

Static Chamber Method for Measuring Soil Greenhouse Gas Fluxes

K. Kahmark, N. Millar and G. P. Robertson

W.K. Kellogg Biological Station, Michigan State University

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INTRODUCTION

This protocol covers the design, construction, deployment, and gas sampling procedures for the static (closed cover) stainless steel chambers used to measure soil greenhouse gas (GHG) fluxes at Michigan State University's W.K. Kellogg Biological Station (KBS) in southwest Michigan. The sampling procedure is appropriate for soil nitrous oxide (N_2O), methane (CH_4), and carbon dioxide (CO_2) fluxes. Detailed instructions on the design and construction of our cylindrical static chamber are covered in part one of this document; field deployment and gas sampling procedures are covered in part two.

Many reviews and publications discuss sampling protocols and procedures for determining soil gas fluxes using various chamber designs and systems, and readers are encouraged to conduct their own review and research on these. The design described herein is compliant with those used by national and international networks^{1,2,3}. We have successfully used this design (or a close variant) for several decades and share it with the notion that others might benefit from our experience and save some time and effort when deploying their own chambers. Modifications should be straightforward as needs dictate.



Figure 1. Static chamber as deployed in the field prior to (left) and during (right) gas sampling.

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¹ Holland, E. A., G. P. Robertson, J. Greenberg, P. M. Groffman, R. D. Boone, and J. R. Gosz. 1999. Soil CO_2 , N_2O , and CH_4 exchange. Pages 185-201 in G. P. Robertson, D. C. Coleman, C. S. Bledsoe, and P. Sollins, editors. *Standard Soil Methods for Long-term Ecological Research*. Oxford University Press, New York, New York, USA.

² Parkin, T. B. and R. T. Venterea. 2010. USDA-ARS GRACenet project protocols. Chapter 3. Chamber-based trace gas flux measurements. Pages 3.1-3.39 in R. F. Follett, editor. *Sampling protocols*. Available at: www.ars.usda.gov/anrds/gracenet/gracenet-protocols/.

³ De Klein, C. a. M. and M. J. Harvey, editors. 2015. *Nitrous oxide chamber methodology guidelines*. Global Research Alliance on Agricultural Greenhouse Gases, Available at www.globalresearchalliance.org.

PART ONE: CHAMBER CONSTRUCTION

Basic Equipment and Materials

- Chambers: 304 stainless steel (16 gauge [thickness = 0.063" or 1.60 mm]) cylinder with final dimensions of 28.5 cm ID × 22.8 cm Ht (~11 ¾" × 9")
- Chamber lid with O-ring seal: 12.7 mm (0.5") thick HDPE plastic (e.g., from Alro Plastics <https://www.alro.com/>) machined to 29.8-30 cm (~11 ¾") circles with grooves and seal
- 1/8" (3 mm) Swagelok bulkhead union
- Seals/O-rings for all hardware connections
- Other equipment and materials as covered in the procedure

Chamber Construction Procedure

1. The chamber base is constructed from 16 gauge 304 stainless steel according to the drawings in Fig.
2. We contract with a local mechanical fabrication company (metal shop) to stamp holes and weld the cylinder (base). The top edge of the cylinder is folded prior to welding for additional strength.

