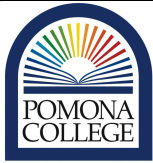


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|---|---|--|--------------------|
|  | Environmental Analysis Teaching and Research Laboratory | Date: 8/28/2018 | Number: 38 v.03MLH |
| | Standard Operating Procedure | Title: Greenhouse Gas Measurements w/Picarro | |
| | Approved By: Los Huertos | Revision Date: August 14, 2019 | |

1. Scope and Application

- 1.1** Covers how to install Eosense soil gas flux chambers (PN: 10089) and connect them to multiplexer (PN: 10197) and a Picarro G2508 (Santa Clara, USA) gas analyzer.
- 1.2** Originally, this SOP was developed for greenhouse gas emissions from strawberry fields in NorCal. It has been modified as new projects come on line that rely on these instruments.

2. Summary of Method

- 2.1** This SOP describes how to 1) set up the Picarro, Multiplexer, Dynamic Soil Chambers, and Vacuum Pump and 2) obtain and download data from the Picarro spectrometer computer.

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3. Acknowledgements

We thank the work of Isaac Medina, Neha Vaingankar, and Bailey Lai who worked with the instrument early on and developed early versions of the SOP.

4. Definitions

- 4.1 Picarro G2508 is the latest Picarro analyzer that measures N₂O concentration along with CH₄, CO₂, NH₃ and H₂O.
- 4.2 EosAC soil flux chambers (Eosense Inc, Dartmouth, Canada) is coupled with the eosMX-P multiplexer (Eosense Inc, Dartmouth, Canada). The eosMX multiplexer connects up to 12 eosAC chambers to the gas analyzer.
- 4.3 Recirculation pump (PN: A0702) provides the vacuum required for sample gas sequencing into and out of the Analyzer.

5. Biases and Interferences

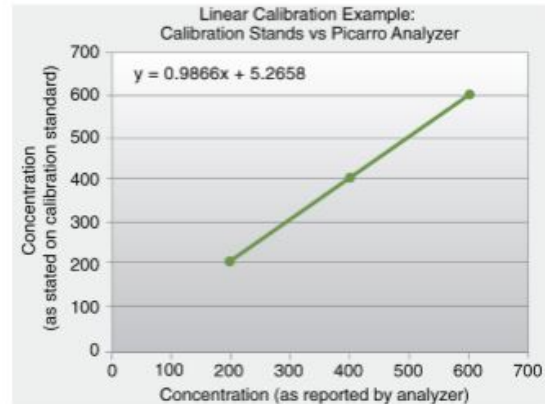
5.1 Biases and interferences

- Ambient temperature can prevent the analyzer from stabilizing properly.
- Trapping high moisture gas samples can cause condensation damage to the instrument. To prevent such damage, a flow of clean, relatively dry gas should always be directed through the instrument for several minutes prior to shutdown.

5.2 Calibration Changes

- The analyzer's calibration is intended to be done infrequently. It is only necessary to use three calibration standards to calibrate each gas or isotopic species (two points define the calibration line and a third intermediate point is used for verification).
- When calibrating, the exact value of each calibration standard is not of particular importance as long as they span a representative range of values over which the analyzer will typically be operated. It is reasonable to use a concentration of zero for the low calibration value, for example.
- To perform a calibration or verification of calibration, the user simply introduces the first calibration standard into the analyzer for an interval long enough for the analyzer to yield a stable measurement of that sample.
- The stated concentration of the calibration sample (a permeation tube or calibrated gas bottle, for example) and the value the analyzer reads for that sample are recorded for each calibration standard used.

| | Value given by analyzer | Value of calibration standard |
|----------------------|-------------------------|-------------------------------|
| Calibration point #1 | 200.1 | 202.7 |
| Calibration point #2 | 600.3 | 597.6 |
| Calibration point #3 | 400 | 400 |



- These values can then be plotted (in a spreadsheet, for example, as shown above) to determine the linear relationship between the known calibration values and the analyzers reported values.
- A linear best-fit equation can be calculated from the data. It is important to plot the analyzers reported concentration on the horizontal axis and the gas standards stated concentrations on the vertical axis.
- The slope and intercept of the best-fit line through these points are the two values that are used to calibrate the analyzer. By determining what the linear relationship is between the known calibration values and the analyzers reported concentration values in this way, a calibration offset (slope and intercept) can be calculated so as to add a correction term to the analyzers factory or previous calibration.

