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Field inter-comparison of two high-accuracy fast-response spectroscopic sensors of carbon dioxide

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Abstract

Tunable diode laser absorption (TDL) and cavity ring-down spectroscopic (CRDS) sensors for atmospheric carbon dioxide were co-deployed during summer and fall of 2010 in the field at Los Alamos National Laboratory. Both sensors were characterized for accuracy and precision for ambient carbon dioxide measurements at ground level and are compared using both laboratory and atmospheric data. After a four point laboratory cross calibration, the mean $[^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}} = 392.05 \pm 8.92$ ppm and $[^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}} = 392.22 \pm 9.05$ ppm between 29 July and 16 August 2010 (mean difference = 0.04 ± 0.04 ppm). The slope of the cross-calibrated linear regression analysis between the two sensors is 1.000. The CRDS sensor is capable of measuring ambient $[^{12}\text{C}^{16}\text{O}_2]$ to a relative precision of 23 ppb Hz^{-1/2} for a 1-min time constant and this decreases to 6.5 ppb Hz^{-1/2} for a 58-min time constant. At one and 58-min time constants, the TDL exhibits precisions of 29 ppb Hz^{-1/2} and 53 ppb Hz^{-1/2}. The CRDS is compact, fast, and stable. The TDL is larger and requires frequent calibrations that limit its time resolution. Field observations show that 1-min averaged data measured by the two instruments agree almost perfectly, for the 19-day period the CRDS/TDL ratio is a Gaussian distribution at $x_0 = 1.000 \pm 3.38 \times 10^{-5}$. The sensors also exhibit consistent hourly averaged diurnal values underscoring the interplay of biological, anthropogenic, and transport processes regulating CO₂ at the site.

1 Introduction

Sensors based on optical spectroscopy are important tools for rapid, accurate in situ measurements of greenhouse gases for biosphere-atmosphere flux measurements and source attribution applications. Sensors using mid-IR and IR laser sources or high finesse optical cavities are the state of the art for continuously sensing greenhouse gases with precision(s) approaching that of isotope ratio mass spectrometry for isotopic

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analysis schemes (Powers et al., 2010; Karlon et al., 2010; Brown, 2003; Chen et al., 2010). Numerous laser-based sensors are undergoing rapid development to study greenhouse gases, thus it is important to conduct instrument inter-comparisons to establish their relative precisions under field conditions. The World Meteorological Organization/International Atomic Energy Agency recommends laboratory inter-comparison agreement of ± 0.1 ppm for $^{12}\text{C}^{16}\text{O}_2$ between operational techniques and further recommends the CO₂ mixing ratios be measured for dry gases (WMO, 2005). We inter-compare a commercially available cavity ring-down absorption analyzer (CRDS) with a tunable diode laser absorption (TDL) system for monitoring carbon dioxide [$^{12}\text{C}^{16}\text{O}_2$].

We conduct the study in both the laboratory and field settings to establish accuracy and precision for the two sensors. In the laboratory, the CRDS analyzer was run on the TDL analyzer's operational calibration protocol so both sensors measured the same standard gas in the laboratory. In the field, we compare the ambient carbon dioxide data sets obtained from the sensors obtained during a 19-day period in late summer 2010. For ambient measurements, the CRDS analyzer and TDL sensors were set up at the same field site and were run with their respective sampling protocol to get the best data possible from each sensor. Both the CRDS and TDL sensors are used throughout the climate and ecosystem research and environmental sensing communities and it is important to directly compare the results of laser-based optical absorption sensors operating via related principles but different technique to ensure data sets from either sensor are in agreement with references and each other. The purpose of this paper is to compare [$^{12}\text{C}^{16}\text{O}_2$] obtained operating the CRDS and TDL sensors under their optimal operational protocols.

