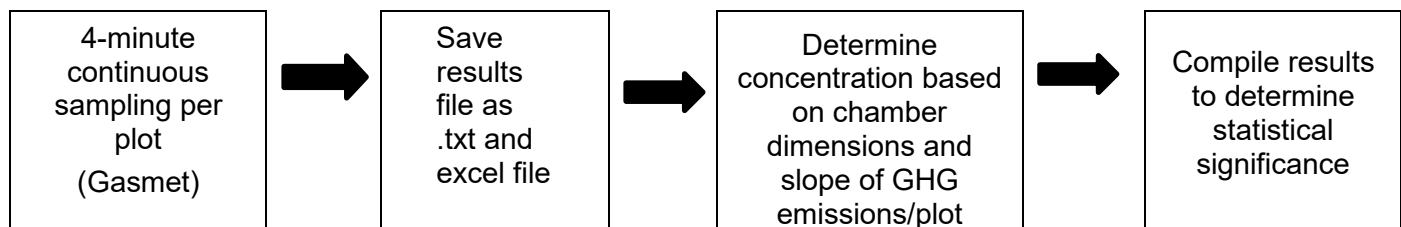


SOP: Greenhouse Gas Emissions –On-site Measurement Using Closed-Chamber Protocol

Overview:

The following standard operating procedure (SOP) for the use of in-situ measurements of greenhouse gases (GHG), including the operation of the Gasmeter GT–5000 portable infrared analyzer for manual on-site measurements with closed chambers and for converting values into a concentration basis. Collection of greenhouse gases samples is done through at least 4 minutes of continuous sampling, following suggested protocol.¹ Values are then converted to flux rates of greenhouse gas emissions through R code, given measurements associated with the chamber and collar area and volume.



Safety:

All standard safety protocols and online safety training via UIUC [Division of Research Safety \(DRS\)](#) are required.

Personal protection (PPE) for this procedure include:

Eye Protection: Safety goggles

Body Protection: Outdoor coat

Hand Protection: Work gloves

Particularly hazardous substances: Batteries used for machine operation, if broken or used incorrectly, could leak. Acid from battery leakages is considered hazardous to the environment and can pose health risks from exposure.

Instrumentation:

Gasmet Analyzer and Equipment

- Gasmet battery packs
 - Location: C414, at Gasmet charging stations
 - **Batteries will last for 2 hours if kept in proper condition.** Before field measurements, determine how long sampling will take for the total number of plots that will be completed that day.
 - Assuming 4-minute sampling intervals, also accounts for preparation time during N₂ background calibration, and include an extra 1-2 minutes per plot for traveling across the field.
- Flow meter
 - Location: C414 or attached to N₂ gas valve.
- Chamber head
 - Attached to chamber head: 2 tubing, one H₂O vapor filter (top of chamber), one temperature probe.
- Laptop OR tablet connected to specific Gasmet Machine
 - This will be used for controlling the analyzer.
 - Recommended setting is WiFi Access Point ("TCP").
- Filters
- Tube fittings

Other

- Chamber tops and collars (Fig. 1)
- Chamber thermometer
- Soil temperature and soil moisture probes
- Stopwatch (to time sampling period)
- Measuring the height of the collar (for converting to flux rates)

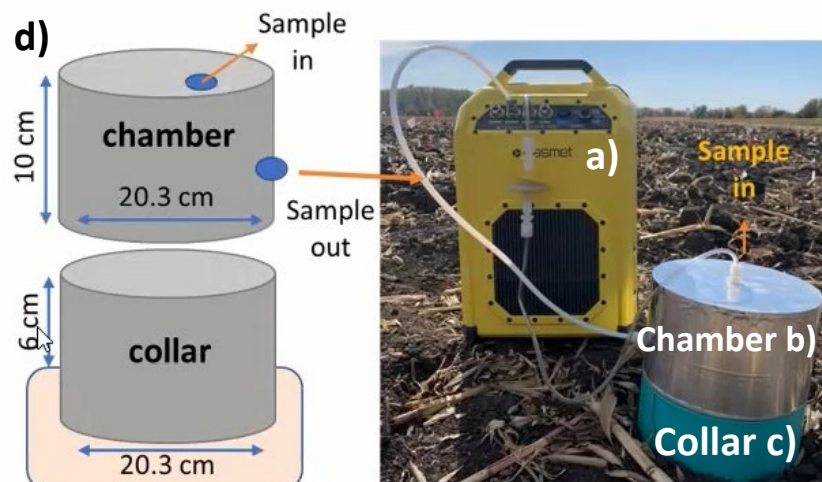


Figure 1. Picture of a) Gasmet machine in use, with b) chamber top attached with tubing and c) collar installed into field plot. Close up diagram of d) chamber and collar including dimensions.

