	C ₁₄ H ₁₄ O ₈	4.02×10 ³
C ₁₇ H ₂₂ O ₆	6.63×10 ³	C ₂₀ H ₄₀ O ₂	4.60×10 ³
C ₁₈ H ₂₆ O ₅	6.28×10 ²	C ₁₂ H ₁₀ O ₁₀	4.00×10 ³
C ₁₉ H ₃₂ O ₄	1.58×10 ³	C ₁₃ H ₁₆ O ₉	8.34×10 ³
C ₁₅ H ₁₈ O ₈	1.65×10 ³	C ₁₉ H ₂₆ O ₄	4.96×10 ³
C ₁₃ H ₁₂ O ₁₀	8.85×10 ³	C ₁₇ H ₂₂ O ₆	1.05×10 ³
C ₁₄ H ₂₀ O ₉	8.44×10 ²	C ₁₃ H ₁₂ O ₁₀	5.46×10 ³
C ₁₆ H ₂₈ O ₇	9.89×10 ³	C ₁₃ H ₁₈ O ₁₀	4.68×10 ³
C ₁₅ H ₂₆ O ₈	2.18×10 ³	C ₁₅ H ₁₂ O ₉	4.22×10 ³
C ₁₀ H ₈ O ₁₃	6.33×10 ³	C ₁₀ H ₁₂ O ₁₃	5.00×10 ³
C ₁₈ H ₂₄ O ₆	6.06×10 ²	C ₂₂ H ₂₈ O ₃	8.88×10 ³
C ₁₁ H ₁₄ O ₁₂	7.70×10 ²	C ₁₉ H ₂₆ O ₆	1.54×10 ³
C ₂₁ H ₂₂ O ₄	4.80×10 ³	C ₁₆ H ₂₀ O ₉	1.64×10 ³
C ₂₀ H ₃₄ O ₄	2.53×10 ³	C ₁₅ H ₁₈ O ₁₀	5.00×10 ³
C ₂₃ H ₃₂ O ₂	2.12×10 ³	C ₁₆ H ₂₂ O ₉	1.69×10 ³
C ₁₈ H ₃₂ O ₆	3.68×10 ²	C ₁₈ H ₂₂ O ₈	3.32×10 ³
C ₁₇ H ₃₀ O ₇	7.46×10 ³	C ₁₂ H ₁₆ O ₁₃	4.00×10 ³
C ₁₄ H ₂₂ O ₁₀	4.04×10 ³	C ₂₀ H ₃₂ O ₆	8.21×10 ³
C ₂₁ H ₃₆ O ₄	1.36×10 ⁴	C ₁₆ H ₁₈ O ₁₀	4.50×10 ³
C ₁₇ H ₃₀ O ₈	4.68×10 ²	C ₁₆ H ₂₀ O ₁₀	5.20×10 ³
C ₁₂ H ₁₆ O ₁₃	2.43×10 ³	C ₁₉ H ₂₄ O ₈	8.21×10 ³
C ₁₁ H ₁₄ O ₁₄	4.46×10 ²	C ₂₀ H ₂₈ O ₇	2.38×10 ³
C ₁₈ H ₃₀ O ₈	4.46×10 ²	C ₁₇ H ₂₀ O ₁₀	4.16×10 ³
C ₁₆ H ₂₆ O ₁₀	7.44×10 ²	C ₂₁ H ₃₆ O ₆	8.03×10 ³
C ₁₇ H ₂₀ O ₁₀	2.12×10 ³	C ₁₆ H ₂₆ O ₁₁	1.16×10 ³
C ₁₆ H ₂₄ O ₁₁	1.48×10 ³	C ₁₇ H ₂₆ O ₁₁	1.32×10 ³
C ₂₀ H ₂₄ O ₈	3.96×10 ³	C ₁₈ H ₁₈ O ₁₁	4.02×10 ³
C ₁₇ H ₂₂ O ₁₁	2.48×10 ³	C ₁₈ H ₂₂ O ₁₁	4.54×10 ³
C ₂₁ H ₃₄ O ₈	1.28×10 ⁴	C ₁₈ H ₂₆ O ₁₁	1.49×10 ³
C ₁₃ H ₂₂ O ₁₅	4.06×10 ²	C ₂₂ H ₂₈ O ₈	4.62×10 ³
C ₁₉ H ₃₂ O ₁₀	5.30×10 ²	C ₁₅ H ₁₈ O ₁₄	4.08×10 ³
C ₂₂ H ₃₂ O ₈	5.90×10 ³	C ₂₀ H ₃₂ O ₁₀	5.97×10 ³
C ₂₀ H ₂₈ O ₁₀	1.53×10 ³	C ₁₇ H ₂₂ O ₁₃	5.10×10 ³
C ₁₈ H ₁₈ O ₁₃	4.49×10 ³	C ₂₁ H ₂₈ O ₁₀	4.25×10 ³
C ₁₉ H ₂₄ O ₁₂	1.49×10 ⁴	C ₁₉ H ₂₂ O ₁₂	5.44×10 ³
C ₁₉ H ₃₀ O ₁₂	6.10×10 ²	C ₂₂ H ₃₄ O ₉	7.52×10 ³
C ₁₅ H ₁₈ O ₁₆	1.14×10 ³	C ₂₁ H ₃₄ O ₁₀	2.12×10 ³
C ₂₃ H ₃₈ O ₉	4.34×10 ²	C ₁₄ H ₂₄ O ₁₆	4.80×10 ³
C ₃₂ H ₄₄ O ₂	8.96×10 ²	C ₁₅ H ₂₂ O ₁₆	4.04×10 ³
C ₂₁ H ₃₆ O ₁₁	3.74×10 ²	C ₁₇ H ₃₀ O ₁₄	3.51×10 ³
C ₁₄ H ₂₆ O ₁₇	1.00×10 ³	C ₂₂ H ₃₆ O ₁₀	4.02×10 ³
C ₂₀ H ₂₆ O ₁₃	1.26×10 ⁴	C ₁₈ H ₂₄ O ₁₄	4.44×10 ³