6. Health and Safety

Special care should be taken when using gas cylinders.

Safety and Personnel Protective Equipment

7. Personnel & Training Responsibilities

Researchers training to use the Eosense chambers and Picarro analyzer include the following components:

Researchers using this SOP should be trained for the following SOPs:

- SOP03 Field Safety
- SOP04 Electrical Power in the Field

Unanswered Picarro Questions

7.1 Set up tool / Data Logger setup: what is difference between dry and regular and timed Data columns

7.2 What is etalon temp (part of sensors in data source)

7.3 We need an SOP for calibration, including parts to order

7.4 Recommendations for car-mounting? Is okay to stack the multiplexer on top?

7.5 Is there are way to turn on computer without turning on the gas analyzer and vacuum?

7.6 What files do we need to send to marc for him to reprocess them.

7.7 N₂O is very noisy. Is there a way to reduce the noise?

Picarro Data Source/ sensors: allows you to see all sensor readings on picarro controller: zoom in/out= right and left mouse click

Forunner Multiplexer Questions

7.8 In the chamber data processor what does (L) or (E) mean in displaying fluxes for graphs?...for example Flux CO₂ (L) or Flux CO₂ (E)

7.9 In options for measurements chart in the data processor what is dead band range and what is chamber offset?

7.10 Are we supposed to measure chamber offset for each of the chambers we set out? (distance between bottom of chamber and soil due to collar)

8. Required Materials and Apparati

8.1 Picarro G2508 S/N 2143-JFAADS2048

8.2 Eosense (Forerunner) Multiplexer S/N ??

8.3 Chambers with Cables and Teflon Tubing (X meters in length)

8.4 In addition, we have identified the following customer support contacts:

- Picarro Tech support: Melissa, 408-962-3978
- Scott (408-962-3987)
- Karrin Alstad Applications — get calibration stuff from her. 408-962-3991
- Chance is pretty helpful at Eosense

8.5 Eosense (East Coast Time Zone)

- Customer support: 888.352.8313 (toll-free)

Table 1: EosAC soil flux chambers (Eosense Inc, Dartmouth, Canada)

| Chamber S?N | Filed ID # (2015-16) |
|-------------|----------------------|
| 1001501 | |
| 1001502 | 5 |
| 1001503 | 2 |
| 1001504 | 3 |
| 1001505 | 4 |
| 1001506 | |
| 1001507 | 1 |
| 1001508 | |
| 1001509 | |
| 1001510 | |
| 1001511 | |
| 1001512 | |

- Email: support@eosense.com

9. Reagents and Standards

9.1 Driorite

10. Estimated Time

- 10.1** Setting up the Picarro can take between 1-1 1/2 hours depending on your experience. Depending on the ambient temperature, the Picarro can take 30 minutes to reach operating temperatures. To be efficient, start the Picarro as soon as possible.

11. Sampling Design

- 11.1** The cable and gas tubing is XX meter.

- 11.2** If the generator is used, place it downwind at a minium of 100 feet.

12. Set Up

- 12.1** Connect pump to Analyzer VACUUM port using the convoluted metal hose. Hand tighten and use wrench to seal connection.

- 12.2** Connect multiplexer to Picarro through USB ports on Picarro (does not matter which port used)



Figure 1: Pump to Multiplexer

12.3 Connect monitor, mouse, and keyboard to Picarro via the remaining USB ports (note: there are two extra USB ports on the front of the Picarro)

12.4 Connect blue monitor plug to Picarro and monitor power plug to outlet.

12.5 Ensure all machines are in the off position (the circle signifies off position)

12.6 Connect power plugs for CRDS, multiplexer, and pump

12.7 Connect pump to multiplexer

- Connect control and data ports on the back of the pump to the Picarro ensuring white triangles are facing up on the back of the pump
- Insert other cable end of these connections into any USB port on the Picarro

12.8 Connect clear tubes to an inlet port on the pump, connect other end to the inlet on the front of the multiplexer

- Inlet on multiplexer on right (labeled)
- Inlet on vacuum (on left – clear tube)



eosAC (rear): INLET, OUTLET, and COMM.

