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*Supplement of*

## **Measuring and modeling mercury in the atmosphere: a critical review**

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## Supplemental Information for

### Measuring and modeling mercury in the atmosphere: A critical review

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#### *Lumex*

Using an older Lumex 915 instrument the following was found to occur at UNR. After startup, the instrument baseline drifted downward asymptotically for 1 to 3 hours before leveling off at a stable value, after which a meaningful zero baseline value could be established. Laboratory tests show after stabilization, a mean blank concentration of  $-0.13 \pm 0.34 \text{ ng m}^{-3}$  could be maintained for at least 3 hours while operating in the internal zero reference mode, with no indication of baseline drift (linear regression coefficient of 0.00004). External source injections of GEM in zero air at calculated concentrations of 240, 120, and 60  $\text{ng m}^{-3}$  resulted in mean recoveries of 99.7, 97.9, and 95.9 %, respectively. If the recovery efficiency trend is extrapolated to 1  $\text{ng m}^{-3}$  the Lumex would under report by about 15%.

#### *Sheu and Mason (2001) methods and detailed results*

The filter pack system used consisted of in-series two Teflon filters in front of three cation exchange membranes. They assumed the first Teflon filter collected PBM and the second was a blank, the first cation exchange membrane collected GOM, the second was a blank, and the third kept GOM from diffusing from the pump. They slightly altered the mist chamber from the design of Lindberg and Stratton (1998).

The mist chamber collected ~34% more GOM than the cation exchange membrane. Sheu and Mason suggested this may be an artifact of the mist chamber in marine air. The denuders in general indicated higher concentrations than the filter pack measurements. They suggested this was due to an inherent blank problem or due to GEM being oxidized at the KCl surface. They tested whether there were reactions in the mist chamber when exposed to light and found this did not occur. PBM was also measured on Teflon filters and concentrations ranged from  $<9$  to  $147 \text{ pg m}^{-3}$ .

#### *Cation exchange membrane*

The original material used for the cation exchange membrane “ICE 450” manufactured by Pall has been discontinued and replaced with a polyethersulfone membrane (“Mustang S”). In laboratory and field tests collection of GOM in ambient air were not significantly different; however, in the field the ICE 450 in general had higher collection efficiency, although they were not significantly different, suggesting it may collect some forms of GOM that the Mustang S does not (Huang and Gustin, 2015). Recent field tests indicated there was no significant loss when surrogate surfaces and UNR passive sampler for GOM concentrations (box sampler) were deployed for 2 to 4 weeks (Huang and Gustin, 2015). In addition, use of an automated analytical system (Tekran 2600) for membrane analyses significantly improved the method detection limit (Huang and Gustin, 2015).

### ***TAM, TGM vs GEM comparison***

Limited field work has indicated that the Tekran® 2537 alone does collect all TGM with a soda lime and particulate filter in-line. This has not been adequately tested. Whether the soda lime captures and retains GOM is not known. If the sampling line is long and not covered, and the air is dry the Tekran 2537 could be measuring TGM. Temme et al. (2002) compared data from two Tekran systems in Antarctica, one measuring TGM using Tekran® 2537 only and the other using the Tekran® 1130/1135/2537 to measure GEM, GOM and PBM. They suggested that GOM and GEM were collected as TGM by the 2537. Based on 7 months of data from Mercury Deposition Network site MDN 98 in Nevada, Gustin (2011) found the sum of the GEM and GOM was more similar to TGM than GEM alone. This comparison was in an environment with relatively dry air and similar comparisons in humid air may yield more comparable concentrations since humidity can reduce GOM collected on surfaces to GEM. During RAMIX the 2537 operated by the University of Miami measured TGM (Figure SI 3).

