



Selection of the most suitable sampling time for static chambers for the estimation of daily mean N₂O flux from soils

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ABSTRACT

Soil N₂O fluxes are frequently assessed by the use of static chambers with a single daily sampling. In this study, two experiments were conducted in two contrasting climatic locations, one in Edinburgh, UK, and the other at Seropédica, Rio de Janeiro State, Brazil. Soil N₂O fluxes were monitored every 6 h for 30 days during the summer in Edinburgh by the use of an automatic chamber system, and every 3 h for 5 days at Seropédica, using a manually-sampled static chamber. Air and soil temperatures were also measured at the same time as the N₂O fluxes. The principal driver of N₂O flux within any diurnal period was found to be soil temperature. Regression analysis showed that, for both places, the evenings (21:00–22:00 h) and mornings (09:00–10:00 h), were the times that the flux best represented the daily mean. The ability to work in daylight make the morning period the preferred one.

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1. Introduction

Static chambers are widely used for measuring greenhouse gas fluxes from soils. The most common static chamber procedure involves manual sampling of chamber headspace gas using syringes (e.g. Ball et al., 1999; Du et al., 2006; Jantalia et al., 2008) or more advanced systems such as the use of vacuum pumps or automated flux monitoring systems (e.g. Akiyama et al., 2000; Dobbie and Smith, 2003).

The high spatial variability of N₂O fluxes, related to hotspots of production in soil, requires many chamber replicates to evaluate N₂O fluxes with reasonable precision. Moreover, it is considered a good practice to take four or five successive air samples (at 5 or 10 min intervals for example) after chamber deployment to examine possible deviations from linearity of N₂O flux measurements with time (Rochette and Eriksen-Hamel, 2008). It is also advisable that sampling regimes should be intensified after any

event that raises mineral N levels in the soil or that might create an oxygen limitation in the pore space (Smith and Dobbie, 2001). However, compromises often have to be made in order to limit the number of samples to manageable quantities, so soil N₂O daily emission calculations are usually based on the extrapolation of a single daily measurement during a short period to represent the mean flux for a full 24 h period.

In most environments N₂O formation in soil is controlled mainly by available C and mineral N, soil O₂ concentration in the soil pore space and temperature (Granli and Bockman, 1994). Available soil C and N are not expected to vary significantly during a period of one day, unless crop residues and fertilizers are added to the soil. However, soil O₂ concentration can decrease rapidly after rainfall events or irrigation, and soil temperature is likely to follow the diurnal fluctuation of air temperature. The N₂O flux generally increases exponentially with soil temperature, with high Q₁₀ values sometimes observed (e.g. Brumme, 1995; Flessa et al., 2002; Dinsmore et al., 2009), which can be explained by a combination of an expansion in anaerobic zones triggered by the acceleration of soil respiration, and the increasing denitrification rate per unit of anaerobic volume (Smith et al., 2003). The saturation of soil pore space with water also leads to exponential changes in soil N₂O fluxes, but the effect seems not to be so rapid (Russow et al., 2000) as that demonstrated for changes in soil temperature.

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Several studies have found a close relationship between diurnal variations in air temperature and N₂O fluxes, with a general pattern of higher fluxes during the day and lower fluxes during the night, accompanying the trends of soil temperature (Ryden et al., 1978; Denmead et al., 1979; Akiyama et al., 2000; Livesley et al., 2008). However, other studies failed to find this relationship (Blackmer et al., 1982; Chao et al., 2000; Du et al., 2006) or have demonstrated a substantial lag between temperature and flux maxima (Thomson et al., 1997). Ryden et al. (1978) suggested that N₂O fluxes could be measured at any time of the day and night as long as the afternoon peak coinciding with maximal daily temperature was avoided.

The diurnal air temperature fluctuation generally follows a sinusoidal path and is described by well-established models (Parton and Logan, 1981; Ephraim et al., 1996). Hence the mean temperature for the day occurs sometime after sunrise and after sunset. If the air temperature is a powerful driver of the changes in N₂O fluxes observed during the 24 h of the day, it can be hypothesized that there are two times in the day when the chance of the observed N₂O flux is most representative of the mean N₂O flux for the day. We investigated this issue and tested the hypothesis at two sites in contrasting climates (Scotland, UK and Rio de Janeiro, Brazil).

2. Material and methods

2.1. Sites

The experiments were carried out under field conditions at two sites with contrasting climates. A first experiment was set up in Edinburgh, Scotland, UK, at 55° 56'58"N and 3°9'37"W, with a mean daylight of 17 h in the summer (June to August) which was the season when measurements were made. Another experiment was carried out at Seropédica, Rio de Janeiro State, Brazil, at 22°45'28"S 43°40'54"W, with a mean daytime of 12 h at the beginning of autumn (April). According to the World Meteorological Organization (www.worldweather.org) mean daily minimum and maximum temperatures for Edinburgh are about 9 °C and 18 °C, respectively, during June and July. Monthly mean rainfall is similar over the whole year, varying from 51 to 57 mm during the summer, with an average of 13 days with rain. In the case of Seropédica, Rio de Janeiro State, mean monthly minimum and maximum temperatures for April are 22 °C and 28 °C, respectively, and mean rainfall is approximately 138 mm with 10 days of rain.

