



# Automation of soil flux chamber measurements: potentials and pitfalls

Carolyn-Monika Görres<sup>1,2</sup>, Claudia Kammann<sup>2</sup>, and Reinhart Ceulemans<sup>1</sup>

<sup>1</sup>Center of Excellence PLECO (Plant and Vegetation Ecology), University of Antwerp, Universiteitsplein 1, 2610 Wilrijk, Belgium

<sup>2</sup>WG Global Change Research in Special Crops, Department of Soil Science and Plant Nutrition, Hochschule Geisenheim University, Von-Lade-Str. 1, 65366 Geisenheim, Germany

Correspondence to: Carolyn-Monika Görres (carolyn.goerres@hs-gm.de)

Received: 15 July 2015 – Published in Biogeosciences Discuss.: 7 September 2015

Revised: 2 February 2016 – Accepted: 25 February 2016 – Published: 30 March 2016

**Abstract.** Recent technological advances have enabled the wider application of automated chambers for soil greenhouse gas (GHG) flux measurements, several of them commercially available. However, few studies addressed the challenges associated with operating these systems. In this contribution we compared two commercial soil GHG chamber systems – the LI-8100A Automated Soil CO<sub>2</sub> Flux System and the greenhouse gas monitoring system AGPS. From April until August 2014, the two systems monitored in parallel soil respiration (SR) fluxes at a recently harvested poplar (*Populus*) plantation, which provided a bare field situation directly after the harvest as well as a closed canopy later on. For the bare field situation (15 April–30 June 2014), the cumulated average SR obtained from the unfiltered data sets of the LI-8100A and the AGPS were 520 and 433 g CO<sub>2</sub> m<sup>-2</sup> respectively. For the closed canopy phase (1 July–31 August 2014), which was characterized by a higher soil moisture content, the cumulated average SR estimates were not significantly different with 507 and 501 g CO<sub>2</sub> m<sup>-2</sup> for the AGPS and the LI-8100A respectively. Flux quality control and filtering did not significantly alter the results obtained by the LI-8100A, whereas the AGPS SR estimates were reduced by at least 20%. The main reasons for the observed differences in the performance of the two systems were (i) a lower data coverage provided by the AGPS due to technical problems; (ii) incomplete headspace mixing in the AGPS chambers; (iii) lateral soil CO<sub>2</sub> diffusion below the collars during AGPS chamber measurements; and (iv) a possible overestimation of nighttime SR fluxes by the LI-8100A. Additionally, increased root growth was observed within the LI-8100A col-

lars but not within the AGPS collars, which might have also contributed to the observed differences. In contrast to the LI-8100A, the AGPS had the gas sample inlets installed inside the collars and not the chambers. This unique design feature enabled for the first time the detection of disturbed chamber measurements during nights with a stratified atmosphere, resulting in unbiased nighttime SR estimates. Thus besides providing high temporal frequency flux data, automated chamber systems offer another possibility to greatly improve our understanding of SR fluxes.

## 1 Introduction

The majority of soil greenhouse gas (GHG) flux data have been obtained using manually operated closed static chambers (Pumpanen et al., 2004; Levy et al., 2011). These chambers are placed airtight on a small soil area (typically < 1 m<sup>2</sup> and < 1 h) and gas samples are collected from the chambers during the closure time. The gas samples are subsequently analysed by gas chromatography or other analytical techniques and the flux is calculated from the rate of gas concentration change over time (Levy et al., 2011; Collier et al., 2014). The chamber design and measurement protocol are highly flexible and can be adjusted for different ecosystems or land use types, and this at relatively low costs (Pumpanen et al., 2004; de Klein and Harvey, 2012). A major drawback, however, is the low temporal resolution since working with manual closed chambers is very laborious, and measurements are thus only performed at low or irregular frequency

(every few days or weeks) (Savage et al., 2014; Koskinen et al., 2014). As a result, our knowledge not only of short-term responses of soil GHG flux dynamics to perturbations such as rain events, irrigation and fertilisation but also of the diurnal cycles of soil GHG fluxes and associated time lags is still very limited (Carbone and Vargas, 2008; Vargas et al., 2011; Hopkins et al., 2013; Phillips et al., 2013). 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 (Giltrap et al., 2010; FAO, 2014; Olander et al., 2014; Savage et al., 2014).

One approach to obtain high temporal frequency soil GHG flux data is the automation of chamber measurements. Automated chambers have been in use since the 1970s (Denmead, 1979) and different systems have been developed over the years (e.g. Breuer et al., 2000; Ambus et al., 2010; Koskinen et al., 2014; Savage et al., 2014). The total number of studies with automated chambers remains, however, quite low and the majority of them only deal with soil CO<sub>2</sub> fluxes. The latter is mainly due to a lack of available field gas analysers for CH<sub>4</sub> and N<sub>2</sub>O in the past (Venterea et al., 2008; Savage et al., 2014). The requirements for a larger infrastructure and for intensive maintenance as compared to manual chamber measurements have prevented the widespread application of automated systems. Therefore, only a few studies actually address the difficulties and challenges associated with running these systems (Koskinen et al., 2014).

In general chambers provide an invasive method and, depending on the design, they alter soil and microclimatic conditions to a degree that can potentially bias the measured fluxes. Potential biases introduced by different chamber designs and sampling procedures have been quantified in numerous studies (Pumpanen et al., 2004; Christiansen et al., 2011; Pihlatie et al., 2013; Görres et al., 2014), and the elimination of these biases is an ongoing debate (de Klein and Harvey, 2012). Several studies have compared the data quality of automated chamber systems with manually operated chambers (Savage et al., 2014), with soil gas concentration profile measurements (Jassal et al., 2005; Roland et al., 2015) and with the eddy covariance method (Wang et al., 2013). However, different automated chamber systems have never been compared in the field as has been done for eddy covariance flux systems (Janssens et al., 2000; Peltola et al., 2013).

Due to technological advances, more automated chamber systems are commercially available, and an increasing number of custom-made systems are being designed and deployed for soil GHG flux measurements (de Klein and Harvey, 2012). Comparative analyses are important to guarantee high-quality data collection with these systems and a high comparability among studies using different systems (Janssens et al., 2000; Creelman et al., 2013). Here we present a detailed field comparison of two automated soil GHG flux systems – the LI-8100A Soil CO<sub>2</sub> Flux System (LI-COR Biosciences, Lincoln, NE, USA) and the automated gas sampling system AGPS (UIT, Dresden, Germany). The

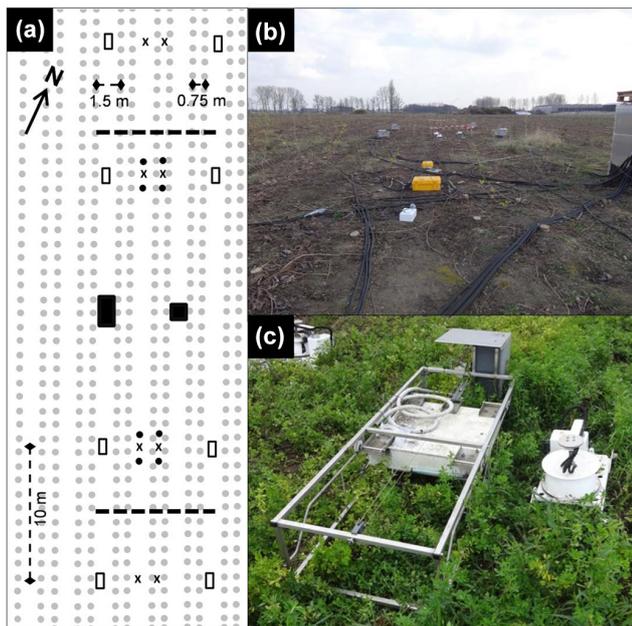
LI-8100A is a fully automated chamber system including multiplexer, gas analyser and flux calculation software. The AGPS is a commercially available automated vial collector system in which each automated chamber operates as an autonomous unit and the collected gas samples have to be subsequently analysed by gas chromatography (Kitzler et al., 2006). For this study, the AGPS has been equipped with a multiplexer and gas analysers for the first time, resembling a fully automated custom-made chamber system with continuous gas analysis in the field. In parallel, both systems were monitoring soil respiration (SR) in a coppiced poplar plantation. The poplar plantation had the advantage that it provided open field conditions as well as closed canopy conditions within one vegetation period. In addition to the chamber measurements, CO<sub>2</sub> concentrations were monitored in the topsoil to give insights into the potential range of soil CO<sub>2</sub> fluxes at the site and to better understand the performance of the chambers under different soil moisture conditions. The aim of this study was not to understand the processes driving SR or soil CO<sub>2</sub> efflux at the poplar plantation per se since these have already been discussed amongst others by Verlinde et al. (2013) and Zenone et al. (2015). The results presented here serve as the comparison of the performance of the two chamber systems in quantifying SR fluxes under a wide range of different environmental conditions.

## 2 Materials and Methods

### 2.1 Field site and experimental design

This study was conducted in a short-rotation poplar (*Populus*) bioenergy plantation located in Flanders, Belgium (51°06'44" N, 3°51'02" E). The plantation had been established in spring 2010 in a double-row planting system, i.e. the distance between two adjacent rows of poplar trees alternated between 0.75 and 1.50 m (hereafter referred to as narrow and wide rows respectively). Within a row, the poplars were planted 1.10 m apart. The soil was a loamy sand. More information on the design, the layout and the management of the plantation can be found in Broeckx et al. (2012) and Berhongaray et al. (2015).

The part of the plantation in which this study took place was coppiced for the second time in March 2014. The poplar stems were cut manually about 10 cm above the soil surface. The experimental set-up of the automated GHG flux monitoring intercomparison campaign is shown in Fig. 1a and b. The measurement set-up consisted of eight automated chambers located only in wide rows due to their size (AGPS, Umwelt- und Ingenieurtechnik GmbH (UIT), Dresden, Germany), eight automated chambers which were evenly distributed in narrow and wide rows (LI-8100A, LI-8150, LI-8100-104, LI-COR Biosciences, Lincoln, NE, USA) and eight soil gas concentration profile plots – each consisting of two soil gas samplers (ML 131099, Mikrolab, Højbjerg, Denmark) – which were also evenly distributed in narrow and



**Figure 1.** Schematic drawing of the field site (a), view of the southern half of the field site on 25 March 2014 shortly after the harvest (b), and size comparison of an AGPS chamber (left, chamber open) and a LI-8100A chamber (right, chamber closed) (c). In (a), the big black-filled rectangle shows the location of the housing for the Los Gatos analysers and the AGPS multiplexer; the small black-filled rectangle indicates the location of the LI-8100A gas analyser and multiplexer; hollow rectangles represent AGPS chambers; black circles represent LI-8100A chambers; crosses represent soil gas concentration measurement nests; grey circles indicate the position of the poplars. The dashed black lines indicate the soil sampling transects.

wide rows. A detailed description of each soil GHG sampling device is presented in Sects. 2.2 and 2.3 below. The intercomparison campaign took place from 15 April until 31 August 2014. During this period the coppiced poplar stools regrew to a height of about 3.40 m. Canopy closure was achieved at the beginning of July 2014.

## 2.2 Automated soil flux chamber systems

The AGPS and LI-8100A were both closed dynamic chamber systems with the chambers operating in sequence. The technical specifications of the two chamber systems are displayed in Table 1, and Fig. 1c shows a close-up of an AGPS chamber and a LI-8100A chamber.

The AGPS chambers ran on rails and were moved to and from the collar by an attached steel cable. The average time for closing the chamber was about 40 s. Once closed, the chamber rested directly on the collar rim. The tubing inlet and outlet went through one of the collar walls and were positioned 5 cm above the soil surface inside the measurement plot together with a non-shaded air temperature sensor. This design caused additional disturbance of the soil during collar

installation because a small hole had to be dug in one corner of the measurement plot to put the tubing and the sensor cable into the ground. Each chamber was equipped with a combined soil sensor for temperature and moisture at 5 cm depth outside the collar. The chambers were connected to a multiplexer which was housed in an air-conditioned box (2.10 m length  $\times$  1.21 m wide  $\times$  1.55 m high, 20–23 °C). Air was circulated in a closed loop between the chambers and the multiplexer with a pump installed inside the multiplexer. For gas concentration analyses, gas analysers were connected in a small closed loop with the multiplexer, continuously subsampling from the big sample loop with their own internal pump. Any type and number of gas analysers could be connected to the multiplexer in parallel as long as their combined flow rate did not exceed 2.5 L min<sup>-1</sup>. The AGPS can be bought pre-configured as described in the introduction, but for this study the entire AGPS set-up had been completely custom-designed by UIT, including all the specifications listed in Table 1. Not included in the set-up were the gas analysers. Here, we report CO<sub>2</sub> data measured by a Fast Greenhouse Gas Analyser (FGGA, Los Gatos Research, Mountain View, CA, USA). All data were logged on a central computer and managed with the software SENSOWeb (UIT). The computer was also the access point for remotely controlling the AGPS and the FGGA. The AGPS had continuously been deployed in the poplar plantation since May 2013 and was only shortly removed during the harvest (January–March 2014). Reinstallation of the AGPS took place during the first half of March 2014 in exactly the same locations that were used prior to the coppice operation. Due to extensive problems with condensing water inside the tubes in 2013, the tubing was equipped with a heating system during reinstallation. During the flux monitoring, weeds were manually removed from inside the collars and around the chambers about every 2 weeks.

