Automation of soil flux chamber measurements: potentials and pitfalls

C.-M. Görres¹, C. Kammann², and R. Ceulemans¹

¹Center of Excellence PLECO (Plant and Vegetation Ecology), University of Antwerp, Universiteitsplein 1, 2610 Wilrijk, Belgium
²WG Global Change Research in Special Crops, Department of Soil Science and Plant Nutrition, Hochschule Geisenheim University, Von-Lade-Str. 1, 65366 Geisenheim, Germany

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Correspondence to: C.-M. Görres (carolyn.goerres@uantwerpen.be)

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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, only few studies addressed the difficulties and challenges associated with operating these systems. In this contribution we compared two commercial soil GHG chamber systems—the LI-8100A Automated Soil CO$_2$ Flux System and the Greenhouse Gas Monitoring System AGPS. From April 2014 until August 2014, the two systems monitored in parallel soil respiration (SR) fluxes at a recently harvested poplar 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 datasets of the LI-8100A and the AGPS were 520 and 433 g CO$_2$ m$^{-2}$, respectively. For the closed canopy phase (01 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$_2$ m$^{-2}$ 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$_2$ diffusion below the collars during AGPS chamber measurements; (iv) increased root growth within the LI-8100A collars; and (v) a possible overestimation of nighttime SR fluxes by the LI-8100A. 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 has been obtained using manually operated closed static chambers (Pumpanen et al., 2004; Levy et al., 2011). These chambers are placed air-tight on a small soil area (typically $< 1 \text{ m}^2$ and $< 1 \text{ h}$) 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 large 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 of short-term responses of soil GHG flux dynamics to perturbations such as rain events, irrigation and fertilization, 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 deals with soil CO$_2$ fluxes. The latter is mainly due to a lack of available field gas analysers for CH$_4$ and N$_2$O in the past (Venterea et al., 2008; Savage et al., 2014). The requirements for a larger infrastructure and for in-
tensive 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 CO₂ Flux System and the Automated Gas Sampling System AGPS. 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 au-
tomated custom-made chamber system with continuous gas analysis in the field. In parallel, both systems were monitoring soil respiration 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, CO₂ concentrations were monitored in the topsoil to give insights into the potential range of soil CO₂ 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 soil respiration or soil CO₂ efflux at the poplar plantation per se since these have already been discussed amongst others by Verlinden et al. (2013) and Zenone et al. (2015). The results presented here serve the comparison of the performance of the two chamber systems in quantifying soil respiration 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 lay-out 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 inter-comparison 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, UIT Umwelt- und Ingenieurtechnik GmbH 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 wide rows. A detailed description of each soil GHG sampling device is presented in Sects. 2.2 and 2.3 below. The inter-comparison campaign took place from 15 April 2014 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 Greenhouse Gas Monitoring System AGPS (UIT Umwelt- und Ingenieurtechnik GmbH Dresden, Germany) and the LI-8100A Automated Soil CO$_2$ Flux System (LI-COR Biosciences, Lincoln, NE, USA) 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 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 × 1.21 m wide × 1.55 m high, 20–23°C). Air was circulated in a closed loop between the cham-
bers and the multiplexer with a pump installed inside the multiplexer. For gas concentra-
5 tion 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\(^{-1}\). 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 (Umwelt- und Ingenieurtechnik
GmbH Dresden, Germany), including all the specifications listed in Table 1. Not in-
cluded in the set-up were the gas analysers. Here, we report CO\(_2\) data measured by
10 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 Umwelt- und Ingenieurtechnik GmbH). The computer was also the
access point for remotely controlling the AGPS and the FGGA. The AGPS had con-
15 tinuously 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 removed from inside the collars and around the
20 chambers about every two weeks.

The AGPS sampling protocol consisted of the following steps: (i) 30 min tube heat-
ing, (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, the multiplexer pump auto-
25 matically 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), (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 Automated Soil CO₂ Flux System 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 sub-zero 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 did not rest 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 running 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 CO₂ concentration measurements