2 Methods

The carbon dioxide sensors used in this study are a cavity ring-down analyzer (Picarro 1301-m, Picarro, Inc. CA, USA) (Crosson, 2008) and a TDL absorption sensor (TGA100, Campbell Scientific, Logan, UT). The CRDS sensor measures $^{12}\text{C}^{16}\text{O}_2$,

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CRDS data begins at 1 s (black trace) and at 15 s for the TDL (red trace). Using 16 h of data at $[^{12}\text{C}^{16}\text{O}_2] = 405.923 \pm 0.121$ ppm, we estimate the precision of the CRDS sensor to be $29 \text{ ppb Hz}^{-1/2}$ at 30 s integration and $23 \text{ ppb Hz}^{-1/2}$ at 60 s integration time. The same statistics for the TDL at 30 and 60 s integration time are $34 \text{ ppb Hz}^{-1/2}$ and $29 \text{ ppb Hz}^{-1/2}$. At 58 min (3500 s integration time), the precision of the CRDS sensor is $6 \text{ ppb Hz}^{-1/2}$ and the same statistic for the TDL is $53 \text{ ppb Hz}^{-1/2}$. Figure 2 shows the Allan variance plot for the CRDS (black trace) and TDL (red trace) sensors taken from the laboratory data set. We clearly see the stability of the CRDS sensor does not show a sharp “V” shape exhibited in Allan variance plots for the TDL but a slower transition from the so-called white noise to drift noise regions of the Allan variance plot (Werle et al., 1993). This behavior is similarly exhibited by the quantum cascade laser absorption spectrometer (QCLAS) methane sensors described by Tuzson et al. (2010). The CRDS sensor exhibits stability at considerably longer integration times than does the TDL sensor, the minimum detection limit ($6 \text{ ppb Hz}^{-1/2}$) is observed at 3500 s (58 min) signal integration time, opposed to 30 s ($23 \text{ ppb Hz}^{-1/2}$) for the TDL, which corresponds to two calibration cycles in its measurement protocol. The CRDS detection limit at 58 min is in close agreement with the prototype CRDS sensor from the manufacturer (Van Pelt, 2011), and is here independently verified.

3.2 Continuous ambient carbon dioxide monitoring

Both the TDL and CRDS sensor were housed in a laboratory at the Los Alamos National Laboratory Environmental Research Park for the ambient carbon dioxide inter-comparison study. The CRDS sensor was operated without in situ calibration for the 19-day study. The TDL CO₂ sensor measures ¹²CO₂ absorption near 2308.225 cm^{-1} , pressure and temperature in the TDL optical cavity were maintained at 15.0 Torr and 30 °C, respectively. The TDL sampling protocol calls for use of a Nafion dryer, ensuring that the sample measured by the instrument has similar partial pressure of H₂O as the calibration gases in the measurement cell. The TDL CO₂ analyzer requires frequent

calibration to maintain its stated precision. The sample stream was switched to both a high or low reference calibration gas for 30 s, then measured ambient $[\text{CO}_2]$ for 1 min. The first 15 s of data at each stage of this cycle is ignored to account for flushing and sample equilibration in the TDL optical cavity (Powers et al., 2010).