The AGPS sampling protocol consisted of the following steps: (i) 30 min tube heating; (ii) 5 min sampling of atmospheric air at 50 cm height outside the multiplexer for flushing the gas analyser; (iii) 2 min purging of the tubes between the chamber and the multiplexer; (iv) 1 min in which the chamber closed, with the multiplexer pump automatically turned off during this time; (v) 10 min measurement with 1 Hz gas sampling frequency; (vi) 1 min in which the chamber opened (multiplexer pump turned off); and (vii) step ii repeated for 11 min. Each chamber was sampled every 4 h resulting in total in 48 measurements per day. The chambers did not move when the air temperature dropped below 2 °C (built-in freeze protection).

The LI-8100A was an off-the-shelf product. It consisted of three main components: the gas analyser hosted in an analyser control unit (LI-8100A), a multiplexer (LI-8150) and the automated long-term chambers (8100-104) (LI-COR Biosciences, 2010). Both the analyser control unit and the multiplexer had their own weather-proof casing, requiring no additional air-conditioning. Neither tube heating nor freeze protection had been implemented; chambers operated at subzero

**Table 1.** Technical specifications of the two tested automated chamber systems.

		Greenhouse gas monitoring system AGPS	LI-8100A Automated Soil CO <sub>2</sub> Flux System
Chamber	Dimensions <sup>a</sup>	200 cm L × 80 cm W × 50 cm H	48 cm L × 38 cm W × 33 cm H
	Headspace volume	25 000 cm <sup>3</sup> , square	4076 cm <sup>3</sup> , round
	Material	stainless steel frame with white FOREX box	white coated stainless steel
	Sealing	1 continuous hollow and soft PVC strip per chamber side, 1 cm thick, transparent	1 neoprene gasket plus 1 neoprene collar gasket, black
	Vent Fan	20 cm long tube on the outside, 1 cm I.D. <sup>b</sup>	special vent design <sup>c</sup>
Collar	Dimensions	48 cm × 48 cm <sup>d</sup> /58 cm × 58 cm <sup>e</sup>	20.3 cm I.D./21.3 cm O.D.
	Enclosed soil area	2304 cm <sup>2</sup>	318 cm <sup>2</sup>
	Insertion depth	~ 3 cm	~ 7 cm
	Offset <sup>f</sup>	2.1 ± 0.7 cm	4.1 ± 1.1 cm
	Material	stainless steel	PVC, green
Tubing <sup>g</sup>	Length	11–25 m	15 m
	Diameter	6.0 mm I.D.	3.2 mm I.D.
	Material	PTFE, protected inside a black plastic tube	Bev-a-line, protected inside a black plastic tube
Flow rate	3.0–3.2 lpm <sup>g</sup> /0.4–0.5 lpm <sup>h</sup>		2.4–2.9 lpm <sup>g</sup> /1.7 lpm <sup>h</sup>
Multiplexer pump	diaphragm		diaphragm
Gas analyser	Principle	off-axis integrated cavity output spectroscopy	Non-dispersive Infrared
	Measurement range	200–4000 ppm <sup>i</sup> , 7000–70 000 ppm <sup>j</sup>	0–20 000 ppm <sup>i</sup> , 0–40 mmol mol <sup>-1</sup> <sup>j</sup>
	Uncertainty	total uncertainty: < 0.25 % of reading <sup>i,j,m</sup> precision <sup>n</sup> CO <sub>2</sub> : 150 ppb precision <sup>n</sup> H <sub>2</sub> O: 100 ppm	accuracy: 1.5 % of reading <sup>i,j</sup> RMS noise CO <sub>2</sub> : < 1 ppm <sup>k</sup> RMS noise H <sub>2</sub> O: < 0.01 mmol mol <sup>-1</sup> <sup>l</sup>
Total gas volume	30 294–33 719 cm <sup>3</sup>		5372–6294 cm <sup>3</sup>
Operational range	Chamber	> 2 °C, RH: non-condensing <sup>o</sup>	–20 to 45 °C, 0 to 95 % RH (non-condensing)
	Gas analyser	0 to 45 °C, < 98 % RH (non-condensing)	–20 to 45 °C, 0 to 95 % RH (non-condensing)
Accessories	Air temperature	Easytemp TMR31, Pt100 A	thermistor, accuracy ±0.5 °C
	Soil temperature	SPADEP, DS18B20 digital thermometer, accuracy ±0.5 °C	thermistor, accuracy ±1.0 °C
	Soil moisture	SPADEP, ring oscillator, relative accuracy ±4 %	Decagon ECH <sub>2</sub> O model EC-5, ±3% VWC, most mineral soils
	Air pressure	not implemented	1.5 % accuracy
Power requirement	max. 2000 W		max. 60 W

<sup>a</sup> The entire supporting structure, not only the chamber itself; <sup>b</sup> according to Parkin and Venterea (2010); <sup>c</sup> Xu et al. (2006); <sup>d</sup> internal; <sup>e</sup> rim included; <sup>f</sup> collar height above the soil surface; <sup>g</sup> chamber to multiplexer; <sup>h</sup> multiplexer to gas analyser; <sup>i</sup> CO<sub>2</sub>; <sup>j</sup> H<sub>2</sub>O; <sup>k</sup> at 370 ppm with 1 s signal averaging; <sup>l</sup> at 10 ppt with 1 s signal averaging; <sup>m</sup> without calibration; <sup>n</sup> 1σ, 5 s signal averaging; <sup>o</sup> incorporated freeze protection which automatically puts the system into standby when ambient air temperature drops below 2 °C; however, the chambers could also work at lower temperatures; <sup>p</sup> the soil temperature and soil moisture sensor are incorporated into one device (Qu et al., 2013); L: length, W: width, H: height, RH: relative humidity, I.D.: inner diameter, O.D.: outer diameter.

temperatures. The chambers were moved by a non-flexible arm. The time needed to close a chamber was between 11 and 15 s during which the multiplexer pump did not turn off. Once closed, the chamber rested not directly on the collar rim but on a metal plate surrounding the collar, leaving the collar undisturbed. Tubing inlet and outlet were installed inside the chamber. Soil sensors were installed the same way as for the AGPS. All measurement data were stored inside the analyser control unit on a compact flash card which could be accessed and controlled remotely via the central computer. The measurement protocol for each chamber consisted of a 2 min tubing pre-purge period, a 3 min measurement with 1 Hz gas sampling frequency and 2 min tubing post-purge time. Each chamber was sampled every 2 h. The LI-8100A had been run-

ning at a different location in the plantation since March 2011 (Verlinden et al., 2013) and received a factory check-up in spring 2014. Reinstallation after the harvest took place at the beginning of March 2014. Weeding in and around the chambers followed the same routine as for the AGPS.

### 2.3 Soil CO<sub>2</sub> concentration measurements

Each soil CO<sub>2</sub> concentration sampler consisted of a 16 mm thick, corrosion-resistant steel tube with a 10 mL sampling cell (12 mm diameter) at its lower end. The length of the sampler depended on the sampling depth. The sampling cell was connected to the surrounding soil via a 3 mm diameter opening in the steel tube. The opening was covered

with a  $12 \times 0.5 \text{ mm}^2$  silicone disc to allow only the diffusion of gases between the soil and the cell. For sampling, the steel tube contained two smaller tubes made from stainless steel needles (18G, inner diameter 0.8–0.875 mm) which connected the sampling cell with the soil surface after installation (ML 131099, Mikrolab, Højbjerg, Denmark). The samplers were installed by pre-drilling a hole of the same diameter as the sampler to about 5 cm above the intended measurement depth. The samplers were inserted into the hole and carefully pushed to the measurement depth, aided by a 30 mm long, hardened PVC tip at the bottom of the sampler. At each soil gas concentration profile plot, two samplers were installed – one at 5 and one at 15 cm depth.

Soil  $\text{CO}_2$  was sampled about every 2 weeks between 10:00 and 14:00. A plastic syringe containing 10 mL  $\text{N}_2$  and 50 ppm  $\text{C}_2\text{H}_4$  was connected via a two-way valve to one of the small tubes inside the sampler. An empty 10 mL glass syringe (SIGMA-ALDRICH, Diegem, Belgium) and a 12 mL pre-evacuated Exetainer (Labco Ltd, Lampeter, UK) were connected to the other tube via a three-way valve. The  $\text{N}_2/\text{C}_2\text{H}_4$  mixture was injected into the diffusion cell flushing the 10 mL soil gas sample via the second tube into the glass syringe. The glass syringe was then emptied into the Exetainer. Finally, 12 mL  $\text{N}_2$  was injected into the Exetainer to create an overpressure needed for the subsequent gas analysis. The concentration of  $\text{C}_2\text{H}_4$  recovered in the collected sample was used to calculate the dilution of the original sample, which occurred while replacing it with  $\text{N}_2$  in the sampling cell, and to correct the measured  $\text{CO}_2$  concentration accordingly. The correction was performed with the assumption that there was full equilibrium between the diffusion cell and the inlet and outlet tube. During the sampling, diffusive loss of  $\text{C}_2\text{H}_4$  via the silicone membrane to the soil atmosphere was considered negligible. After the sampling, the diffusion cell and the sampling tubes were flushed with 60 mL  $\text{N}_2$  to remove remaining traces of  $\text{C}_2\text{H}_4$ . For more details on the sampler design and the  $\text{C}_2\text{H}_4$  correction see Petersen (2014).

The gas samples were analysed on a Bruker custom greenhouse analyser (Bruker Daltonik GmbH, Bremen, Germany) equipped with a thermal conductivity detector (TCD) for the analysis of  $\text{CO}_2$  and  $\text{C}_2\text{H}_4$ . The TCD channel was equipped with a HayeSep column. Temperatures of the inlet, column and TCD were 50, 50 and 200 °C respectively. Helium at  $20 \text{ mL min}^{-1}$  was used as reference flow. Total flow was  $60 \text{ mL min}^{-1}$ . Concentrations were quantified with reference to three calibration gases with an accuracy of 2 %: (i)  $50 \mu\text{L L}^{-1}$   $\text{C}_2\text{H}_4$  in  $\text{N}_2$ , (ii)  $799 \mu\text{L L}^{-1}$   $\text{CO}_2$  in synthetic air and (iii) 5.04 %  $\text{CO}_2$  in synthetic air.

## 2.4 Soil sampling

To assess the impact of the permanently installed chambers on soil properties which potentially control soil  $\text{CO}_2$  concentrations and flux rates, as well as to assess the comparability

between the flux measurement plots, soil samples were taken before and after the intercomparison campaign. In February 2014, undisturbed topsoil samples were taken along two transects (Fig. 1a). At each transect, three samples were taken per row type and per sampling depth. For soil C and dissolved organic carbon (DOC), soil was sampled at 0–10 and 10–20 cm depth with an auger ( $\sim 2 \text{ cm}$  diameter). Separate samples were taken with stainless steel cylinders ( $100 \text{ cm}^3$ ) (Eijkelkamp Agrisearch equipment, Giesbeek, the Netherlands) at 0–5 and 10–15 cm depth for dry bulk density. Soil sampling was repeated at the beginning of September 2014, but this time in each of the 16 chamber collars and within each soil gas sampling plot.

About 9 g field moist soil of each auger sample was shaken in 35 mL 0.5 M  $\text{K}_2\text{SO}_4$  for 1 h. This suspension was filtered with Whatman filter paper (grade 42, ashless, 150 mm) and the filtered liquid analysed for DOC with continuous flow analysis (San++ Automated Wet Chemistry Analyzer, Skalar Analytical, Breda, the Netherlands). The rest of the auger samples were dried at 50 °C and ground, and three subsamples per sample were analysed by dry combustion with a NC element analyser (NC-2100, Carlo Erba Instruments, Italy) and their means were reported. Out of necessity the February 2014 samples had to be aggregated by row type and sampling depth prior to the grinding. The steel cylinder samples were dried at 105 °C to constant weight for dry bulk density determination.

The soil data from February 2014 were grouped by row type, and the data from September 2014 by row type and measurement device. One-way omnibus ANOVA and the Tukey honest significant difference test were used to compare group means ( $\alpha = 0.05$ ). Normality for each group and homogeneity of variance of the groups were tested with the Shapiro–Wilk test and the Levene test respectively. The soil data analysis as well as any other data analysis for this study was conducted with the software R (version 3.1.1) (R Core Team, 2014). The only exception was the chamber flux calculation for the AGPS (Sect. 2.5) which had to be conducted with R version 3.0.2 due to a package incompatibility.