### ***PBM detailed information***

Keeler et al (1995) used Teflon and quartz fiber filters to measure PBM in the Great Lakes area and showed PBM had a bimodal distribution. A paper by Kim et al. (2012) showed a bimodal size distribution with the strength of this distribution varying by season and location, and PBM was measured for 0.18 to 18  $\mu\text{m}$  using an eight-stage MOUDI (Micro Orifice Uniform Deposit Impactors, Midel M 100R, MSP Corp) with Zeflour filters (Pall corp.) and a cascade impactor with glass fiber filters. Particles across all size ranges were measured.

Keeler et al. (1995) reported 60-100% of PBM was found in fine particle fraction in Detroit. A significant fraction of PBM can be found in coarse particle at coastal sites (Engle et al., 2008; Malcolm et al., 2007). Kim et al. (2012) found PBM in the winter in Chuncheon and Seoul Korea were in the < 2.5  $\mu\text{m}$  fraction, however, in the summer, in Seoul, there was a constant distribution across all particulate sizes, and in Chuncheon, concentrations in the summer were higher in the 2 to 10  $\mu\text{m}$  fraction. This indicates the grain size fraction measured by the Tekran® is not adequate due to the need for an elutricator to keep particles of > 2.5  $\mu\text{m}$  from entering the denuder.

Rutter et al. (2008) used quartz fiber filters followed by KCl-coated quartz fiber filters in-series to collect PBM and GOM and found that concentrations on the first filter were higher,

and the latter comparable to that measured with a Tekran® system. Since work with the DOGHS instrument demonstrated that GOM was collected on quartz wool then some GOM was most likely collected on the quartz fiber filter. PBM measured by the quartz filter was 2.7 times higher than that measured by the Tekran® 1135. Since RM is semi-volatile (Rutter and Schauer, 2007), Rutter et al. (2008) suggested that since the particulate module is maintained at 50°C, RM could be lost. Rutter et al. (2008) suggested that evaporative loss could be augmented by the presence of other semi-volatile compounds such as NH<sub>3</sub>NO<sub>3</sub> or other semi-volatile organics. Malcolm and Keeler (2007) observed less PBM collected on quartz filters for 12 versus 4 h, and suggested a negative sampling artifact that could be due to reduction due to reaction with gases in the air (such as O<sub>3</sub>) or relative humidity.

Talbot et al. (2011) measured PBM with a Millipore® 1 µm fluoropore membrane to compare with data collected using the Tekran® 1135 unit and found the filter method collected 21% higher PBM and 85% of the data disagreed by 25% or more. In addition, the Tekran® measurement had a high percentage of data below the detection limit while the particulate filter (average blank concentration 25 pg and blank for the Tekran® measurement was 0 pg m<sup>-3</sup>) always had detectable Hg. The filter method was more responsive to pollution episodes associated with a combustion source. It is also possible the filters were collecting some GOM.

### ***Detailed discussion on the potential for different chemical compounds in the air in Florida***

There are several important points not presented in the Peterson et al., (2012) and Gustin et al. (2012) Florida Hg papers. First, in Peterson et al. (2012), the discrepancy between modeled dry deposition using a multi-resistance model, and measured deposition varied by location and across different seasons. This supports the contention that there are different compounds in air. At these sites modeled deposition was weakly correlated with measured deposition ( $r^2 = 0.24$  and  $0.28$   $p < 0.5$ , and  $r^2 = 0.16$   $p < 0.10$ ). This indicates at times there were GOM compounds in the air that the Tekran® system was more efficiently capturing, and at other times this measurement was less efficient, or the model was not accurately capturing deposition.

The constant model parameterization and similar environmental conditions across all sites (cf. Peterson et al., 2012) provides a basis for comparison of measured deposition across all sites. In all cases measured deposition was significantly higher (2 to 18 times) than modeled values (Figure SI 1).