Both the CRDS and TDL sensors sampled ambient air from a single tube that was run out of the building to a small tower approximately 4 m above ground outside the building at the Los Alamos Environmental Research Park. The tube was connected to a manifold and the CRDS and TDL sensors sampled from the manifold continuously at 500 ml min^{-1} and 200 ml min^{-1} , respectively. The sensors were run independently of each other, using their own operational sampling protocols. We characterize the agreement between the two sensors for quantitative CO_2 measurement at ground level by comparing their temporal relationships; the linear regression between their temporal signatures, and the calculated ratio and difference for their response to ambient CO_2 for the 19 day observation period. The 1-min temporal response of both sensors to ambient CO_2 near Los Alamos, NM is shown in Fig. 3. The $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}'$ mixing ratio is shown on top, $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$ is shown on the bottom of the plot. The ambient $[\text{}^{12}\text{C}^{16}\text{O}_2]$ signal varies between 378 and 440 ppm. The diurnal variation is $\sim 60 \text{ ppm day}^{-1}$ at ground level. Linear regression analysis between $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}'$ and $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$ is shown in Fig. 4. The linear regression analysis of the 1-min averaged signal between the sensors yields $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}' = 1.00 \pm 3.5 \times 10^{-5} [\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$, with $R^2 = 0.96$ for the ambient data. The mean ratio calculated $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}' / [\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$ for the sampling period is 1.000 ± 0.005 . Also shown in Fig. 4 is a histogram of the ratio $[\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}' / [\text{}^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$ for the 1-min data. The peak of the gaussian fit to the histogram data is centered at $x_0 = 1.003 \pm 3.28 \times 10^{-5}$. We note the importance of synchronizing the time axis for proper inter-comparison between the two sensors. For example, on 16 August 2010 there was some drifting (both forward and backward) between the clocks on CRDS and TDL analyzers. The data had to be separated into periods that exhibited linear time relationships, analyzed for correlation

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separately, and subsequently concatenated. Originally the correlation analysis for 16 August showed $[^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}} = 0.979 \pm 0.008 [^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}} + (13.10 \pm 2.95)$, a y-intercept that is statistically different from zero (and indeed may be interpreted as a 13 ppm offset in $[\text{CO}_2]$). The data on 16 August was separated into AM and PM periods and time synchronized separately. This data was merged and re-analyzed to $[^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}} = 1.003 \pm 0.007 [^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}} + (3.67 \pm 2.6)$, which we interpret as a zero-intercept with respect to a quantitative $[^{12}\text{C}^{16}\text{O}_2]$ offset between the two sensors. Indeed we force a y-intercept $b = 0$ for in our final analysis because both sensors should respond to a sample absent of carbon dioxide with 0 ppm, similar to Tuzson et al. (2010).

3.3 Diurnal cycle of carbon dioxide

The cross-calibration and 1-min time resolution agreement between the data sets are robust factors underlying longer time averaged data to describe the diurnal pattern of the $[^{12}\text{C}^{16}\text{O}_2]$ atmospheric background signal. The hourly averaged diurnal pattern of CO_2 is an important statistic to understand local biogenic respiration/photosynthesis processes and effects transport (including anthropogenic CO_2) in the regional CO_2 background. Raw data was averaged to 1-h time constants for each hour of the day (0–23 h) for the 19-day ambient observation study to create hourly averaged diurnal $^{12}\text{C}^{16}\text{O}_2$ profiles. Table 1 shows the median diurnal $[^{12}\text{C}^{16}\text{O}_2]$ for each hour of the day between 29 July and 16 August 2010 from the CRDS (top trace) and TDL (bottom trace) sensor. Nightly increases (hours 0–6 and 20–23) in $[^{12}\text{C}^{16}\text{O}_2]$ (both in magnitude and variability) are due to respiration and daily (hours 7–19) uptake of $[^{12}\text{C}^{16}\text{O}_2]$ by photosynthesis is evident in data sets from both sensors. Ambient temperature is included in the third column. The fourth column in Table 1 shows the difference between the diurnal median $[^{12}\text{C}^{16}\text{O}_2]$ for each hour. The mean difference between the CRDS and TDL diurnal median values is much smaller (1.80 ± 1.50) than either of their variabilities (75th–25th percentile difference), 4.85 ± 2.40 and 5.17 ± 2.56 for CRDS

