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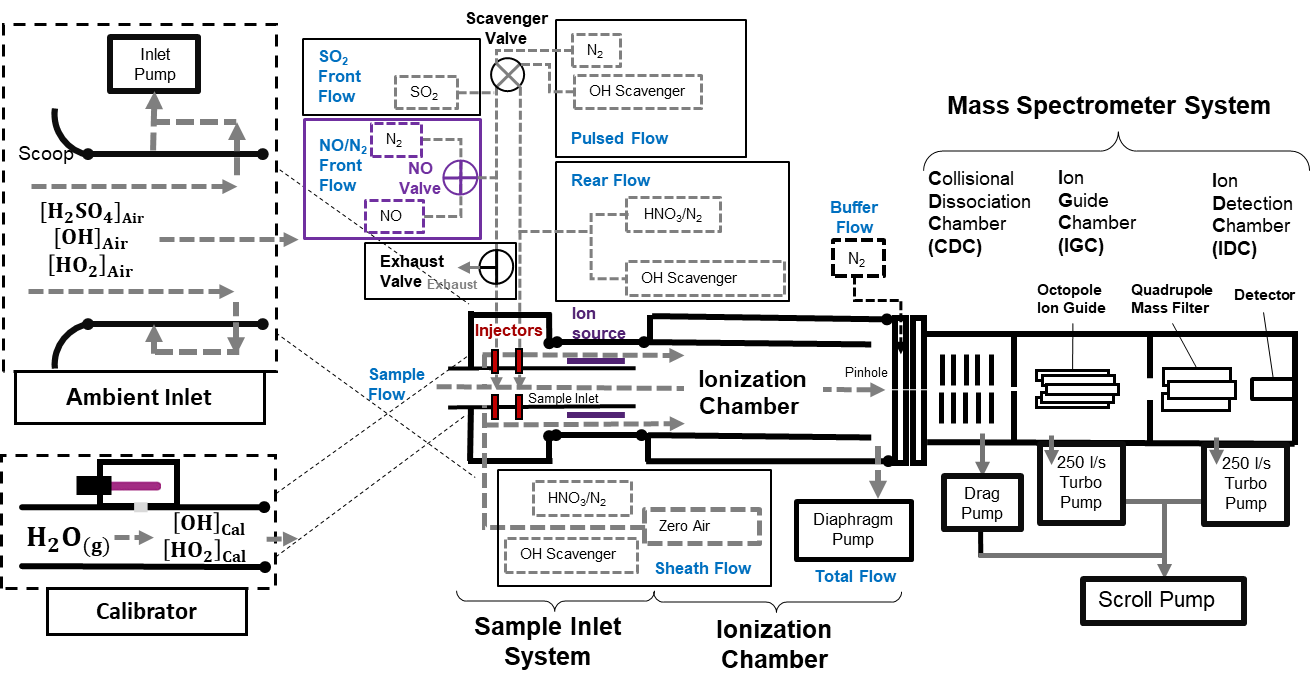
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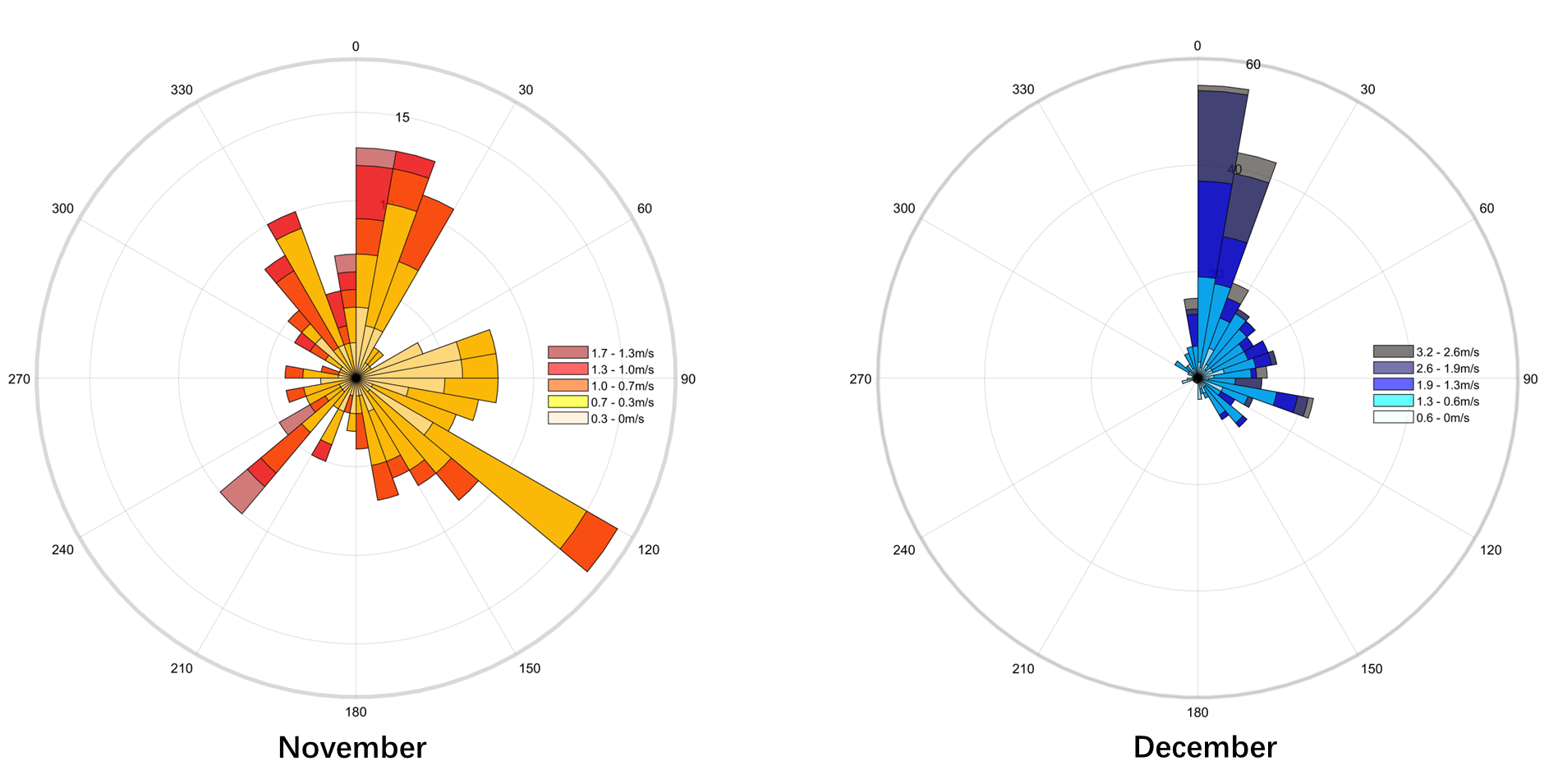
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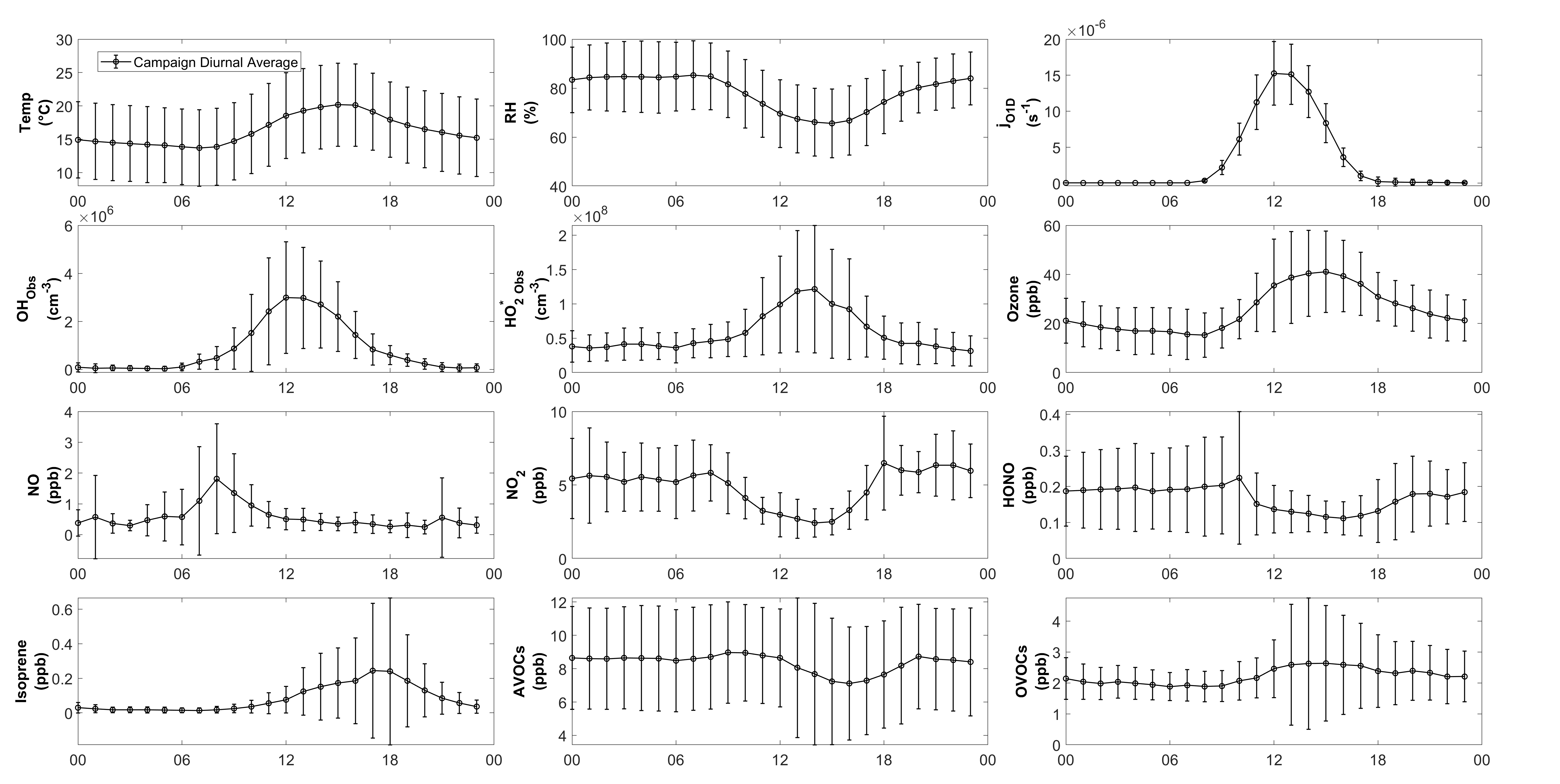
**Figure S1** Schematic of the ROx family’s photochemical pathway. Photolysis reactions are highlighted in yellow, reactions contributing to secondary aerosol production are marked in brown; and reactions associated with photochemical pollution are indicated in purple. The chemical reactions (R1 to R21) referenced in Table S1.