Detailed Procedure:

I. Preparing Gasmet for field measurements

1. Preparations before going to the field:

- i. **Schedule:** use the GHG-Group calendar to schedule your time to use the machine. Scheduling is on a first-come, first-serve basis, and should be made at least 3 days in advance before use.
- ii. **Check N₂ Gas:** Check the amount remaining in the N₂ gas. If the gas tank reads as 1/3 full, check with lab manager about replacement of the gas tank.
- iii. **Locate Flow regulator:** Use of N₂ gas for background calibration will need to be done at 2-10 lpm, which requires the use of a fixed flow regulator. This should already be attached to the N₂ tank. Flow is adjusted on the N₂ tank valve (leftmost valve, turn clockwise)
- iv. **Use sign-in/sign-out sheet:** Record when you are using the N₂ gas tank and Gasmet number before heading out to the field.
- v. **Check machine and program:** Familiarize yourself with the machine you would like to use, and the associated laptop/tablet that contains the Calcmnet program to use. Make sure you can log into the laptop/tablet and check to see if the connection with the Gasmet is working properly.

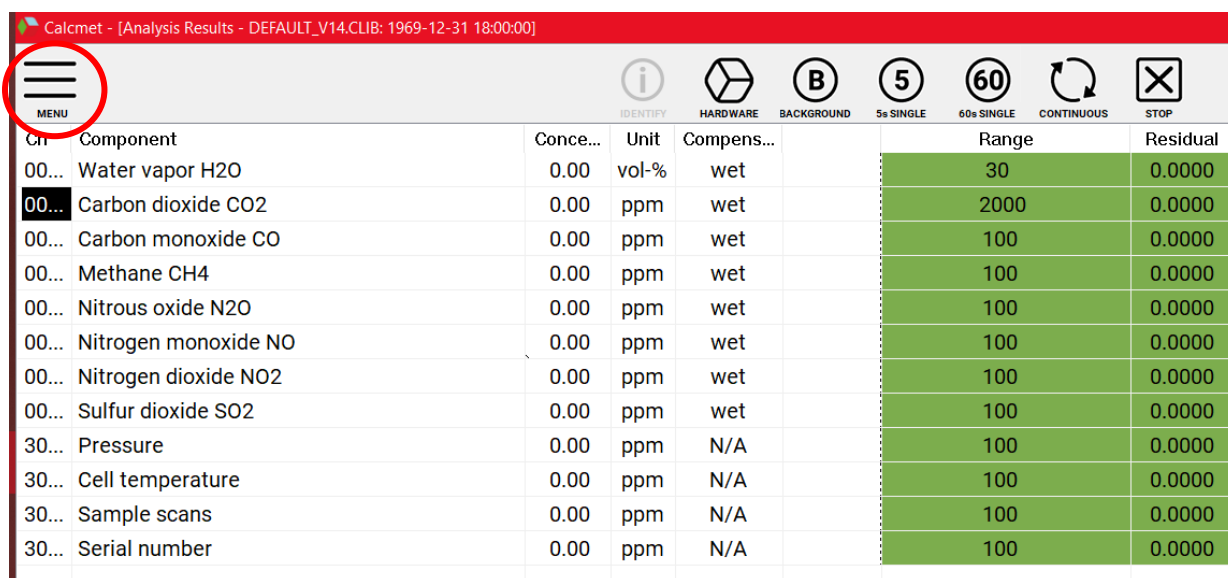
1. **Check machine, batteries, laptop/tablet are FULLY CHARGED.** This is important to ensure that all equipment will last for the entire duration of the sample. Pack extra batteries if necessary for sampling (see “Gasmet Battery Packs” under Instrumentation).
2. **Check Save Locations:** Results will be saved under “C:/SN#####/CalcmnetResults/”, then select the date you have sampled for all files from your plot measurements. If each plot had a renamed save file, all files should appear there. The SN##### will change depending on the Gasmet machine, as each unit has a unique 5 number identifier (E.g., SN43590 for Gasmet 1).

vi. Check Chamber Top

1. Chamber top must be able to **connect one tube to “SAMPLE IN” and another tube to “SAMPLE OUT”.**
2. Chamber top **must have filter attached to “SAMPLE IN” tubing.** This is important to avoid any water vapor buildup in

the machine.

2. For troubleshooting, refer to Gasmet guide or Appendix in this SOP.



Calcmeter - [Analysis Results - DEFAULT_V14.CLIB: 1969-12-31 18:00:00]

Top bar icons: MENU (circled), IDENTIFY, HARDWARE, BACKGROUND, 5s SINGLE, 60s SINGLE, CONTINUOUS, STOP.