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between these sensors underscores their fast, quantitative [$^{12}\text{C}^{16}\text{O}_2$] capability for atmosphere-biosphere exchange and ambient carbon dioxide (ground, mobile, and flight) measurements. For 1-min data, the mean difference between [$^{12}\text{C}^{16}\text{CO}_2$]_{CRDS} and [$^{12}\text{C}^{16}\text{O}_2$]_{TDL} was 0.04 ± 0.4 ppm for dry gas [$^{12}\text{C}^{16}\text{O}_2$] measurement over the 19-day period, hence we demonstrate that the CRDS and TDL instruments are in agreement with the WMO/IAEA recommendation of ± 0.100 ppm for dry [$^{12}\text{C}^{16}\text{O}_2$], once they are cross-calibrated. Operational in situ calibration of the CRDS system is needed infrequently (especially for ground based sensing), but periodic calibration with high precision standards should be performed to ensure linearity of its response under ambient [CO_2] conditions. Over 12 months of operation, we have not observed non-linear behavior for either the CRDS or TDL sensors in a wide variety of applications. Both sensors provide valuable data for carbon dioxide monitoring and their additional data streams put the $^{12}\text{C}^{16}\text{O}_2$ data stream in different contexts. Inter-comparison for isotopic speciation sensors (e.g., $^{13}\text{C}^{16}\text{O}_2$ and $^{18}\text{O}^{12}\text{C}^{16}\text{O}$) should be investigated for appropriate sensors to compare their performance. Our study will be especially valuable for analysis of experiments where multiple high precision fast response instruments are measuring greenhouse gases and differences may need to be interpreted and diagnosed (Wofsy, 2011).

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Table 1. Hourly diurnal median $[\text{CO}_2]_{\text{CRDS}}'$, $[\text{CO}_2]_{\text{TDL}}$, ambient temperature ($^{\circ}\text{C}$), and the absolute value of $\Delta[\text{CO}_2]$ (CRDS – TDL) measured between 29 July and 16 August 2010 near Los Alamos, NM.

Hour	$[\text{CO}_2]_{\text{CRDS}}'$	$[\text{CO}_2]_{\text{TDL}}$	Temp. ($^{\circ}\text{C}$)	$\Delta[\text{CO}_2]$
0	398.20	399.41	19.6	1.21
1	399.50	400.98	18.3	1.30
2	400.15	401.18	18.0	1.02
3	401.28	402.30	17.7	1.02
4	402.54	401.53	17.3	1.01
5	401.77	403.41	17.0	1.64
6	404.10	398.86	16.3	5.23
7	399.10	393.67	17.0	5.41
8	393.37	390.47	17.7	2.90
9	391.07	388.55	19.0	2.52
10	388.97	386.19	21.0	2.78
11	386.35	384.21	22.3	2.14
12	384.35	382.59	23.7	1.75
13	382.67	382.89	25.0	0.22
14	382.66	381.94	25.3	0.72
15	381.97	381.75	25.7	0.22
16	382.13	381.96	26.0	0.17
17	382.28	383.62	25.7	1.34
18	383.23	386.25	25.7	3.02
19	391.12	389.83	23.5	3.71
20	391.23	393.85	22.3	2.62
21	394.66	394.06	21.0	0.60
22	394.34	393.83	21.0	0.51
23	394.13	394.04	20.0	0.09
Mean	391.55 ± 7.52	391.92 ± 7.66		1.80 ± 1.50

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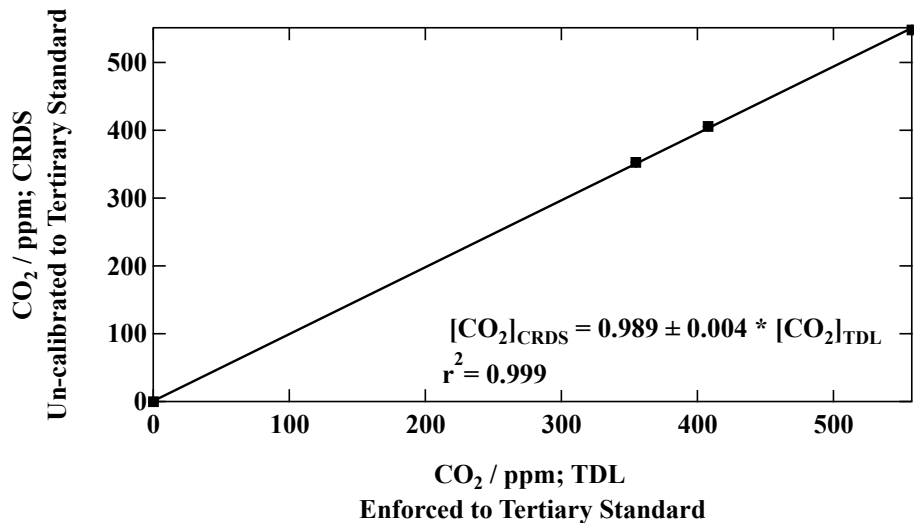