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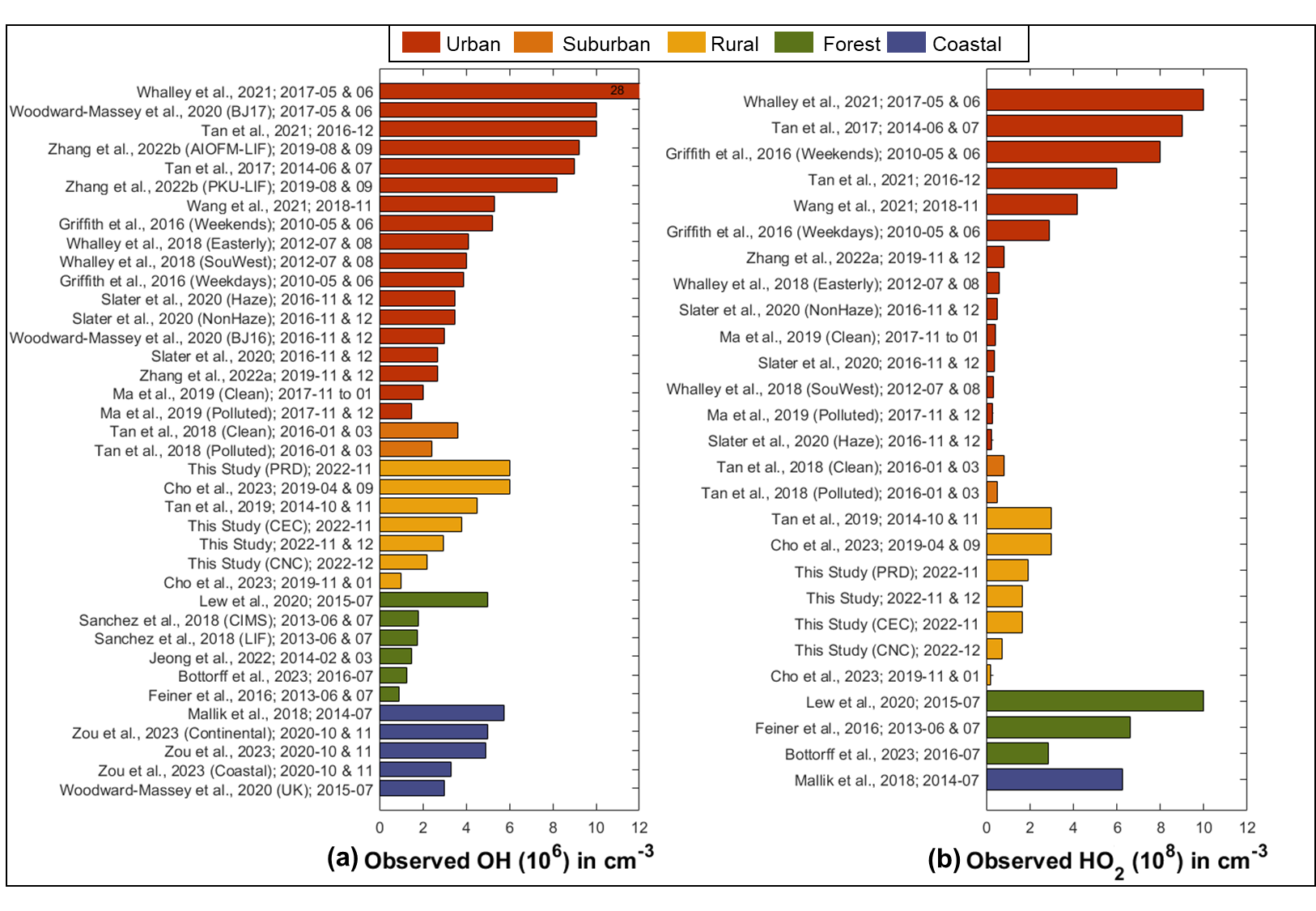
**Figure S2** Schematic diagram of the PolyU-CIMS system. The CIMS is composed of two detachable components: the ambient inlet and the calibrator; and the main body, which includes the sample inlet system, ionization chamber, and the mass spectrometer system. The frames labeled in purple highlight the additional valve incorporated for HO2 measurement. Further details on setup, measurement principles of the CIMS are available in a previous study (Zou et al., 2023) and Text S1.