Ch	Component	Conce...	Unit	Compens...	Range	Residual
00...	Water vapor H2O	0.00	vol-%	wet	30	0.0000
00...	Carbon dioxide CO2	0.00	ppm	wet	2000	0.0000
00...	Carbon monoxide CO	0.00	ppm	wet	100	0.0000
00...	Methane CH4	0.00	ppm	wet	100	0.0000
00...	Nitrous oxide N2O	0.00	ppm	wet	100	0.0000
00...	Nitrogen monoxide NO	0.00	ppm	wet	100	0.0000
00...	Nitrogen dioxide NO2	0.00	ppm	wet	100	0.0000
00...	Sulfur dioxide SO2	0.00	ppm	wet	100	0.0000
30...	Pressure	0.00	ppm	N/A	100	0.0000
30...	Cell temperature	0.00	ppm	N/A	100	0.0000
30...	Sample scans	0.00	ppm	N/A	100	0.0000
30...	Serial number	0.00	ppm	N/A	100	0.0000

Figure 2. Example of Calcmeter, the program used to interact with the Gasmet analyzer. "MENU" button is circled in the top left.

3. Turning on Gasmet Machine and Calcmeter program

- i. The laptop/tablet associated with the machine should have Calcmeter installed and ready for use.
- ii. Machine: turn to "Bluetooth" or "WIFI Access Point)
 1. Machine should already have Bluetooth connection to the laptop/tablet as the default setting. To change this, go to "Menu" -> "Configuration".
- iii. Press "Hardware" on the top right bar.



Figure 3. Close-up of Gasmet machine. Connection for gas lines, cable port, and communication modes are circled and labeled. Note latch mechanism on gas lines,

which must be pressed before inserting tubing. Care should be taken when inserting gas lines.

4. N₂ background calibration before sampling

- i. Once hardware status reads OK, hook up N₂ tank to “ZERO GAS” inlet.
- ii. Open N₂ tank and check the flow regulator attached has 2-10 liters per minute (lpm). Recommended lpm is at 2-3.
- iii. Once flow is regulated to appropriate amount, press “Background” on the top right of the Calcmeter program.
- iv. Background calibration should take approximately 5 minutes, and the background spectrum will appear on the screen once completed.
- v. Once background is completed, sampling can begin.
- vi. Close laptop or tablet and turn off the Gasmeter when transporting to the field for sampling to conserve battery life.

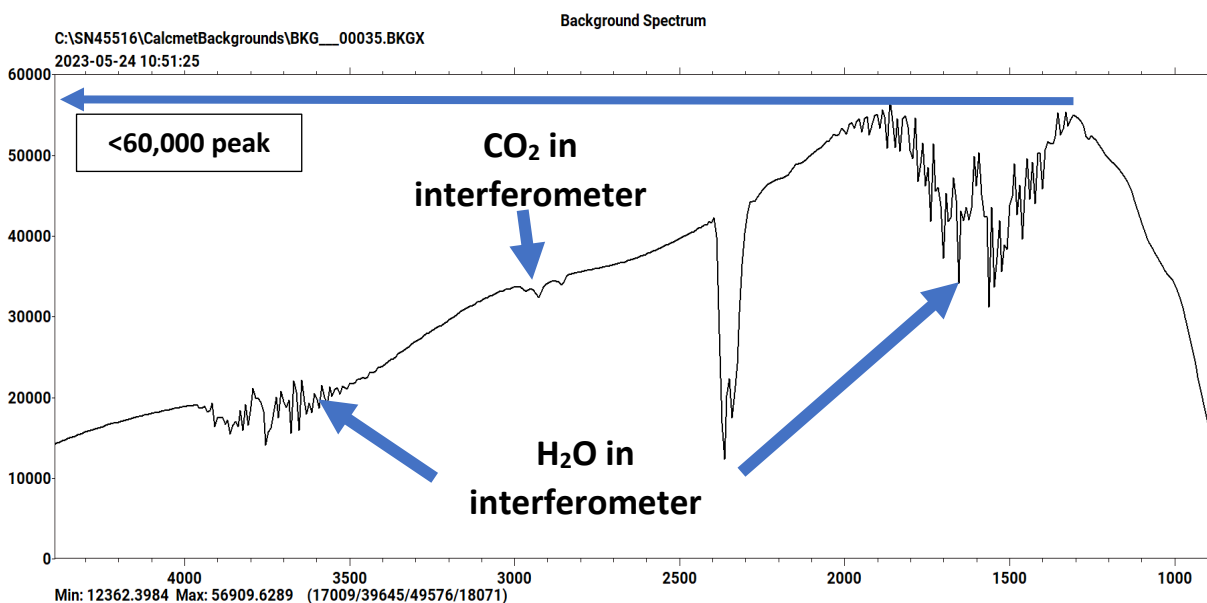


Figure 4. Screenshot of a typical background calibration, with arrows pointing towards peaks or troughs across the spectrum. The highest peak should occur between 2000-1000 nm in the spectrum, reaching a maximum close to 60,000.