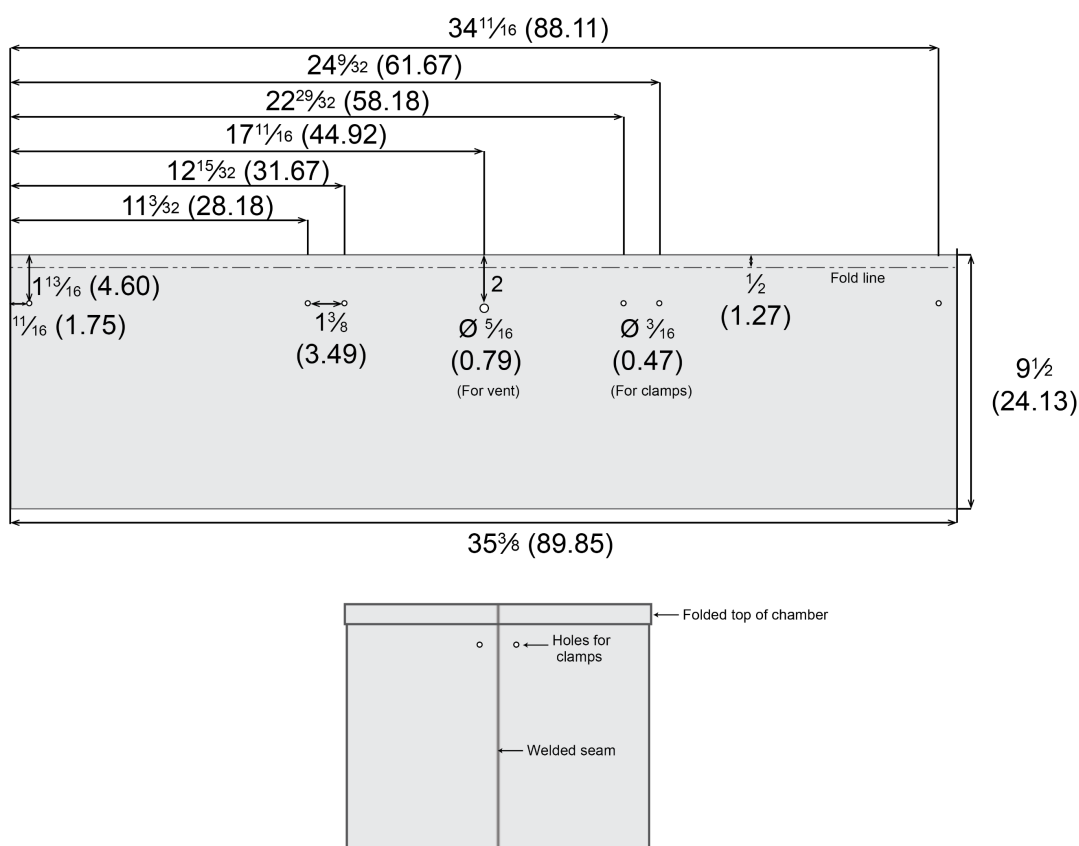


Figure 2. Stainless steel chamber template, in inches (nearest centimeter equivalents given in parentheses).

- The chamber lid is fabricated in our shop from $\frac{1}{2}$ " (12.7 mm) HDPE white plastic, cut into 12" (30.5 cm) squares. Several router templates were made to aid in the completion of the lid design (Fig. 3). Figures 4 and 5 show the design and dimensions of a completed lid (top and bottom views).