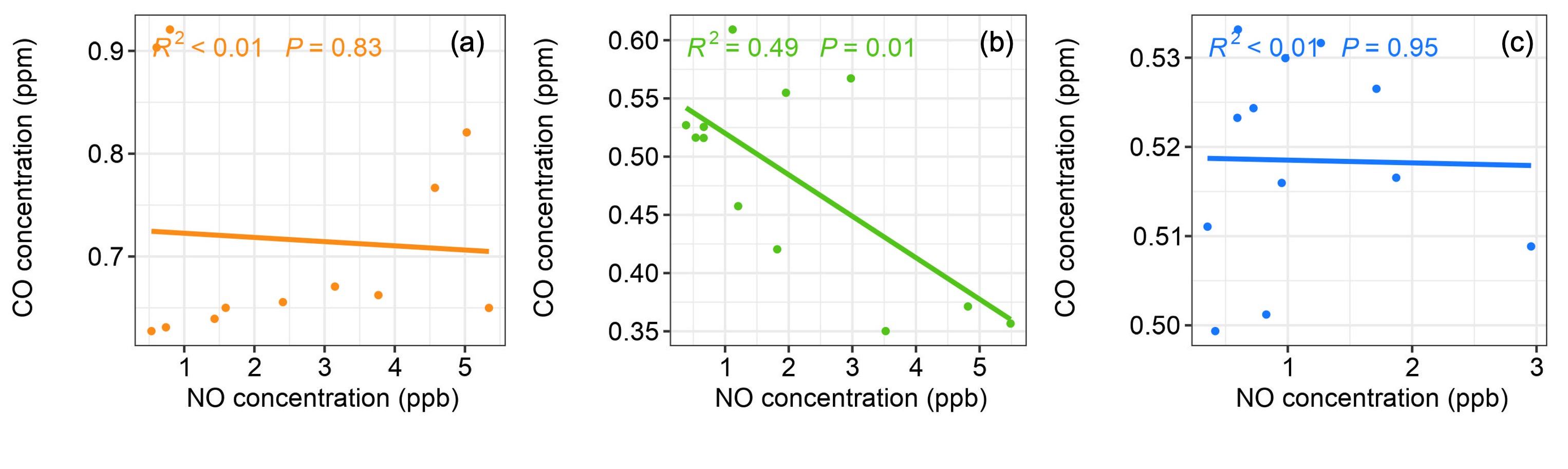
**Figure S3** The wind rose for November and December.