II. Begin Sampling

1. In the field, make sure the Gasmeter and the Calcmeter software are on and communicate among themselves. This could be ensured by doing a checking “Hardware” status again before sampling. The hardware status should be “ok”.

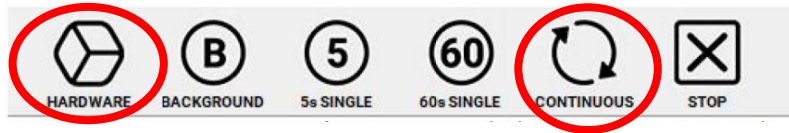


Figure 5. Screenshot of Calcmnet easy toolbar, located on the top right of the program. “HARDWARE” and “CONTINUOUS” buttons are circled.

2. Label save file for each plot: Rename Autosave File Name under “Autosaving” setting. Before sampling any plot, rename the file. This will ensure each plot will have a separate text file (.TXT) for its measurements.

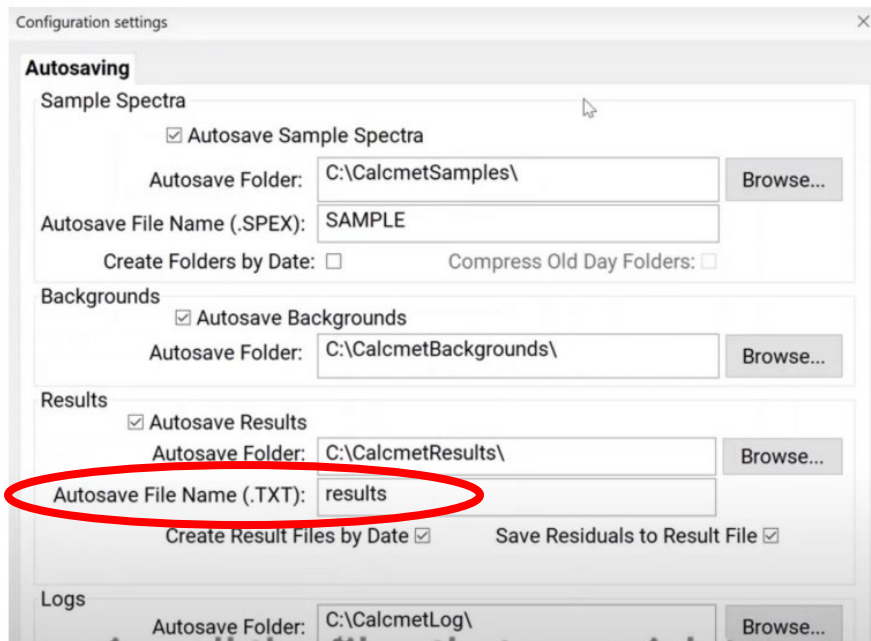


Figure 6. Screenshot of how to change file name under “Menu” -> “Autosaving”. The circled portion indicates where to input the new file name for the specific plot being sampled. This should be changed before sampling a new plot.

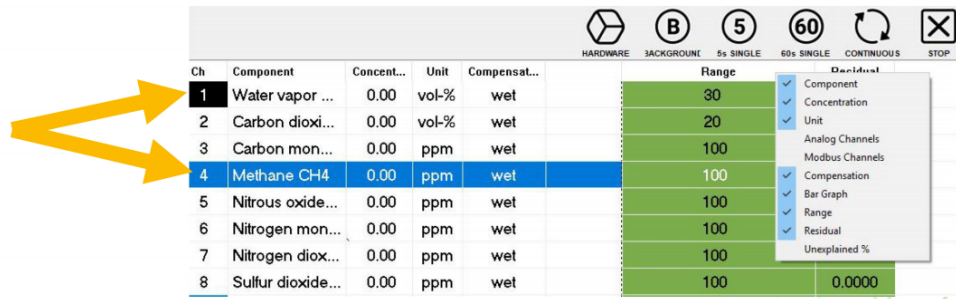
3. Place temperature probe inside the collar and chamber space, leaving the probe lying on the soil surface. The recorded temperature once sampling is finished will be used to correct flux rates of greenhouse gas emissions.
4. Place the chamber top on top of the collar and attach tubing to “SAMPLE IN” and “SAMPLE OUT”. Check to ensure the chamber completely closes over the collar.
5. Press “CONTINUOUS” to begin sampling. Machine will sample every 20 seconds, and the program page with refresh to show previous reading.

6. Reading real-time results: Click the top right arrows to flip through the different results of each sample and sample points.
 - i. The TREND VIEW page shows all sample points over time. In the results table, click the first column to appear on the trend view graph.