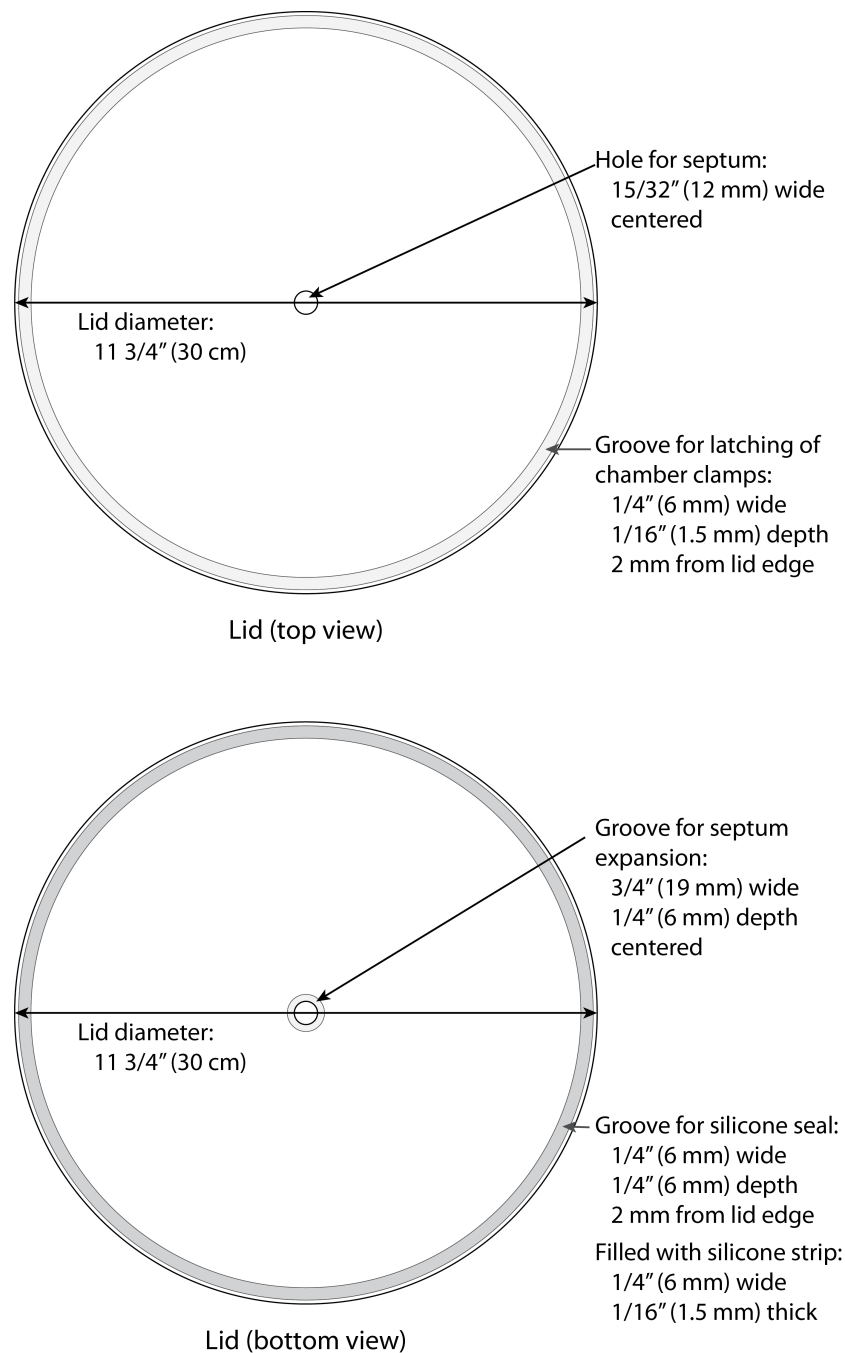


Figure 3. Completed HDPE lid (top and bottom views).