**Figure S4** Diurnal profiles of average concentration of HO2\*, OH, meteorological data and trace gases of the whole campaign. The shaded error bars represent standard deviations of the averaged data.



**Figure S5** Typical daily averaged maximum concentration of (a) OH and (b) HO2 observed in various geophysical regions, including coastal (blue), forest (green), rural (yellow) and urban (red).



**Figure S6** Relationship between NO and CO concentrations from 7:00 to 10:00 in (a) PRD, (b) CEC and (c) CNC. The solid lines depict the linear regression fits, with the corresponding equations R2 and p-values annotated on the plot.

**Table S1** The HOX related reactions in the model.



**Table S2** Summary of instruments used, and species measured during the field campaign.



**Tabel S3** Configurations of the PolyU CIMS in Hok Tsui 2020 and Conghua 2022 campaigns. The changes from the last configuration were labelled by red color.

Notes: B/S Ratio - background to signal ratio.



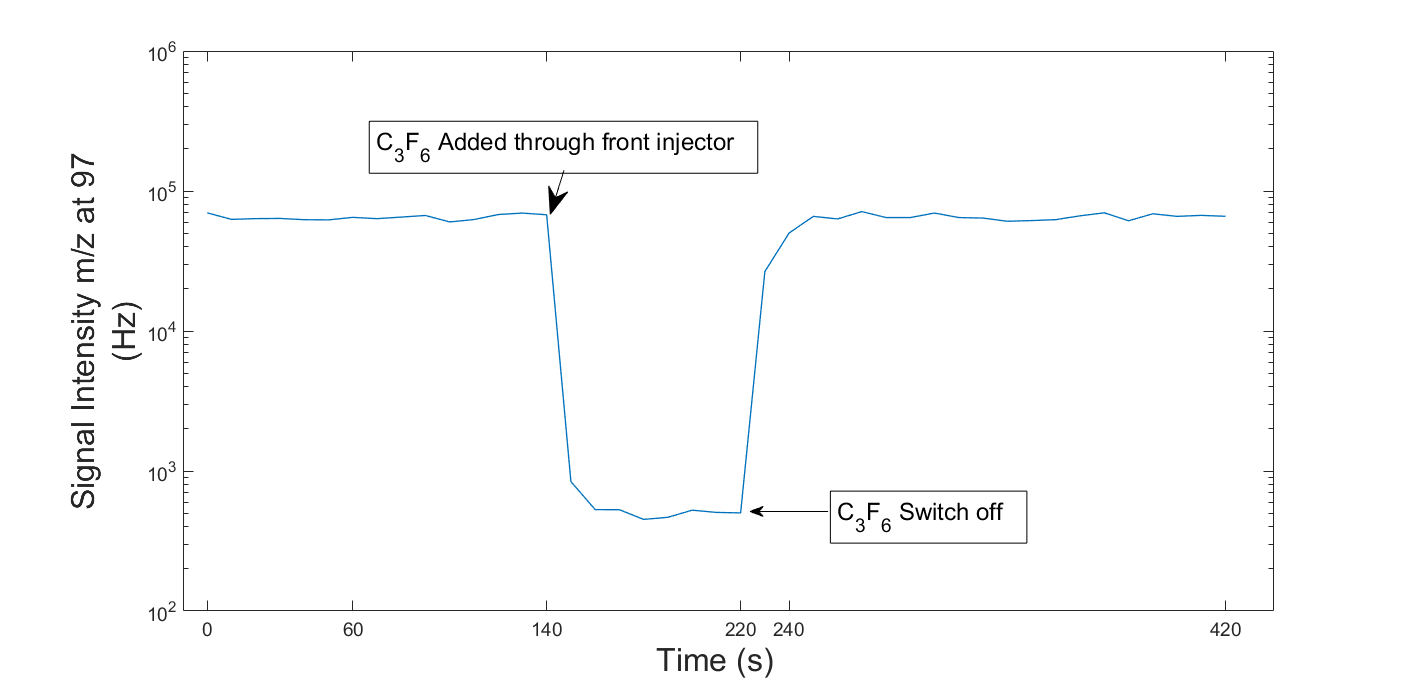
**Table S4** Average mixing ratios (ppbv) and standard deviations of measured VOCs that are constrained in the box model in the entire campaign and in different cases.



**Text S1 The Background mode and scavenge efficiency**

In the background mode, the scavenger gas C3F6 is introduced into the sample flow along with SO2. Since the concentration of C3F6 is 100 times higher than that of SO2, ambient OH and any HO2 converted to OH are scavenged by C3F6, rather than converted to H2SO4.

To determine the amount of C3F6 needed to achieve complete OH scavenging, we gradually increased the C3F6 concentration added to high levels of OH and HO2 ([HOx] ≈ 109 cm-3) generated from the calibrator in synthetic air until no further reduction in the measured signal, which indicates complete scavenging of OH. This point defines the background noise, which is attributed to any Criegee intermediates and ambient sulfuric acid. In our setup, residual C3F6 is present after the CIMS switches from background to signal mode, but it does not affect the measurement results. As shown in Figure S7, after switching off C3F6, the measurement signals rapidly return to their initial levels within 20 seconds. Data affected by residual C3F6 is excluded to minimize its impact on the measurements.



**Figure S7** Variation of signal intensity m/z at 97 before C3F6 addition with time, after addition and switching off of C3F6 in synthetic air containing OH of ~5×108 cm-3.

**Text S2 Calibration procedures**

The calibration of the Chemical Ionization Mass Spectrometer (CIMS) involves the generation of OH and HO2 radicals through photolysis of water vapor by 184.9 nm light, as outlined in Reaction R30. The concentration of radicals produced during calibration is determined from the known concentration of water vapor [H2O], which is calculated from water vapor pressure, the relative humidity and temperature. Other essential parameters include the photolysis cross-section of water vapor (𝜎𝐻2𝑂 = 7.14 × 10-20 cm2; Cantrell et al., 1997), the photolysis quantum yield (, assumed to be 1, Kürten et al., 2012) and the photon flux (*It* value, see details about *It* value determination on Kürten et al., 2012). The generated radical concentrations ([OH] and [HO2]) are calculated using the following equation:

From these values, the calibration factors for OH and HO2 (COH and CHO2) are calculated using the signals obtained during calibration (SOHcal and SHO2cal), as expressed in the transformed form of Equations E1 and E2:

(E1, transformed)

(E2, transformed)

The calibrator produced OH and HO2 concentrations in the range of 3 × 107 to 1 × 109 cm-3 depending on RH conditions in 10 LMP synthetic air. More detailed information on calculation procedures is given in our previous study (Zou et al., 2023).