> Right click on headers to remove certain columns

Select components on the first column:

They will be assigned a color assigned and displayed in the 'trend view'



The screenshot shows a table with 8 columns: Ch, Component, Concent..., Unit, Compensat..., Range, and a final column for residuals. The first column (Ch) is highlighted with a blue background. A context menu is open over the first column, showing options: Component, Concentration, Unit, Analog Channels, Modbus Channels, Compensation, Bar Graph, Range, Residual, and Unexplained %. The 'Range' column contains values: 30, 20, 100, 100, 100, 100, 100, 100. The 'Residual' column contains the value 0.0000.

Ch	Component	Concent...	Unit	Compensat...	Range	Residual
1	Water vapor ...	0.00	vol-%	wet	30	
2	Carbon dioxi...	0.00	vol-%	wet	20	
3	Carbon mon...	0.00	ppm	wet	100	
4	Methane CH4	0.00	ppm	wet	100	
5	Nitrous oxide...	0.00	ppm	wet	100	
6	Nitrogen mon...	0.00	ppm	wet	100	
7	Nitrogen diox...	0.00	ppm	wet	100	
8	Sulfur dioxide...	0.00	ppm	wet	100	0.0000

Figure 7. Screenshot Calcmet program, with instructions on how to select different components to view in "Trend View".

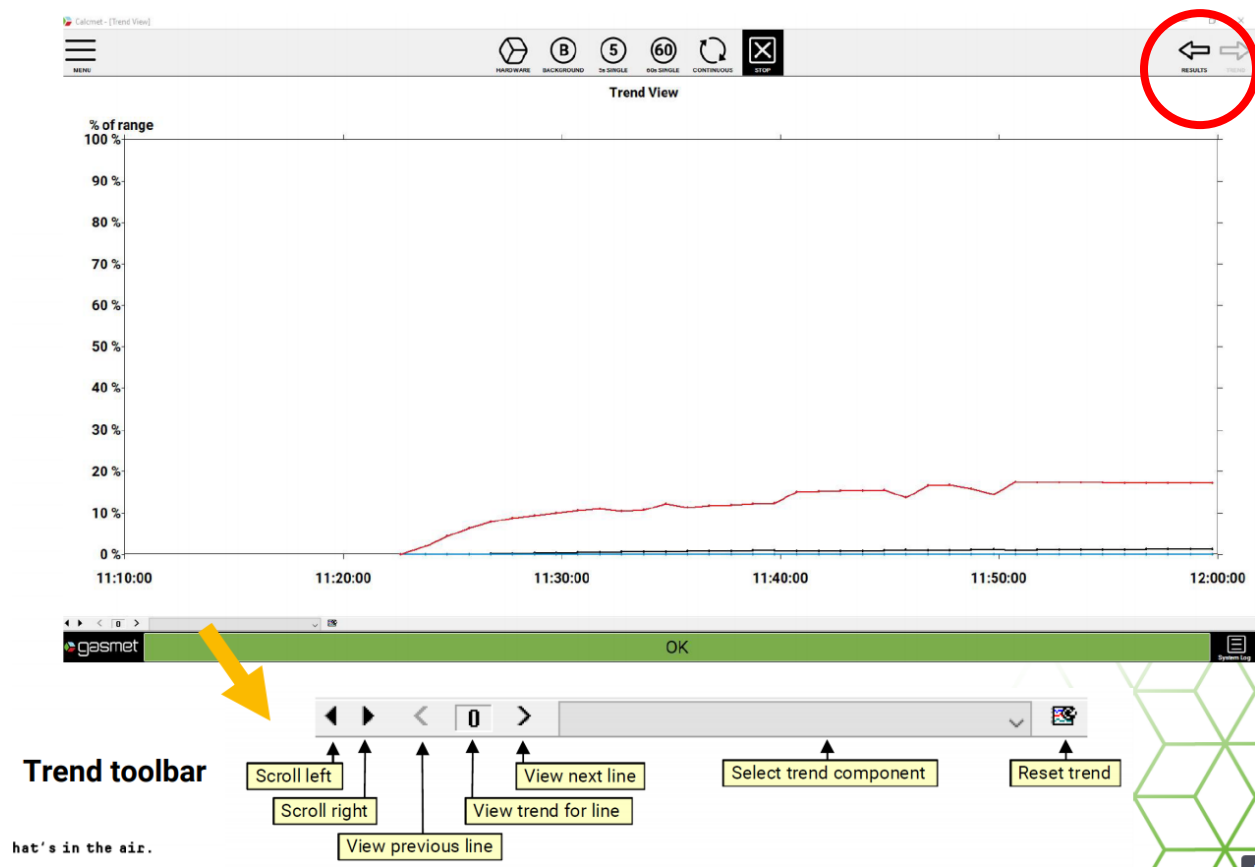


Figure 8. Screenshot of the TREND VIEW tab, and the toolbar (bottom “Trend toolbar”) to navigate the graph. The circled portion indicates how to swap back from TREND VIEW to the results table.

