



Automatic high-frequency measurements of full soil greenhouse gas fluxes in a tropical forest

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Abstract. Measuring *in situ* soil fluxes of carbon dioxide (CO₂), methane (CH₄), and nitrous oxide (N₂O) continuously at high frequency requires appropriate technology. We tested the combination of a commercial automated soil CO₂ flux chamber system (LI-8100A) with a CH₄ and N₂O analyzer (Picarro G2308) in a tropical rainforest for 4 months. A chamber closure time of 2 minutes was sufficient for a reliable estimation of CO₂ and CH₄ fluxes (100% and 98.5% of fluxes were above 25 Minimum Detectable Flux – MDF, respectively). This closure time was generally not suitable for a reliable estimation of the low N₂O fluxes in this ecosystem but was sufficient for detecting rare major peak events. A closure time of 25 minutes was more appropriate for reliable estimation of most N₂O fluxes (85.6% of measured fluxes are above $MDF \pm 0.002 \text{ nmol m}^{-2} \text{ s}^{-1}$). Our study highlights the importance of adjusted closure time for each gas.

30 1 Introduction

Carbon dioxide (CO₂), methane (CH₄) and nitrous oxide (N₂O) are the three main greenhouse gases (GHGs) in terms of radiative forcing. Increases in these GHG concentrations in the atmosphere is driving anthropogenic global warming. Understanding the magnitude of GHG fluxes in natural ecosystems has recently become a priority in the study of GHG balances (Merbold et al., 2015). Tropical intact forests cover 1392 Mha globally and represent about 70% of the total tropical forest 35 area (1949 Mha), which accounts for the largest area of global forest biomes (~50%). Very few reliable long term datasets on full GHG balances are available from tropical ecosystems, despite their known importance for the global cycles of these three GHGs (Dutaur and Verchot, 2007). This is in part due to the challenges of designing and operating continuous, multi-gas flux analysis systems in tropical forests. Soil processes in particular are responsible for an important part of GHGs that are produced or consumed in tropical ecosystems (Oertel et al., 2016). Soil physical, chemical, and biological characteristics are linked to



variation in GHG emissions from soils, which in turn can display very high spatial and temporal variability (Arias-Navarro et al., 2017; Silver et al., 1999).

Historically, soil GHG fluxes (emission or consumption) have been measured using the static chamber method. This involves closing chambers manually for a known period of time, usually 30-60 minutes, and repeated collection of air samples for further analysis via gas chromatography (Verchot et al., 1999, 2000). Fluxes are then computed from the increase (or decrease) in gas concentration per unit time, per surface area enclosed by the chamber, and corrected by the volume of the chamber. While these labor-intensive and time-consuming manual measurements are well adapted to capture high spatial flux variability (Arias-Navarro et al., 2017; Pumpanen et al., 2004), they do not capture high temporal variation, which is necessary for the accurate estimation of annual GHG budgets. Moreover, short term, transient spikes in the emission or consumption of these GHGs likely remains undetected with static chamber methods, imposing a lost opportunity to fully understand the production or consumption processes of GHGs and their response to rapidly changing environmental conditions. One of the key challenges of contemporary GHG flux research is to close these knowledge gaps in order to improve the quantitative prediction of GHG fluxes (Merbold et al., 2015).

The use of automatic chambers is one approach to obtain continuous estimation of soil GHG flux data at high temporal frequency (several measurements per days) at various sampling points. Since the 1970s (Denmead, 1979), a variety of technical solutions for automated flux sampling have been developed (Ambus et al., 2010; Breuer et al., 2000; Görres et al., 2016; Kostyanovsky et al., 2018; Petrakis et al., 2017a; Savage et al., 2014), particularly for soil CO₂ fluxes. However, accurate detection of CH₄ and N₂O fluxes from soils via flow through systems is more difficult than CO₂ due to significantly lower background concentrations and lower flux rates (Kostyanovsky et al., 2018). The budgetary requirements for large infrastructure and intensive maintenance as compared to manual chamber measurements have prevented the widespread application of automated systems. The use of automated and continuous methods to estimate full GHG budgets *in situ* remains scarce, especially in complex biomes with extreme climate such as tropical forests. Therefore, only a few studies actually address the difficulties and challenges associated with operating these systems under field conditions (Görres et al., 2016; Koskinen et al., 2014).

Recent technological advances have now made more automated chamber systems commercially available, and an increasing number of custom-made systems are being designed and deployed for soil GHG flux measurements [17,18]. Here, we present a detailed field deployment of a custom built, automated soil GHG flux system – the LI-8100A Soil CO₂ Flux System (LI-COR Biosciences Inc., Lincoln, NE, USA) running in line with a Picarro G2308 (Picarro Inc., Santa Clara, CA, USA). Using a 4 month dataset of continuous measurements of CO₂, CH₄, and N₂O fluxes simultaneously under tropical forest conditions, we present an optimized sampling protocol for the estimation of the full GHG budget in this ecosystem.

2 Methods

2.1 Measurement site



This study was conducted in the Paracou research station (5°15'N, 52°55'W), located in the coastal area of French Guiana, South America. The automated soil GHG flux system was deployed in the footprint of the Guyaflux site, which holds a 55 m-tall tower upon which canopy CO₂, H₂O and energy fluxes have been monitored since 2004 using the eddy covariance technique (Aguilos et al., 2018; Bonal et al., 2008). The site is covered with tropical pristine forest and located in the northernmost part of the Guiana shield. It is characterized by a succession of small, elliptical hills rising to 10–40 m a.s.l., sometimes associated with plateaus of similar altitude.

The soils are mostly nutrient-poor acrisols (FAO-ISRICISS, 1998) with pockets of sandy ultisols developed over a Precambrian metamorphic formation called the 'Bonidoro series', and composed of schist and sandstone, sporadically traversed by veins of pegmatite, aplite and quartz (Bonal et al., 2008). The forest around the tower is characteristic of a tropical pristine forest with both high tree density (~ 620 trees with a dbh>10 cm ha⁻¹) and species richness (~ 140 species ha⁻¹). The climate is highly seasonal due to the north/south movement of the Inter-Tropical Convergence Zone. The wet season, characterized by heavy rain events, lasts for 8 months (December–July) and alternates with a 4 month dry period (August–November) during which precipitation is typically lower than 100 mm per month. For the period 2004–2015, annual rainfall quantities were on average 3103 mm year⁻¹, relative extractable water (an index of soil water availability (Wagner et al., 2011)) varied from 0.93 in the wet season to 0.46 in the dry season and soil temperature was on average 25.1 with little seasonal nor diurnal variation (Aguilos et al., 2018).

