# InGOS – Integrated non-CO2 Observing System

1. **N2O Chamber intercomparison campaign 2014**

**Mark Lee, SRUC, Hestan House, The Crichton, Dumfries, DG1 4TA, UK.**

**E:** [**mark.lee@sruc.ac.uk**](mailto:mark.lee@sruc.ac.uk)**, T: +44 (0)7500 107457.**

**Proposed visiting dates:** 8th -11th July 2014.

**Local organizers:** Janne Korhonen**;** Mari Pihlatie**;** Jukka Pumpanen**;** Mika Korkiakoski, Mari Rämö and Elisa Halmeenmäki.

**Campaign dates:** 16th June - 30th July 2014.

**Location:** Warm machinery hall, Hyytiälä Forestry field station, Juupajoki, Finland.

**Instrumentation:** N2O lasers and calibration tank.

1. **Background**
   1. Significance of the research

Chamber measurements are associated with systematic and random errors. The major error sources are systematic and random error of the estimation of the flux based single chamber measurement and the error associated by the large spatial variability of the soil flux and the low spatial coverage of the measurements. Systematic errors of CO2 chambers have been quantified by [Pumpanen et al. (2004](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_11)) and systematic errors of static CH4 by [Pihlatie et al. (2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)) and [Christiansen et al. (2011](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_1)). In this study, we aim to gain more knowledge on the errors, and also provide methods to control them. Until recently, soil N2O fluxes have been measured based on static chambers and analyzing air samples on a gas chromatograph (CG). Detecting small changes of N2O concentration is difficult with a CG, and thus in many cases, the analysis of gas concentrations has been the largest source of error in soil N2O flux estimations. Recently N2O LASER instrumentation has become available soil N2O flux measurement, and has practically eliminated this problem of random error when estimating the flux for a single chamber. However it must be noted that the N2O LASER instrumentation is currently relatively expensive, and thus only few research groups currently have N2O laser instrumentation. The random error of soil N2O efflux associated with high spatial variability cannot be solved by N2O LASER instrumentation. Low-cost methods for estimating the soil N2O efflux from various spatial locations would be a solution for this problem. One option would be to sample the N2O concentration in the soil airspace periodically, and estimate the flux based on this. While this method is probably not very accurate, it could give information about the range and variability of the flux. If the systematic errors of this method are small, the random errors can be got rid of by large number of samples. One purpose of this study is to evaluate the possibilities of using the information of soil N2O concentration for estimating soil N2O efflux. The same principles could be used for other compounds, such as CO2 and CH4 as well. Systematic errors related to estimating the flux for a single chamber. Another option is to support the N2O laser measurements by measuring spatial variability of the N2O soil flux based on manual chamber measurements. Such a measurement is laborious if systematic errors are to be avoided. However, if the amount of systematic error is known, it can be corrected. We also want to study this, and in conjunction with soil N2O concentration measurements, a low-cost method for eliminating the uncertainty caused by high spatial variability could be developed. Like in the case of spatial variability, N2O laser instrumentation cannot alone solve the problems of systematic errors of estimating the soil N2O flux from a single chamber. These errors are in general similar for other compounds, such as CO2 and CH4 as well. For CO2 and CH4 many kinds of systematic errors are reported. Typically these errors are related to pressure changes inside the chamber, effects of wind speed, or leaking of the chamber. The chamber measurements require the assumption that the soil gas storage under the chamber does not change, and the pressure and wind effects are violating this assumption. In an ideal case, the gas flux during the enclosure saturates, an exponential function is fitted to the gas concentration data against time, and the flux is calculated in the beginning of the enclosure using the fitted parameters of the exponential function. However, if gas is accumulating in the soil, the rate of change of gas concentration in the chamber headspace is too low in the beginning, causing erroneous curvature for the exponential function. This naturally leads to underestimation of the flux. If the case is the opposite, for example is the fan is ventilating the chamber headspace too efficiently, then using exponential fit causes overestimation to the flux. In this study, we want to find out how much the storage change can change the flux estimation, how to identify this in data analysis, and how to correct the flux for the storage effect. In addition, in this study we will measure the leaking of the chambers.