## 2.5 Chamber flux calculation and quality control

For the AGPS, descriptive statistics and water-corrected  $\text{CO}_2$  fluxes were calculated with a self-written R script incorporating the “gasfluxes” script (Roland Fuß, Institute of Agricultural Climate Research, Johann Heinrich von Thünen Institute, Braunschweig, Germany, version 0.98.int) and the HMR package (Pedersen et al., 2010) and additionally by employing the packages “zoo”, “xts” and “xtsExtra” (Zeileis and Grothendieck, 2005; Ryan and Ulrich, 2014; Weylandt, 2014). For each AGPS measurement, the flux was calculated with linear regression, robust linear regression with a Huber M estimator (RLM) (Huber, 1981) and a modified Hutchinson–Mosier non-linear function (HMR) (Pedersen et al., 2010). This procedure was performed twice for each mea-

surement – for a closure time of 4 and 9 min respectively. Prior to each flux calculation, the first minute of the CO<sub>2</sub> concentration curves was discarded (= deadband) to account for the time needed to establish steady headspace mixing as well as any disturbances caused by the chamber placement at the beginning of the measurement (Christiansen et al., 2011; Koskinen et al., 2014). For each flux calculation, the “gas-fluxes” script selected the HMR flux if (i) the Akaike information criterion (AIC) of HMR was smaller than the AIC of the linear fit, (ii) the *p* value of the flux calculated with HMR was smaller than the *p* value of the flux calculated with linear regression and (iii) the flux calculated with HMR was not more than 4 times higher/lower than the flux calculated with RLM. In all other cases, RLM was chosen as the best-fitting model. The fluxes calculated by linear regression and RLM were the same, except that RLM was robust against outliers in the CO<sub>2</sub> concentration curves. Fluxes were converted from  $\mu\text{L m}^{-2} \text{s}^{-1}$  to  $\mu\text{mol m}^{-2} \text{s}^{-1}$  using the ideal gas law (Parkin and Venterea, 2010) with air temperature and pressure provided by the AGPS and the LI-8100A respectively.

For the LI-8100A, water-corrected mass CO<sub>2</sub> fluxes and descriptive statistics were automatically provided by the LI-8100 File Viewer version 3.0.0 (LI-COR Biosciences). For each chamber measurement, the flux was calculated with either a linear or an empirical exponential regression (LI-COR Biosciences, 2010). The software compared for each measurement the normalised sums of the squares of the residuals (SSN) of the linear and the exponential fit to find the best-fitting model. The first 25 s of each 3 min CO<sub>2</sub> concentration curve were discarded before the flux calculation.

Fluxes were discarded from the two data sets by applying in sequence the following quality control criteria: (i) negative fluxes, (ii) fluxes with the SSN of the linear fit > 1.0 ppm CO<sub>2</sub> (equivalent to a root mean square error threshold of 1.0 ppm CO<sub>2</sub>, Görres et al., 2014), (iii) decrease in headspace temperature during the closure time by more than 0.5 °C or increase by more than 1.0 °C, (iv) difference in the atmospheric CO<sub>2</sub> concentration 5 cm above the collar directly before the chamber closure and after a deadband of 1 min of less than 0.0 ppm (i.e. decreasing CO<sub>2</sub> concentration) and (v) mean relative humidity (RH) inside the closed chamber higher than 100 %. The first criterion detected chamber measurements with large leaks, whereas smaller leakages and other measurement disturbances could be detected by selecting an appropriate noise level threshold in the second criterion. CO<sub>2</sub> flux measurements can be very sensitive to changes in environmental conditions; thus criteria (iii)–(v) removed measurements for which the CO<sub>2</sub> concentration increase curve looked okay, but which might still have been biased by changes in environmental conditions too large to guarantee continuous identical diffusion conditions during chamber closure.

## 2.6 Soil diffusivity and gradient-based CO<sub>2</sub> flux calculation

Changes in topsoil CO<sub>2</sub> concentration dynamics for each collar and each soil gas sampling plot throughout the intercomparison campaign were approximated by calculating the effective soil diffusion coefficient ( $D_s$ ), which is the product of the CO<sub>2</sub> diffusion coefficient in free air ( $D_a$ ) and the gas tortuosity factor  $\xi$ .  $D_a$  was corrected for temperature and air pressure by

$$D_a = D_{a0} \left( \frac{T}{293.15} \right)^{1.75} \left( \frac{P}{101.3} \right), \quad (1)$$

where  $T$  is soil temperature at 5 cm depth (K),  $P$  the air pressure from the LI-8100A (kPa) and  $D_{a0}$  a reference value of  $D_a$  at 20 °C (293.15 K) and 101.3 kPa given as  $14.7 \text{ mm}^2 \text{ s}^{-1}$  (Jones, 1992). The empirical Millington–Quirk model was used for estimating  $\xi$  (Millington and Quirk, 1961):

$$\xi = \frac{(\phi - \text{VWC})^{10/3}}{\phi^2}, \quad (2)$$

where VWC is the volumetric water content at 5 cm depth and  $\phi$  the total porosity ( $\text{m}^3 \text{ m}^{-3}$ ). Total porosity was calculated by dividing the averaged topsoil dry bulk density for each measurement plot by the particle density. Particle density was empirically adjusted for the C content at each measurement plot according to Eq. (12) in Rühlmann et al. (2006), assuming a C content in the organic matter of 55 %.

Additionally, soil CO<sub>2</sub> fluxes were calculated via Fick’s first law of diffusion by multiplying the CO<sub>2</sub> concentration gradients between 5 and 15 cm depth obtained from the soil gas sampling plots with the respective  $D_s$  (Roland et al., 2015). Prior to the flux calculation, soil CO<sub>2</sub> concentrations in ppm were converted to  $\mu\text{mol m}^{-3}$  by multiplying them by the molar volume of a gas at standard temperature and pressure ( $0.04462 \mu\text{mol L}^{-1}$ ; Brummell and Siciliano, 2011). Soil temperature and soil moisture values at 5 cm depth were obtained from the nearest chamber in the same row type.

## 2.7 Comparison of the CO<sub>2</sub> flux data sets

The AGPS and LI-8100A soil CO<sub>2</sub> fluxes were directly compared for four different environmental conditions, namely (i) daytime, constant atmospheric CO<sub>2</sub> concentration, (ii) daytime, fluctuating atmospheric CO<sub>2</sub> concentration, (iii) nighttime, constant atmospheric CO<sub>2</sub> concentration and (iv) nighttime, fluctuating atmospheric CO<sub>2</sub> concentration. Daytime and nighttime fluxes were separated based on local sunrise and sunset times. Atmospheric CO<sub>2</sub> concentration was considered as constant when the CO<sub>2</sub> concentration measured at 50 cm height above the soil surface had a standard deviation < 1.0 ppm (3 min measurements). Constant ambient CO<sub>2</sub> concentrations were seen as a proxy indicator of a well-mixed atmosphere, i.e. wind perturbation.

Additionally, two modelling approaches were applied for the comparison of the two flux data sets. Firstly, average CO<sub>2</sub> flux rates for each automated chamber system and their respective 95 % confidence intervals were estimated using generalized additive models (GAM) with a nonlinear smooth (thin plate regression spline) for time and with random smooths (factor smooth interactions) over time for each chamber (R packages “itsadug” and “mgcv”, function “bam”) (Wood, 2006; van Rij et al., 2015). Autocorrelation was accounted for by including an AR1 model. Input data were the unfiltered and filtered CO<sub>2</sub> fluxes respectively, averaged by day and by chamber. Secondly, the chamber flux data sets were quantitatively compared by using the common approach of modelling SR according to Lloyd and Taylor (1994) (Eq. 11):

$$SR = R_{10} \exp E_0 \left( \frac{1}{56.02} - \frac{1}{T - 227.13} \right), \quad (3)$$

where  $R_{10}$  is the respiration rate at 10 °C ( $\mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$ ),  $E_0$  the temperature sensitivity coefficient (K) and  $T$  the soil temperature at 5 cm depth (K). SR was modelled separately for each combination of chamber system and row type. Each model was also fitted once with the unfiltered and the filtered data set respectively, using nonlinear regression (R function “nls”). Input data were the single measured CO<sub>2</sub> fluxes. A part of the chamber measurements were excluded from the modelling due to missing soil temperature values (sensor malfunctions). Cumulated CO<sub>2</sub> fluxes for the monitoring period were calculated by running the different fitted SR models with average hourly time series of soil temperature at 5 cm depth. For the construction of the average hourly time series, the time series with the least number of gaps was chosen as a reference to which all other chamber soil time series were linearly correlated. Any remaining gaps in the time series were linearly interpolated and the time series subsequently averaged for each combination of chamber system and row type. Model runs were performed with the R function “predictNLS” (package “propagate”) which calculated 95 % confidence intervals for the fitted values by using Monte Carlo simulation and taking into account the error in the model parameter estimates as well as the standard deviation of the averaged soil temperature time series.

### 3 Results

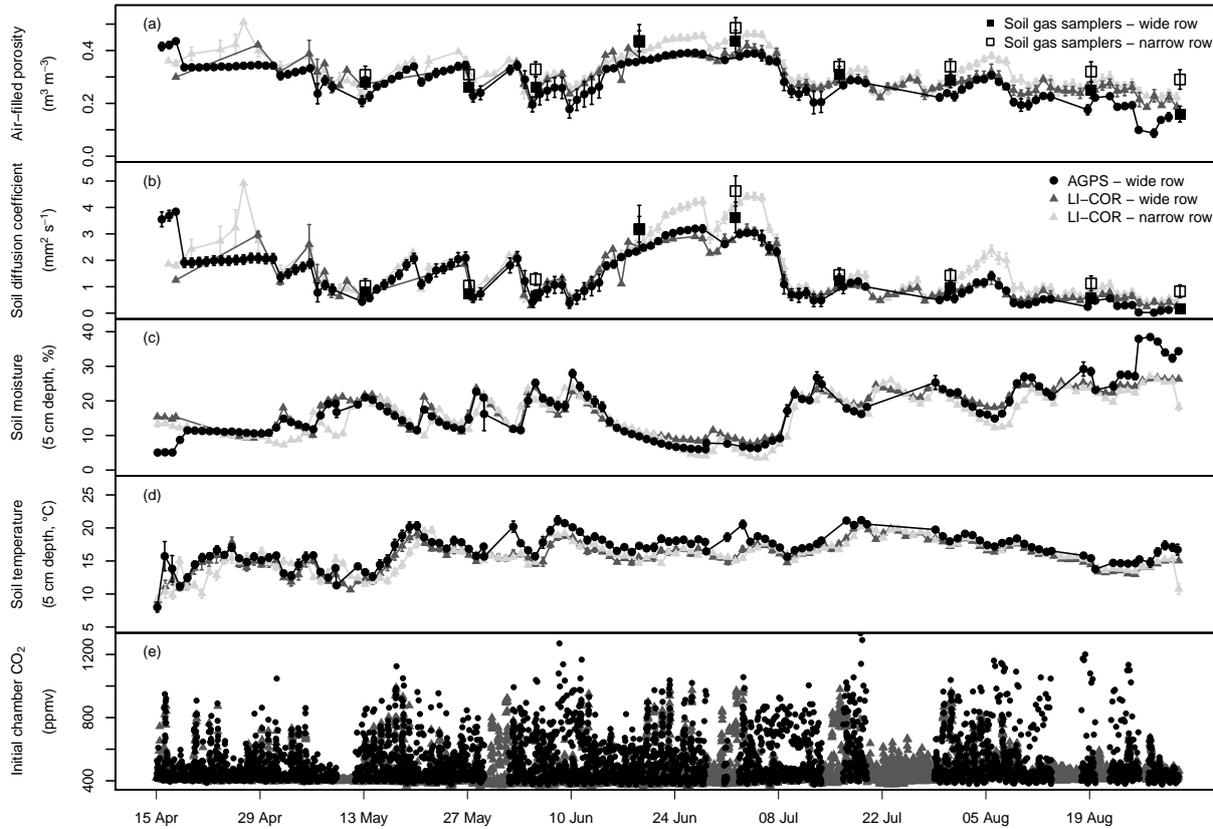
#### 3.1 Variability in environmental conditions

In February 2014 the dry bulk density in the undisturbed top soil was  $1.41 \pm 0.11 \text{ g cm}^{-3} \text{ dry soil}^{-1}$  (average  $\pm$  SD,  $n = 24$ ), the C content  $1.21 \pm 0.17 \%$  ( $n = 8$ , pre-analysis sample pooling) and the DOC content  $32.07 \pm 10.03 \mu\text{g g}^{-1} \text{ dry soil}^{-1}$  ( $n = 24$ ), with no significant differences between wide and narrow poplar rows. The soil sampling results at the end of the chamber intercomparison

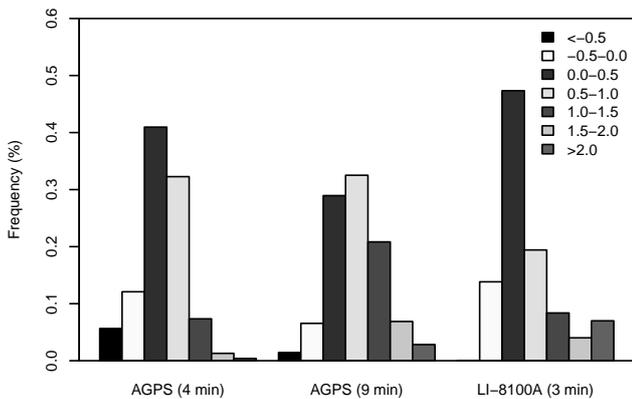
campaign did not differ significantly from these values. After the end of the flux monitoring, the inner walls of the LI-8100A collars were covered with a loose mat of new grown roots (Fig. S1 in the Supplement). Such a mat was not observed during the removal of the AGPS chambers and soil gas samplers. However, no significant differences in dry bulk density, C and DOC between the devices were found within a row type. Between row types, only the dry bulk density inside the AGPS collars in the wide rows ( $1.44 \pm 0.07 \text{ g cm}^{-3} \text{ dry soil}^{-1}$ ,  $n = 16$ ) differed significantly from the LI-8100A chambers ( $1.32 \pm 0.12 \text{ g cm}^{-3} \text{ dry soil}^{-1}$ ,  $n = 8$ ) and the soil gas samplers ( $1.24 \pm 0.13 \text{ g cm}^{-3} \text{ dry soil}^{-1}$ ,  $n = 5$ ) installed in the narrow rows. Thus, a methodological comparison of the soil CO<sub>2</sub> flux dynamics captured by the flux measurement devices within a row type was regarded as feasible.