2.2 Automated sampling system

A schematic view of the automatic sampling system is shown in Figure 1(A). The system consisted of four main components: sixteen automated long-term chambers (8100-104, LI-COR Biosciences), a multiplexer to link one chamber at a time to the gas analyzers (LI-8150, LI-COR Biosciences), an infrared gas analyzer (IRGA) to measure CO₂ concentrations (LI-8100A, LI-COR Biosciences), and a cavity ring down spectroscopy (CRDS) instrument to measure CH₄ and N₂O concentrations (G2308, Picarro) that was fitted with an external pump. Both the IRGA and CRDS systems were necessary to measure all three GHG concentrations due to the different abundances and flux rates of CO₂, CH₄ and N₂O. The IRGA methodology is accurate and precise enough to detect small CO₂ concentration changes at high background concentrations (approximately 400 ppmv; parts per million in volume units). However, the detection of small changes in CH₄ and N₂O concentrations, even at their low background atmospheric concentrations in the order of 2000 ppbv (ppbv=parts per billion in volume units) and 300 ppbv, respectively, requires higher accuracy and precision levels that can only be detected with the CRDS.

Power supply was delivered through a 12 kVa generator (Perkins STORM15) fitted with batteries located 400 m away from the instruments. Both the CO₂ analyzer control unit and the multiplexer (LI-COR) had their own weather-proof casing, requiring no additional protection in the field. Nonetheless, in consideration of the high precipitation at the site, these devices were placed under a wooden shelter for added protection. The CH₄ and N₂O analyzer (Picarro), its external pump and a computer monitor were housed in a waterproof shelter that was specifically designed to host them (Figure 1(C)). The Li-8100 and the G2308 computers were connected through ethernet connection to ensure time synchronization. The sixteen automated



soil chambers (8100-104, LI-COR Biosciences) were installed in a grid in the forest (Figure 1(B)) covering in total an area of approximately 300 m² (15 m x 20 m). Each chamber was only closed during individual chamber measurement periods, and was fully open when not sampling. The PVC collars that were provided with the 8100-104 automatic chambers were inserted in the soil one month prior to the first measurement (20.3 cm inner diameter/21.3 cm outer diameter; enclosed soil area ~ 318
5 cm²; insertion depth ~ 7cm; offset ~ 4cm; green PVC). When the chambers close, they are automatically lowered so that they cover each soil collar and ensure a fully sealed chamber. The chamber lid does not directly rest on the collar rim, but on a metal plate surrounding the collar, leaving the collar undisturbed and minimizing lateral leaks (Hupp et al., 2009).

The 16 chambers were connected via 15 m Bev-a-line tubing (8 mm inner diameter) with the multiplexer (LI-8150), which allows for switching between each of the 16 chambers in any given sequence. Soil temperature (at a depth of 10 cm)
10 was monitored with 8100-201 Ω thermistor probes (Omega Engineering Inc., Stamford, CT, USA), and soil volumetric water content (0-10 cm) was monitored with 8100-202 ECH₂O Model EC-5 soil moisture sensors (Decagon Devices Inc., Pullman, WA, USA). Soil temperature and soil volumetric water content were recorded by the Licor system using the same time step.

Each chamber was purged for 15 sec prior to each measurement and 45 sec after each measurement in order to flush the lines and restore background gas levels in the system. The flow rate during the purging and the measurements was ~2.8 L
15 min⁻¹ between the Li-8150 and the chambers, which ensures sufficient air mixing in the chamber headspace during the measurements (Görres et al., 2016). The LI-8100 software provided the rate of CO₂ concentration increase in the chamber which was used to quantify the flux of CO₂ from the soil surface into the atmosphere (taking into account the enclosed soil surface area and the total system volume). A subsampling loop was inserted after the analyzer (LI-8100A) and before the multiplexer (LI-8150), to pull the air sample through the Picarro G2308 CRDS analyzer for the determination of CH₄ and N₂O
20 concentrations and flux estimations, before going back to the chamber (Figure 1(A)). All three gas concentrations were recorded every second over the sampling periods.

2.3 Flux calculations

All fluxes estimation were done by using commercially available Soil Flux pro software (LI-COR Biosciences). An R script
25 (Appendix File 1) was created to merge all the Picarro files from a given week in order to import them into the Soil flux Pro software. The Picarro creates one file per hour and when Picarro files are not merged, Soil flux Pro software is not able to deal with measurements overlapping between two distinct Picarro files (e.g. when a single measurement is done from 9:50 am to 10:15 am) leading to incorrect estimation of CH₄ and N₂O fluxes. CO₂, CH₄ and N₂O fluxes were measured as linear changes in gas concentration with time using Soil flux Pro software and include a 60 sec dead band to account for soil surface pressure
30 disturbances due to the closing of the chamber.

2.4 Minimum Detectable Fluxes



The minimum detectable flux (MDF) for each gas was estimated by using a metric originally developed by Christiansen et al. (2015), which was modified by Nickerson (2016) to make it more suitable for high-frequency measurements (Christiansen et al., 2015; Nickerson, 2016):

$$MDF = \left(\frac{A_a}{t_c \sqrt{n}} \right) \left(\frac{VP}{SRT} \right)$$

5 Where A_a is the analytical accuracy of the analyzer (25 ppb for N_2O and 10 ppb for CH_4 with the Picarro G2308 and 600 ppb for CO_2 with the Li8100), t_c is the closure time of the chamber in seconds, n is the number of points that are available to compute the flux (i.e. t_c divided by the sampling periodicity, every 1 second in this study), V is the chamber volume (0.0040761 m^3), P is the atmospheric pressure (101325 Pa), S is the chamber surface area (0.03178 m^2), R is the ideal gas constant (8.314 $m^3 Pa K^{-1} mol^{-1}$) and T is the ambient temperature (298.15 K). We computed the MDF of each gas for closure
10 times from 2 minutes to 30 minutes in order to select the optimal chamber closure time for each gas in our integrated system (Table 1).

2.5 Closure time

Selecting the best length of time for soil GHG measurements and accurate flux calculation in an integrated CO_2 , CH_4 and N_2O
15 automated measurement system requires careful consideration. At low fluxes, longer measurement periods are needed to reach reliable measurements of real concentration changes, while at high fluxes possible storage and saturation effects in the chamber headspace might result in non-linear concentration increases and thereby underestimated fluxes. In order to maximize the detectable percentage of fluxes for N_2O and CH_4 without impeding spatial coverage and temporal resolution, we built a combined program with two different closure times. Each week, four out of sixteen chambers were programmed to stay closed
20 for a longer measurement period to ensure a reliable estimation of low fluxes while the other twelve chambers were programmed to stay closed for a shorter period to capture diel variation and detect high fluxes. For the short closure time (SHORT hereafter), we used a 2 minute measurement period because (1) this is a standard closure time for soil CO_2 flux calculations (Janssens et al., 2000), even in tropical forests (Epron et al., 2006; Sayer et al., 2007) of this region where MDF for CO_2 flux is typically low (Bonal et al., 2008; Bréchet et al., 2009; Courtois et al., 2018), (2) corresponding MDFs of CH_4
25 (0.04 $nmol m^{-2} s^{-1}$ or) and N_2O (0.1 $nmol m^{-2} s^{-1}$) are compatible with the detection of emission or consumption peaks of these two gases in this region (Courtois et al., 2018; Petitjean et al., 2015). For the long closure time (LONG hereafter), we decided to use a 25 minute measurement period in order to optimize the trade-off between a reliable estimation of low N_2O fluxes (Table 1) and a program length that allows for a sufficient number of flux measurements per chamber and per day.