* 1. Previous research relevant to the topic and how the proposed project links to this

My colleague Stephanie Jones from SRUC (previously SAC) attended Hyytiälä Forestry field station in previous years. Stephanie contributed our chambers and methodology as part of a CH4 chamber intercomparison campaign. This work has been published recently as [Pihlatie et al. (2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)).

* 1. Links with current research of the applicant

I am currently working on the GREENHOUSE project ([www.greenhouse-gases.org.uk/greenhouse](http://www.greenhouse-gases.org.uk/greenhouse)). Here, we are using chambers, eddy covariance towers and aircraft to measure greenhouse gas emissions over a two year period, across different scales and at different locations whilst quantifying the effects of weather and management (e.g. grazing and fertilisation). It would be interesting to compare our chamber methodology in intensively managed grassland with those that are used by our project partners who are using different methodologies in different ecosystems e.g. arable and forests. This will help us to understand some of the methodological drivers of inter-site variation in N2O fluxes.

1. **Objectives**

To evaluate the importance of storage effects to the systematic error of the flux estimate driven by disturbance in installing the chamber (Christiansen et al. 2011), wind (pressure effects, effect on chamber and soil airspace volume, heating and venting tubes ([Xu et al. 2006](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_16)). We will be calculating transport coefficient during the enclosure period ([see also Lai et al. (2012](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_7)) and calculating the transport coefficient for the end of the measurement (Korhonen et al. 2013). We will evaluate the methods of extrapolating the flux to undisturbed flux by extrapolating to t0, extrapolating to ambient concentration and extrapolating using the knowledge of transport coefficient and soil concentration, when known (Korhonen et al 2013). We will compare the ways of estimating the saturation of the flux during chamber measurement by measuring time transport coefficient from exponential fit (aka time constant), calculating transport coefficient based on soil porosity and calculating transport coefficient based on soil and air concentrations and flux rate continuously (Korhonen et al. 2013). We will also test if it makes sense to use linear fit for flux estimation, and correcting the systematic error (Korhonen et al. 2010).

1. **Methods and materials (legal and ethical issues)**

Control flux is measured based on the concentration change inside the tank and chamber flux is measured based on the concentration change inside the chamber. One chamber per time in measurement, because control flux is equal to flux from sandbed plus chamber flux (both fluxes weighed by surface area, typically chamber covers only about 20% of the pumpeli surface area). This allows measuring gas concentration below the chamber, in addition to outside the chamber in sand bed. The sand depth can be adjusted (with the new calibration tank). Gas concentration inside the Pumpeli (calibration tank) does not affect the systematic error of the flux estimation. This is according to theory, and also verified by [Pihlatie et al. (2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)). In last campaign ([Pihlatie et al., 2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)) a lot of time was used by changing the flux level and waiting the system to stabilize. We do not need several “flux levels”, but good to have some replicate measurements. The reference flux and gas concentration in the chamber decrease with time. The concentration in the sand bed must be stabilized between the measurements. Stabilization time must be checked from the sand concentration data. The time depends on sand depth. It would be nice to have different soil (sand) types. In last campaign ([Pihlatie et al., 2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)) a lot of time was used by changing the sand, especially to fine and wet sand. The differences of the transport coefficients between the sands were small and so we can change the depth of the sand to change the transport coefficient. There will be 3 replicates at 3 wind speeds using 3 sand height totally 27 measurements per chamber.

Schedule:

* + Tuesday: Day of the arrival and set-up
  + Wednesday-Thursday: 2 days for the basic measurements
  + Friday: Final tests and departure
  + Visit in total 4 days (3.5 working days).

1. **Implementaton: timetable, budget, distribution of work**

Typical daily timetable:-

DAY1 - Wednesday (Thursday equal to Wednesday except different sand depth), Friday = additional tests.