7. Measure for 4 minutes per plot, or however long determined by your group or project needs.
 - i. Sampling time is based on common procedure for closed chamber gas sampling. At least 3 time points at intervals equally spaced in time (e.g., 0 s, 20 s, 30 s) should be collected².
 - ii. Other longer time intervals are by minutes, typically 20-60 minutes as the maximum sampling time for a given chamber. Discuss with your group on intervals, and plan out how long sampling will take for the number of chambers you must sample in a day.
 - iii. Additional sampling for non-GHG emissions can be done while waiting for 4 minutes, such as soil moisture. Record each measurement per plot.
8. Once time has run out, press “STOP” to halt sampling. Remove chamber top from collar, and record final temperature from probe for each plot.
9. Move to next chamber, rename save file and press continuous when chamber is securely placed onto collar.
 - i. Wait for the first 20 seconds sampling to pass, as this will flush out gases from the previous plot.
10. Changing batteries: turn off machine before switching out batteries.
 - i. Open battery hatch, remove battery and replace with fully charged battery.
 - ii. Store uncharged battery pack in bag or case, then charge when returning from field.
11. Once done with sampling, turn off the machine.

BATTERY HATCH



Figure 9. Images of side battery hatch, including where the battery is open and how to charge the machine directly with the power inlet.

III. Return from Sampling: Maintenance of Equipment After Use

1. Turn machine back on and turn on “CONTINUOUS” to allow air-conditioning to run through machine.
2. Run N₂ gas through machine and continue to sample until CO₂ ppm reaches <1 ppm.
 - i. This should especially be done when field conditions are very wet, or when water vapor is higher than usual.
3. Return all equipment and machines back to their respective locations.
4. Laptop/Tablet: retrieve data and save it to your personal USB drive/OneDrive.
 - i. Results will be saved under “C:/SN#####/CalcmeterResults/”, then select the date you have sampled for all files from your plot measurements. If each plot had a renamed save file, all files should appear there.
5. Charge all batteries, and the laptop or tablet used. This is important to ensure that the next users will have all equipment charged for next use.
6. Maintenance of Gasmet Equipment

- i. Check frequently if sample-in or sample-out tubing has buildup of water. This could mean water trap/filter is not working properly, which should be examined.
 - 1. Check for any leaks in the tubing, which may be letting in moisture.
 - 2. If water droplets are suspected to have entered the Gasmeter machine, immediately flush with N₂ gas or air.
 - 3. If water trap/filter appears to be discolored, this could mean a replacement filter is needed.
- ii. Clean equipment regularly
 - 1. Includes batteries if soil particles/crop residue dirty the batteries. Wipe down after use and before charging.
 - 2. The outer yellow case of the Gasmeter may be dirtied after constant use, wiping down after each use can reduce buildup of residue.

IV. Calculations

Volume of cylinder;	$V = \pi r^2 h$
Chamber vol;	$v_1 = \pi (10.15)^2 \times 10 \text{ cm}^3$
Collar vol;	$v_2 = \pi (10.15)^2 \times 6 \text{ cm}^3$
Sample (in + out) vol.;	$v_3 = \pi (0.2159)^2 \times (64 + 80) \text{ cm}^3$
Analyzer cell vol.;	$v_4 = 500 \text{ cm}^3$
Total volume;	$v_t = v_1 + v_2 + v_3 + v_4 \text{ cm}^3$
Vol. / Area ratio;	$R = \frac{v_t}{\pi (10.15)^2} \text{ cm}$

1. Flux calculations

Result file(s) from the analyte have units in ppm. The units, ppm, are typically on a volume per volume basis (which is the same as a mole per mole basis). Volumetric parts per million (ppm(v)) has units of μL trace gas L^{-1} total gas. For example, a 1 ppm (vol/vol) N_2O standard will contain $1\mu\text{L}$ N_2O / L of gas.

- Determine the linear relationship (i.e., slope; α_v) between the concentration and the sampling time, in ppm (v)/sec. This is equivalent to $\mu\text{L} / \text{L} / \text{sec}$.
- Since slope (α_v) have units based on volume, convert units based on mass (α_m) and correct for field temperature using the Gas Law:

$$\alpha_m = \frac{\alpha_v \times M \times P}{R \times T}$$

where,

α_m is expressed in μg CO_2 or N_2O / L / sec,

M = molecular weight of GHG (44 μg CO_2 or N_2O / μmol CO_2 or N_2O),

P = 1 atm = atmospheric pressure,

R = universal gas constant = 0.0821 $\mu\text{L-atm} / \mu\text{mol-K}$,

T = chamber temperature, in $^\circ\text{K} = ^\circ\text{C} + 273$,

Calculate the flux (f_m) of GHG flux rate:

$$f_m = \frac{\alpha_m \times V \times 3600 \text{ sec/hr}}{A}$$

where,

f_m is expressed in μg CO_2 or N_2O / m^2 / hr

α_m = as above, in μg CO_2 or N_2O / L / min

V = volume of gas in chamber, in L (1 $\text{cm}^3 = 0.001$ L)