Fig. 1. Four point calibration plot of CRDS sensor with TDL tertiary standards showing linear range of calibration between 375 and 560 ppm CO₂.

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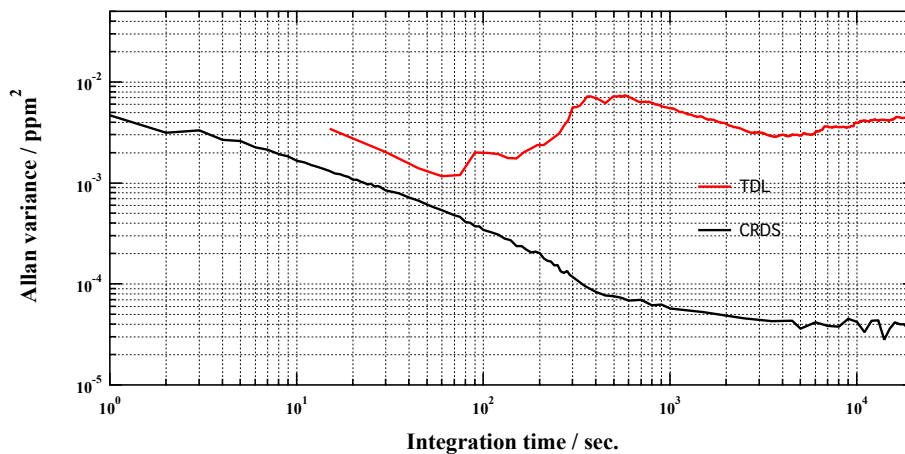


Fig. 2. Allan variance plot for CRDS sensor (black trace) and TDL (red trace) for 16 h of data shown as a log-log plot of signal variance vs. signal integration time. The minimum variance is observed at 58-min signal integration time for the CRDS sensor and at 60 s for the TDL sensor.

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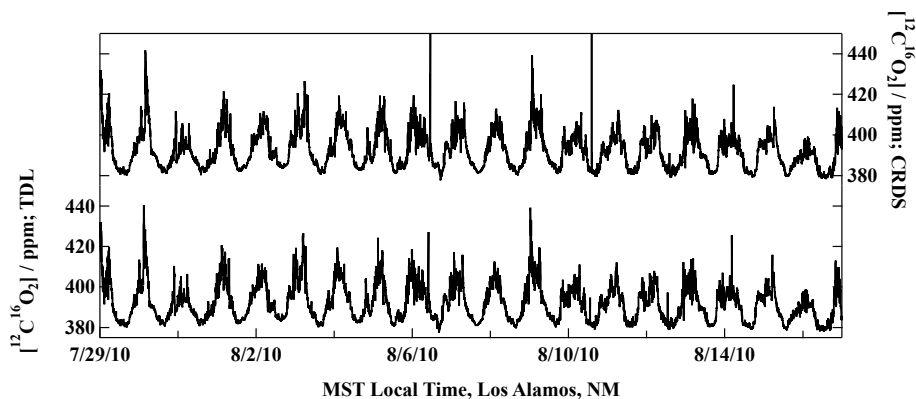


Fig. 3. Temporal profile of $[^{12}\text{C}^{16}\text{O}_2]$ mixing ratio measured near Los Alamos, NM with CRDS and TDL sensors. The CRDS signal has been cross-calibrated, as described in the text.