We therefore programmed the multiplexer for 2.5-h cycles (9-10 measurements per chamber per day), which included
30 four chambers with LONG measurements and twelve chambers with SHORT measurements. Each week, the program was modified manually so that the four LONG measurements were rotated across the chambers. Each chamber was therefore measured with the LONG closure time for one 7 consecutive day period per month (4 weeks).



2.6 System maintenance and data processing

The automated sampling system was installed on June 1st 2016 and operated until September 29th 2016 (4 months), totaling 17652 individual measurements for each gas (4326 with LONG closure time and 13326 with SHORT closure time). Coarse wood debris was removed weekly but small litter, such as leaves, fruits, and twigs, was left in the collar area. Every week, 5 living plants growing inside the collars, and the dead leaves on the chambers, were carefully removed by hand.

For CO₂, we observed a strong concentration saturation effect when using the LONG closure time (25 minutes), leading to an underestimation of fluxes (Figure A1). All CO₂ flux estimates were therefore based on 2 minute regressions only, using either full concentration measurements of the SHORT closure time or the 2 first minutes of the LONG closure time. The R² value of the linearly increasing CO₂ over 2 minutes was used as an indicator that the system was functioning correctly and 10 not impeded by debris (Görres et al., 2016; Savage et al., 2014). When the R² of the regression between time and CO₂ concentration was lower than 0.9, we considered this as an indication that there may have been an issue with the chamber closing and sealing correctly and removed the flux measurement for all three gases from our analysis. For CH₄, we observed only a slight saturation effect when using the LONG closure time (Figure A1). Variation in the flux calculations did not differ 15 between the SHORT and LONG chamber closure measurements. All fluxes above or below (for negative fluxes) MDF were considered as reliable and were calculated using the full data available (2 minutes measurement for the SHORT closure time and 25 minutes for the LONG closure time). N₂O flux calculations were much more variable when measuring with the SHORT closure time compared to the LONG closure time (Figure A2). Even if fluxes were above the detection limit, the low fluxes estimated with the SHORT closure time were not reliable. We therefore decided (1) to keep all the fluxes estimated with the 20 LONG closure time even with low R² for the regression between time and N₂O concentrations and (2) to consider all fluxes estimated with the SHORT closure time with a R² lower than 0.8 as unreliable (Savage et al., 2014).

3 Results and discussions

A cleaning frequency of once a week was necessary and sufficient to remove falling leaves and branches from the automatic chamber system, prevent leaks and generate a continuous dataset of soil GHG fluxes from this tropical forest. The automatic 25 chamber system worked well most of the time, but some data gaps did exist. Over the 17724 individual flux estimations, 276 (1.5 %) had to be discarded because of (1) problems in the connection between the chamber and the multiplexer (196 measurements, 1% of data points); (2) imperfect chamber closing, which was detected by an insufficient increase of CO₂ (103 measurements, 0.6% of data points).

3.1 CO₂ fluxes

CO₂ fluxes were on average $7.34 \pm 3.21 \mu\text{mol m}^{-2} \text{s}^{-1}$ with a high variability among chambers (Table 2). The minimum flux measured during the study period was $0.19 \mu\text{mol m}^{-2} \text{s}^{-1}$ (Table 2) and the majority of fluxes were between 3 to $10 \mu\text{mol m}^{-2} \text{s}^{-1}$ (Figure 2). All two-minute measurements of CO₂ fluxes from the four-month study period were therefore above the MDF of $2.39 \text{ nmol m}^{-2} \text{ s}^{-1}$ for the LI8100 analyzer (Table 1). No saturation effect was detected using the SHORT closure time and



estimation of CO₂ over a shorter time period is not recommended (Davidson et al., 2002). CO₂ fluxes using the LONG closure time would be underestimated due to the buildup of high CO₂ concentrations due to large fluxes over this long time period (Figure A1), and are not recommended. We therefore conclude that a 2 minute sampling time should be used for CO₂ flux calculations since the MDF of this short measurement period allowed for the retention of 100% of the data. When the chambers stay closed longer for accurate detection of N₂O fluxes, only the first two minutes of data should be used for CO₂ flux calculations. The use of 16 automated flux chambers allowed for the capture of spatial and temporal variability of soil respiration over this four month period, which is needed to constrain ecosystem carbon budgets (Figure 3).

3.2 CH₄ fluxes

CH₄ fluxes were on average 1.06 ± 4.52 nmol m⁻² s⁻¹ with a high variability among chambers (Table 2). Minimum detectable fluxes for CH₄ were ± 0.04 nmol m⁻² s⁻¹ using the SHORT closure time and ± 0.001 nmol m⁻² s⁻¹ using the LONG closure time (Table 1). 98.5 % and 99.9% of fluxes measured with the SHORT and LONG closure times, respectively, were retained in our quality control data processing over the four-month study period. These measurement periods, therefore, allowed for the retention of a large majority of CH₄ emission or consumption fluxes in our data analysis. The frequency of negative CH₄ fluxes (consumption) was greater than positive fluxes (emission) during this period (Figure 2). Most of the time, soils were either consuming or emitting small amounts of CH₄, but transient, large emission peaks were periodically detected at individual chamber locations during the study period (Figure 4).

3.3 N₂O fluxes

N₂O fluxes were on average 0.038 ± 0.537 nmol m⁻² s⁻¹ with a high variability among chambers (Table 2). Most N₂O fluxes (85.6%) with the LONG closure time were above or below MDF (± 0.002 nmol m⁻² s⁻¹, Table 1) and varied between -2 to 2 nmol m⁻² s⁻¹ (Figure 2). When measured over 25 minutes, N₂O fluxes in our site could therefore be considered as reliable. Using the SHORT closure time, most flux estimations had to be discarded because they led to unreliable flux estimations (Figure A2). Only 7.9% of the measurements using the SHORT closure time were retained after our quality control checks for the N₂O flux data. Nonetheless, the high frequency of the SHORT closures still allowed the detection of one high N₂O emission event (up to 15 nmol m⁻² s⁻¹) that was detected during the study period (Figure 5, chamber 7). The high variability in N₂O fluxes that we detected over four months with our automated system are in agreement with the typical high variability in N₂O fluxes measured from tropical soils over space and time using static chambers (Arias-Navarro et al., 2017; Courtois et al., 2018).