**Text S3 Measurement interferences**

**3.1 Sampling Loss**

Wall losses in the ambient inlet were evaluated by varying the distance between the calibration lamp and the inlet to assess potential signal attenuation. The instrument was calibrated in two distinct configurations: initially, the lamp was positioned close to the CIMS sample inlet (Figure S6a), and subsequently, moved away from the CIMS sample inlet (Figure S6b). By comparing the observed signals from these two configurations, we were able to calculate the wall losses associated with the ambient inlet. The results indicated no significant difference (<1%) between the two measurements, suggesting negligible wall losses in the sampling system.

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**Figure S8** Calibration process during ambient sampling in (a) close and (b) far positions.

Furthermore, the potential for radical-radical loss after the radicals exit the calibrator and enter the sampling inlet was considered. Given the flow speed of the ambient inlet (12.2 m/s) the sample inlet (55 cm/s), along with the short distances involved—the calibration lamp is less than 1 cm from the sample inlet, and the sampling port to the front injectors is 1 cm—the calculated transport time for radicals to reach the front injectors is less than 20 ms. This short transport time is sufficiently short to prevent significant radical-radical losses. Additionally, since the sample inlet draws from the central part of the airflow within the ambient inlet, and the flow is laminar, wall losses at this stage are also considered negligible.

**3.2 OH interference**

The OH interference in the PolyU-CIMS, resulting from ambient NO + HO2 recycling (R11) and ionization process (R24, artificial OH), was accounted for and included in the measurement uncertainty, as outlined by Zou et al. (2023). To assess and correct this interference, model simulations were conducted. Initially, we constrained all measured species (except OH) in the model, and a three-day spin-up was used to simulate the chemical conditions of the sampled air during measurement. Subsequently, the outputs (except OH) were utilized as inputs for another simulation that included the injection of 12 ppm SO2, to simulate chemical reactions in the inlet with reaction time of 47 ms - matching the reaction time in the CIMS. Photolysis frequencies were maintained at zero to simulate the dark environment of the inlet. The modeled H2SO4 concentrations reflect interference from NO + HO2 reactions, with an average value of 2.91 × 104 and range from 104 to 105 cm-3throughout the whole campaign. These calculated interferences were subtracted from the ambient results.

In this study, the PolyU-CIMS encountered additional interference from residual NO in the injectors when switching from NO (used for HO2 measurement) to N2 (used for OH measurement). To prevent residual NO buildup, the inlet was cleaned daily, and a one-hour calibration was performed at both the start and end of day’s measurements to monitor NO residuals. Monitoring results showed that the NO residual time for the PolyU - CIMS was approximately 26 mins, similar to values reported in earlier studies (Edwards et al., 2003; Sjostedt et al., 2007). Consequently, data collected during the residual period (30 mins after switching the measurement target from HO2 to OH) were discarded to eliminate NO residual interference from the final dataset. Details about how the duty cycle was configured to monitor NO residual interference are provided in Text S5.

**3.3 HO2 interference**

The concentration of injected NO is the primary source of HO2 measurement interference in this study. High NO concentrations convert ambient RO2, particularly alkene and aromatic-related RO2, into HO2 and then OH, leading to a positive bias in HO2 measurements (Fuchs et al., 2014). To assess HO2 interference, we conducted model analysis using the same settings as those used for OH interference, including observational data input (except HO2\* and OH), reaction time, and dark conditions, while adjusting specific parameters to address HO2-related reactions. The modeled ambient RO2 concentrations (blue line in Figure S9a) were used as inputs for additional simulations assessing RO2 interference with HO2 via reaction with added NO (1.2 ppm) and SO2 (12 ppm) in the CIMS inlet. The H2SO4 concentrations resulting from the model simulation represent the interference in HO2 measurements attributed to RO2 (blue line in Figure S9b). By comparing these results with the modeled H2SO4 concentrations (red line in Figure S9b) when modelled ambient HO2 concentrations (red line in Figure S9a) are constrained, we determine that the average daytime (08:00-16:00) RO2 interference was estimated to contribute 56%, 54%, and 59% of the HO2\* signal during the PRD, CEC and CNC case, respectively. Throughout the entire campaign, the contribution ranged from 44% to 69%. These results indicate substantial positive interference from RO2 in HO2 measurements under the CIMS configurations adopted in the field study.

We note that the MCM model has inherent uncertainties on RO2 chemistry, such as incomplete representations of RO2 species and the assumption that most of RO2 species share the same reaction rate constant with NO. In addition, HO2 was over-simulated by the model (Figure S9a). For these reasons, we use the modeled interferences only for sensitivity tests and qualitative discussion rather than correcting the measured HO2\*. Nonetheless, these values indicate the potential magnitude of positive interference in HO2\* measurements caused by RO2.