SD = sand depth, SD05 = 5 cm, SD10 = 10 cm, SD15 = 15 cm, SD20 = 20 cm

WS = wind speed, WS0 = 0 m/s, WS5 = 5 m/s

* + Previous evening: Preparation of the sand for sand depth (SD) 1, injection of gases to the chamber. Installation of chamber collars. Over-night measurement of chamber leaking (letting the chamber concentration go to steady state). In the middle of the night, fans start and/or chamber is replaced.
  + 07:00-07:15 Removal of chambers.
  + 7:15-7:30 Injection of gases
  + 07:30-08:45 Breakfast, stabilization of the calibration tank.
  + 08:45-10:15: SD20, WS0: Chamber(s) : 3 replicate measurements 15min measurement + 15min stabilization = 1,5h.
  + 10:15-11:45 SD20, WS5: Chamber (s): 3 replicate measurements 15min measurement + 15min stabilization = 1,5h.
  + 11:45-12:15 Lunch
  + 12:15-13:45 SD20, WS0: Chamber(s) : 3 replicate measurements 15min measurement + 15min stabilization = 1,5h.
  + 13:45-14:00 Coffee break
  + 14:15-15:45 SD20, WS5: Chamber (s): 3 replicate measurements 15min measurement + 15min stabilization = 1,5h.
  + 15:45-17:15 SD20, WS?/ADDITIONAL TESTS: Chamber (s): 3 replicate measurements 15min stabilization + 15min measurement = 1,5h. If someone wants to sample chamber headspace with syringes to be analyzed at their own institutes by GC, this can be done here.
  + 18:00-19:45 Changing sand bed, preparations for next day

**Estimated budget:-**

Return air fare (Manchester – Helsinki) - EUR250

Return train fare (Helsinki – Hyytiala) - EUR150

Total travel = EUR400

Accommodation/food - 4 x EUR50

Total Accommodation/food = EUR200

Total = EUR600

1. **Expected results and possible risks**
   1. Expected scientific impact of the research: more accurate measurements of N2O fluxes, improved quantification of the errors between methodologies and recommendations of improvements to existing chamber methodologies to improve flux estimates.
   2. Global application.
   3. It is expected that there will be at least one research paper on N2O inter-comparison as per [Pihlatie et al. (2013](file:///C:\Users\MLee\AppData\Local\Microsoft\Windows\Temporary%20Internet%20Files\Content.Outlook\7XK10Z87\Chamber%20calibration%20campaign%202014%20plan_12%2005%202014.doc#_ENREF_10)).
   4. Data is available to all participants
2. **Key literature**

Christiansen, J. R., Korhonen, J. F. J., Juszczak, R., Giebels, M., and Pihlatie, M.: Assessing the effects of chamber placement, manual sampling and headspace mixing on CH4 fluxes in a laboratory experiment, Plant and Soil, 343, 171-185, DOI 10.1007/s11104-010-0701-y, 2011.

Conen, F., and Smith, K. A.: A re-examination of closed flux chamber methods for the measurement of trace gas emissions from soils to the atmosphere, Eur J Soil Sci, 49, 701-707, DOI 10.1046/j.1365-2389.1998.4940701.x, 1998.

Conen, F., and Smith, K. A.: An explanation of linear increases in gas concentration under closed chambers used to measure gas exchange between soil and the atmosphere, Eur J Soil Sci, 51, 111-117, DOI 10.1046/j.1365-2389.2000.00292.x, 2000.

Creelman, C., Nickerson, N., Risk. C., 2013. Quantifying Lateral Diffusion Error in Soil Carbon Dioxide

Respiration Estimates using Numerical Modeling. Soil Sci. Soc. Am. J. 77:699–708

Hossler, K., and Bouchard, V.: The joint estimation of soil trace gas fluxes, Soil Sci Soc Am J, 72, 1382-1393, DOI 10.2136/sssaj2007.0232, 2008.

Hutchinson, G. L., and Livingston, G. P.: Vents and seals in non-steady-state chambers used for measuring gas exchange between soil and the atmosphere, Eur J Soil Sci, 52, 675-682, DOI 10.1046/j.1365-2389.2001.00415.x, 2001.

