PINEMAP Tier III Soil Greenhouse Gas (CO_2, N_2O, CH_4) Flux Protocol

Introduction

Forest ecosystems can help mitigate greenhouse gas (GHG) emissions, particularly via photosynthetic fixation of CO_2 in plant biomass and subsequently in soils. Although large potential atmospheric C sinks are available in the forest, the role of forest management practices in offsetting other GHG emissions (i.e., CH_4 and N_2O) is less familiar. Understanding the interaction of climate drivers and forest management on GHG fluxes is an essential step in reducing GHG emissions through forest management. This may be particularly true where N is added through fertilization. Fertilization has been shown to increase the biosphere-atmosphere flux of N_2O, while simultaneously decreasing the CH_4 sink strength of upland forest soils. Future GHG mitigation strategies involve the identification of fertilizer and other management practices that increase N and C use efficiency.

Objectives

1. Quantify the independent and combined impacts of fertilization and reduced throughfall on soil GHG fluxes.
2. Estimate the impacts of fertilization and reduced throughfall on soil nutrient availability.

Methods

Objective 1: *Quantify the independent and combined impacts of fertilization and reduced throughfall on soil GHG fluxes.*

Overview

Three primary approaches are traditionally employed to quantify gas fluxes at the soil-atmosphere interface (Hutchinson and Livingston, 2002): 1) estimates from diffusion theory combining measures of a diffusion coefficient of a gas with estimates of its concentration gradient based on Fick's law of diffusion, 2) micrometeorological techniques, like eddy-flux towers, which can integrate over larger spatial scales but can be prohibitively expensive to replicate across multiple sites, and 3) chamber methods where changes in gas concentrations can be measured by restricting the volume of air available for gas exchange.

Chamber methods are ideal where discrete temporal and spatial observations are desired, and are adaptable to a wide variety of studies and sampling environments (Hutchinson and Livingston, 2002). Further, they are relatively inexpensive, mechanically simplistic, yet powerful options to address the physical, chemical, and biological controls over soil-atmosphere gas flux (Holland et al., 1999). Chambers can be divided into two categories: static, or non-steady-state; and flow-through, or steady-state. Where the increased measurement sensitivity of very small fluxes is desired (e.g., CO_2, N_2O, and CH_4), static chambers are optimal because they prevent gas diffusion into the atmosphere (Hutchinson and Livingston, 2002). Where larger fluxes are to be measured at the same location for an extended period of time (e.g., NH_3 volatilization following urea fertilization), flow-through chambers are ideal. Thus, this protocol details a vented static chamber method to quantify soil GHG (CO_2, CH_4, and N_2O) fluxes.

Of note, it is not possible to accurately model annual soil GHG fluxes, and thus the global warming potential (GWP) of the system, without monthly, or preferably sub-monthly, sampling intervals. At present, the quarterly sampling occurring at the PINEMAP Tier III sites can only be

used as a method for determining if treatment differences exist, and not the magnitude of those differences as they relate to climate feedbacks.

Chamber design and installation

- Soil collars were constructed of white, Sch. 40, polyvinyl chloride (PVC) pipe with a 25.2 cm (10 in) inner diameter and a wall thickness of 1 cm.
- Collars were 17.5 cm (~7 in) high and on one end the outer edge was beveled at ~45° angle to facilitate field installation.
- In the field, three collars per plot were installed to a depth of ~ 5 cm (2 in) or until the collar was firmly seated and created a good seal at the soil atmosphere interface. Coarse roots were manually cut where necessary to facilitate this.
- Collars were installed in the field one month before start of the gas sampling.
- Collar lids used for sampling (shown below) were also constructed of white, Sch. 40, PVC pipe end caps. Each lid contained a sampling septum of grey butyl rubber and a vent with the soil surface area and chamber volume specific considerations detailed in Hutchinson and Livingston (2002).



GHG Sampling and Analysis

- Sampling should not take place during windy conditions, as the Venturi effect will more rapidly draw headspace gasses out of the vent tube. Again, Hutchinson and Livingston (2002) provide a good discussion of wind speed considerations relative to the size of the vent opening.
- Sampling should take place between 10:00am to 4:00pm when fluxes are expected to be at the maximum and diurnal variation in the flux is minimal.
- At the time of sampling, lids are placed on the collars and sealed at the collar-lid junction. Duct tape, bicycle tire tubes, and latex physical therapy bands (shown above) are all candidate materials to ensure a good seal.
- Using a disposable syringe, headspace gas samples are collected at 0, 20, 40, and 60 min after covering chamber with a lid and stored in a 10 ml crimp-sealed pre-evacuated vials for subsequent analysis.
- Immediately adjacent to the collar, soil moisture and temperature are collected at 10cm depth simultaneously with gas sampling.
- Gas samples are analyzed for CO_2, N_2O, and CH_4 on a modified Shimadzu GC-2010 gas chromatograph (with an ECD, FID, and methanizer) fitted with Shimadzu AOC-5000 Plus autosampler.
- Specific gas fluxes (i.e., g CO_2-C m^{-2} d^{-1}, mg CH_4-C m^{-2} d^{-1}, mg N_2O-N m^{-2} d^{-1}) are calculated by fitting a linear regression model to the data for gas concentration over time after the chamber was closed using the equation given below (Jacinthe et al., 1997; Shrestha et al., 2009):