Air-filled porosity and the derived soil diffusion coefficient showed a high variability throughout the monitoring time. They were on average slightly higher in the narrow rows than in the wide rows (Fig. 2a and b). This variability was driven by several heavy rain events resulting in sharp soil moisture increases (Fig. 2c). From July 2014 onwards standing water was observed in parts of the wide rows following precipitation but never in the narrow rows, which drained much faster despite no significant differences in dry bulk density between row types.

The AGPS collars received more direct sunlight than the LI-8100A collars, resulting in higher air and subsequently constantly higher soil temperatures (Fig. 2d). This was an effect of the weeding, since the collar area which could be potentially shaded by the vegetation still surrounding the chamber decreased with increasing collar area. The average daily soil temperature difference between the AGPS and the LI-8100A was generally less than 1 °C when the fraction of shading by the vegetation was homogenous throughout the study site. However, during the transition period from an open to a closed poplar canopy the soil temperature difference was constantly higher than 1 °C. This transition period from the beginning of June until the middle of July also coincided with the warmest and the driest monitoring period. Canopy closure above the AGPS collars was reached about a week later in comparison to the LI-8100A chambers because the larger structure of the AGPS chambers hindered the growing poplar stems from leaning towards each other (see Fig. S2 in the Supplement for the different vegetation stages). The high air temperatures observed above the AGPS collars were also partly an artefact of the non-shaded sensors. During a small proportion of the AGPS measurements, the temperature inside the chamber decreased by more than 0.5 °C (Fig. 3). This phenomenon was mainly observed above an ambient air temperature of 20 °C and was regarded as an indicator for the cooling down of an overheated sensor. Overall, the insulation of the chambers worked well, with more than 68 and 80 % of the AGPS and LI-8100A measurements respectively, fulfilling quality control criterion 3.



**Figure 2.** Comparison of different environmental variables measured by the AGPS and the LI-8100A before each chamber closure. Panels (a–d) show daily averages with their respective standard deviations. For air-filled porosity and the soil diffusion coefficient only those measurements have been included for which both soil temperature and moisture data were available from the specific chamber at the time of the measurement. All single measurements are shown for the initial CO<sub>2</sub> concentration (panel e), which is equivalent to the CO<sub>2</sub> concentration at time = 0 s of the flux measurement. They are measured by the AGPS prior to the chamber closure and calculated for the LI-8100A by its internal software.



**Figure 3.** Air temperature change inside the chambers during the closure time. For the AGPS, temperature change is shown for the first 4 min of the closure time and for 9 min closure time. The LI-8100A had a closure time of 3 min.

Variability in air and soil temperature decreased after the canopy closure at the beginning of July, but the opposite was

true for the atmospheric CO<sub>2</sub> concentration measured 50 cm above the soil surface. Constant atmospheric CO<sub>2</sub> concentrations at that height were only observed before the canopy closure and mainly during daytime as one would expect with a well-mixed boundary atmosphere (unstable atmospheric layering). For more than 70 % of the flux measurements, the atmospheric CO<sub>2</sub> concentration 50 cm above the soil surface fluctuated by more than 1.0 ppm prior to the chamber closure. Atmospheric CO<sub>2</sub> concentrations measured 5 cm above the collars at the time of chamber closure matched well between the AGPS and the LI-8100A most of the time (Fig. 2e). However, the AGPS recorded a number of atmospheric CO<sub>2</sub> concentrations at chamber closure above 500 ppm which were not observed by the LI-8100A. Two-thirds of the AGPS CO<sub>2</sub> values above 500 ppm were measured during nighttime.

### 3.2 Technical reliability of the two chamber systems

During the intercomparison campaign, the LI-8100A conducted 12 874 chamber measurements (wide rows: 6253; narrow rows: 6621) of which only one measurement had to be

**Table 2.** Number of discarded CO<sub>2</sub> fluxes after each filter step for the two automated chamber systems (SSN<sub>Lin</sub> is the normalised sum of squares of residuals for linear fit;  $\Delta T_{\text{air}}$  is the change in air temperature inside the closed chamber during the closure time;  $\Delta\text{CO}_2$  is the difference in the atmospheric CO<sub>2</sub> concentration 5 cm above the collar directly before chamber closure and after a deadband of 1 min; RH is relative humidity; NA indicates information not available for that chamber system). Data sets were grouped by time of the day and stability of the atmospheric CO<sub>2</sub> concentration at 50 cm above the soil surface. Day and night were based on sunrise and sunset times. Atmospheric CO<sub>2</sub> concentration was considered as constant when the standard deviation for a 3 min measurement prior to the chamber closures was  $\leq 1.0$  ppm. The AGPS total closure time was 10 min. Fluxes were once calculated for the first 4 min of the closure time (left of the vertical line) and once for 9 min closure time (right of the vertical line), each with a 1 min deadband.

	Total		Day (constant)		Day (fluctuating)		Night (constant)		Night (fluctuating)	
	LIN	EXP	LIN	EXP	LIN	EXP	LIN	EXP	LIN	EXP
AGPS										
Unfiltered <sup>a</sup>	2806 4140	2105 771	580 824	492 248	1211 1780	875 306	197 276	141 62	818 1260	597 155
Negative fluxes	120 97	31 30	49 57	17 19	31 18	4 3	18 12	5 7	22 10	5 1
SSN <sub>Lin</sub> > 1.0 ppm	1717 3510	1165 719	321 699	276 223	858 1583	577 298	86 215	58 46	452 1013	254 152
$-0.5 < \Delta T_{\text{air}} > 1.0$ °C	146 192	138 3	29 14	29 0	44 33	36 2	0 15	2 0	73 130	71 1
$\Delta\text{CO}_2 < 0.0$ ppm	88 53	58 5	8 3	8 1	30 30	20 2	14 2	1 2	36 18	29 0
RH > 100 %	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Filtered	735 288	713 14	173 51	162 5	248 116	238 1	79 32	75 7	235 89	238 1
LI-8100A										
Unfiltered	5640	7233	1376	888	2781	3233	313	437	1170	2675
Negative fluxes	0	1	0	0	0	1	0	0	0	0
SSN <sub>Lin</sub> > 1.0 ppm	201	191	66	31	68	69	13	21	54	70
$-0.5 < \Delta T_{\text{air}} > 1.0$ °C	1328	1102	533	263	663	386	11	24	121	429
$\Delta\text{CO}_2 < 0.0$ ppm	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
RH > 100 %	74	61	14	8	37	28	14	10	9	15
Filtered	4037	5878	763	586	2013	2749	275	382	986	2161

<sup>a</sup> Twenty-one measurements discarded prior to the filtering because of missing air temperature measurements for the flux calculation.

discarded due to technical problems with the chamber closing mechanism. Overall, the LI-8100A showed a high robustness despite having previously operated continuously in the poplar plantation for 3 years. It recorded only 62 suspicious atmospheric air pressure readings and 206 readings of RH inside the closed chambers of more than 100 %, indicating conditions of water condensation. The AGPS conducted 78 % of the 6296 theoretically possible chamber measurements. A negligible amount of measurements did not take place due to system maintenance ( $n = 37$ ), activation of the freeze protection ( $n = 10$ ) and chamber malfunctions ( $n = 111$ ). Chamber malfunctions were all caused by the steel cables which moved the AGPS chambers. These cables did expand or contract depending on the air temperature and, as a consequence, their tension had to be checked and corrected once per week or at least every 2 weeks to prevent chambers from getting stuck.

Two main issues prevented the AGPS from operating continuously in the field. Firstly, the air filters inside the multiplexer became clogged up with liquid water during heavy rain events, preventing 602 potential measurements. That could have probably been avoided had the inlets at the collars been equipped with air filters the same way the LI-8100A chambers are. Secondly, 609 chamber measurements could not be analysed because the gas analyser froze. Each time these two issues occurred, it was possible to get the AGPS operational again in less than 2 h. The large amount of lost

data was mainly attributable to the fact that most of the time someone had to be present in the field for maintenance which was not always possible on the very day the problems occurred.

Another issue with the gas analyser was that the internal software did not save the measured data continuously at 1 Hz. For the 4 min closure time and the 9 min closure time only 1070 (22 %) and 328 (7 %) measurements provided a data set at 1 Hz frequency respectively. The median number of data points for the short and long flux calculation period (i.e. chamber closure time excluding 1 min deadband period) was 167 and 328 respectively. During the first half of June 2014, the number of data points per measurement even dropped below 50 and 100 respectively.

### 3.3 Flux quality

In total 23 % of the LI-8100A CO<sub>2</sub> flux measurements were discarded, mainly because of headspace temperature changes (Table 2). During the open canopy phase, this problem was mainly encountered during the day, whereas equal amounts of fluxes were discarded from the daytime and nighttime data set based on headspace temperature changes once the canopy was closed. With respect to row type, headspace temperature problems were more often encountered in the wide rows, whereas chamber measurements in the narrow rows were more likely to have a SSN<sub>Lin</sub> > 1.0 ppm or a RH > 100 % than those in the wide rows. More than 50 % of the LI-8100A con-

centration curves were best-fitted exponentially, especially under fluctuating atmospheric CO<sub>2</sub> concentrations.

For the AGPS data set, a higher proportion of the concentration curves were best-fitted linearly regardless of daytime or atmospheric CO<sub>2</sub> concentration variability. Only for the 4 min closure time in the filtered data set were the two flux calculation models about equally distributed. In total, 71 and 94 % of the AGPS fluxes using the short and the long flux calculation period respectively did not pass the quality control (Table 2). No correlation was found between the amount of discarded data and the tubing length of the chambers. For the unfiltered data set, the fluxes calculated for 9 min closure time were  $0.21 \pm 0.50 \mu\text{mol m}^{-2} \text{s}^{-1}$  (average  $\pm$  SD) lower than the fluxes calculated for a 4 min closure time. In the filtered data set, this was reduced to  $0.18 \pm 0.18 \mu\text{mol m}^{-2} \text{s}^{-1}$ . Due to the low data quality for the long flux calculation period, only the fluxes calculated for the 4 min closure time were considered in the remaining result sections below.

For the 4 min closure time, 2882 flux measurements had a  $\text{SSN}_{\text{Lin}} > 1.0$  ppm. This included almost all data from the first half of June when we had the severe gas analyser logging problem. The  $\text{SSN}_{\text{Lin}}$  criterion also already filtered out 60 and 79 % of the flux measurements with headspace temperature problems and with a decrease of the CO<sub>2</sub> headspace concentration during the deadband period respectively. The latter criterion filtered out most of the flux measurements which had shown a large discrepancy in initial chamber CO<sub>2</sub> concentration as compared to the LI-8100A in Fig. 2e. Measurements which did not pass this criterion had a median pre-closure atmospheric CO<sub>2</sub> concentration at 5 cm height above the collar of 600 ppm, whereas measurements passing this criterion had a median pre-closure atmospheric CO<sub>2</sub> concentration of 433 ppm.

The  $\text{SSN}_{\text{Lin}}$  criterion would have also detected 83 % of the negative CO<sub>2</sub> fluxes. Negative CO<sub>2</sub> fluxes were clearly associated with severely leaking chambers. Insufficient airtight sealing was also probably a problem for a part of the fluxes with a high  $\text{SSN}_{\text{Lin}}$ . In contrast to the LI-8100A chambers, the AGPS chambers had no mechanism which additionally pressed them onto the collar once the sealing and the collar rim came in contact. The AGPS chamber and the collar had to be perfectly aligned to achieve an airtight sealing, which was challenging and required regular re-adjustments of the collars throughout the monitoring. However, the noise in the AGPS flux data set was large regardless of the environmental conditions, and the noise was lower at constant atmospheric CO<sub>2</sub> concentrations, i.e. windy conditions. All of this pointed to an inherent technical problem with the system (see Discussion below).