Each soil CO₂ 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 × 0.5 mm² 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 CO$_2$ was sampled about every two weeks between 10:00 and 14:00. A plastic syringe containing 10 mL N$_2$ and 50 ppm C$_2$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 extainer (Labco Ltd, Lamber, UK) were connected to the other tube via a three-way valve. The N$_2$ / C$_2$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 extainer. At last, 12 mL N$_2$ were injected into the extainer to create an overpressure needed for the subsequent gas analysis. The concentration of C$_2$H$_4$ recovered in the collected sample was used to calculate the dilution of the original sample which occurred while replacing it with N$_2$ in the sampling cell, and to correct the measured 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 C$_2$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 N$_2$ to remove remaining traces of C$_2$H$_4$. For more details on the sampler design and the C$_2$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 CO$_2$ and C$_2$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 mL min$^{-1}$ was used as reference flow. Total flow was 60 mL min$^{-1}$. Concentrations were quantified with reference to three calibration gases.
with an accuracy of 2%: (i) 50 µL L\(^{-1}\) C\(_2\)H\(_4\) in N\(_2\), (ii) 799 µL L\(^{-1}\) CO\(_2\) in synthetic air, and (iii) 5.04 % 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 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 inter-comparison 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 (\(\sim 2\) cm diameter). Separate samples were taken with stainless steel cylinders (100 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 K\(_2\)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 (CFA) (San++ Automated Wet Chemistry Analyzer, Skalar Analytical, Breda, The Netherlands). The rest of the auger samples were dried at 50°C, ground and three subsamples per sample were analysed by dry combustion with a NC element analyser (NC-2100, Carlo Erba Instruments, Italy) and means 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.
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 were 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 CO₂ 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 measurement— for a closure time of 4 and 9 min, respectively. Prior to each flux calculation, the first minute of the CO₂ 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 “gasfluxes” 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 if (iii) the flux calculated with HMR was not more than four 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 CO₂ concentration curves. Fluxes were converted from µL m⁻² s⁻¹ to a µmol m⁻² 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$_2$ 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 either calculated with a linear or an empirical exponential regression (LI-COR Biosciences, 2010). The software compared for each measurement the normalized 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$_2$ concentration curve were discarded before the flux calculation.

Fluxes were discarded from the two datasets 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$_2$ (equivalent to a root mean square error threshold of 1.0 ppm CO$_2$, 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$_2$ 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$_2$ 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$_2$ flux measurements can be very sensitive to changes in environmental conditions, thus criteria (iii)–(v) removed measurements for which the CO$_2$ 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$_2$ flux calculation

Changes in topsoil CO$_2$ concentration dynamics for each collar and each soil gas sampling plot throughout the inter-comparison campaign were approximated by calculating the effective soil diffusion coefficient ($D_s$) which is the product of the CO$_2$ diffusion coef-
efficient in free air ($D_a$) and the gas tortuosity factor $\xi$. $D_a$ was corrected for temperature and air pressure by

$$D_a = D_{a0} \left( \frac{T}{293.15} \right)^{1.75} \left( \frac{P}{101.3} \right)$$  \hspace{2cm} (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 mm$^2$ s$^{-1}$ (Jones, 1992). The empirical Millington-Quirk model was used for estimating $\xi$ (Millington and Quirk, 1961):

$$\xi = \frac{(\phi - VWC)^{10/3}}{\phi^2}$$  \hspace{2cm} (2)$$

where VWC is the volumetric water content at 5 cm depth and $\phi$ the total porosity (m$^3$ 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$_2$ fluxes were calculated via Fick’s first law of diffusion by multiplying the CO$_2$ 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$_2$ concentrations in ppmv were converted to µmol m$^{-3}$ by multiplying them by the molar volume of a gas at standard temperature and pressure (0.04462 µmol 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$_2$ flux datasets

First, the AGPS and LI-8100A soil CO$_2$ fluxes were descriptively compared for four different environmental conditions, namely (i) daytime, constant atmospheric CO$_2$ concentra-
centration, (ii) daytime, fluctuating atmospheric CO$_2$ concentration, (iii) nighttime, constant atmospheric CO$_2$ concentration, and (iv) nighttime, fluctuating atmospheric CO$_2$ concentration. Daytime and nighttime fluxes were separated based on local sunrise and sunset times. Atmospheric CO$_2$ concentration was considered as constant when the CO$_2$ concentration measured at 50 cm height above the soil surface had a standard deviation < 1.0 ppm (3 min measurements). Constant ambient CO$_2$ concentrations were seen as a proxy indicator of a well-mixed atmosphere, i.e. wind perturbation.

Secondly, the chamber flux datasets were quantitatively compared by using the common approach of modelling soil respiration (SR) according to Lloyd and Taylor (1994) (Eq. 11):

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

where $R_{10}$ is the respiration rate at 10°C ($\mu$mol CO$_2$ m$^{-2}$ 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 fitted once with the unfiltered and the filtered dataset, respectively, using nonlinear regression (R function “nls”). Cumulated CO$_2$ 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 5% 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 ± 0.11 g cm\(^{-3}\) dry soil\(^{-1}\) (average ± STD, \(n = 24\)), the C content 1.21 ± 0.17 % (\(n = 8\), pre-analysis sample pooling), and the DOC content 32.07 ± 10.03 µg g\(^{-1}\) 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 inter-comparison 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, 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 ± 0.07 g cm\(^{-3}\) dry soil\(^{-1}\), \(n = 16\)) differed significantly from the LI-8100A chambers (1.32 ± 0.12 g cm\(^{-3}\) dry soil\(^{-1}\), \(n = 8\)) and the soil gas samplers (1.24 ± 0.13 g cm\(^{-3}\) dry soil\(^{-1}\), \(n = 5\)) installed in the narrow rows. Thus, a methodological comparison of the soil CO\(_2\) 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 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. However, 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.