4 Conclusions

Our unique system coupled a Li8100 CO₂ analyzer and multiplexor with a Picarro G2308 CH₄ and N₂O analyzer to sample 16 automated soil flux chambers with a rotation of SHORT and LONG closure times for the accurate monitoring of three GHG fluxes over four months with high spatial and temporal resolution. The sampling system of SHORT and LONG closure times



with a weekly rotation presented here has three major advantages, which ultimately can provide high confidence in the estimation of annual the full GHG budgets of tropical soils: (1) the LONG closure time allows a reliable estimation of the low N₂O fluxes in this ecosystem, which was clearly not achieved using a shorter closure time, (2) the number of data points per day are sufficiently high (9 to 10 measurements per day) to capture potential diurnal variation (Nicolini et al., 2013; Rubio and Detto, 2017) of the three gases with good spatial replication (16 chambers), (3) periodic extreme events of high N₂O fluxes can still be detected with the SHORT closure time period, which occurs at higher frequency than the LONG closure measurements. Our study underlines the importance of appropriate closure time for each GHG gas for accurate estimation of GHG budgets.

We demonstrate here that the combination of a commercial soil GHG chamber system – the LI-8100A Automated Soil CO₂ Flux System – running in line with a Picarro G2308, enables the continuous, long-term measurement of CO₂, CH₄, and N₂O simultaneously. Similar configurations have been recently implemented in temperate climates (Petrakis et al., 2017b, 2017a), but to our knowledge, this is the first time that this experimental set up is described and tested under tropical field conditions. Additionally, our study determined the optimal chamber closure time for each GHG. This information is crucial for the calculation of accurate soil fluxes at diurnal timesteps and for the estimation of annual GHG budgets. This combination of automated closed dynamic chambers and advanced GHG analyzers allows for, (1) accounting of short-term variability in GHG fluxes while taking into account spatial variability, (2) estimating annual GHG budgets at these locations, (3) tracking the variability in GHG fluxes along hours, days, seasons and years, and (4) studying the impact of climatic change on soil GHG budgets.

Author contribution. JVB and NA designed the experiments and EAC, CS, BB and DB carried them out. EAC and CS prepared the manuscript with contributions from all co-authors.

Competing interests. The authors declare that they have no conflict of interest.

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References



- Aguilos, M., Hérault, B., Burban, B., Wagner, F. and Bonal, D.: What drives long-term variations in carbon flux and balance in a tropical rainforest in French Guiana?, *Agric. For. Meteorol.*, 253, 114–123, 2018.
- Ambus, P., Skiba, U., Drewer, J., Jones, S., Carter, M. S., Albert, K. R. and Sutton, M.: Development of an accumulation-based system for cost-effective chamber measurements of inert trace gas fluxes, *Eur. J. Soil Sci.*, 61(5), 785–792, 2010.
- 5 Arias-Navarro, C., Díaz-Pinés, E., Klatt, S., Brandt, P., Rufino, M. C., Butterbach-Bahl, K. and Verchot, L.: Spatial variability of soil N₂O and CO₂ fluxes in different topographic positions in a tropical montane forest in Kenya, *J. Geophys. Res. Biogeosciences*, 122(3), 514–527, 2017.
- Bonal, D., Bosc, A., Ponton, S., GORET, J., Burban, B., Gross, P., BONNEFOND, J., Elbers, J., Longdoz, B. and Epron, D.: Impact of severe dry season on net ecosystem exchange in the Neotropical rainforest of French Guiana, *Glob. Change Biol.*, 10 14(8), 1917–1933, 2008.
- Bréchet, L., Ponton, S., Roy, J., Freycon, V., Coûteaux, M.-M., Bonal, D. and Epron, D.: Do tree species characteristics influence soil respiration in tropical forests? A test based on 16 tree species planted in monospecific plots, *Plant Soil*, 319(1–2), 235–246, 2009.
- Breuer, L., Papen, H. and Butterbach-Bahl, K.: N₂O emission from tropical forest soils of Australia, *J. Geophys. Res. Atmospheres*, 105(D21), 26353–26367, 2000.
- 15 Christiansen, J. R., Outhwaite, J. and Smukler, S. M.: Comparison of CO₂, CH₄ and N₂O soil-atmosphere exchange measured in static chambers with cavity ring-down spectroscopy and gas chromatography, *Agric. For. Meteorol.*, 211, 48–57, 2015.
- Courtois, E. A., Stahl, C., Van den Berge, J., Bréchet, L., Van Langenhove, L., Richter, A., Urbina, I., Soong, J. L., 20 Peñuelas, J. and Janssens, I. A.: Spatial Variation of Soil CO₂, CH₄ and N₂O Fluxes Across Topographical Positions in Tropical Forests of the Guiana Shield, *Ecosystems*, 1–14, 2018.
- Davidson, E., Savage, K., Verchot, L. and Navarro, R.: Minimizing artifacts and biases in chamber-based measurements of soil respiration, *Agric. For. Meteorol.*, 113(1), 21–37, 2002.
- De Klein, C. and Harvey, M.: Nitrous oxide chamber methodology guidelines, *Glob. Res. Alliance Agric. Greenh. Gases* 25 *Minist. Prim. Ind. Wellingt. N. Z.*, 2012.
- Denmead, O.: Chamber systems for measuring nitrous oxide emission from soils in the field, *Soil Sci. Soc. Am. J.*, 43(1), 89–95, 1979.
- Dutaur, L. and Verchot, L. V.: A global inventory of the soil CH₄ sink, *Glob. Biogeochem. Cycles*, 21(4), 2007.
- Epron, D., Bosc, A., Bonal, D. and Freycon, V.: Spatial variation of soil respiration across a topographic gradient in a 30 tropical rain forest in French Guiana, *J. Trop. Ecol.*, 22(05), 565–574, 2006.
- Görres, C.-M., Kammann, C. and Ceulemans, R.: Automation of soil flux chamber measurements: potentials and pitfalls, *Biogeosciences*, 13(6), 1949–1966, 2016.