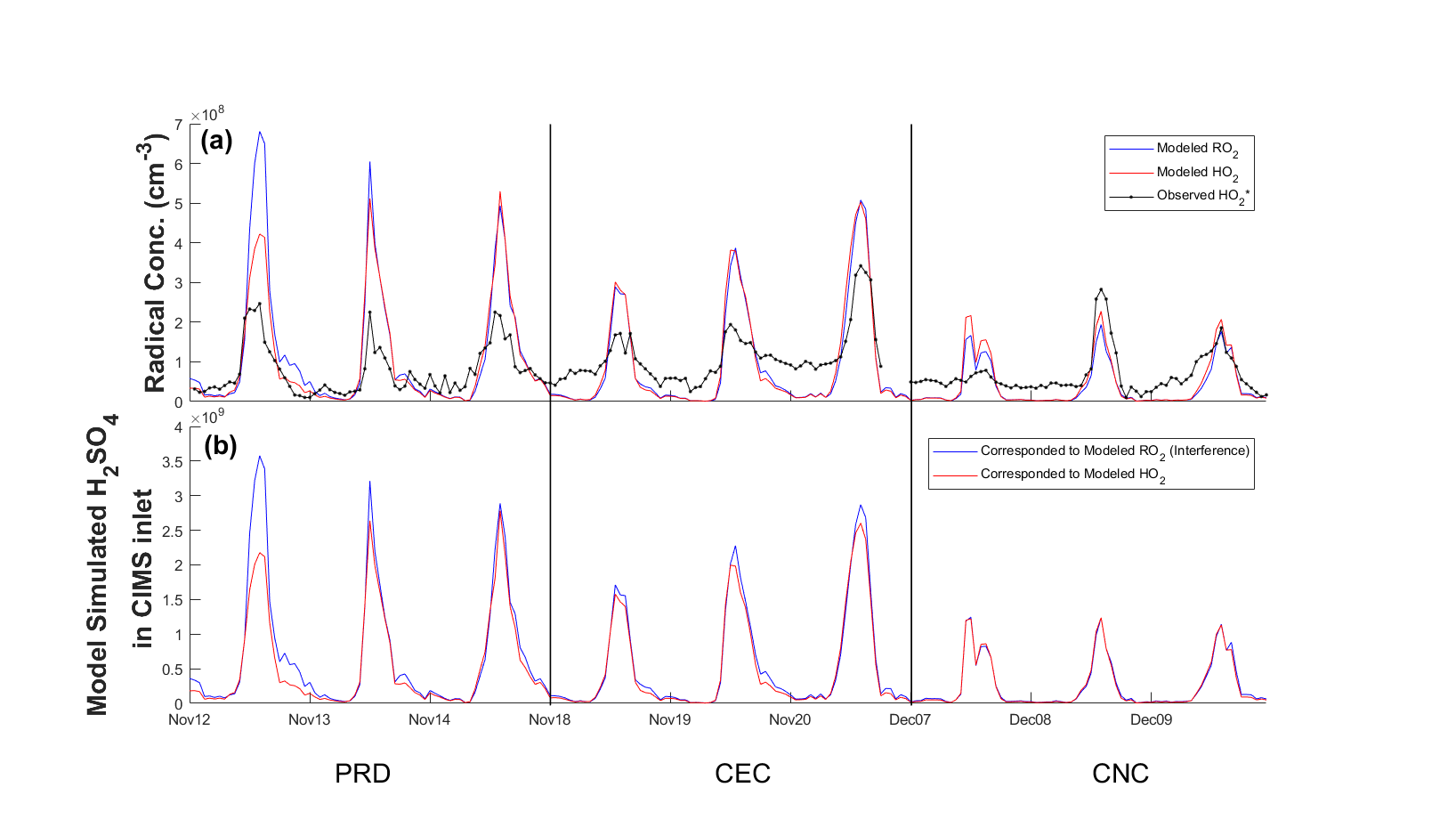


Figure S9: Modeled peroxyl radical concentrations and resulting H2SO4 simulations. (a) Temporal profiles of modeled RO2 and HO2 concentrations alongside observed HO2\* for the PRD, CEC, and CNC periods. (b) Simulated H2SO4 concentrations corresponding to RO2 and HO2 in the CIMS inlet for the same periods.

**Text S4 The measurement duty cycle of CIMS**

As detailed in Section 2.2, the PolyU-CIMS was configured to sequentially measure HO2, H2SO4, and OH within each hour during the field study, corresponding to changes in the injection gases. Table S3 outlines the hourly measurement schedule and associated injection gases, while Figure S10 illustrates the 1-hour duty cycle.

Table S5 Duty cycle and injection gases for targeted chemical analysis.



|  |
| --- |
| Notes: |
| Front and Rear Injectors - The injector pairs as demonstrated in Figure S2  SIG & BKG – the signal and background modes. |
| Sca - scavenger gas, C3F6 in this study. |
| Sca(p) - scavenger gas added through the pulsed flow. |
| N2(p)- nitrogen gas, added through the pulsed flow. |