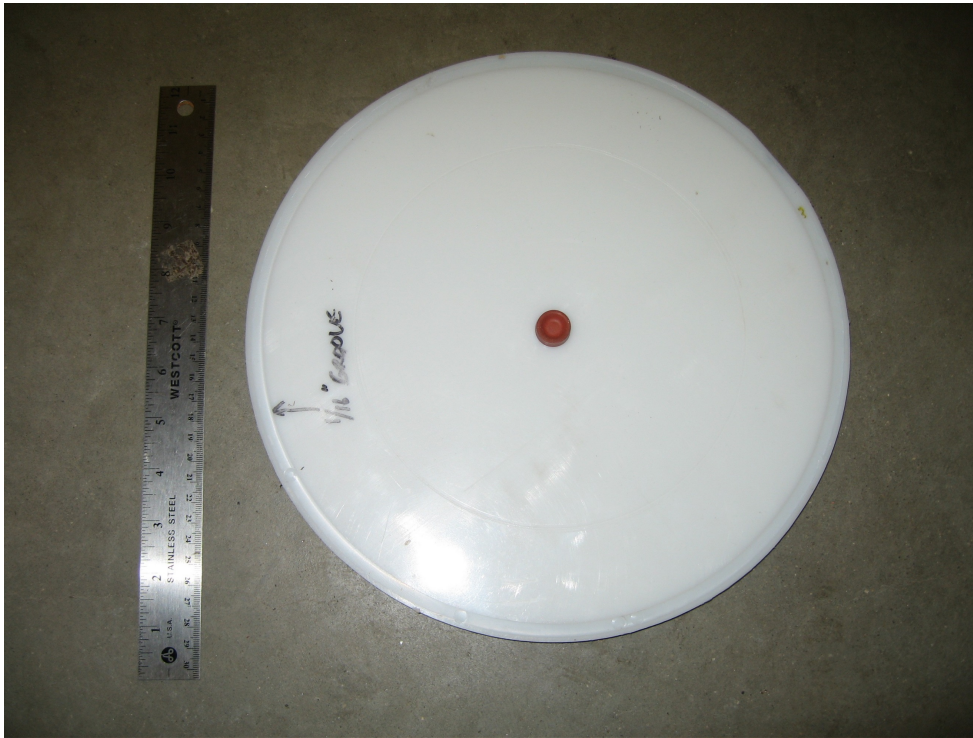


Figure 4. Lid, top view, with $1/4'' \times 1/16''$ depth groove and rubber septum in center hole.



Figure 5. Lid, bottom view, with dimensions. Black ring is the silicone strip seal.

3. The lid is routed to size using a template that cuts a circle from the HDPE square to a 29.8 (+/- 0.1) cm diameter using a ¼" (6 mm) straight routing bit (Fig. 6).

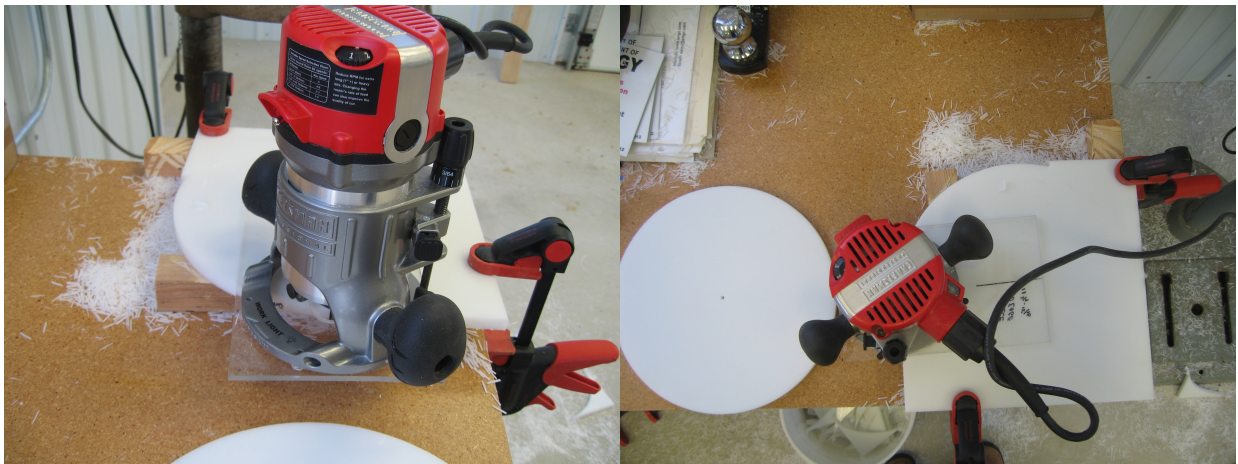


Figure 6. Routing the lid outer dimension.

4. A groove is made on the bottom surface of the lid to house a silicone seal. The groove is positioned 2 mm from the lid edge and is routed to ¼" (6 mm) depth with the ¼" (6 mm) routing bit using a template (Fig. 7). The seal is a high temperature silicone ¼" (6 mm) strip with adhesive backing. To install the strip first cut the free end of the strip at an angle, then insert into the groove, and set firmly in the groove all the way around the lid (Fig. 8). Wait 24 hr to ensure the seal will not shrink after stretching during installation. The end of the strip is then cut to length at an angle that matches the start end. Make sure the two end pieces are joined flat for an airtight seal, then seal the joint with a dab of silicone gasket material.