- Hupp, J. R., Garcia, R. L., Madsen, R. and McDermitt, D. K.: Measurement of CO₂ Evolution in a Multiplexed Flask System, vol. 44, pp. 1143–1143, AMER SOC HORTICULTURAL SCIENCE 113 S WEST ST, STE 200, ALEXANDRIA, VA 22314-2851 USA., 2009.
- Janssens, I. A., Kowalski, A. S., Longdoz, B. and Ceulemans, R.: Assessing forest soil CO₂ efflux: an in situ comparison of
5 four techniques, *Tree Physiol.*, 20(1), 23–32, 2000.
- Koskinen, M., Minkkinen, K., Ojanen, P., Kämäräinen, M., Laurila, T. and Lohila, A.: Measurements of CO₂ exchange with an automated chamber system throughout the year: challenges in measuring night-time respiration on porous peat soil, *Biogeosciences*, 11(2), 347, 2014.
- Kostyanovsky, K., Huggins, D., Stockle, C., Waldo, S. and Lamb, B.: Developing a flow through chamber system for
10 automated measurements of soil N₂O and CO₂ emissions, *Measurement*, 113, 172–180, 2018.
- Merbold, L., Wohlfahrt, G., Butterbach-Bahl, K., Pilegaard, K., DelSontro, T., Stoy, P. and Zona, D.: Preface: Towards a full greenhouse gas balance of the biosphere, *Biogeosciences*, 12(2), 453–456, 2015.
- Nickerson, N.: Evaluating gas emission measurements using Minimum Detectable Flux (MDF), Eosense Inc Dartm. N. S. Can., 2016.
- 15 Nicolini, G., Castaldi, S., Fratini, G. and Valentini, R.: A literature overview of micrometeorological CH₄ and N₂O flux measurements in terrestrial ecosystems, *Atmos. Environ.*, 81, 311–319, 2013.
- O’Connell, C. S., Ruan, L. and Silver, W. L.: Drought drives rapid shifts in tropical rainforest soil biogeochemistry and greenhouse gas emissions, *Nat. Commun.*, 9(1), 1348, 2018.
- Oertel, C., Matschullat, J., Zurba, K., Zimmermann, F. and Erasmi, S.: Greenhouse gas emissions from soils—A review,
20 *Chem. Erde-Geochem.*, 76(3), 327–352, 2016.
- Petitjean, C., Hénault, C., Perrin, A.-S., Pontet, C., Metay, A., Bernoux, M., Jehanno, T., Viard, A. and Roggy, J.-C.: Soil N₂O emissions in French Guiana after the conversion of tropical forest to agriculture with the chop-and-mulch method, *Agric. Ecosyst. Environ.*, 208, 64–74, 2015.
- Petrakis, S., Seyfferth, A., Kan, J., Inamdar, S. and Vargas, R.: Influence of experimental extreme water pulses on
25 greenhouse gas emissions from soils, *Biogeochemistry*, 133(2), 147–164, 2017a.
- Petrakis, S., Barba, J., Bond-Lamberty, B. and Vargas, R.: Using greenhouse gas fluxes to define soil functional types, *Plant Soil*, 1–10, 2017b.
- Pumpanen, J., Kolari, P., Ilvesniemi, H., Minkkinen, K., Vesala, T., Niinistö, S., Lohila, A., Larmola, T., Morero, M. and Pihlatie, M.: Comparison of different chamber techniques for measuring soil CO₂ efflux, *Agric. For. Meteorol.*, 123(3),
30 159–176, 2004.
- Rubio, V. E. and Detto, M.: Spatiotemporal variability of soil respiration in a seasonal tropical forest, *Ecol. Evol.*, 7(17), 7104–7116, 2017.
- Savage, K., Phillips, R. and Davidson, E.: High temporal frequency measurements of greenhouse gas emissions from soils, *Biogeosciences*, 11(10), 2709, 2014.



- Sayer, E. J., Powers, J. S. and Tanner, E. V.: Increased litterfall in tropical forests boosts the transfer of soil CO₂ to the atmosphere, *PLoS One*, 2(12), e1299, 2007.
- Silver, W. L., Lugo, A. and Keller, M.: Soil oxygen availability and biogeochemistry along rainfall and topographic gradients in upland wet tropical forest soils, *Biogeochemistry*, 44(3), 301–328, 1999.
- 5 Verchot, L. V., Davidson, E. A., Cattânio, H., Ackerman, I. L., Erickson, H. E. and Keller, M.: Land use change and biogeochemical controls of nitrogen oxide emissions from soils in eastern Amazonia, *Glob. Biogeochem. Cycles*, 13(1), 31–46, 1999.
- Verchot, L. V., Davidson, E. A., Cattânio, J. H. and Ackerman, I. L.: Land-use change and biogeochemical controls of methane fluxes in soils of eastern Amazonia, *Ecosystems*, 3(1), 41–56, 2000.
- 10 Wagner, F., Hérault, B., Stahl, C., Bonal, D. and Rossi, V.: Modeling water availability for trees in tropical forests, *Agric. For. Meteorol.*, 151(9), 1202–1213, 2011.



Table 1: Minimum Detectable Fluxes (MDF) for each gas and for closure times from 2 to 30 minutes. The two closure times that were used in this study (2 minutes and 25 minutes) are highlighted in bold.

Closure time (minutes)	N ₂ O (nmol m ⁻² s ⁻¹)	CH ₄ (nmol m ⁻² s ⁻¹)	CO ₂ (nmol m ⁻² s ⁻¹)
2	0.100	0.040	2.393
5	0.025	0.010	0.605
10	0.009	0.004	0.214
15	0.005	0.002	0.117
20	0.003	0.001	0.076
25	0.002	0.001	0.054
30	0.002	0.001	0.041



Table 2: Mean, standard deviation (SD), minimum (Min) and maximum (Max) values of each gas and each chamber over the study period.

	CO₂ ($\mu\text{mol m}^{-2} \text{s}^{-1}$)				CH₄ ($\text{nmol m}^{-2} \text{s}^{-1}$)				N₂O ($\text{nmol m}^{-2} \text{s}^{-1}$)			
	Mean	SD	Min	Max	Mean	SD	Min	Max	Mean	SD	Min	Max
Chamber 1	6.36	0.68	1.37	8.87	8.78	7.02	-2.08	23.35	0.081	0.069	-0.021	0.534
Chamber 2	7.10	1.19	4.00	24.47	-1.38	1.59	-4.09	11.18	0.007	0.048	-0.066	0.247
Chamber 3	5.44	0.96	1.51	11.24	0.24	2.74	-2.64	28.17	0.034	0.160	-0.032	2.538
Chamber 4	7.11	1.22	4.36	10.91	-1.80	1.19	-3.63	4.79	0.039	0.048	-0.035	0.205
Chamber 5	3.87	0.87	0.53	8.93	0.62	2.38	-2.20	10.08	0.131	0.257	-0.019	2.596
Chamber 6	8.24	1.79	2.79	18.47	-1.10	1.46	-3.20	5.84	-0.009	0.021	-0.055	0.085
Chamber 7	13.13	3.15	0.89	23.83	0.69	2.56	-2.63	13.98	0.640	1.881	0.041	17.605
Chamber 8	6.92	1.15	2.03	19.14	-0.15	2.09	-3.37	13.95	0.025	0.047	-0.046	0.226
Chamber 9	4.08	2.27	0.35	24.81	1.88	3.40	-2.14	23.96	0.023	0.058	-0.039	0.484
Chamber 10	5.23	1.55	0.23	18.78	0.46	1.70	-1.91	7.94	0.037	0.098	-0.049	0.686
Chamber 11	10.54	1.55	6.13	17.18	5.67	6.96	-1.06	29.56	0.038	0.045	-0.029	0.227
Chamber 12	7.67	2.29	3.15	24.23	1.08	5.37	-3.29	28.36	0.025	0.033	-0.056	0.171
Chamber 13	5.45	1.73	0.19	23.06	3.18	4.76	-1.45	24.15	0.083	0.135	-0.033	1.408
Chamber 14	5.25	1.12	0.63	18.08	1.79	4.97	-2.48	30.66	0.092	0.104	-0.017	0.444
Chamber 15	13.57	2.13	8.35	24.50	-0.48	1.68	-3.25	6.22	0.010	0.043	-0.052	0.160
Chamber 16	6.47	0.65	3.92	8.54	-1.24	1.28	-3.60	2.94	-0.004	0.024	-0.052	0.065
All Chambers	7.24	3.21	0.19	24.81	1.06	4.52	-4.10	30.66	0.038	0.537	-2.345	17.605