### 3.4 Comparison of the CO<sub>2</sub> flux data sets

Until the beginning of July 2014, CO<sub>2</sub> concentrations at 5 and 15 cm depths in the narrow rows were  $4702 \pm 762$  ppm (average  $\pm$  SE,  $n = 16$ ) and  $12\,565 \pm 2145$  ppm ( $n = 15$ ) re-

spectively and in the wide rows  $6664 \pm 1108$  ppm ( $n = 14$ ) and  $12\,251 \pm 1512$  ppm ( $n = 15$ ) respectively. Afterwards, CO<sub>2</sub> concentrations increased at 5 and 15 cm depth in the narrow rows to on average  $11\,797 \pm 2365$  ppm ( $n = 20$ ) and  $27\,071 \pm 3615$  ppm ( $n = 19$ ) respectively. In the wide rows, CO<sub>2</sub> at 5 cm depth reached the same concentrations as in the narrow rows, whereas at 15 cm depth it increased even further ( $38\,008 \pm 4574$  ppm,  $n = 19$ ). The increasing steepness of the soil CO<sub>2</sub> concentration gradient was probably partly the result of CO<sub>2</sub> accumulation in the soil due to the reduced air-filled porosity as the magnitude of the surface CO<sub>2</sub> fluxes measured with the chambers did not increase strongly during this period (Fig. 4). Contrastingly, the CO<sub>2</sub> fluxes based on the flux gradient method were unrealistically high in July and August 2014 (wide row:  $8.9 \pm 1.5 \mu\text{mol m}^{-2} \text{s}^{-1}$ ,  $n = 19$ ; narrow row:  $10.8 \pm 1.5 \mu\text{mol m}^{-2} \text{s}^{-1}$ ,  $n = 17$ ), whereas prior to the rewetting they were in the same range as the chamber CO<sub>2</sub> fluxes (wide row:  $2.6 \pm 0.5 \mu\text{mol m}^{-2} \text{s}^{-1}$ ,  $n = 12$ ; narrow row:  $3.9 \pm 0.6 \mu\text{mol m}^{-2} \text{s}^{-1}$ ,  $n = 15$ ). The soil depth resolution chosen in this study for the flux gradient method was very likely too low to realistically approximate the soil CO<sub>2</sub> concentration profiles and soil diffusion coefficients during high soil moisture conditions. Short-term fluctuations in the soil CO<sub>2</sub> concentration profiles due to heavy precipitation events were unlikely to be the main cause for the failure of the flux gradient method in July and August 2014 because the soil CO<sub>2</sub> concentration samplings were performed at least 3 days after such events, except for the last sampling.

Daily average CO<sub>2</sub> fluxes estimated from the unfiltered flux data sets did not differ significantly between the two chamber systems; however, the daily AGPS flux rates tended to be lower than the flux rates obtained with the LI-8100A during the open canopy phase (Fig. 4, top panel). With regard to the single unfiltered CO<sub>2</sub> flux measurements, the data set from the AGPS showed higher flux variability throughout the intercomparison campaign than the unfiltered CO<sub>2</sub> flux data set from the LI-8100A chambers installed in the same (wide) rows (Fig. 4). This difference in flux variability disappeared with the filtering except for a number of very low fluxes observed only by the AGPS during the open canopy phase (Fig. 5). In contrast to the AGPS, filtering mainly thinned out the LI-8100A data set of the open canopy phase since the biggest problem for these chambers was overheating. LI-8100A chambers installed in the wide rows were more subjected to this problem than those in the narrow rows (Fig. S3). The fit of the GAM for the LI-8100A data set was only slightly changed by the filtering whereas the fit for the AGPS changed significantly. This led to a distinct separation of the two GAM curves for the daily CO<sub>2</sub> fluxes (Fig. 5, top panel) with the AGPS flux estimates being constantly lower in comparison to the LI-8100A. However, both chamber systems still seemed to be able to capture the same temporal flux dynamics although the AGPS model curve was slightly shifted to the right in comparison to the LI-8100A model curve.

**Table 3.** Number of measurements ( $N$ ), regression parameters ( $E_0$  is the temperature sensitivity coefficient;  $R_{10}$  is the soil respiration rate at 10 °C soil temperature at 5 cm depth) and residual standard errors (RSEs) for the Lloyd and Taylor model fits presented in Fig. 6, and cumulated average soil respiration (cSR). Data are shown for the entire monitoring period (E, 15 April–31 August 2014), the open canopy phase (OC, 15 April–30 June 2014) and the closed canopy phase (CC, 1 July–31 August 2014) respectively. The standard errors for the regression parameters and the 95 % confidence intervals for the average cSR respectively are shown in brackets.

Chamber	Row type	Filtered	$N$			$E_0$			$R_{10}$			RSE			Average cSR		
			E	OC	CC	E	OC	CC	E	OC	CC	E	OC	CC			
AGPS	Wide	No	3378	2333		198	177	307	1.17	1.10	1.10	0.93	0.80	1.07	897	433	507
						(10.7)	(10.9)	(28.1)	(0.03)	(0.03)	(0.07)				(838–956)	(409–458)	(449–567)
AGPS	Wide	Yes	1049	743		156	125	282	0.99	0.96	0.88	0.68	0.64	0.70	698	347	308
						(18.3)	(20.1)	(53.0)	(0.04)	(0.04)	(0.10)				(655–742)	(327–367)	(334–430)
LI-8100A	Wide	No	4601	2367		279	222	369	1.24	1.32	1.07	0.75	0.66	0.81	1018	520	501
						(7.5)	(8.5)	(13.6)	(0.02)	(0.02)	(0.03)				(931–1108)	(482–558)	(448–557)
LI-8100A	Wide	Yes	3335	1445		326	226	406	1.10	1.28	0.93	0.69	0.61	0.73	974	507	469
						(9.5)	(13)	(14.4)	(0.02)	(0.03)	(0.03)				(878–1074)	(468–546)	(415–527)
LI-8100A	Narrow	No	6588	3616		230	198	263	1.77	1.77	1.76	0.87	0.80	0.91	1376	687	691
						(5.9)	(6.8)	(10.8)	(0.02)	(0.02)	(0.04)				(1319–1433)	(662–713)	(658–724)
LI-8100A	Narrow	Yes	4811	2262		285	243	285	1.57	1.60	1.62	0.76	0.64	0.84	1338	668	661
						(7.0)	(9.1)	(11.2)	(0.02)	(0.03)	(0.04)				(1270–1406)	(638–699)	(627–695)

Modelling CO<sub>2</sub> fluxes along the soil temperature gradient revealed discrepancies between the data sets of the AGPS and the LI-8100A similar to those visualised by the GAM. Filtering the LI-8100A data set changed only slightly the model fits of the Lloyd and Taylor model and had no significant effect on the subsequently calculated SR balances (Table 3, Fig. 6). The opposite was observed for the AGPS data set. The regression parameters based on the AGPS data set were lower than the ones based on the LI-8100A data set from the wide rows (Table 3); the discrepancies between the regression lines increased with increasing soil temperature and the data filtering (Fig. 6). Similar SR balances and  $R_{10}$  values were only obtained by the two chamber systems during the closed canopy phase. Regardless of chamber type, row type and environmental conditions, the filtering led to a decrease in the SR balance estimates, but it also improved the model fit (see Fig. S4 in the Supplement for the distribution of the residuals). The  $SSN_{Lin}$  and the headspace temperature criteria filtered out fluxes mainly above  $1.5 \mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$ . Only criterion (iv) mainly removed positive fluxes below  $1.5 \mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$ , but this criterion was not applicable to the LI-8100A chambers since these chambers did not provide an undisturbed pre-closure atmospheric CO<sub>2</sub> concentration.

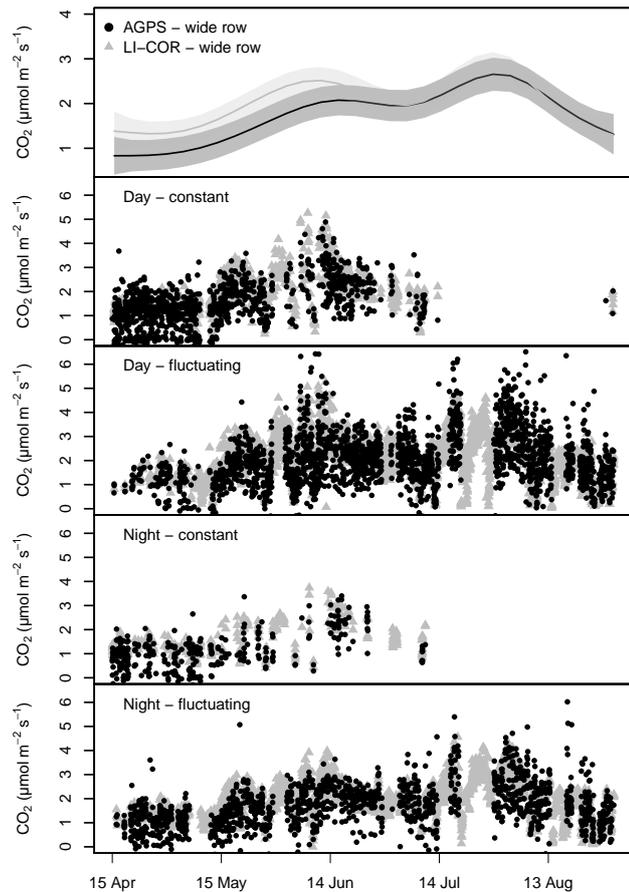
The tendency that the absolute differences in the CO<sub>2</sub> flux rates between the two data sets varied throughout the monitoring period was also visible when looking just at the measurement periods with the highest data quality (Fig. 7). During the open canopy phase, the AGPS flux rates were  $0.31 \pm 0.03 \mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$  (average  $\pm$  SE,  $n = 47$ ) lower than the ones obtained by the LI-8100A, whereas during the closed canopy phase the opposite was observed, with the AGPS flux rates being  $0.08 \pm 0.06 \mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$  ( $n = 15$ ) higher.

## 4 Discussion

The chamber methodology is based on the simple principle of diffusion, but it is an invasive method and seemingly small changes in the chamber design, the measurement protocol and the data analysis can lead to significant biases in the measured fluxes (Davidson et al., 2002; de Klein and Harvey, 2012). These biases have been quantified for different chamber types under controlled laboratory conditions, and this has already led to significant improvements in the methodology (Pumpanen et al., 2004; Christiansen et al., 2011; Pihlatie et al., 2013). However, laboratory tests have the drawback that they can only cover a limited and simplified set of environmental conditions. The field site of this study offered the unique possibility to study the chosen automated chamber systems in a wide range of environmental conditions within a short period of time. The following sections address real alterations of the field SR introduced by the presence of the chamber systems, measurement artefacts caused by environmental conditions that affect chamber performance and biases introduced by the subsequent data analysis.

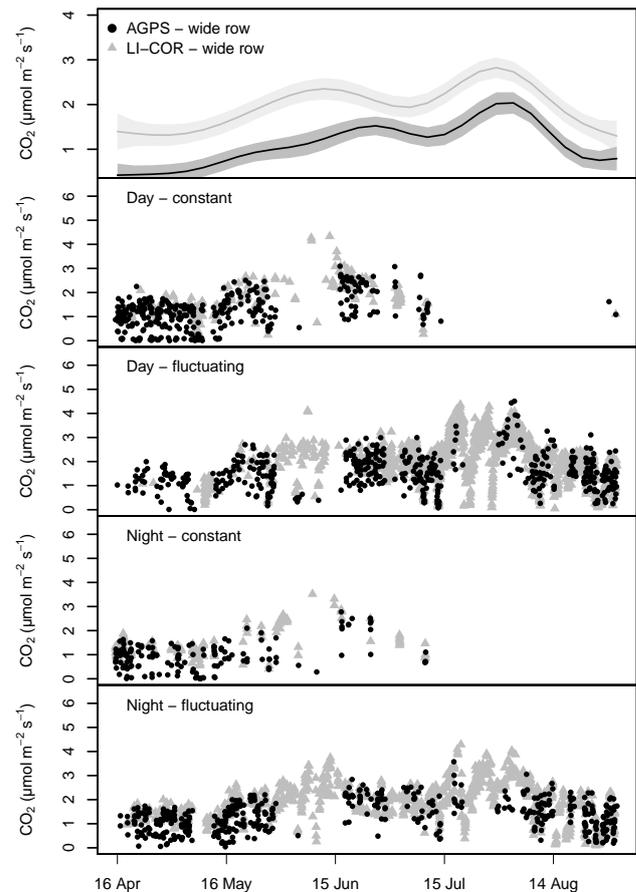
### 4.1 Effect of chambers on environmental conditions

The impact of the automated chamber systems on the vegetation structure increased with the size of the chamber itself and additionally with the size of the frame needed for the support and the movement of the chamber. To guarantee unrestricted movement and airtight closure of automated chambers, the support structure has to be kept free of vegetation. Additionally, the height of the chambers restricts the height to which vegetation can be allowed to grow inside the collars. At the poplar plantation, this subsequently altered the environmental conditions for each chamber system in two ways. First, the smaller LI-8100A was able to cover a wider range of environmental conditions since it could also be installed in the narrow rows. Including the narrow rows increased the



**Figure 4.** Unfiltered chamber CO<sub>2</sub> flux data sets for the entire monitoring period (wide rows only). The top panel shows the average daily CO<sub>2</sub> flux and its respective 95 % confidence interval for each chamber system estimated using generalized additive models (GAM) (deviance explained: AGPS 64.9 %,  $n = 896$ ; LI-8100A 58.6 %,  $n = 527$ ). In the other panels, the single measured CO<sub>2</sub> fluxes over time were grouped by time of the day and stability of the atmospheric CO<sub>2</sub> concentration at 50 cm above the soil surface. The data sets were divided into day and night based on sunrise and sunset times. Atmospheric CO<sub>2</sub> concentration was considered as constant when the standard deviation for a 3 min measurement prior to the chamber closures was  $\leq 1.0$  ppm. The AGPS CO<sub>2</sub> fluxes were calculated from the first 4 min of the closure time (including 1 min deadband).