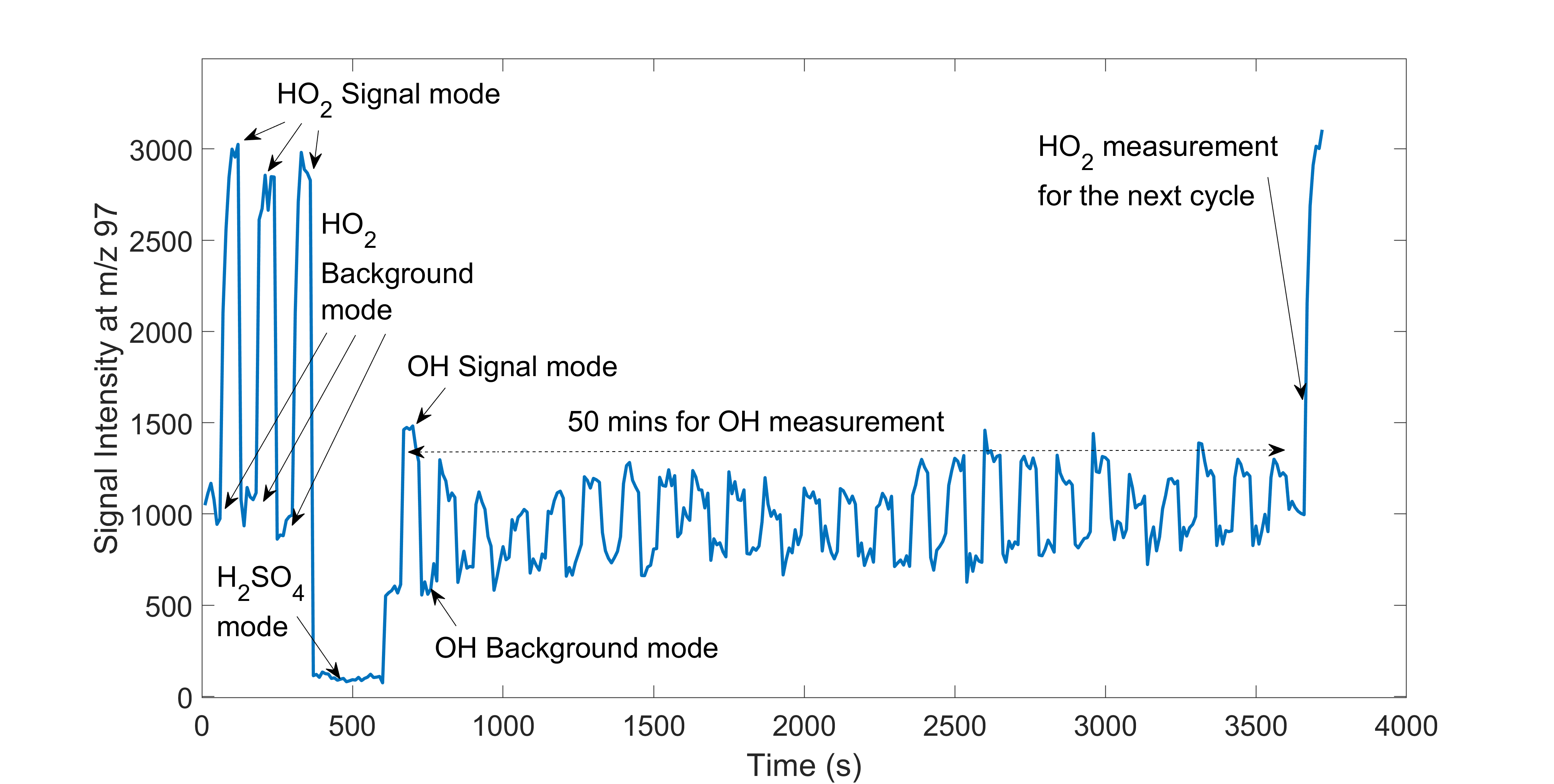


Figure S10 Variation of signal intensity at m/z 97 during a 1-hour duty cycle of CIMS measurement.

**Text S5 Modification for HO2 measurement**

To measure HO2, a valve was added to switch the injection gas between NO and N2 as indicated by the purple frame in Figure S2. When N2 is added, the CIMS operates in OH measurement mode. When NO is added to the sample flow, the CIMS switches to HO2 mode for total HOx measurement.

During HO2 measurement, ambient HO2 is converted by NO to OH. It should be noted that in HO2 mode, the increasing NO concentration can enhance HO2 conversion to OH (R11). However, excessive NO levels trigger the HONO formation when reacts with OH (R15), competing with the OH conversion process by SO2 (R21) and lowering the detection efficiency for OH. Consequently, the NO-to-SO2 concentration ratio is crucial for HO2 measurements. Sensitivity tests revealed an optimal [NO]/[SO2] ratio of 0.1 for the PolyU-CIMS, achieving 100% conversion of HO2 in the laboratory ([H2SO4]/[HO2] = 1), aligning with prior research recommendations (Edwards et al., 2003; Sjostedt et al., 2007). Because the concentrations of both SO2 and NO injected into the sample flow are maintained at levels over 100-1000 times higher than ambient levels, and the injection flow rates are fixed, the efficiency of the HO2-to-OH conversion remains stable and is believed to be complete.

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