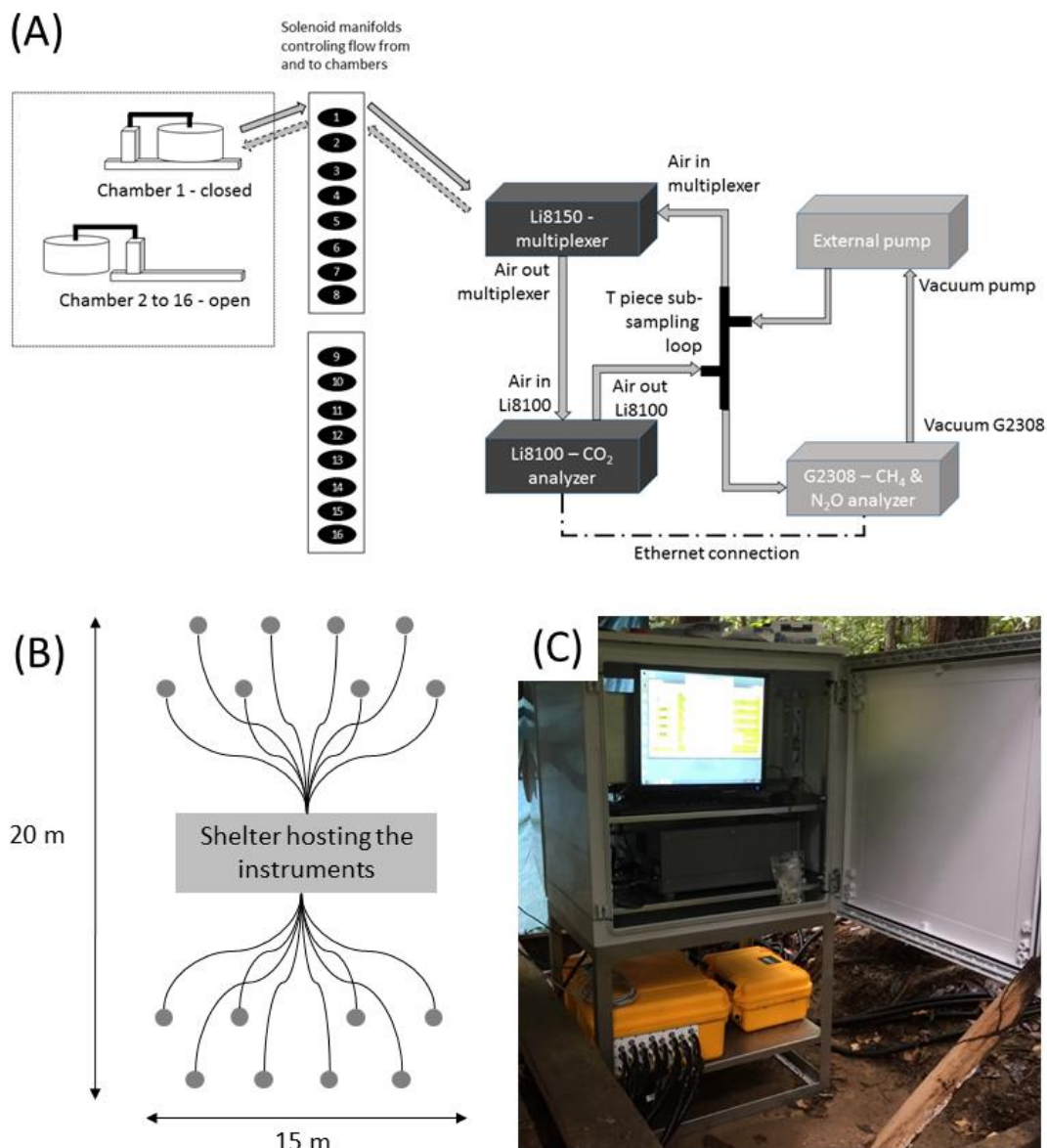
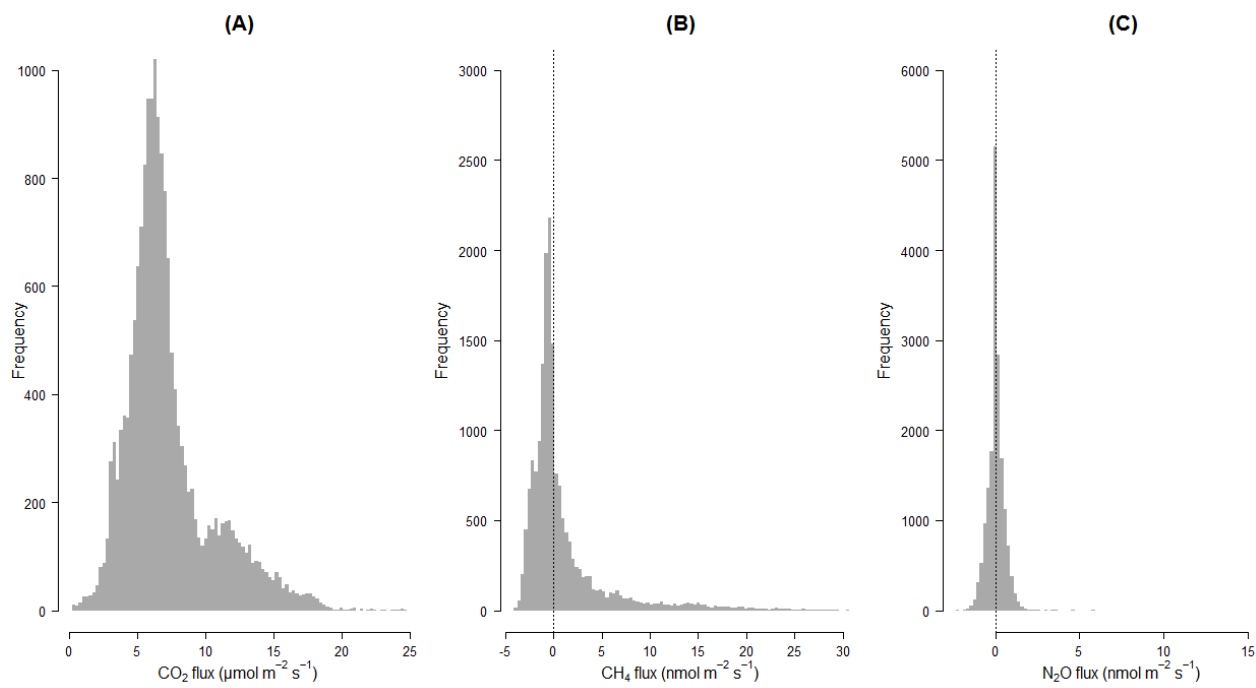