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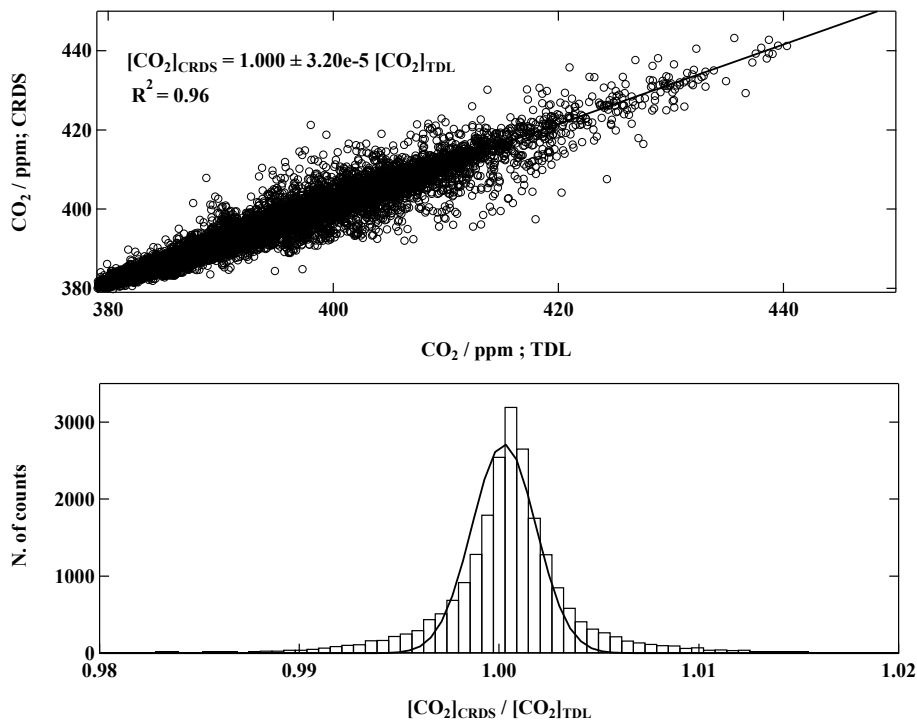


Fig. 4. Linear regression analysis of $^{12}\text{C}^{16}\text{O}_2$ measured with commercial CRDS and TDL analyzers after cross calibration using 1-min time resolution. The histogram plot of the ratio between the CRDS and TDL measurements and the gaussian fit of the $[\text{CO}_2]_{\text{CRDS}}/[\text{CO}_2]_{\text{TDL}}$ ratio is centered on an x_0 value of 1.003.

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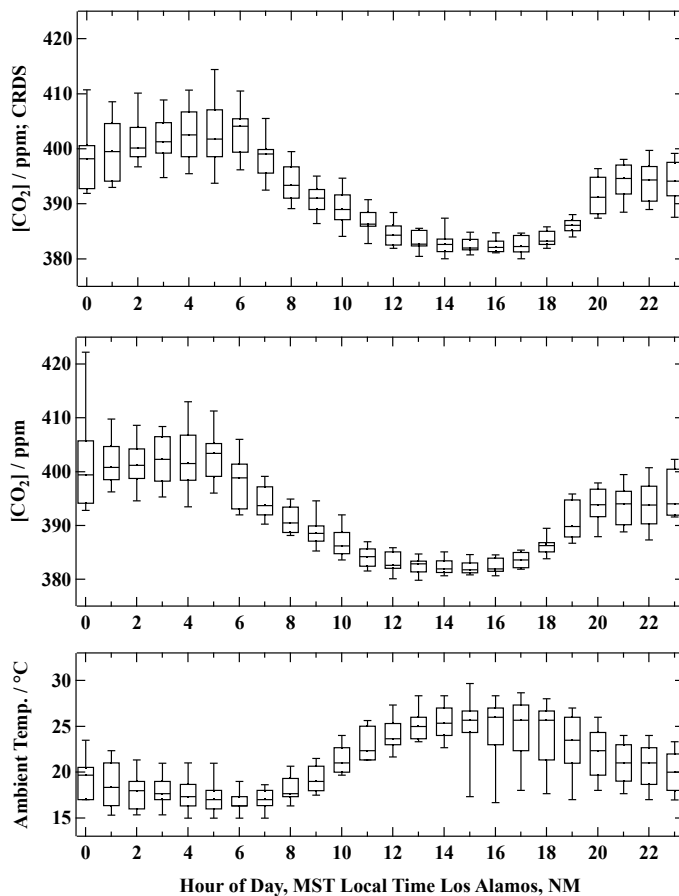