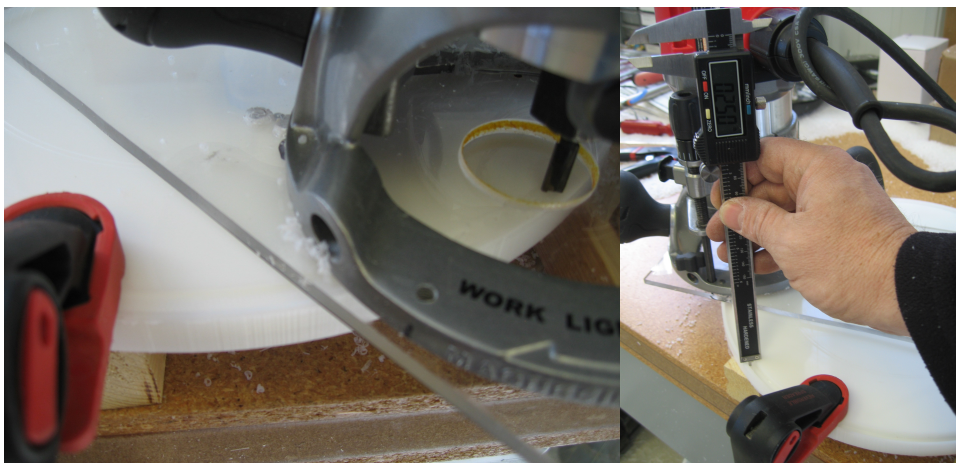


Figure 7. Routing the groove on the bottom surface of lid to house the silicone strip.



Figure 8. Seal installation into the lid groove on bottom surface.

5. A groove is also routed on the top surface of the lid to securely latch the chamber clamps to the lid and to withstand enough force to provide a firm seal. The groove is routed with a $\frac{1}{4}$ " (6 mm) straight router to a $\frac{1}{16}$ " (1.5 mm) depth (Fig. 9).



Figure 9. Groove in top surface of lid routed to $\frac{1}{16}$ " (1.5 mm) depth.

6. Create a centered hole for septa installation using a $\frac{15}{32}$ " (12 mm) drill bit (Fig. 10a). On the bottom surface of the lid (seal side), drill a $\frac{3}{4}$ " (19 mm) recessed groove $\frac{1}{4}$ " (6 mm) in depth (Fig. 10b) to allow for expansion of septa (septas from a Vacutainer serum vial; Becton Dickinson, #366430) after they are installed from the top of the chamber (Fig. 10c). This ensures that septa fit firmly in place and create a gas tight seal. The septum serves as the sampling port for needle insertion during gas sampling (Fig. 10d).



Figure 10. Installation of sampling septum.

7. Use a permanent marker to mark several places around the outside of the chamber at 5 cm (2") above the bottom edge of the chamber (Fig. 11). Inserting chambers into the soil to a depth up to this mark delineates an internal headspace volume of approximately 11.7 liters (L). Actual chamber headspace volumes are determined under field conditions and are discussed later.
8. Install a vent made from a 1/8" (3mm) swagelok bulkhead union and a 6" (15 cm) long coiled copper tube (Fig. 12). The vent allows for pressure equilibration with the atmosphere when the chamber is closed. Place a buna washer/O-ring between the inside chamber wall and the bulkhead union nut to prevent leakage around the fitting.

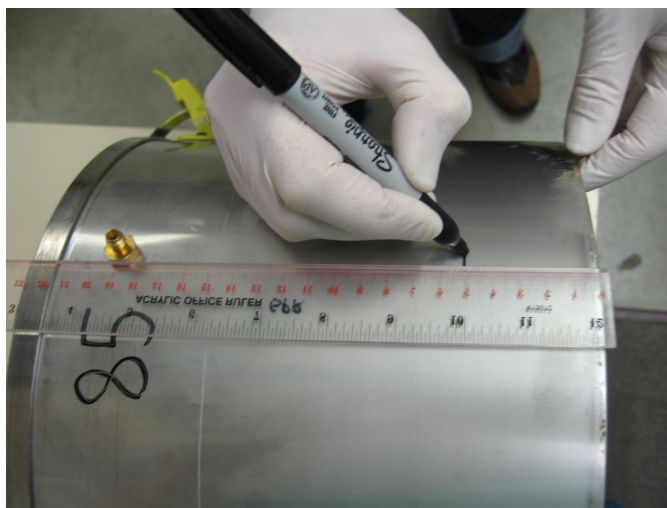


Figure 11. Marking the standard depth to which to insert the chamber into soil.