At all sites there was a pattern in the ratio of measured:modeled deposition with a decline in the summer and increase in fall. Differences between the measured and modeled values were similar for all sites in the winter. The Davie Site near Fort Lauderdale (DAV) site had the lowest ratio of measured: modeled deposition during the other seasons, suggesting the denuder was more efficient at collecting GOM compounds at this location, or that the model was better at capturing deposition at this site. Ratios were highest at Tampa (TPA) in the spring and fall relative to the other sites. At Outlying Landing Field (OLF), the most background site, measured: modeled values were higher in the spring (time periods when long range transport occurs) and in the fall, a time of year when mobile source pollution would be higher. An alternate explanation it is possible that the model bias varies seasonally because of errors in meteorology, surface/land type, PBL height, etc. and this

could explain some of the variability in the measured:modeled ratio. These data demonstrate there are different forms of GOM in the air.

Data of Peterson et al (2012), presented in Figure SI 2, shows that the surrogate surface-derived deposition velocities are highest for the TPA site that is primarily impacted by mobile sources, and overlap for DVE (local point source impact and mobile source impacted), and were intermediate for OLF (most background site with a long range transport impact). Despite the fact these were calculated using the Tekran® GOM data, since there were similar conditions across the sites, this also indicates there are different forms in the air at these sites. Dry deposition measured by the surrogate surface will be influenced by the deposition velocity or the chemical compound. Lin et al. (2006), using the model of Wesely (1989), reported the deposition velocity of HgO was 2-fold higher than that for HgCl<sub>2</sub>. Thus, if there are different forms in the air, data collected by the surrogate surface will vary across space and time.

If we assume:

1- the cation exchange collects GOM quantitatively then the collection efficiency of KCl-coated denuder (in a Tekran 1130) for different GOM compounds is HgBr<sub>2</sub> (1.6)>HgSO<sub>4</sub> (2.3)=HgCl<sub>2</sub> (2.4)>HgO (3.7) >Hg(NO<sub>3</sub>)<sub>2</sub> (12.6) (Values in parenthesis represent concentrations measured by the cation exchange membrane: concentrations reported by the Tekran® denuder using the laboratory manifold system in zero air); and

2- based on the work presented in Gustin et al. (2012) that deposition associated with natural background is 0.23 ng m<sup>-2</sup> h<sup>-1</sup> (corrected by adding 0.2 ng m<sup>-2</sup> h<sup>-1</sup> due to the lack of the need of the correction factor (Huang and Gustin, 2015); deposition associated with oxidation of GEM due to mobile source emissions at TPA, DAV, and OLF was 0.3, 0.3, and 0.23 ng m<sup>-2</sup> h<sup>-1</sup>, respectively; deposition from long range transport was 1.0 ng m<sup>-2</sup> h<sup>-1</sup> at all sites in the spring; and deposition at DVE associated with direct emission or formation due to associated oxidants was 0.3 ng m<sup>-2</sup> h<sup>-1</sup>, there could be different sources associated with each specific source. For example, natural background could be due to reactions with Br and perhaps S compounds; deposition associated with mobile sources could be Hg-O or Hg-N associated compounds; that due to long range transport in the spring could be HgO related or HgBr<sub>2</sub> compounds; and that associated with local point sources could be, Hg-S, Hg-O or Hg-N related compounds (cf. Timonen et al., 2013). Since collection efficiency for HgSO<sub>4</sub> is relatively good, this might explain the better agreement between the measured and modeled deposition at DVE since this site is impacted by oil electrical generation facilities. These ratios could also be influenced by ozone and humidity (see Lyman et al., 2010; McClure et al. 2014; Huang and Gustin, this issue, and discussion below). The atmospheric constituents at all sites are more complicated than that in the laboratory manifold (discussed below). Recent work by Huang et al. (this issue) based on thermal desorption of the nylon membranes, at OLF there are different compounds depending on the time of the year.