12.9 Repeat process for the outlet tubes

- Outlet on Picarro
- Outlet on vacuum (on right – silver tube)

12.10 Attach clear tube inlet of Picarro to labeled outlet on multiplexer

12.11 Attach one end of power/data cable to the COMM port in the pump and the other end to the matching COMM port on the multiplexer

Power Up Sequence

12.12 Power pump

12.13 Switch in Eosense Multiplexer

12.14 Switch on Picarro

****Never turn off pump when Picarro is on****

Selection the Proper Location for Instruments

12.15 Machine serves as base location.

12.16 Use random number generator to determine location for each of the chambers.

Installing Chamber Rings

12.17 This will vary based on the design of your experiment.

Picarro Start up Procedure

12.18 Connect to power supply

12.19 Make sure all instruments are in the off position and turn on power strip

12.20 Turn-on recirculation pump (apparently there is no wait time)

12.21 Pump must be turned on before starting G2508 analyzer. Never disconnect vacuum will analyzer is running.

12.22 Keep ambient temperature below 35°C

12.23 Switch on Picarro – Boot up sequence initializes CRDS software and analyzer

12.24 Switch on Multiplexer. Warming up for 30 minutes – warning error until everything needs to be heated.

Place Chambers on Rings

12.25 Place chambers onto bases, pressing ring down to get a good seal. Try to avoid disturbing the chamber after it has been installed.

12.26 Connect all hoses while Picarro is warming up. –place tubing along the bed (dont let them get into the furrow)

12.27 After all chambers have opened up, do a second check for foliage that might get in the way

Data Collection (when alarm is green)

12.28 Check Picarro Conditions

12.29 Ambient should be below 35°C (95°F). (DAS – if DAS goes up to 45°C (113°F), should be turned off or cooled – Use fan to cool air around the Picarro.

12.30 Check for typical atmospheric concentrations:

- N₂O 0.3 ppm
- CH₄ 2.4 ppm
- C₂O 300-600 ppm
- NH₃?
- H₂O 1-5%

Start Multiplexer Software

12.31 FP-Monitor??

12.32 Uncheck default cycle

12.33 Load cycle in user folder. The following cycles are available

straw 5 chamber ??? names?

Full 12

Full 12 minus #4

12.34 When Picarro G2508 is warm, initiate cycle – "start cycle"??

12.35 Check chambers to ensure good seals

12.36 Check fuel every two hours, top off each time – be careful to avoid spilling. Be sure to release pressure before putting nozzle into filler throat. Open valve for gas when nozzle is inserted nearly into generator filler throat. Press green button for 3-5 sec intervals, checking between filling to determine if the gas is at or near the red line. Try to get at least 3 cycles minimum. Ideal is 2 cycles before irrigation, 2 cycles during irrigation and 2 cycles after.

Shut-Down Procedure

12.37 After at least two cycles after irrigation, begin retrieving chambers after each one has completed measurements (starting at 1 usually).

12.38 Cap all sets of tubes with glove and tape.

12.39 Carefully coil each set of tubes and wires to limit twisting and scratching of teflon. Zip/velcro together and stack in order 1-5 on the ground.

12.40 Remove chambers from sampling location

12.41 Separate based from chamber and repackage them into boxes.

12.42 When final chamber measurement has been completed, "end" ? sampling cycle

12.43 Connect spare chamber to channel X and refresh chamber...

12.44 select "Desciccate" method and run dry air through Picarro for 10 minutes.

12.45 shut down.

12.46 Shut down Picarro — Select option for moving instrument.

12.47 After Picarro shuts down, manually switch off:

1. Multiplexer
2. Picarro
3. Vacuum Pump
4. Power Strip

13. Data Analysis and Calculations

13.1 Once the Picarro startup procedure has been completed and the Picarro desktop is opened, run the "Data Analysis Software" program. The title should be "eosAnalyze-AC" on the top left of the window.