Figure 1: Experimental Design: (A) Schematic view of the installation composed of four main components: sixteen automated long-term chambers (8100-104, LI-COR Biosciences), a multiplexer to link one of these chambers to the gas analyzers (LI-8150, LI-COR Biosciences), an infrared gas analyzer (IRGA) to measure CO₂ concentrations (LI-8100A, LI-COR Biosciences), and a cavity ring down spectroscopy (CRDS) instrument to measure CH₄ and N₂O concentrations (G2308, Picarro) that was fitted with an external pump. (B) Schematic representation of the grid with the shelter housing the equipment in the middle and the 16 chambers (grey dots) linked to the Li-8150 multiplexer with 15 meters cables (black lines). (C) Picture of the instruments in the field.



Figure

2: Distribution of fluxes: Histogram of (A) CO₂, (B) CH₄ and (C) N₂O fluxes over the study period. For (B) and (C), the dotted line represents null fluxes.

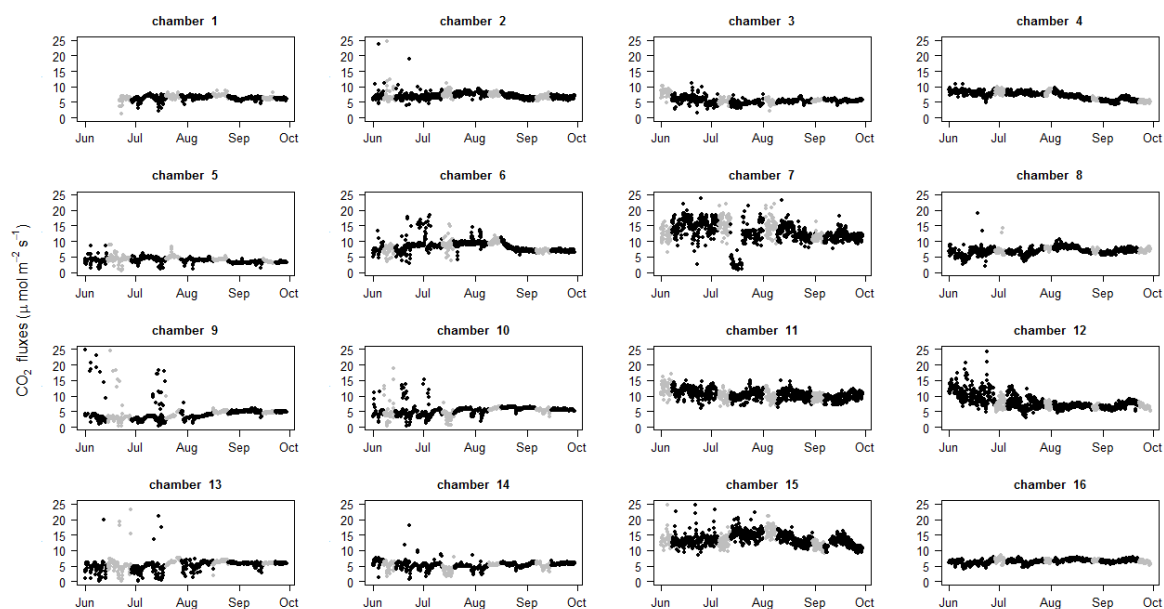


Figure 3: CO₂ fluxes through time: CO₂ fluxes for each chamber (1 to 16) over the study period with fluxes estimated with SHORT (2 minutes) closure time in black and fluxes estimated with the 2 first minutes of the LONG (25 minutes) closure time in grey. All panels have the same limits on the y axis (from 0 to 25 $\mu\text{mol m}^{-2} \text{s}^{-1}$)

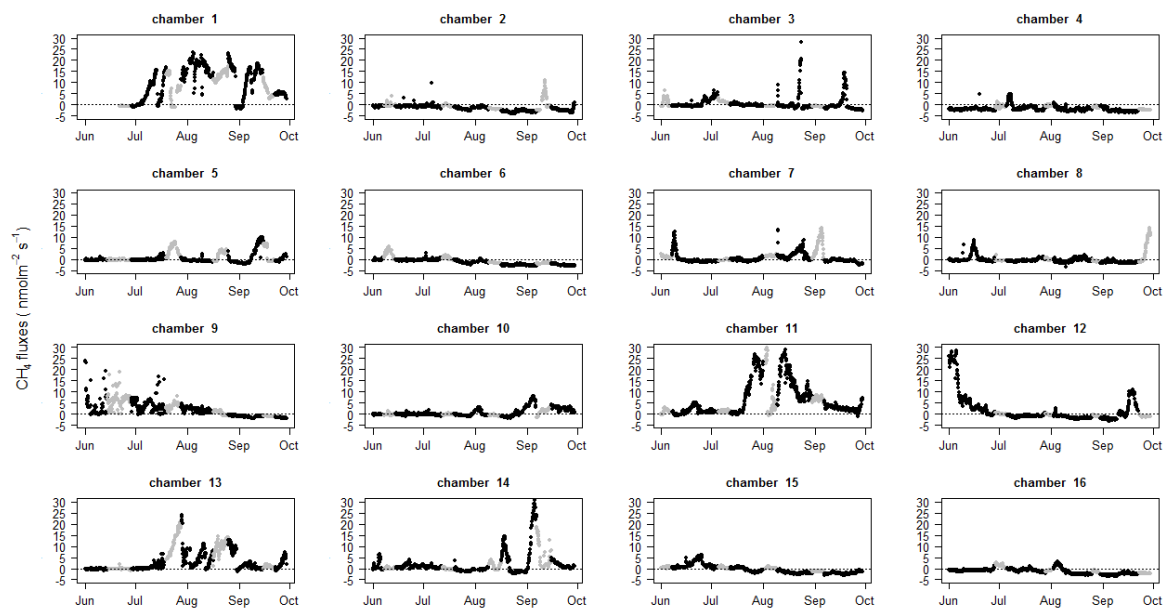


Figure 4: CH₄ fluxes through time: CH₄ fluxes for each chamber (1 to 16) over the study period with fluxes estimated with SHORT (2 minutes) closure time in black and fluxes estimated with LONG (25 minutes) closure time in grey. The dotted line displays the zero flux line. All panels have the same limits on the y axis (from -5 to 30 nmol m⁻² s⁻¹)