### ***Detailed discussion of RAMIX data***

Ambient air RM concentrations measured during RAMIX by the DOHGS were typically higher than the Tekran® derived RM. Although data are noisy, RM was typically higher as measured by the DOHGS at midnight to late morning (up to 350 pg m<sup>-3</sup>), relative to the Tekran® system. At 12:00, values measured by the DOHGS compared with the Tekran® systems were higher by ~75 pg m<sup>-3</sup>. The variation in the discrepancy between DOHGS and Tekran® RM measured over 24 h points to different GOM compounds in the air and production of GOM in the manifold.

Another piece of evidence for different GOM compounds in the air and indicates fast reactions that were hypothesized to occur in the manifold, is presented in Figure SI 3 from Gustin et al. (2013). The timing of the hourly peaks in GOM as measured are similar to those observed for ozone (one at 1200 h and another later in the afternoon). The first is attributed to mixing of air pollution to the surface from the free troposphere. The second is due to a reactions associated with local pollution (see Gustin et al., 2013). This latter could be due to the reaction suggested by Calvert and Lindberg (2005) for Hg and O<sub>3</sub>:

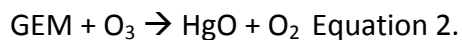
GEM + O<sub>3</sub> → HgO<sub>3</sub> (unstable) → OHgOO → O<sub>2</sub> and HgO. Equation 1.

They also indicated that HgO<sub>3</sub> would deposit on aerosols and other surfaces where it may form HOHgOH, and this state Hg could be transformed into a variety of Hg (II) compounds with other ions. For additional discussion of atmospheric chemistry see the discussion below.

Thus, the RAMIX experiment demonstrated that the Tekran® KCl-coated denuder measurement for GOM was biased low, there were different chemical forms in the air, and reactions were occurring in the manifold (Gustin et al., 2013). The latter has implications for reactions that could occur in ambient air. Although there were some limitations with each method applied during the experiment, the project also demonstrated: 1) using GEM spikes, measurements made by two carefully calibrated Tekran® 2537 units could significantly differ and the only way to explain this was a discrepancy between the instruments; 2) GOM was significantly underestimated by the denuder (17 to 24% recovery of HgBr<sub>2</sub> spikes in ambient air in Reno); and 3) the DOHGS measured different RM forms than the Tekran® KCl-coated denuder.

### ***Detailed discussion of atmospheric chemistry***

Hynes et al (2009) summarized the laboratory work that has been done on the Hg + O<sub>3</sub> reaction by Hall (1995), Pal and Aryia (2004a and b), Summer et al.(2005), Rutter et al. (2012). All of these studies found O<sub>3</sub> to participate in a reaction with GEM. Based on an analyses of the electronic configuration, Tossell (2003), and Shepler and Peterson (2003) indicated that HgO is a weakly bound molecule. Hynes et al (2009) calculated a rate coefficient for the following equation:

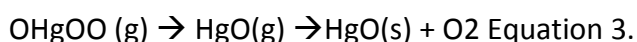


They indicated the rate coefficient was unreasonably high and suggested this reaction was unlikely to occur by homogenous process. Hynes et al (2009) noted that if the Hg + O<sub>3</sub>

reaction does exist it must occur as a weakly bound complex; and “In laboratory experiments such a complex could diffuse to the reactor surface to form solid mercuric monoxide, possibly by way of oligomer formation. Such a process would be energetically favorable because of the exothermicity of oligomer formation and the lattice energy associated with formation of the solid.”

Peterson et al (2009) indicated that the rate coefficient for O<sub>3</sub> production developed by Pal and Ayria (2004a) was sufficient to produce 5 to 10 pg of HgO per h; and using the rate coefficient developed by Pal and Ayria (2004b) and a concentration of OH of 1 x 10<sup>6</sup> molecules per cm<sup>3</sup> (Seinfeld and Pandis, 1996) approximately 1 to 10 pg of HgO could be produced per h.