9. Three draw latch clamps are installed on the outside of the chamber to firmly hold the lid in place (Fig. 13). The clamp plate is first bent slightly to fit the curve of the chamber; it is then tool dipped to prevent corrosion. Clamps are securely fastened to the chamber wall with stainless or black oxide machine screws along with a rubber washer on each side of the chamber to prevent leakage around the screws. Clamps are purchased from McMaster-Carr, Inc. (Part# 1863A21).



Figure 12. Inside view of a chamber vent.



Figure 13. Clamp design and installation.

PART TWO: CHAMBER DEPLOYMENT AND GAS SAMPLING

Basic Sampling Equipment and Materials

- Stainless vented chamber and lid
- Sample vials: Exetainer vials, 5.9 ml flat bottom with septum cap and septa (Labco, <https://www.labco.co.uk/>)
- Chamber lid septa: Septum from Vacutainer serum vial, 10 ml, 16 × 100 mm (Becton Dickinson, #366430)
- Syringe: General purpose, Luer-lok tip, 10 ml (Becton Dickinson, #309604)
- Needles: Hypodermic, 22 gauge, 1" (Becton Dickinson, #305155)
- Stopwatch
- Pencils/pens
- Data recording sheets

PREPARATION

Chamber Deployment (minimum of one day before gas sampling)

1. Position the chamber on the soil surface in an area representative of the ecosystem of interest (Fig. 14). Spatial variability should be discussed prior to installation. For the GLBRC Biofuels Cropping System Experiment, one site is selected per plot.



Figure 14. Chamber position should represent the ecosystem tested.

2. To install the chamber, place a suitable sized piece of wood or board (e.g., 18x18" or 45x45cm by $\frac{3}{4}$ " or 2 cm thick plywood) on top of the chamber and tap evenly around the chamber circumference until the chamber is inserted into the soil up to the marked 5 cm line (Fig. 15). Depending on soil conditions, a fair amount of force may be necessary for proper insertion. Chamber deployment should result in as little soil disturbance as possible. And it must be done prior to a gas sampling campaign, typically at least one day before.
3. If soil disturbance occurs during chamber deployment and results in vertical 'gaps' between the soil surface and the chamber walls, either inside or outside the chamber, then it is necessary to gently tamp the soil and close the gaps to ensure a good seal between the soil and chamber walls. The chamber should now be firmly in place with no leaks. If this is not possible, remove the chamber and deploy elsewhere.
4. Check the copper tubing of the vent for potential plugging and place the tube opening about 1" (2.5 cm) above the soil surface.
5. Depending on specific research objectives, all plant litter on the soil surface should either be left within the chamber area during deployment (see Fig. 11) or carefully removed and re-positioned inside and outside the chamber following deployment.



Figure 15. Installing the chamber

Field Gas Sampling Campaign (typically one day prior)

1. Attach new septa to the sampling vial caps and screw onto vials (Fig. 16). Label the vials as appropriate for the particular sampling campaign— see below for more details.
2. Take spare vials, needles and syringes to the field.
3. Use new, sealed needles for each sampling day.
4. Ensure seals on syringe plungers are gas tight and plunger action is smooth – replace as necessary.
5. Prepare data recording sheets (see page 15 for example), and bring sheet covering and hard base for writing.
6. Check all chambers for damage (rodent, weather etc.) prior to sampling. Remove and replace if time allows, otherwise remove from sampling campaign on that day.
7. Check integrity of septa on chamber lids and replace if needed - damage by repeated needle insertion occurs. Typically replace after three sampling day events.
8. For convenience, place chamber lids and labeled sample vials next to matching chambers.



Figure 16. Sample vials, caps and septa.

GAS SAMPLING (day of gas sampling)

Lid Deployment

1. Check the lid seal for any defects, debris, or dirt that may prevent an effective seal.
2. Record the time of day the sampling regime starts.
3. Place an individual needle into the chamber lid septum to serve as a vent to relieve any induced pressure changes inside the chamber during lid placement.
4. Be careful not to step close to the chamber – working close to the chamber may result in increased soil compaction and alter gas flux. Make sure the chamber vent tubing is just above the soil surface.
5. Clamp the lid onto the chamber (Fig. 17) making sure lid is firmly and completely attached.
6. Start the stopwatch IMMEDIATELY after the first chamber lid is attached.
7. Remove the needle from the septum.