- (a) If you are only downloading data and not using the Picarro for experimental cycles, you do not necessarily need to wait for the Picarro system that pops up on the screen to warm up.
- (b) If the program is not on the desktop, open the Start Menu and search "eosAnalyze-AC." Open the "eosAnalyze-AC-v3.4 .2" folder. In the folder, open the .exe application with the same name.

13.2 In the eosAnalyze-AC software, click on "Collect Data." Put in the range of start and end times for the you want to download. You must repeat this for each cycle you are analyzing. Trying to put multiple tests in at once, ex. putting in 3 different tests over 3 days, will combine all tests together and will result in a single data table with huge time increments in between each cycle. It is also important to ensure the time zone is correct, Pacific Time Zone is UTC-8:00.

13.3 The data will load into the program under "Measurements". Double-check that the correct times and dates are obtained and the data graphs on the left look accurate once all measurements are done loading. .

13.4 Once the data you want to export is loaded into the software, click "Export Data Table". Export the data table onto the desktop or an easily accessible file location.

13.5 If you need more than one data table, close and reopen the program to create each new data table export.

13.6 The exported data tables should be .CSV files which can be loaded onto a USB or hard drive for analyses on another computer. The .CSV files should be loadable in Excel as a spreadsheet or in R as a data table.

You should see subfolders named by year. Click OK, then retry collecting data.

- If you still cannot get data, use Windows Explorer on the taskbar to search for the data logs. Double-check folder for .DAT files. The naming convention should be something like this: JFAADS2048-20180803-131415-DataLog.Forerunne r.dat

1. JFAADS20048: Instrument Serial Number -written behind the Picarro.
2. 20180803: YYYY MM DD ex. 2018 August 03.
3. 131415: HH MM SS when file started in 24-hour format ex. 1:14PM, 15 sec.

14. QC/QA Criteria

15. Trouble Shooting

15.1 Reading data from Picarro (must be emailed): The Chamber Data Processor program is expecting to find all of the relevant

files in its own folder. You can freely move these out between sites, but as they contain information about the chamber sequencing, when you are processing a certain date range, you will need to have to correct FRMonitor logs present (the program will start its search with log 0000 and go until it cannot find the next in the sequence). The path for raw analyzer data should always be the same, and should look like:

You can set this from the Data menu in the Chamber Data Processor (Data-> Analyzer Data Path).

15.2 Days with unexpected data: These days (159 and 175 or June 8th and June 24th) appear to contain valid chamber measurements. The Julian Day metric is pulled directly from the raw analyzer data and there don't appear to be any cases where a measurement got moved from the proper day. I would check to see if the system and/or local time on the analyzer have been changed, as this could potentially cause problems. You could also compare the processed measurements against your field notes to see if there is an obvious time period with missing data that seems to match with these days.

15.3 Days that should have more data: I noticed several errors in the FRMonitor logs that you uploaded. I've fixed these files and attached them to this email. Back up the older versions and try using these instead: I noticed several additional measurements appear once I had updated them. As for where these errors came from,

the log files suggest that, at some points at least, chambers were disconnected from the Multiplexer while it was actively running a measurement cycle? If so, I would strongly advise against this, as it can cause the Chamber Data Processor to overlook measurements (especially if the chamber was closed when it was removed). Also, are you using the 1.6.0 or 1.6.2 version of the FRMonitor software? Make sure to use the newer version to schedule measurements.

15.4 Days where some chambers are missing: I saw several days where Chambers 1 and/or 2 do not appear to be sending any data. This indicates either a communication issue between the Multiplexer and Analyzer, or that the chambers were not connected during this time period. Has the FRMonitor software been recognizing all of the connected chambers on start-up? If not, then they will not record data (even if they open and close). The System Info button will show you a break down of chamber-specific events, including successfully logged measurements and communication problems. Disconnecting and reconnecting chambers during a measurement cycle can also cause these errors.

Find the folder named....

Diagnostics Folder

start the program called

Stop Instrument

select shut down...with drivers...

Middle...

16. References

16.1 APHA, AWWA. WEF. (2012) Standard Methods for examination of water and wastewater. 22nd American Public Health Association (Eds.). Washington. 1360 pp. (2014).