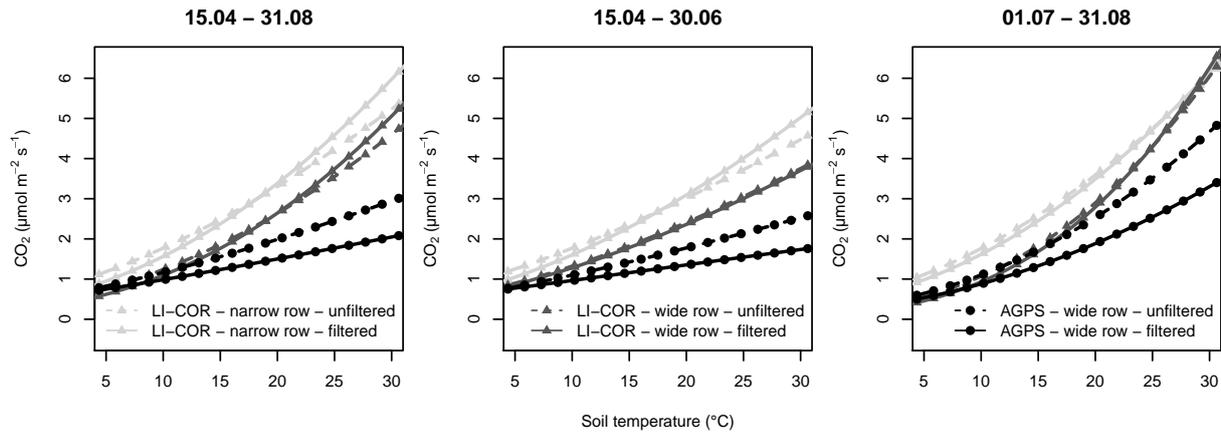
overall SR balance of the site obtained by the LI-8100A flux measurements by about 20 %. Soil respiration at this site was higher in the narrow rows as compared to the wide rows due to the higher fine root biomass and better aeration (Verlinden et al., 2013). Second, the larger size of the AGPS chambers required more weeding, and it also prevented the resprouting poplar stems to lean towards each other early in the growing season, thus slightly delaying canopy closure. The resulting reduced shading made a larger proportion of the measurement plot susceptible to soil heating and drying, but also precipitation events had a more immediate effect on



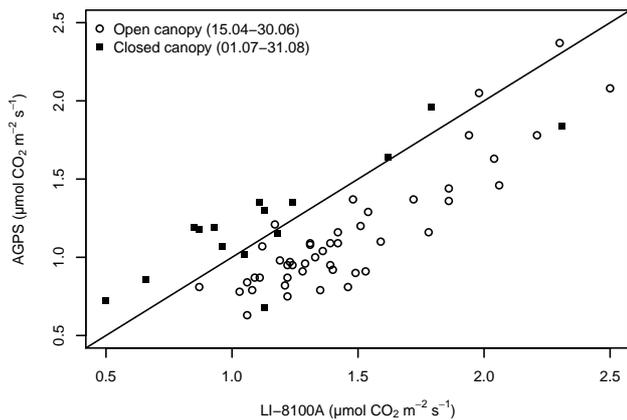
**Figure 5.** Filtered chamber CO<sub>2</sub> flux data sets for the entire monitoring period (wide rows only). Modelling and grouping of the data is the same as in Fig. 4 (deviance explained for the GAM: AGPS 71.1 %,  $n = 582$ ; LI-8100A 57.0 %,  $n = 526$ ).

the soil surface since less precipitation was intercepted by the vegetation canopy (lower leaf area index) in comparison to the LI-8100A. Biological processes in the soil have temperature and moisture optima (Schipper et al., 2014; Zhou et al., 2014). Thus, differences in temperature and moisture could have led either to lower or higher SR fluxes from the AGPS in comparison to the LI-8100A depending on the time of the measurement. However, it is not possible to resolve these differences in more detail in this study.

Potential alterations of environmental conditions due to the presence of automated chamber systems are not restricted to the aboveground part of the ecosystem. A problem shared by manual and automated chambers is the effect of the collar presence on soil conditions. During installation, collars can cut roots and disturb the soil structure, leading to significant alterations of SR fluxes. The risk of altering SR dynamics increases with collar insertion depth. However, the common consensus in the literature is that these disturbances are only temporary and can be largely overcome by installing the collars long before the actual start of the chamber measurements



**Figure 6.** The Lloyd and Taylor model fitted with the different CO<sub>2</sub> flux data sets for the entire monitoring period (15 April–31 August 2014), and separately for the open and closed canopy phase (15 April–30 June 2014 and 1 July–31 August 2014 respectively) using soil temperature at 5 cm depth.



**Figure 7.** Direct comparison of average CO<sub>2</sub> fluxes obtained with the automated chamber systems LI-8100A and AGPS. AGPS fluxes were averaged for each complete measurement cycle which consisted of eight chambers run in sequence within a 4 h window. Only those 4 h windows were included in the figure where at least five of the eight chambers passed the quality control protocol. Filtered LI-8100A fluxes were averaged for the matching 4 h windows ( $n = 4$ –8). Standards errors varied between 0.08 and 0.37  $\mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$  for the AGPS and between 0.03 and 0.34  $\mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$  for the LI-8100A respectively.

(Hutchinson and Livingston, 2001; Davidson et al., 2002; de Klein and Harvey, 2012). The current recommendation for minimising disturbance of environmental conditions by the presence of automated chambers is to have at least two collars per replicate plot and to move the chambers regularly between the collars. It does not include the regular relocation of the collars itself (de Klein and Harvey, 2012). To our knowledge we are the first to report the restriction of horizontal root growth by collars and the subsequent build-up of root mats along the interior collar walls. Root respiration is an im-

portant component of the total SR flux (Vargas et al., 2011; Heinemeyer et al., 2011). The development of root mats only in the LI-8100A collars, which had a deeper insertion depth than the AGPS collars, might have contributed to the higher SR observed in the wide rows by the LI-8100A in comparison to the AGPS. The small size of the LI-8100A chambers allows the system to cover a wider range of microsites in the field and it makes it easy to relocate the chambers. However, the contribution of any type of collar edge effect to the total SR flux increases with an increasing collar perimeter to collar area ratio and is thus more of a problem for smaller chambers.

#### 4.2 Effect of environmental conditions on chamber performance

Collars have the purpose of providing an airtight system during chamber measurements by (i) offering a smooth contact surface for the chamber to rest on which can be sealed using either rubber or water seals and (ii) preventing lateral soil gas diffusion and thus leakages in the soil during chamber deployment (Hutchinson and Livingston, 2001). Chamber leakages can lead to negligible or significant flux underestimation depending on the environmental conditions and soil properties (Hutchinson and Livingston, 2001). For example, the collar insertion depth necessary to reduce the error due to lateral soil gas diffusion increases with increasing air-filled porosity (Hutchinson and Livingston, 2001; Heinemeyer and McNamara, 2011; Creelman et al., 2013). The increase in soil CO<sub>2</sub> concentrations during the closed canopy phase in the poplar plantation, which was not accompanied by a change of magnitude in the chamber CO<sub>2</sub> fluxes, and the unrealistic SR estimates obtained with the soil gradient flux method during this period were indicative of a significant decrease in air-filled porosity and thus diffusivity (Turcu et al., 2005; Hashimoto and Komatsu, 2006). The application of the

flux gradient method has been shown to be problematic in soils which are near water saturation because of the difficulties estimating low soil diffusion coefficients with high certainty (Maier and Schack-Kirchner, 2014). The AGPS was more prone to lateral diffusive soil gas losses than the LI-8100A due to its shallower collar insertion depth. Thus, lateral soil gas diffusion losses likely played a significant role in the larger discrepancy observed in the SR estimates between the two automated chamber systems during the open canopy phase (before coppice) in comparison to the closed canopy phase with its higher soil moisture conditions.

Flux underestimation caused by leakages in the above-ground seal was certainly also an issue for the AGPS as could be seen from the high maintenance needs necessary to keep the seal properly aligned to the collar and the large noise in the data set. The LI-8100A flux data set had a very low noise level regardless of the wide range of environmental conditions encountered at the poplar plantation, especially regardless of the wind protection. This is a good indicator that the chambers had no issue with the airtightness of the rubber sealing. Under windy conditions, one can expect to see more noise in the CO<sub>2</sub> concentration curves obtained during chamber closure if the chamber seal is not perfectly airtight (Bain et al., 2005). However, the AGPS data set had a high noise level throughout the entire inter-comparison campaign, and it was highest during calm conditions. Additionally, high SSN<sub>Lin</sub> values were often associated with higher fluxes ( $> 1.5 \mu\text{mol CO}_2 \text{ m}^{-2} \text{ s}^{-1}$ ). These are indicators that the sample air flow rate between the multiplexer and the chambers was not high enough to sufficiently mix the chamber headspace during the measurements (Liu and Si, 2009; Christiansen et al., 2011). Insufficient headspace mixing leads also to flux underestimation (Liu and Si, 2009; Christiansen et al., 2011).

Chamber design-induced flux estimation errors can be reduced by shortening the chamber closure time (Venterea and Baker, 2008). For the AGPS, the average flux estimate decreased and the noise in the data set increased with increasing closure time. It is a well-known phenomenon that even a perfectly designed non-steady-state chamber will show increasing flux underestimation with increasing closure time due to the chamber's feedback on the soil gas concentration profile if it is not corrected for in the flux calculation (Creelman et al., 2013). Regarding the increasing noise level, Koskinen et al. (2014) reported for their automated chamber system that the SR flux curves became erratic in several cases after a closure time of more than 300 s; this might have been related to wind gusts or CO<sub>2</sub> saturation effects.

### 4.3 Effect of data processing on flux rates

Based on Fick's first law of diffusion, the GHG flux rate should decline with increasing chamber deployment time due to a decreasing diffusion gradient between the air-filled soil pore space and the chamber headspace (Davidson et al.,

2002). Thus, theoretically, gas concentration curves obtained by non-steady-state chambers are always nonlinear. However, whether nonlinearity can be detected with sufficient statistical significance depends on the length of the measurement time, the number of sampling points during the measurement and the precision of the gas concentration measurement (Kutzbach et al., 2007; Pedersen et al., 2010). As a consequence of the high noise in the AGPS flux data set, the majority of the flux measurements were best-fitted linearly whereas most of the LI-8100A fluxes were best-fitted nonlinearly. However, the use of a linear fit may result in a significant underestimation of the flux by at least a few percent in most soils (Davidson et al., 2002; Kutzbach et al., 2007; Pedersen et al., 2010). Creelman et al. (2013) have demonstrated in a model simulation that an exponential fit yields much better results over a wide range of soil types and air diffusivities. A linear fit only seems to be suitable for soils with a low diffusivity or for measurements with a closure time of less than 3 min (Jassal et al., 2012; Creelman et al., 2013). This evidence suggests again that the discrepancies observed between the AGPS and the LI-8100A flux data set are to a large extent caused by CO<sub>2</sub> flux underestimation of the AGPS.