Note: Since multiple chamber samples are typically taken, THE STOPWATCH SHOULD RUN CONTINUOUSLY THROUGHOUT GAS SAMPLING OF ALL CHAMBERS. DO NOT RESET THE STOPWATCH AT EACH CHAMBER. Rather, keep the stopwatch running and write down the time each lid is deployed and each time a sample is taken – see below for more details.



Figure 17. Chamber lid deployment using needle vent.

Gas Sampling

1. Select the correct sample vial in the sampling sequence and insert an individual needle into its septum to act as a vent.
2. Insert the needle of the sampling syringe into the chamber lid septum and mix the chamber air three times by pulling out and then pushing in the syringe plunger (Fig. 18) – the needle should remain securely inserted in the sampling septum during this mixing. About 10 ml should be extracted then reintroduced each time to mix the chamber air prior to sampling.
3. After mixing, withdraw a 10 ml sample of chamber air, remove the syringe from the chamber septum and inject the sample into the vial with the vent needle in place (Fig. 19). Listen for gas escaping from the vent needle. After flushing remove the vent needle from the vial. This procedure flushes the vial with chamber air, in preparation for the actual sample injection.
4. Reinsert the syringe with attached needle into the lid septum and mix chamber air an additional three times as described above.
5. Draw a 10 ml sample from the chamber, remove the needle from the septum and inject sample into the vial. **RECORD THE SAMPLING TIME AS THE TIME WHEN THE AIR SAMPLE IS DRAWN UP INTO THE SYRINGE.** Inject the 10ml sample into the 5.9 ml flushed sample vial (Fig. 20). The over



Figure 18. Chamber mixing and headspace sampling.



Figure 19. Vial flush.



Figure 20. Gas sampling collection.

pressure helps prevent sample contamination and is corrected for before analysis. In our GC analysis, overpressurization by at least 3 ml is a must.

6. Repeat sampling procedure for all chambers in a given plot / treatment / block, etc.
7. Record chamber and vial ID and lid deployment and sampling times for all sampling events.
8. Include sampling of ambient air at the same stage in each sampling round of your sampling campaign. Also include duplicate samples using the same gas sampling procedure to sample the chamber air twice for a selected chamber in your sampling campaign.
9. Record soil temperature and moisture next to chamber at a consistent time during sampling regime – see below for more details on ancillary measurements.

Notes: 1) Although vials should be clearly labeled, to avoid incorrect vial venting or duplicate sampling, turn the vials upside down in the vial storage tray after sampling. 2) Listen for any gas leaks from the vials and septa, and replace and re-label new vial as necessary. 3) If rain is expected, data sheets can be photocopied onto rainproof paper or covered with suitable transparent sheeting.

Post Gas Sampling

1. Collect all sample vials from site. If there is an expected prolonged period of time between sampling and analysis, vials should be stored with the caps/septa submerged underwater to reduce pressure loss from the vial.
2. Remove all chamber lids and store appropriately. At this time, check sampling septa for damage and replace as necessary.
3. Headspace chamber volumes should be calculated as frequently as possible, ideally following each sampling event, but at least following chamber removal and replacement due to agronomic practice and/or damage. Measure the vertical height from the soil surface within the chamber to the top of the chamber lip (without attached lid) at a number of points (we use 4) around the chamber circumference. Calculate the average chamber height and use it to determine the headspace volume needed in gas flux calculations.

Sampling Time and Frequency

Prior to any prolonged sampling campaign at a particular site, N_2O fluxes from the various treatments at the site should be examined for linearity (i.e., a linear increase (or decrease) in concentration of N_2O over the time period to be used for chamber closure, e.g., 60 minutes, should be determined).

As far as possible, sampling should be carried out during the same time period for each gas sampling campaign (e.g., between 10 – 11 am, 2 - 3 pm, etc). In all cases the time of sampling (start and finish) should be recorded. Sampling of all chambers two or more times during an individual day should be carried out to help determine the daily (diurnal) variation of N_2O fluxes at the site, and therefore help in interpreting flux data if sampling times vary between days.