A = chamber surface area, in m^2

R-script for calculating GHG flux from the raw data

For detailed explanation and example on use of R code calculations, please refer to video demonstration ("GHG SOP Calculations Explanation").

```
library(dplyr)
library(readxl)

vol<- read_excel("C:\\Users\\js142\\OneDrive - University of Illinois -
Urbana\\My projects\\ISA\\Data Analysis\\Raw
data\\Ewing\\03132023\\vol.xlsx")[, c(1, 2, 12)] # This code reads plot id,
chamber temperature (centigrade) and vol/area ratio. Volume (includes collar,
chamber, sample in, sample out, analyzer cell).This spreadsheet should be
prepared in advance.

tbl<- list.files(path = "C:\\Users\\js142\\OneDrive - University of Illinois
- Urbana\\My projects\\ISA\\Data Analysis\\Raw data\\Ewing\\03132023", #
Where files are stored
                pattern = "*.txt",
                full.names = T) %>%
  lapply(., read.delim, header=TRUE) %>%
  lapply(., function(x){x[c(10, 18, 26)]}) %>% # Only CO2, N2O and CH4 are
selected.
  lapply(., function(x) {x <- x[-c(1, 2), ]}) %>% # This will remove first
two rows (first two sampling points)
  lapply(., function(x){cbind(x, time=seq(0, by=20, length.out=nrow(x)))}) #
Providing 20 sec. points

fun1<- function(x) {
  coef(lm(Carbon.dioxide.CO2 ~ time, data=x))[2] # 2 is slope
}

fun2<- function(x) {
  summary(lm(Carbon.dioxide.CO2 ~ time, data=x))$r.squared # R. square
values
}

fun3<- function(x) {
  coef(lm(Nitrous.oxide.N2O ~ time, data=x))[2] # 2 is slope
}

fun4<- function(x) {
  summary(lm(Nitrous.oxide.N2O ~ time, data=x))$r.squared # R. square values
}

fun5<- function(x) {
  | SOP: Greenhouse Gas Emissions –On-site Measurement Using Closed-Chamber Protocol |
  | UIUC Soils Lab | Last revised 24 Apr 2023
```

```

    coef(lm(Methane.CH4 ~ time, data=x))[2] # 2 is slope
  }

fun6<- function(x) {
  summary(lm(Methane.CH4 ~ time, data=x))$r.squared # R. square values
}

f1<- lapply(tbl, fun1) # slope CO2
f2<- lapply(tbl, fun2) # R. square CO2 values
f3<- lapply(tbl, fun3) # slope N2O
f4<- lapply(tbl, fun4) # R. square N2O values
f5<- lapply(tbl, fun5) # slope CH4
f6<- lapply(tbl, fun6) # R. square CH4 values

d1<- do.call(rbind, Map(data.frame, slope= f1, r.sq= f2)) # CO2
d2<- do.call(rbind, Map(data.frame, slope= f3, r.sq= f4)) # N2O
d3<- do.call(rbind, Map(data.frame, slope= f5, r.sq= f6)) # CH4

# Merging dataframes
d1<- cbind.data.frame(vol, d1)
d2<- cbind.data.frame(vol, d2)
d3<- cbind.data.frame(vol, d3)

d1$CO2<- d1$slope*d1$Ratio*.001*44*60*60*(1/ (.0821*(d1$Temp+273))) # Final
CO2 calculations (ugCO2/m2/hr)
d2$N2O<- d2$slope*d2$Ratio*.001*44*1000*60*60*(1/ (.0821*(d2$Temp+273))) #
Final N2O calculations (ngN2O/m2/hr)
d3$CH4<- d3$slope*d3$Ratio*.001*16*1000*60*60*(1/ (.0821*(d3$Temp+273))) #
Final CH4 calculations (ngCH4/m2/hr)

final<- data.frame(d1$ID, d1$CO2, d2$N2O, d3$CH4, d1$r.sq, d2$r.sq, d3$r.sq)
nam<- c('id', 'CO2', 'N2O', 'CH4', 'R.CO2', 'R.N2O', 'R.CH')
names(final)<- nam

write.csv(final, "C:\\Users\\js142\\OneDrive - University of Illinois -
Urbana\\My projects\\ISA\\Data Analysis\\Raw
data\\Ewing\\03132023\\03132023.csv") # Transport to your folder