Ariya et al. (2009) suggested that most atmospheric oxidation reactions occur by multiple steps complicating our ability to experimentally study and calculate reaction rates, and understand reactions. They proposed after the initial reaction between GEM and O<sub>3</sub> that the following occurred;



The last two steps are irreversible. Additional work by the Ariya group showed that the O<sub>3</sub> oxidation rate increases in the presence of CO and relative humidity (Snider et al., 2008). These point to mechanisms for producing HgO. For additional discussion of atmospheric chemistry see Open Discussion comments from Dr. Anthony Hynes.

#### ***Details on the sampling configuration in Weiss-Penzias et al. 2003***

For this study Weiss-Penzias et al., 2003 described GEM only concentrations being measured from May 2001 to May 2002 by a Tekran 2537; GOM measurements began in September 2001 by the 1130; and PBM was only measured at selected times using filters. For the GEM measurement the instrument was housed in a trailer with a black plastic covered line heated to 40°C provided by Tekran and a quartz filter to remove PBM and GOM. The quartz filter likely worked for PBM and possibly some forms of GOM. In addition a soda lime trap was also present in the sampling line and the location was not explicitly stated; and it is not known whether soda lime collects GOM. Once installed the 1130 was configured to collect a sample over 4 hours. Quartz filters were used to collect PBM over 27 hours on 47 mm quartz fiber filters. The samplers collected particulate matter < 2.5 µm, and 2.5 to 10 µm.

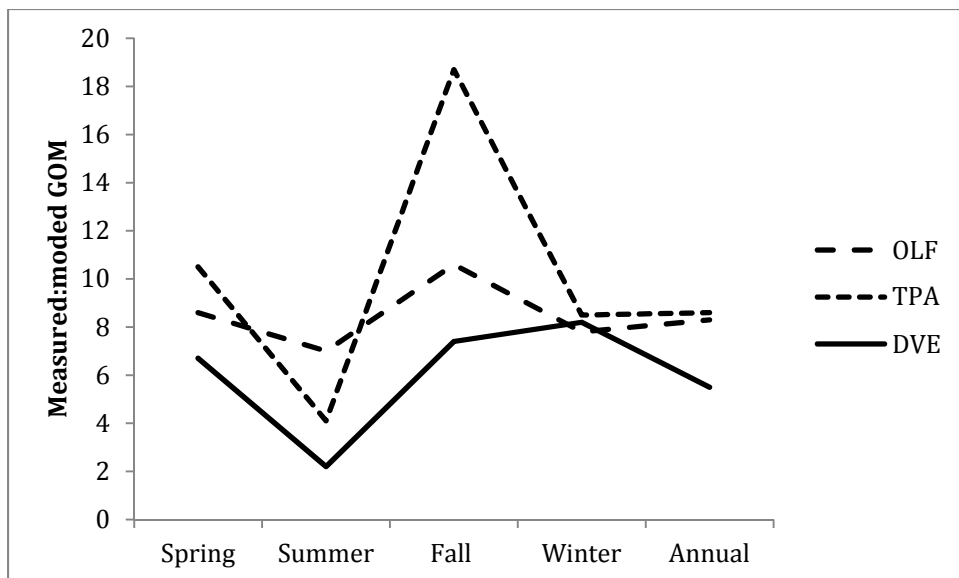


Figure SI 1. Ratio of surrogate surface measured (uncorrected dry deposition value-see section 3.2.2) to modeled values of GOM dry deposition for Spring (March-May ), Summer (June-August), Fall (September-November ), winter (December-January), and for the year. Data were collected from July 2009 to July 2010 at Outlying Landing Field (OLF) near Pensacola, a site just east of Tampa (TPA), and one near Fort Lauderdale (DVE) and the town of Davie (Data from Peterson et al., 2012).