Fig. 5. Hourly diurnal median $[\text{CO}_2]$ between 29 July and 16 August 2010 near Los Alamos NM. The scaled CRDS $[^{12}\text{C}^{16}\text{O}_2]_{\text{CRDS}}$ trace is plotted on top, the $[^{12}\text{C}^{16}\text{O}_2]_{\text{TDL}}$ trace is plotted in the middle and hourly diurnal median ambient air temperature is plotted on the bottom trace.

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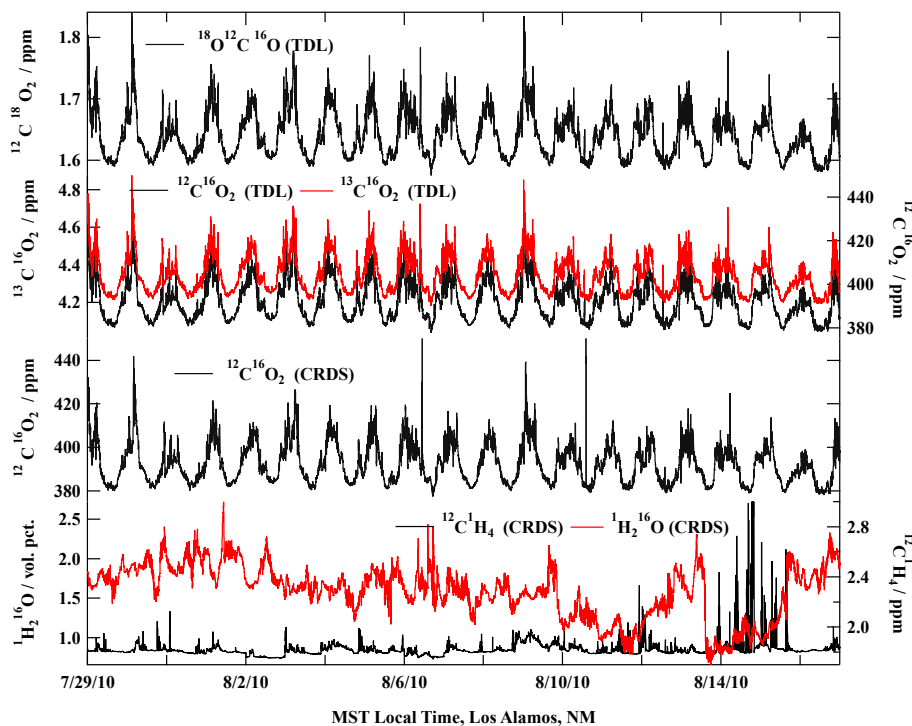


Fig. 6. One-minute traces for all signals available from both sensors used in the study. The $^{18}\text{O}^{12}\text{C}^{16}\text{O}$, $^{13}\text{C}^{16}\text{O}_2$, and $^{12}\text{C}^{16}\text{O}_2$ traces from the TDL are shown in the top of the plot while the $^{12}\text{C}^{16}\text{O}_2$, $^{12}\text{C}^1\text{H}_4$, and $^1\text{H}_2^{16}\text{O}$ traces from the CRDS are shown at the bottom. The $^{13}\text{C}^{16}\text{O}_2$ trace has been offset for clarity.

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