Sampling frequency should be as often as possible during the period of interest. Typically, one sampling per week would be suitable, as well as additional sampling events based on weather and agronomic practices at the site, e.g., immediately following a heavy rainfall and prior to and/or following fertilizer application.

Ancillary Measurements

Soil temperature: Soil temperature should ideally be measured during each sampling campaign and in close proximity to each of the chambers. For example, during gas sampling period T1, probes could be inserted into the soil next to the chamber wall to record temperature during chamber closure. If temperature can be shown to not vary significantly across the site and/or experimental treatments, then representative measurements should be recorded during the time of chamber closure on each gas sampling day.

Soil moisture: Soils should ideally be sampled for soil moisture during each sampling campaign and in close proximity to each of the chambers. If moisture can be shown to not vary significantly across the site and/or experimental treatments, then take representative measurements (e.g., Hydrosense II HS2 from Campbell Scientific) during the time of chamber closure on each gas sampling day.

Chamber air temperature: The potential for a significant change in chamber headspace temperature over external air temperatures during lid closure should be determined. This can be investigated in the field prior to the main gas sampling regime using temperature probes.

Soil nitrogen content: Soil ammonium (NH_4^+) and nitrate (NO_3^-) concentrations should be determined for each treatment on the day of gas sampling. Soil samples used for soil moisture determination can be used for this purpose.

Meteorological data: Air temperature, precipitation and atmospheric pressure should be recorded as close to the experimental site as possible.

FLUX CALCULATION

1. Determine the linear relationship (α_v) between the concentration of each GHG in sampling vials and the sampling time (slope of T0-T3 concentrations over time), in parts per million by volume per minute (ppm_v/min) which is equivalent to microliter per liter per minute ($\mu\text{L}/\text{L}/\text{min}$).
2. Convert α_v with units based on volume to α_m with units based on mass, in microgram per \times liter per minute, and correct for field temperature using the following application of the Ideal Gas Law:

$$\alpha_m = (\alpha_v \times M \times P) / (R \times T)$$

where:

α_m is expressed in $\mu\text{g N}$ or $\text{C}/\text{L}/\text{min}$

M = molecular weight of GHG ($28 \mu\text{g N}/\mu\text{mol N}_2\text{O}$ or $12 \mu\text{g C}/\mu\text{mol CO}_2$ or CH_4)

P = assumed atmospheric pressure = 1 atm

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

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

3. Calculate the flux (f_m) of GHG, as microgram element (N for N_2O ; C for CO_2 and CH_4) per square meter per hour), using the equation:

$$f_m = (\alpha_m \times V \times 60 \text{ min/h}) / A$$

where:

f_m is expressed in $\mu\text{g N}$ or $\text{C}/\text{m}^2/\text{h}$

α_m = as above, in $\mu\text{g}/\text{L}/\text{min}$

V = volume of gas in chamber, in L

A = soil surface area covered by chamber, in m^2

4. Convert hourly flux in square meters to daily flux in square meters by multiplying f_m by 24 h/day.
5. Convert daily flux (f_m) in square meters to grams element per hectare (ha) per day (f_{ha}) by multiplying the flux by 1 gram/1,000,000 micrograms and 10,000 m^2/ha , or:

$$f_{ha} = f_m \times 0.01$$

where:

f_{ha} = is expressed in g N or C/ha/day

6. Fluxes of CO_2 -C are typically much higher than fluxes of N_2O -N and CH_4 -C and can be converted to kilograms CO_2 -C by multiplying the converted flux by 1 kilogram/1000 grams.

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Gas samples for N₂O, CH₄ and CO₂

DATE _____ TIME _____ TECH _____

PLOT: F-1A

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-2A

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-3A

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-4A

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-1B

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-2B

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-3B

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-4B

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-1C

TIME
H M S

LID SHUT: _____

T0 _____

T1 _____

T2 _____

T3 _____

Soil Temp °C
Soil Moist VWC

PLOT: F-2C

TIME
H M S

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T0 _____

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Soil Temp °C
Soil Moist VWC

PLOT: F-3C

TIME
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T0 _____

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Soil Temp °C
Soil Moist VWC

PLOT: F-4C

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Soil Temp °C
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