Over the last years, several advanced nonlinear flux models based on diffusion theory have been developed (Kutzbach et al., 2007; Pedersen et al., 2010; Venterea, 2013). The HMR model selected for the AGPS data set accounts for lateral diffusion losses and chamber leakages (Pedersen et al., 2010; Venterea, 2013). Therefore, it appeared to be well suited for the flux calculation since the detection and subsequent discarding of leaky measurements still present the biggest challenge for the processing of automated chamber data sets. In a study on N<sub>2</sub>O fluxes, the HMR-based flux estimates were indeed less sensitive to chamber leakages and lateral diffusion than other advanced nonlinear flux models, but the model also constantly showed the highest flux underestimation across a wide range of environmental conditions (Venterea, 2013). However, the study was based on model simulations with only five sampling points during the chamber closure time. It still has to be tested whether this underestimation also prevails when fitting the HMR model with high temporal frequency data. A general problem of all nonlinear models is that they are very sensitive to noise at the beginning of the chamber deployment time resulting either in flux over- or underestimation. To avoid this problem, the dead-band has been introduced in the flux calculation procedure, but this initial data discarding leads to inherent flux underestimation (Kutzbach et al., 2007; Forbrich et al., 2010). Thus, flux biases due to the flux calculation method cannot be ruled out for both automated chamber systems, but these biases can only be accurately quantified under laboratory conditions (Pihlatie et al., 2013) or with advanced model simulations (Creelman et al., 2013).

Besides the flux calculation, the other important data processing step is the flux quality control. Currently, there are

no standardised procedures available for checking the quality of chamber flux data sets like they are in the eddy covariance community (Vargas et al., 2011). The challenge is to find criteria which are able to identify faulty measurements of different origins but at the same time not to discard large amounts of good flux measurements as well. The root-mean-square error seems (RMSE) to be such a promising criterion (Christiansen et al., 2011; Jassal et al., 2012; Görres et al., 2014). Since the susceptibility of the chamber methodology to certain measurement artefacts changes with environmental conditions, any filtering can lead to a bias in the temporal resolution of the flux data set and thus change the conclusions of field measurements. In the present study, this was very obvious for the AGPS data set. For the LI-8100A, the filtering also introduced a temporal bias since the chambers were most susceptible to headspace temperature changes and thus most of the data were discarded during the open canopy phase. However, the amount of data collectible with the LI-8100A was so high that even a discard of a quarter of the data did not alter the modelled SR balance significantly. Comparing the unfiltered and filtered data set should be the last step of any flux quality control protocol as it can not only give valuable insights into the performance of the chambers and potential measurement artefacts but also offer a way to check the quality of the filter protocol itself.

#### 4.4 Nighttime chamber measurements

Obtaining reliable nighttime SR fluxes is one of the biggest remaining methodological challenges. During nighttime, atmospheric turbulences tend to calm down. Consequently, CO<sub>2</sub> diffusing out of the soil is not transported away anymore from the emission site, but rather it starts to accumulate on the soil surface leading to a very steep CO<sub>2</sub> gradient between 0 and 100 cm above the soil surface (Schneider et al., 2009; Lai et al., 2012). However, the accumulation of CO<sub>2</sub> on the soil surface leads to a decreasing CO<sub>2</sub> gradient between the soil pore space and the atmosphere, and thus a decreasing diffusive flux. In case of manual chamber measurements, any atmospheric layering is already inevitably disturbed by the presence of the chamber operator and subsequently by the chamber deployment itself. This leads first to a flush of CO<sub>2</sub> into the chamber during chamber placement, when the CO<sub>2</sub> layer directly above the soil surface is broken up, and second to an increasing soil–atmosphere CO<sub>2</sub> gradient (Schneider et al., 2009; Lai et al., 2012; Koskinen et al., 2014). Both effects result in a flux overestimation, and Schneider et al. (2009) have questioned whether it is at all possible to obtain reliable nighttime fluxes with chambers under calm conditions.

This is a serious problem since nighttime chamber measurements have been used to assess the measurement bias of the eddy covariance method which systematically underestimates CO<sub>2</sub> fluxes during calm night conditions (Baldocchi, 2003; Schneider et al., 2009). Solutions to obtain unbiased nighttime flux estimates have focused thus far on the

chamber deployment time, empirical methods to correct biased flux measurements or the use of daytime respiration data instead (Schneider et al., 2009; Lai et al., 2012; Koskinen et al., 2014). We argue that automated chambers have the potential to provide reliable nighttime flux data sets if they fulfil certain design criteria regarding chamber height, direction of chamber movement, chamber closing speed and sample inlet position. The combination of a low chamber height (< 20 cm) and a mainly horizontal movement of the chamber from its parking position to the collar increases the probability that the chamber stays within a stable atmospheric layer which has no steep vertical CO<sub>2</sub> gradient. A gentle chamber movement during the closing procedure reduces the risk of breaking up that stable atmospheric layer and mixing it with overlaying atmospheric layers which have lower CO<sub>2</sub> concentrations. Regarding the position of the sample inlet, the AGPS is to our knowledge the only automated chamber system which has the inlet inside the collar instead of the chamber. This offered the unique opportunity to measure the undisturbed atmospheric CO<sub>2</sub> concentration 5 cm above the soil surface before the chamber moved over the collar. About 17 % of the AGPS measurements showed a decrease in the CO<sub>2</sub> headspace concentration during the 1 min dead-band. The open AGPS chamber which was not flushed before the closure and was parked about 10 cm above the soil surface probably had a lower CO<sub>2</sub> concentration inside than the atmospheric layer less than 10 cm above the soil surface. Closing the chamber and starting the sample air flow broke up the atmospheric layering, mixed the two air layers and led to a dilution of the CO<sub>2</sub> headspace concentration. This dilution is equivalent to the initial CO<sub>2</sub> flush into the chamber observed by Koskinen et al. (2014) who measured the pre-closure CO<sub>2</sub> concentration inside the chamber. Thus, the unique design of the AGPS offers the possibility to directly detect for each measurement plot artificial increases in the soil–atmosphere CO<sub>2</sub> gradient in calm nights and filter out obviously disturbed flux measurements. Moreover, the AGPS measurements have shown that this chamber artefact is indeed mainly a nighttime problem, but it might also affect part of the daytime flux measurements.

The design of the LI-8100A chambers with the sample inlet and outlet positioned inside the chamber did not allow to detect any dilution of the atmospheric CO<sub>2</sub> concentration because no undisturbed pre-closure CO<sub>2</sub> concentration measurement directly above the collar was available. The chamber headspace was already mixed before the chamber closure. It is therefore not possible to say whether part of the LI-8100A nighttime measurements at high ambient CO<sub>2</sub> concentrations have been overestimated.

## 5 Recommendations for automation of soil respiration measurements

The closed dynamic chamber method is an invasive method and biases in soil GHG flux estimates can be introduced by environmental alterations due to the presence of the chamber and alterations of the chamber performance due to changes in environmental conditions, as well as the data processing. Environmental alterations due to the presence of the chamber are a serious concern for automated chamber systems, with the probability of environmental alterations increasing with the size of the chamber structure. It is therefore recommended to regularly move automated chambers between different permanently installed collars to prevent any significant chamber-induced changes for example in soil moisture. However, depending on the ecosystem and the collar insertion depth, this recommendation should not be limited to the aboveground part of the chamber. We showed that collars can restrict horizontal root growth leading to the formation of roots mat along the inside collar walls and thus potentially to artificially increased SR.

Selecting the most appropriate collar insertion depth for a specific study site is always a trade-off between reducing the collar impact on the root system and preventing lateral soil gas diffusion during measurements. Lateral soil gas diffusion due to insufficient collar insertion depth is one of the major causes of significant soil GHG flux underestimation and also one of the processes most affected by changes in environmental conditions, as it increases with increasing air-filled soil pore space. Flux underestimation due to leakages in the chamber system can be reduced by shortening the chamber deployment time and by choosing the appropriate flux calculation model. Shortening the deployment time is no longer a problem with the available field deployable gas analysers which are able to measure at 1 Hz frequency. Regarding the flux calculation, several advanced nonlinear flux calculation models have been developed in recent years, but none of them seem to be able to fully correct flux estimates for leakages. However, the models have mainly been tested against data from manual chambers with only few sample points per measurement.

Accounting for leakages and other measurement artefacts in unsupervised operating automated chamber systems is still a big challenge. Currently, no standardised protocols exist for checking the quality of automated chamber flux data sets. We propose including a comparison of the unfiltered and filtered data set in any flux quality control protocol. Such a comparison can give valuable insights into the performance of automated chamber systems under different environmental conditions and reveal chamber-induced measurement artefacts, but it also offers a way to check the quality of the filter protocol itself. Based on the design of one of the automated chamber systems which had the sample inlet inside the collar instead of the chamber, we included a filter criterion based on the headspace CO<sub>2</sub> concentration change during the deadband

period. The combination of this unique chamber design feature and the filter criterion offered the possibility to detect disturbed chamber measurements during nights with a stratified atmosphere. Obtaining unbiased nighttime respiration measurements is a major challenge and has not been resolved yet. We showed for the first time that automated chamber systems have the potential to solve this issue if certain design criteria are considered. Thus, besides providing high temporal frequency flux data, automated chamber systems would offer another possibility to greatly improve our understanding of soil GHG fluxes.

**The Supplement related to this article is available online at doi:10.5194/bg-13-1949-2016-supplement.**

*Acknowledgements.* The authors thank Nicola Arriga, Joris Cools, Fred Kockelbergh, Kristof Mouton, Jan Segers, UIT, Stefan Vanbeveren and Marc Wellens for their technical assistance at the field site. We also acknowledge Gerald Moser (Justus Liebig University Giessen, Germany) who analysed the soil CO<sub>2</sub> samples, as well as Anne Cools (University of Antwerp, Belgium) for performing the DOC analysis. Funding for this research was provided by ERC Advanced Grant agreement (no. 233366) POPFULL under the EC 7th Framework Programme (FP7/2007–2013), the Flemish Hercules Foundation as Infrastructure contract no. ZW09-06 and the Methusalem Program of the Flemish Government.

Edited by: E. Pendall

## References

- Ambus, P., Skiba, U., Drewer, J., Jones, S. K., Carter, M. S., Albert, K. R., and Sutton, M. A.: Development of an accumulation-based system for cost-effective chamber measurements of inert trace gas fluxes, *Eur. J. Soil Sci.*, 61, 785–792, 2010.
- Bain, W. G., Hutyra, L., Patterson, D. C., Bright, A. V., Daube, B. C., Munger, J. W., and Wofsy, S. C.: Wind-induced error in the measurement of soil respiration using closed dynamic chambers, *Agr. Forest Meteorol.*, 131, 225–232, 2005.
- Baldocchi, D. D.: Assessing the eddy covariance technique for evaluating carbon dioxide exchange rates of ecosystems: past, present and future, *Glob. Change Biol.*, 9, 479–492, 2003.
- Berhongaray, G., Verlinden, M. S., Broeckx, L. S., and Ceulemans, R.: Changes in belowground biomass after coppice in two Populus genotypes, *Forest Ecol. Manag.*, 337, 1–10, 2015.
- Breuer, L., Papen, H., and Butterbach-Bahl, K.: N<sub>2</sub>O emission from tropical forest soils of Australia, *J. Geophys. Res.*, 105, 26353–26367, 2000.
- Broeckx, L. S., Verlinden, M. S., and Ceulemans, R.: Establishment and two-year growth of a bio-energy plantation with fast-growing Populus trees in Flanders (Belgium): Effects of genotype and former land use, *Biomass Bioenerg.*, 42, 151–163, 2012.