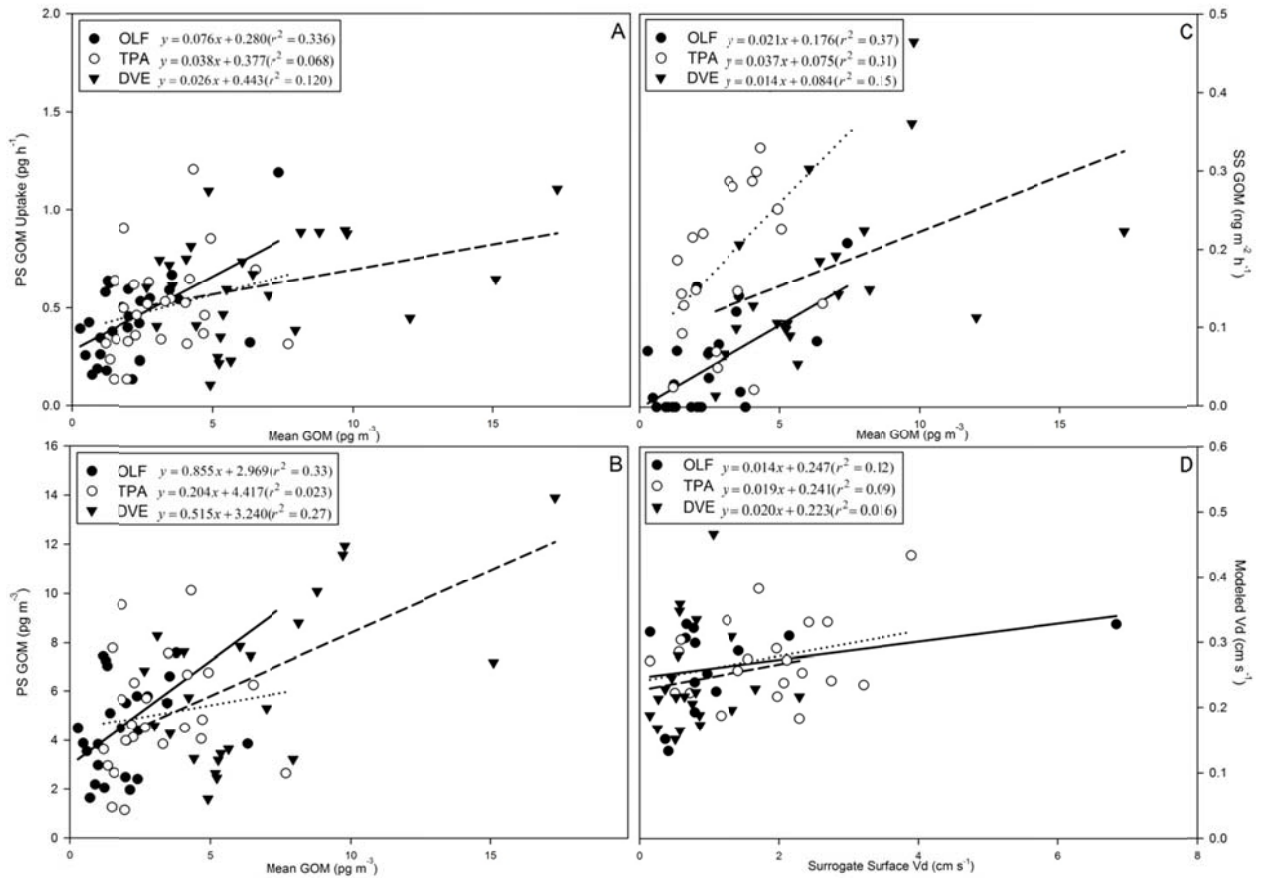


Figure SI 2. Figure 4 from Peterson et al (2012). Reprinted with permission from Peterson et al., 2012, Copyright 1 May 2012, Elsevier Limited.