```

Example calculations:

Example 1. Calculations for measurements of 1) chamber volume, 2) collar volume, sample volume, 4) cell volume, 5) total volume, and 6) the volume to area ratio of the chamber. These values will be used for converting ppm to flux rates of greenhouse gases.

$$1) \text{ Chamber volume}(\text{cm}^3) = \pi \times r^2 \times h = \pi \times 10.15^2 \times 10 \text{ cm}^3$$

$$2) \text{ Collar volume}(\text{cm}^3) = \pi \times r^2 \times h = \pi \times 10.15^2 \times 8 \text{ cm}^3$$

$$3) \text{ Sample volume}(\text{cm}^3) = \pi r^2 h(\text{sample in}) + \pi r^2 h(\text{sample out}) = \pi(64 + 80) \text{ cm}^3$$

$$4) \text{ Cell volume}(\text{cm}^3) = 500 \text{ mL (known amount from Gasmeter Technical Sheet)}$$

$$5) \text{ Total Volume} = \text{Eq. 1} + \text{Eq. 4}$$

$$6) \text{ Vol: Area ratio} = \frac{\text{Total Volume}}{\text{collar area}(\pi r^2)}$$

These calculations are shown in the below spreadsheet. This spreadsheet should be prepared in advance before using R-code to calculate fluxes.

Example 2. Excel spreadsheet showing measurements for chamber and collars.

ID	Temp	Collar height (cm)	Chamber height (cm)	Chamber inner diameter (cm)	Chamber + collar Volum (cm3)	Sample In Length (cm)	Sample Out Length (cm)	Sample In & out Volum (cm3)	Analyzer cell vol (cm3)	Total volum (cm3)	Ratio
101	20.5	7.7	10	20.3	5729	64	80	21.1	500	6250	19.3
102	20.5	6.5	10	20.3	5340	64	80	21.1	500	5861	18.1
103	20.5	8	10	20.3	5826	64	80	21.1	500	6347	19.6
104	20.5	9.3	10	20.3	6247	64	80	21.1	500	6768	20.9

Appendix

Solution: Results showing only “0 PPM” for all components

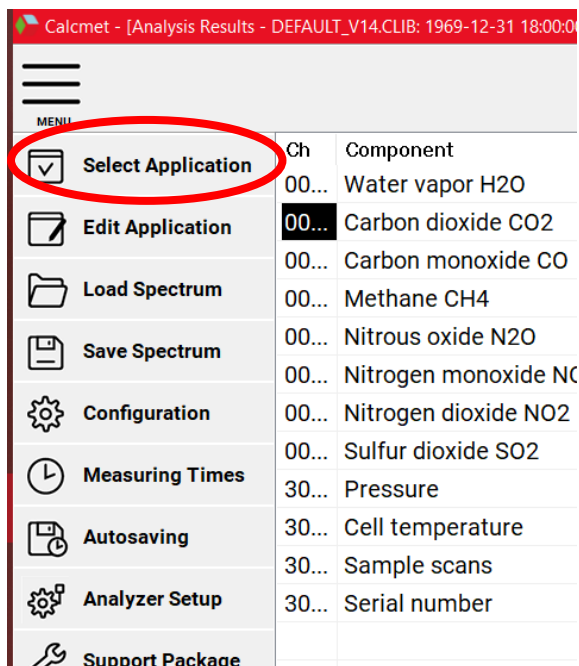


Figure 9. Screenshot of Calcmeter program, with “Select Application” circled in red. Press Select Application, then locate the file named **“GAS-APP-006_SN#####_c14_133_Rev1.CLIB”**. The hash symbol (#) will contain the ID number associated with your Gasmet machine. This file will be located under the folder “GAS-APP-006_SN#####” on the laptop/tablet’s C: Drive. Press open, then retry continuous sampling. The program should now show the correct PPM values.

References:

1. de Klein, E. C., & Harvey, M. (2012). *Nitrous Oxide Chamber Methodology Guidelines*.
2. Parkin, T.B. and Venterea, R.T. 2010. Sampling Protocols. Chapter 3. Chamber-Based Trace Gas Flux Measurements. IN Sampling Protocols. R.F. Follett, editor. p. 3-1 to 3-39. Available at: www.ars.usda.gov/research/GRACEnet

Suggested Readings:

1. Collier, S. M., Ruark, M. D., Oates, L. G., Jokela, W. E., & Dell, C. J. (2014). Measurement of Greenhouse Gas Flux from Agricultural Soils Using Static Chambers. *Journal of Visualized Experiments*, 90, 52110.
<https://doi.org/10.3791/52110>

Video Demonstration Link:

<https://uofi.app.box.com/file/1225467055900>

Citation:

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Accessed at: <https://margenot.cropsciences.illinois.edu/methods-sops/>

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