- Brummell, M. E. and Siciliano, S. D.: Measurement of carbon dioxide, methane, nitrous oxide, and water potential in soil ecosystems, *Method Enzymol.*, 496, 115–137, 2011.
- Carbone, M. S. and Vargas, R.: Automated soil respiration measurements: new information, opportunities and challenges, *New Phytol.*, 177, 295–297, 2008.
- Christiansen, J., Korhonen, J. J., Juszcak, R., Giebels, M., and Pihlatie, M.: Assessing the effects of chamber placement, manual sampling and headspace mixing on CH<sub>4</sub> fluxes in a laboratory experiment, *Plant Soil*, 343, 171–185, 2011.
- Collier, S. M., Ruark, M. D., Oates, L. G., Jokela, W. E., and Dell, C. J.: Measurement of greenhouse gas flux from agricultural soils using static chambers, *J. Vis. Exp.*, 90, e52110, doi:10.3791/52110, 2014.
- Creelman, C., Nickerson, N., and Risk, D.: Quantifying lateral diffusion error in soil carbon dioxide respiration estimates using numerical modeling, *Soil Sci. Soc. Am. J.*, 77, 699–708, 2013.
- Davidson, E. A., Savage, K., Verchot, L. V., and Navarro, R.: Minimizing artifacts and biases in chamber-based measurements of soil respiration, *Agr. Forest Meteorol.*, 113, 21–37, 2002.
- de Klein, C. and Harvey, M.: Nitrous oxide chamber methodology guidelines, Ministry for Primary Industries, New Zealand, available at: [http://globalresearchalliance.org/wp-content/uploads/2015/11/Chamber\\_Methodology\\_Guidelines\\_Final-V1.1-2015.pdf](http://globalresearchalliance.org/wp-content/uploads/2015/11/Chamber_Methodology_Guidelines_Final-V1.1-2015.pdf) (last access: 20 March 2016), 2012.
- Denmead, O. T.: Chamber systems for measuring nitrous oxide emission from soils in the field, *Soil Sci. Soc. Am. J.*, 43, 89–95, 1979.
- FAO: Agriculture, forestry and other land use emissions by sources and removals by sinks: 1990–2011 analysis. FAO Statistics Division Working Paper Series, Rome, Italy, 2014.
- Forbrich, I., Kutzbach, L., Hormann, A., and Wilmking, M.: A comparison of linear and exponential regression for estimating diffusive CH<sub>4</sub> fluxes by closed-chambers in peatlands, *Soil Biol. Biochem.*, 42, 507–515, 2010.
- Giltrap, D. L., Li, C., and Saggart, S.: DNDC: A process-based model of greenhouse gas fluxes from agricultural soils, *Agr. Ecosyst. Environ.*, 136, 292–300, 2010.
- Görres, C.-M., Kutzbach, L., and Elsgaard, L.: Comparative modeling of annual CO<sub>2</sub> flux of temperate peat soils under permanent grassland management, *Agr. Ecosyst. Environ.*, 186, 64–76, 2014.
- Hashimoto, S. and Komatsu, H.: Relationships between soil CO<sub>2</sub> concentration and CO<sub>2</sub> production, temperature, water content, and gas diffusivity: implications for field studies through sensitivity analyses, *J. For. Res.-Jpn.*, 11, 41–50, 2006.
- Heinemeyer, A., Di Bene, C., Lloyd, A. R., Tortorella, D., Baxter, R., Huntley, B., Gelsomino, A., and Ineson, P.: Soil respiration: implications of the plant-soil continuum and respiration chamber collar-insertion depth on measurement and modelling of soil CO<sub>2</sub> efflux rates in three ecosystems, *Eur. J. Soil Sci.*, 62, 82–94, 2011.
- Heinemeyer, A. and McNamara, N. P.: Comparing the closed static versus the closed dynamic chamber flux methodology: Implications for soil respiration studies, *Plant Soil*, 346, 145–151, 2011.
- Hopkins, F., Gonzalez-Meler, M. A., Flower, C. E., Lynch, D. J., Czimczik, C., Tang, J., and Subke, J.-A.: Ecosystem-level controls on root-rhizosphere respiration, *New Phytol.*, 199, 339–351, 2013.
- Huber, P.: *Robust Statistics*, Wiley, New York, USA, 1981.
- 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, 2001.
- Janssens, I. A., Kowalski, A. S., Longdoz, B., and Ceulemans, R.: Assessing forest soil CO<sub>2</sub> efflux: an in situ comparison of four techniques, *Tree Physiol.*, 20, 23–32, 2000.
- Jassal, R., Black, A., Novak, M., Morgenstern, K., Nestic, Z., and Gaumont-Guay, D.: Relationship between soil CO<sub>2</sub> concentrations and forest-floor CO<sub>2</sub> effluxes, *Agr. Forest Meteorol.*, 130, 176–192, 2005.
- Jassal, R. S., Black, T. A., Nestic, Z., and Gaumont-Guay, D.: Using automated non-steady-state chamber systems for making continuous long-term measurements of soil CO<sub>2</sub> efflux in forest ecosystems, *Agr. Forest Meteorol.*, 161, 57–65, 2012.
- Jones, H. G.: *Plants and microclimate: A quantitative approach to environmental plant physiology*, Cambridge University Press, Cambridge, UK, 1992.
- Kitzler, B., Zechmeister-Boltenstern, S., Holtermann, C., Skiba, U., and Butterbach-Bahl, K.: Nitrogen oxides emission from two beech forests subjected to different nitrogen loads, *Biogeosciences*, 3, 293–310, doi:10.5194/bg-3-293-2006, 2006.
- Koskinen, M., Minkkinen, K., Ojanen, P., Kämäräinen, M., Laurila, T., and Lohila, A.: Measurements of CO<sub>2</sub> exchange with an automated chamber system throughout the year: challenges in measuring night-time respiration on porous peat soil, *Biogeosciences*, 11, 347–363, doi:10.5194/bg-11-347-2014, 2014.
- Kutzbach, L., Schneider, J., Sachs, T., Giebels, M., Nykänen, H., Shurpali, N. J., Martikainen, P. J., Alm, J., and Wilmking, M.: CO<sub>2</sub> flux determination by closed-chamber methods can be seriously biased by inappropriate application of linear regression, *Biogeosciences*, 4, 1005–1025, doi:10.5194/bg-4-1005-2007, 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 CO<sub>2</sub> and CH<sub>4</sub> flux measurements in an ombrotrophic peatland, *Biogeosciences*, 9, 3305–3322, doi: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, 2011.
- LI-COR Biosciences: LI-8100A Automated Soil CO<sub>2</sub> Flux System & LI-8150 Multiplexer Instruction Manual, available at: [http://envsupport.licor.com/docs/LI-8100A\\_Manual.pdf](http://envsupport.licor.com/docs/LI-8100A_Manual.pdf) (last access: 20 March 2016), 2010.
- Liu, G. and Si, B. C.: Multi-layer diffusion model and error analysis applied to chamber-based gas fluxes measurements, *Agr. Forest Meteorol.*, 149, 169–178, 2009.
- Lloyd, J. and Taylor, J. A.: On the temperature dependence of soil respiration, *Funct. Ecol.*, 8, 315–323, 1994.
- Maier, M. and Schack-Kirchner, H.: Using the gradient method to determine soil gas flux: A review, *Agr. Forest Meteorol.*, 192–193, 78–95, 2014.
- Millington, R. J. and Quirk, J. P.: Permeability of porous solids, *Trans. Faraday Soc.*, 57, 1200–1207, 1961.
- Olander, L. P., Wollenberg, E., Tubiello, F. N., and Herold, M.: *Synthesis and Review: Advancing agricultural greenhouse gas*

- quantification, *Environ. Res. Lett.*, 9, 75003, doi:10.1088/1748-9326/9/7/075003, 2014.
- Parkin, T. and Venterea, R.: Chapter 3. Chamber-based trace gas flux measurements, in: *Sampling Protocols*, edited by: Follet, R., available at: [www.ars.usda.gov/research/GRACEnet](http://www.ars.usda.gov/research/GRACEnet) (last access: 20 March 2016), 3-1 to 3-39, 2010.
- Pedersen, A. R., Petersen, S. O., and Schelde, K.: A comprehensive approach to soil-atmosphere trace-gas flux estimation with static chambers, *Eur. J. Soil Sci.*, 61, 888–902, 2010.
- Peltola, O., Mammarella, I., Haapanala, S., Burba, G., and Vesala, T.: Field intercomparison of four methane gas analyzers suitable for eddy covariance flux measurements, *Biogeosciences*, 10, 3749–3765, doi:10.5194/bg-10-3749-2013, 2013.
- Petersen, S.O.: Diffusion probe for gas sampling in undisturbed soil, *Eur. J. Soil Sci.*, 65, 663–671, 2014.
- Phillips, R., Griffith, D. W., Dijkstra, F., Lugg, G., Lawrie, R., and Macdonald, B.: Tracking short-term effects of nitrogen-15 addition on nitrous oxide fluxes using fourier-transform infrared spectroscopy, *J. Environ. Qual.*, 42, 1327–1340, 2013.
- 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., Serça, D., Vicca, S., Wolf, B., and Pumpanen, J.: Comparison of static chambers to measure CH<sub>4</sub> emissions from soils, *Agr. Forest Meteorol.*, 171–172, 124–136, 2013.
- Pumpanen, J., Kolari, P., Ilvesniemi, H., Minkkinen, K., Vesala, T., Niinistö, S., Lohila, A., Larmola, T., Morero, M., Pihlatie, M., Janssens, I., Yuste, J. C., Grünzweig, J. M., Reth, S., Subke, J.-A., Savage, K., Kutsch, W., Østreng, G., Ziegler, W., Anthoni, P., Lindroth, A., and Hari, P.: Comparison of different chamber techniques for measuring soil CO<sub>2</sub> efflux, *Agr. Forest Meteorol.*, 123, 159–176, 2004.
- Qu, W., Bogen, H. R., Huisman, J. A., and Vereecken, H.: Calibration of a novel low-cost soil water content sensor based on a ring oscillator, *Vadose Zone J.*, 12, 1–10, 2013.
- R Core Team: R: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria, 2014.
- Roland, M., Vicca, S., Bahn, M., Ladreiter-Knauss, T., Schmitt, M., and Janssens, I. A.: Importance of nondiffusive transport for soil CO<sub>2</sub> efflux in a temperate mountain grassland, *J. Geophys. Res.-Biogeo.*, 120, 502–512, 2015.
- Rühlmann, J., Körschens, M., and Graefe, J.: A new approach to calculate the particle density of soils considering properties of the soil organic matter and the mineral matrix, *Geoderma*, 130, 272–283, 2006.
- Ryan, J. A. and Ulrich, J. M.: xts: eXtensible Time Series: R package version 0.9-7, available at: <http://CRAN.R-project.org/package=xts> (last access: 20 March 2016), 2014.
- Savage, K., Phillips, R., and Davidson, E.: High temporal frequency measurements of greenhouse gas emissions from soils, *Biogeosciences*, 11, 2709–2720, doi:10.5194/bg-11-2709-2014, 2014.
- Schipper, L. A., Hobbs, J. K., Rutledge, S., and Arcus, V. L.: Thermodynamic theory explains the temperature optima of soil microbial processes and high Q<sub>10</sub> values at low temperatures, *Glob. Change Biol.*, 20, 3578–3586, 2014.
- Schneider, J., Kutzbach, L., Schulz, S., and Wilmking, M.: Overestimation of CO<sub>2</sub> respiration fluxes by the closed chamber method in low-turbulence nighttime conditions, *J. Geophys. Res.*, 114, G03005, doi:10.1029/2008JG000909, 2009.
- Turcu, V. E., Jones, S. B., and Or, D.: Continuous soil carbon dioxide and oxygen measurements and estimation of gradient-based gaseous flux, *Vadose Zone J.*, 4, 1161–1169, 2005.
- van Rij, J., Wieling, M., Baayen, R., and van Rijn, H.: itsadug: Interpreting Time Series and Autocorrelated Data Using GAMMs, R package version 1.0.1, 2015.
- Vargas, R., Carbone, M., Reichstein, M., and Baldocchi, D.: Frontiers and challenges in soil respiration research: from measurements to model-data integration, *Biogeochemistry*, 102, 1–13, 2011.
- Venterea, R. T.: Theoretical comparison of advanced methods for calculating nitrous oxide fluxes using non-steady state chambers, *Soil Sci. Soc. Am. J.*, 77, 709–720, 2013.
- 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, 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, 2008.
- Verlinden, M. S., Broeckx, L. S., Wei, H., and Ceulemans, R.: Soil CO<sub>2</sub> efflux in a bioenergy plantation with fast-growing *Populus* trees – influence of former land use, inter-row spacing and genotype, *Plant Soil*, 369, 631–644, 2013.
- Wang, K., Liu, C., Zheng, X., Pihlatie, M., Li, B., Haapanala, S., Vesala, T., Liu, H., Wang, Y., Liu, G., and Hu, F.: Comparison between eddy covariance and automatic chamber techniques for measuring net ecosystem exchange of carbon dioxide in cotton and wheat fields, *Biogeosciences*, 10, 6865–6877, doi:10.5194/bg-10-6865-2013, 2013.
- Weylandt, M. R.: xtsExtra: Supplementary Functionality for xts: R package version 0.0-1/r824, available at: <http://R-Forge.R-project.org/projects/xts/> (last access: 20 March 2016), 2014.
- Wood, S. N.: *Generalized Additive Models: An Introduction with R*, Chapman and Hall/CRC, Boca Raton, FL, USA, 2006.
- Xu, L., Furtaw, M. D., Madsen, R. A., Garcia, R. L., Anderson, D. J., and McDermitt, D. K.: On maintaining pressure equilibrium between a soil CO<sub>2</sub> flux chamber and the ambient air, *J. Geophys. Res.*, 111, D08S10, doi:10.1029/2005JD006435, 2006.
- Zeileis, A. and Grothendieck, G.: zoo: S3 infrastructure for regular and irregular time series, *J. Stat. Softw.*, 14, 1–27, 2005.
- Zenone, T., Zona, D., Gelfand, I., Gielen, B., Camino-Serrano, M., and Ceulemans, R.: CO<sub>2</sub> uptake is offset by CH<sub>4</sub> and N<sub>2</sub>O emissions in a poplar short rotation coppice, *Glob. Change Biol. Bioenergy*, doi:10.1111/gcbb.12269, 2015.
- Zhou, W., Hui, D., and Shen, W.: Effects of soil moisture on the temperature sensitivity of soil heterotrophic respiration: a laboratory incubation study, *PLoS ONE*, 9, e92531, doi:10.1371/journal.pone.0092531, 2014.