$$F = \left(\frac{\Delta C}{\Delta t}\right)\left(\frac{V}{A}\right)k$$

Where, F = gas fluxes (mass of gas m^{-2} d^{-1})
ΔC = change in gas (CO_2, CH_4, N_2O) concentration inside chamber (m^{-3} air)
Δt = change in time (min)
V = chamber volume (m^3)
A = soil surface area covered by chamber (m^2)
k = time conversion factor (= 1440 min day^{-1})

Objective 2: *Estimate the impacts of fertilization and reduced throughfall on soil nutrient availability.*

Overview

This procedure details a method using ion exchange membranes (GE Osmotics, Inc./GE Power and Water, Inc.) to index soil NO_3^- and NH_4^+ availability. Although this protocol focuses on inorganic N, the same preparation, field, and extraction procedures are appropriate for the estimation of other nutrients (e.g., P, Ca, Mg, Mn, Fe, Al, Zn, and B).

Membrane preparation

- Cut independent cation and anion membranes into 5 x 10cm rectangles. Punch hole in membrane to subsequently attach string for retrieval after deployment.
- Thoroughly rinse membranes in deionized H_2O to remove get the glycerol coating used for shipment.
- Shake for 10 min in a fresh 5% (vol/vol) HCl solution.
- Thoroughly rinse membranes in deionized H_2O.
- Shake for 10 min in deionized H_2O.
- Soak in 1*M* NaCl for at least 24 hours (for storage longer than 72 h, keep membranes in 1*M* NaCl at 4°C).

Membrane deployment, collection, and analysis

- Rinse membranes with deionized H_2O prior to taking them to the field.
- Tie membranes with nylon string (e.g., 15-20 lb. test fishing line) so that they can be tied to a flag for retrieval.
- Adjacent to each GHG flux collar, install cation and anion exchange membranes at a 45° angle from the soil surface and leave until next gas sampling.
- Upon collection from soil, place in zip-lock bags for transport.
- Scrape/wash with deionized water to remove any residual soil.
- Combining cation and anion exchange membranes for each location, shake for 1 h in 1*M* KCl (25 ml of 1*M* KCl for each membrane) in an polyethylene or glass bottle.
- Open bottles and remove membranes using tweezers.
- Decant extraction solution for subsequent analysis (filter if needed). If not analyzed immediately, store extractant solution at 4°C prior to analysis.

Membrane cleaning and recharge

- Rinse membranes with deionized H_2O twice.

- Wash membranes in 5% HCl (vol/vol) by shaking them for 10 minutes in the acid solution. Make fresh acid solution for every wash.
- Rinse membranes with deionized H_2O twice.
- Shake membranes for 10 minutes in the deionized H_2O.
- Charge them again with 1*M* NaCl for 24 hours as detailed above and repeat.

References

Holland, E.A., G.P. Robertson, J. Greenberg, P.M. Grofman, R.D. Booner, and J.R. Gosz. 1999. Soil CO_2, N_2O, and CH_4 Exchange. p. 185-201. *In* G.P. Robertson et al. (ed.) Standard Soil Methods for Long-Term Ecological Research. Oxford University Press, New York.

Jacinthe, PA and W.A. Dick. 1997. Soil management and nitrous oxide emissions from cultivated fields in southern Ohio. Soil and Tillage Research 41:221-235.

Hutchinson, G.L., and G.P. Livingston. 2002. Soil-Atmosphere Gas Exchange. p. 1159-1182. *In* Dane, J.H., and Topp, G.C. (ed.) Methods of soil analysis. Part 4. Physical methods. SSSA, Madison, WI.

Shrestha R.K., R. Lal, and C. Penrose. 2009. Greenhouse gas emissions and global warming potential of reclaimed forest and grassland soils. J. Environ. Qual. 38:426–436.

Prepared by Brian Strahm (brian.strahm@vt.edu) and Raj Shrestha (rajs@vt.edu).

Updated on October 5, 2012.