2.2. Edinburgh experiment

The experiment in Edinburgh was set up in a small area of the science campus of the University of Edinburgh that had been used for cropping potatoes and vegetables. For the current experiment, the soil in an area of 3 m² was well mixed with a spade to the depth of 40 cm in order to get a homogenized profile. Gravel, visible roots and other plant parts were manually removed. To avoid excessive soil looseness, some compaction was applied to the soil at 20 cm depth and then at each 5 cm up to the soil surface. During this phase, a thermocouple was buried to a depth of 10 cm and a 30-cm-long CS615 time-domain reflectance (TDR) probe (Campbell Scientific, Edmonton, Alberta, Canada) was inserted diagonally from the soil surface in order to measure the water content of the top 10 cm of the soil. Both probes were connected to a CR10X datalogger (Campbell Scientific, Edmonton, Alberta, Canada). A volume of water equivalent to an irrigation of 20 mm was applied 3 days before starting measurements.

A sample of the soil layer of 0–20 cm presented a sandy-loam texture (58% sand; 38% silt; 4% clay), 4.84% total C, 0.34% total N

and a soil pH of 5.02. The soil bulk density of the 0–10 cm layer was 1.02 Mg m⁻³, after the compaction process.

The N₂O flux measurements were performed every 6 h using one automatic static closed chamber. Samples started to be taken at 03:00 GMT (04:00 British Summer Time) on the first day and thenceforth there were 4 samplings a day during 30 days (17 June – 16 July 2005).

The chamber design was exactly the same as that described by Dobbie and Smith (2003). Briefly, the automated chamber was of the base-lid type, made of galvanized steel. The base was a frame of 70 × 70 cm in area and 30 cm height, with the bottom edges of the walls inserted into the soil to a depth of 8 cm. The chamber lid was also made of galvanized steel with the same area dimensions as the base. When the lid was in the closed position, it compressed a rubber gasket cemented to its underside against a horizontal flange at the top of the base walls, thus providing a gas seal. A control unit contained an air flow pumping system, and a timer/programmer unit to control the opening and closure of the chamber. A second module accommodated a set of Tedlar bags and a switching valve that allowed evacuation of the bags and the pumping in of sufficient chamber head space air for the N₂O analysis. Immediately after lid closure, a sample of the headspace air was pumped to one of the empty Tedlar bags, and another sample into a second bag after 40 min, before the chamber lid was raised. The air sample in each Tedlar bag was later transferred to 20 mL pre-evacuated chromatography vials using gastight syringes and then analysed with a gas chromatograph (GC) fitted with an electron capture detector as described in Dobbie and Smith (2003).

Fluxes of N₂O were calculated on the basis of an analytical curve of N₂O standards in nitrogen used to transform the integrated area of each sample peak into N₂O concentration. Nitrous oxide fluxes were expressed in μg N–N₂O m⁻² h⁻¹ using the equation: N₂O flux = (δC/δt)(M/Vm)V/A, where δC/δt is the change in N₂O concentration (in μL L⁻¹) in the chamber after the incubation time (in hours); M is the molecular weight and Vm is the molecular volume of N₂O at the sampling temperature, and V is the volume of the chamber in litres and A the area in m².

During the sampling period, air and soil temperatures at 10 cm depth below the chamber were monitored hourly along with the soil moisture. An extra thermocouple was fixed above the chamber, but protected from direct sunlight, to record the external air temperature.

2.3. Seropédica experiment

The experiment in Seropédica, Rio de Janeiro State, was performed on a soil covered with the grass *Paspalum notatum* [Flügge] cv. Batatais. No soil preparation was carried out in this area. A sample of the soil layer of 0–20 cm presented a sandy texture (72% sand; 8% silt; 20% clay), 0.94% total C, 0.01% total N and a soil pH of 5.41. The bulk density of the 0–10 cm soil layer was 1.34 Mg m⁻³. The experiment was set up on 9 April 2008, with 5 days of gas sampling every 3 h starting from 01:00 h (Brazilian Standard Time). As the soil was of very low fertility, urea fertilizer was applied at a rate of 10 g N m⁻² two days before starting the measurements, along with a 10 mm irrigation, to stimulate N₂O production.

Five manually-sampled closed static chambers were used for the soil N₂O flux measurement. Each was composed of a rectangular hollow metal frame, 38 cm wide × 58 cm long × 6 cm in height which was inserted 5 cm into the soil and left for the whole experimental period. A trough was made around the top of the frame, and filled with soft rubber to ensure the system could be sealed after coupling the top portion of the chamber. This was a polyethylene tray of the same width and length as the base, 9 cm high, and was only coupled to the base during the periods of gas sampling. The top of each chamber had a three-way tap with Luer

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