Kutzbach, L., Schneider, J., Sachs, T., Giebels, M., Nykanen, H., Shurpali, N. J., Martikainen, P. J., Alm, J., and Wilmking, M.: CO2 flux determination by closed-chamber methods can be seriously biased by inappropriate application of linear regression, Biogeosciences, 4, 1005-1025, 2007.

Lai, D. Y. F., Roulet, N. T., Humphreys, E. R., Moore, T. R., and Dalva, M.: The effect of atmospheric turbulence and chamber deployment period on autochamber CO2 and CH4 flux measurements in an ombrotrophic peatland, Biogeosciences, 9, 3305-3322, 10.5194/bg-9-3305-2012, 2012.

Levy, P. E., Gray, A., Leeson, S. R., Gaiawyn, J., Kelly, M. P. C., Cooper, M. D. A., Dinsmore, K. J., Jones, S. K., and Sheppard, L. J.: Quantification of uncertainty in trace gas fluxes measured by the static chamber method, Eur J Soil Sci, 62, 811-821, DOI 10.1111/j.1365-2389.2011.01403.x, 2011.

Pihlatie, M., Pumpanen, J., Rinne, J., Ilvesniemi, H., Simojoki, A., Hari, P., and Vesala, T.: Gas concentration driven fluxes of nitrous oxide and carbon dioxide in boreal forest soil, Tellus Series B-Chemical and Physical Meteorology, 59, 458-469, DOI 10.1111/j.1600-0889.2007.00278.x, 2007.

Pihlatie, M. K., Christiansen, J. R., Aaltonen, H., Korhonen, J. F. J., Nordbo, A., Rasilo, T., Benanti, G., Giebels, M., Helmy, M., Sheehy, J., Jones, S., Juszczak, R., Klefoth, R., Lobo-do-Vale, R., Rosa, A. P., Schreiber, P., Serca, D., Vicca, S., Wolf, B., and Pumpanen, J.: Comparison of static chambers to measure CH4 emissions from soils, Agricultural and Forest Meteorology, 171, 124-136, DOI 10.1016/j.agrformet.2012.11.008, 2013.

Pumpanen, J., Kolari, P., Ilvesniemi, H., Minkkinen, K., Vesala, T., Niinisto, S., Lohila, A., Larmola, T., Morero, M., Pihlatie, M., Janssens, I., Yuste, J. C., Grunzweig, J. M., Reth, S., Subke, J. A., Savage, K., Kutsch, W., Ostreng, G., Ziegler, W., Anthoni, P., Lindroth, A., and Hari, P.: Comparison of different chamber techniques for measuring soil CO2 efflux, Agricultural and Forest Meteorology, 123, 159-176, DOI 10.1016/j.agrformet.2003.12.001, 2004.

Pumpanen, J., Ilvesniemi, H., Kulmala, L., Siivola, E., Laakso, H., Kolari, P., Helenelund, C., Laakso, M., Uusimaa, M., and Hari, P.: Respiration in boreal forest soil as determined from carbon dioxide concentration profile, Soil Sci Soc Am J, 72, 1187-1196, DOI 10.2136/sssaj2007.0199, 2008.

Rochette, P.: Towards a standard non-steady-state chamber methodology for measuring soil N2O emissions, Anim Feed Sci Tech, 166-67, 141-146, DOI 10.1016/j.anifeedsci.2011.04.063, 2011.

Venterea, R. T., and Baker, J. M.: Effects of soil physical nonuniformity on chamber-based gas flux estimates, Soil Sci Soc Am J, 72, 1410-1417, DOI 10.2136/sssaj2008.0019, 2008.

Venterea, R. T., Spokas, K. A., and Baker, J. M.: Accuracy and Precision Analysis of Chamber-Based Nitrous Oxide Gas Flux Estimates, Soil Sci Soc Am J, 73, 1087-1093, DOI 10.2136/sssaj2008.0307, 2009.

Xu, L. K., Furtaw, M. D., Madsen, R. A., Garcia, R. L., Anderson, D. J., and McDermitt, D. K.: On maintaining pressure equilibrium between a soil CO2 flux chamber and the ambient air, J Geophys Res-Atmos, 111, 10.1029/2005jd006435, 2006.