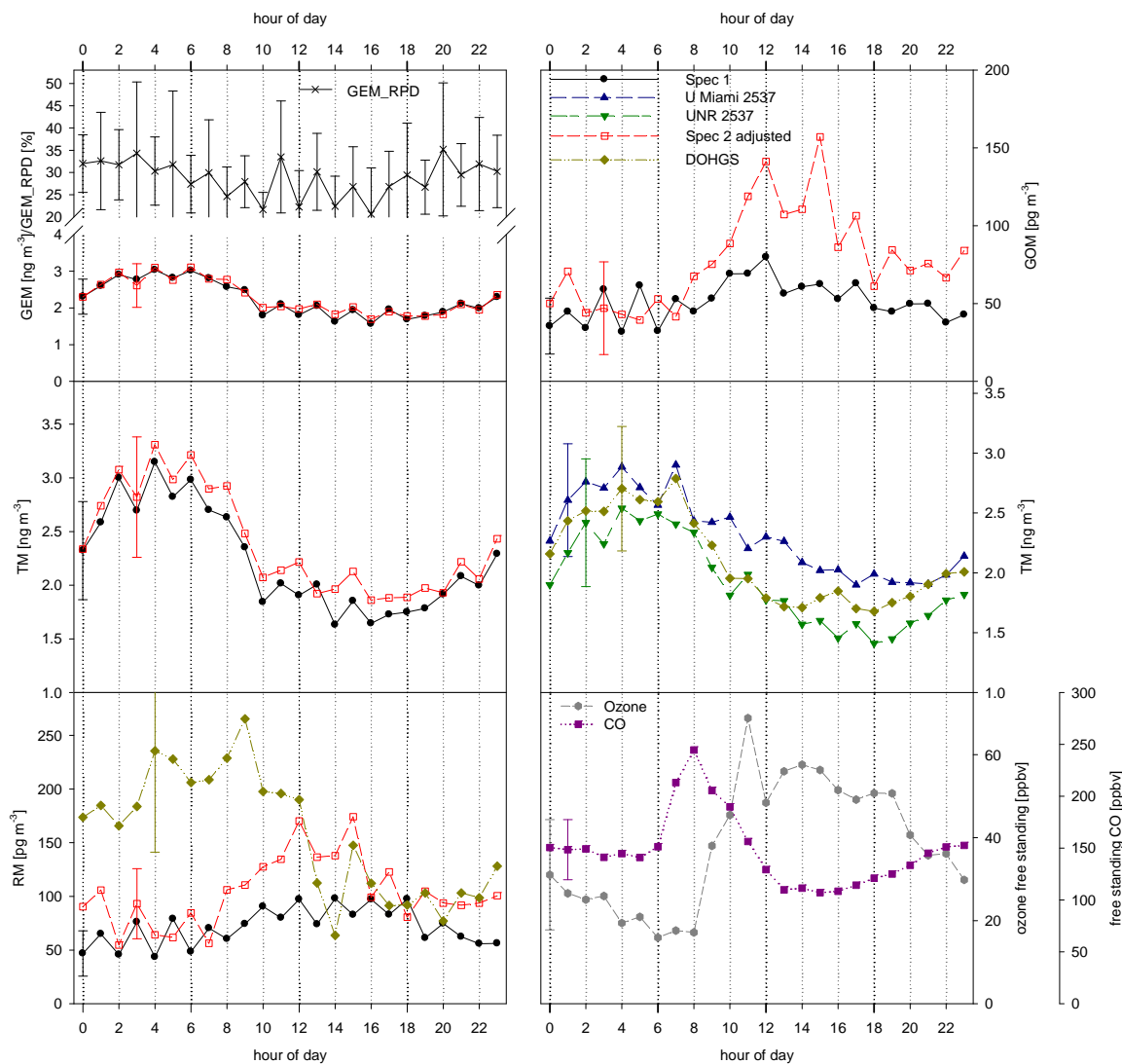


Figure SI 3. Figure 2 from Gustin et al (2013). Reprinted with permission from Gustin et al., 2013, Copyright 1 July 2013 American Chemical Society.

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