$C_{22}H_{34}O_{11}$	1.92×10^3	$C_{19}H_{28}O_{13}$	6.68×10^3
$C_{20}H_{30}O_{13}$	9.36×10^2	$C_{20}H_{22}O_{13}$	3.90×10^3
$C_{18}H_{24}O_{15}$	2.05×10^3	$C_{21}H_{26}O_{12}$	4.48×10^3
$C_{21}H_{38}O_{12}$	9.16×10^2	$C_{22}H_{30}O_{11}$	2.29×10^3
$C_{24}H_{38}O_{10}$	3.78×10^3	$C_{15}H_{24}O_{17}$	4.70×10^3
$C_{16}H_{24}O_{17}$	1.26×10^3	$C_{25}H_{38}O_9$	5.24×10^3
$C_{21}H_{24}O_{14}$	4.80×10^3	$C_{17}H_{26}O_{16}$	5.18×10^3
$C_{20}H_{34}O_4$	4.98×10^2	$C_{21}H_{26}O_{13}$	4.82×10^3
$C_{18}H_{30}O_6$	2.74×10^3	$C_{22}H_{30}O_{12}$	2.47×10^3
$C_{18}H_{28}O_7$	1.53×10^4	$C_{16}H_{24}O_{17}$	5.16×10^3
		$C_{17}H_{28}O_{16}$	6.58×10^3
		$C_{29}H_{44}O_6$	5.82×10^3
		$C_{17}H_{30}O_{16}$	2.06×10^3
		$C_{22}H_{38}O_{12}$	3.86×10^3
		$C_{16}H_{32}O_{17}$	7.04×10^3
		$C_{23}H_{30}O_{12}$	1.26×10^3
		$C_{24}H_{34}O_{11}$	6.82×10^3
		$C_{20}H_{30}O_{10}$	4.14×10^3
		$C_{20}H_{32}O_{11}$	3.41×10^3

Reference

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