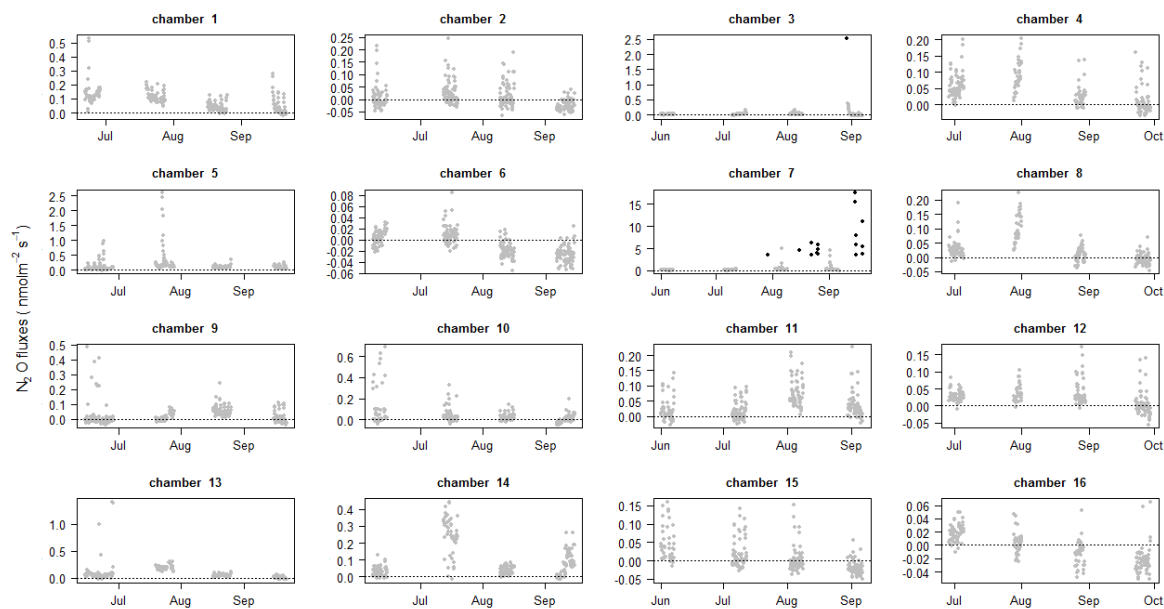


Figure 5: N₂O fluxes through time: N₂O fluxes for each chamber (1 to 16) over the study period with fluxes estimated with the SHORT (2 minutes) closure time in black and fluxes estimated with the LONG (25 minutes) closure time in grey. The dotted line displays the zero flux line. Due to the high differences among chambers, each panel has specific limit on the y axis.

5



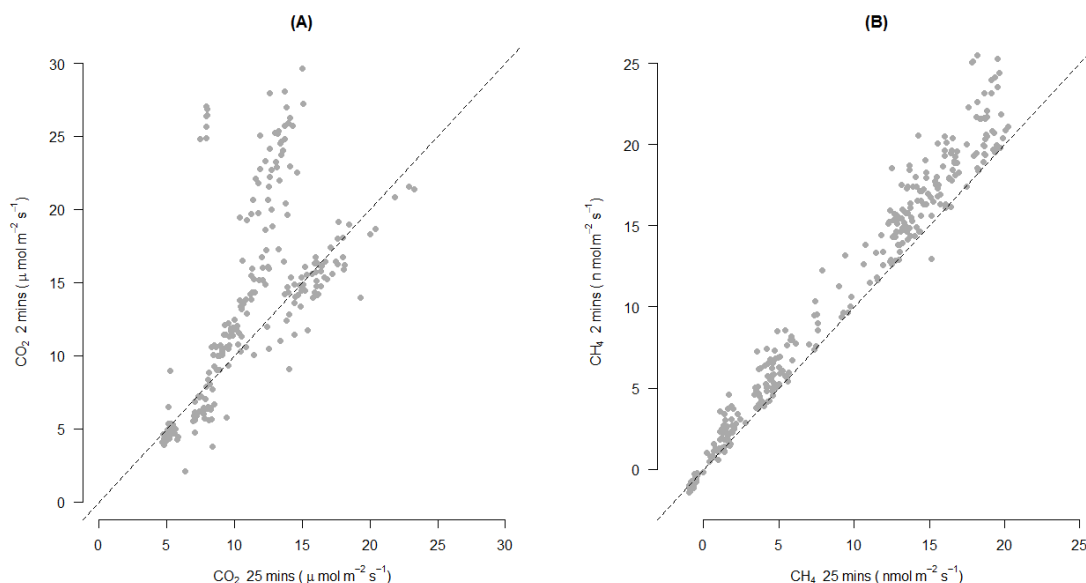
Appendix File 1: R code for merging Picarro files to include them in Soil Flux pro

```
## to list all the days in a given directory (Picarro makes one directory per day)
ListDay<-list.files()
5 Pfile<-list()
## to concatenate all the hourly file in one file per day
for (j in 1:length(ListDay))
{
print(j)
10 ListFilesPicarro<-list.files(ListDay[j])
Data<-read.table(paste(ListDay[j],"/",ListFilesPicarro[1],sep=""))
for (i in 2:length(ListFilesPicarro))
{
temp<-read.table(paste(ListDay[j],"/",ListFilesPicarro[i],sep=""))
15 Data<-rbind(Data, temp)
print(i)
}
Pfile[[j]]<-Data
}
20 ## to concatenante all days and make just one file will all data
MasterData<-Pfile[[1]]
for (k in 2:length(Pfile))
{
MasterData<-rbind(MasterData,Pfile[[k]])
25 print(k)
}
## to write the table in a way that SFP can read it
write.table(MasterData, "MasterData.dat", quote=F)
```

30



Appendix Figure A1: Comparison of (A) CO₂ and (B) CH₄ fluxes when estimated measured over 25 minutes (LONG closure time) or 2 minutes (SHORT closure time). For this, we used measurements made over 25 minutes and recomputed the flux with the two firsts minutes. (A) For CO₂, this effect was tested for the week from August 16th for August 25th where CO₂ fluxes measured on chambers 3, 7, 11 and 15 with LONG closure time are covering almost the whole range of fluxes during the study period. For fluxes higher than 10 $\mu\text{mol m}^{-2} \text{s}^{-1}$, 65 % of individual measurements (131 over 251) were strongly underestimated with the LONG closure time as compare to the SHORT closure time. (B) For CH₄, this effect was tested for the week from August 16th for August 25th where CH₄ fluxes measured on chambers 1, 5, 9 and 13 with LONG closure time are covering almost the whole range of fluxes during the study period. For fluxes higher than 5 $\text{nmol m}^{-2} \text{s}^{-1}$, 89 % of individual measurements (184 over 206) were slightly underestimated with the LONG closure time as compare to the SHORT closure time.





Appendix Figure A2: N₂O fluxes for each chamber (1 to 16) over the study period without R² filtering fluxes estimated over the SHORT closure time. with fluxes estimated with SHORT (2 minutes) closure time in black and fluxes estimated with LONG (25 minutes) closure time in grey.