Variability in air and soil temperature decreased after the canopy closure at the beginning of July, but the opposite was true for the atmospheric CO₂ concentration measured 50 cm above the soil surface. Constant atmospheric CO₂ 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 (= instable atmospheric layering). For more than 70% of the flux measurements, the atmospheric CO₂ concentration 50 cm above the soil surface fluctuated by more than 1.0 ppm prior to the chamber closure. Atmospheric CO₂ 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 CO₂ concentrations at chamber closure above 500 ppm which were not observed by the LI-8100A. Two-thirds of the AGPS CO₂ values above 500 ppm were measured during nighttime.

3.2 Technical reliability of the two chamber systems

During the inter-comparison campaign, the LI-8100A conducted 12874 chamber measurements (wide rows: 6253, narrow rows: 6621) of which only 1 measurement had to be 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 already three 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 theoretically possible 6296 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 two weeks to prevent chambers from getting stuck.

Two main issues prevented the AGPS from operating continuously in the field. First, 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 if the inlets at the collars would have been equipped with air filters the same way as 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 data lost 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 3 min flux calculation period and the 8 min flux calculation period only 1070 (22 %) and 328 (7 %) measurements provided a dataset at 1 Hz frequency, respectively. The median number of data points for the short and long flux calculation 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 CO₂ 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 dataset based on headspace tem-
perature 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_{Lin} > 1.0 ppm or a RH > 100 \% than those in the wide rows. More than 50 \% of the LI-8100A concentration curves were best-fitted exponentially, especially under fluctuating atmospheric CO₂ concentrations.

For the AGPS dataset, a higher proportion of the concentration curves were best-fitted linearly regardless of daytime or atmospheric CO₂ concentration variability. Only for the 4 min closure time in the filtered dataset 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). For the unfiltered dataset, the fluxes calculated for 9 min closure time were 0.21 \pm 0.50 \mu mol m^{-2} s^{-1} (average \pm STD) lower than the fluxes calculated for a 4 min closure time. In the filtered dataset, this was reduced to 0.18 \pm 0.18 \mu mol m^{-2} 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 SSN_{Lin} > 1.0 ppm. This included almost all data from the first half of June where we had the severe gas analyser logging problem. The SSN_{Lin} criterion also already filtered out 60 and 79 \% of the flux measurements with headspace temperature problems and with a decrease of the CO₂ 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 CO₂ concentration as compared to the LI-8100A in Fig. 2e. Measurements which did not pass this criterion had a median pre-closure atmospheric CO₂ concentration at 5 cm height above the collar of 600 ppm, whereas measurements passing this criterion had a median pre-closure atmospheric CO₂ concentration of 433 ppm.

The SSN_{Lin} criterion would have also detected 83 \% of the negative CO₂ fluxes. Negative CO₂ 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 SSN_{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 dataset was large regardless of the environmental conditions, and the noise was lower at constant atmospheric CO\(_2\) concentrations, i.e. windy conditions. All of this pointed to an inherent technical problem with the system (see discussion).

### 3.4 Comparison of the CO\(_2\) flux datasets

Until the beginning of July 2014, CO\(_2\) concentrations at 5 and 15 cm depths in the narrow rows were 4702 ± 762 ppm (average ± SE, \(n = 16\)) and 12 565 ± 2145 ppm (\(n = 15\)), respectively, and in the wide rows 6664 ± 1108 ppm (\(n = 14\)) and 12 251 ± 1512 ppm (\(n = 15\)), respectively. Afterwards, CO\(_2\) concentrations increased at 5 and 15 cm depth in the narrow rows to on average 11 797 ± 2365 ppm (\(n = 20\)) and 27 071 ± 3615 ppm (\(n = 19\)), respectively. In the wide rows, CO\(_2\) at 5 cm depth reached the same concentrations as in the narrow rows, whereas at 15 cm depth, it increased even further (38 008 ± 4574 ppm, \(n = 19\)). The increasing steepness of the soil CO\(_2\) concentration gradient was probably partly the result of CO\(_2\) accumulation in the soil due to the reduced air-filled porosity as the magnitude of the surface CO\(_2\) fluxes measured with the chambers did not increase strongly during this period. Contrastingly, the CO\(_2\) fluxes based on the flux gradient method were unrealistically high in July and August 2014, whereas prior to the rewetting, they were in the same range as the chamber CO\(_2\) fluxes (Fig. 4). The soil depth resolution chosen in this study for the flux gradient method was very likely too low to realistically approximate the soil CO\(_2\) concentration profiles and soil diffusion coefficients during high soil moisture conditions. Short term fluctuations in the soil CO\(_2\) 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$_2$ concentration samplings were performed at least three days after such events – except for the last sampling.

The unfiltered CO$_2$ flux dataset from the AGPS showed higher flux variability throughout the inter-comparison campaign than the unfiltered CO$_2$ flux dataset 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 dataset 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. S2 in the Supplement). Overall, filtering the LI-8100A dataset 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 dataset. The regression parameters based on the AGPS dataset were lower than the ones based on the LI-8100A dataset 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. The SSN$_{Lin}$ and the headspace temperature criteria filtered out fluxes mainly above 1.5 µmol CO$_2$ m$^{-2}$ s$^{-1}$. Only criterion (iv) mainly removed positive fluxes below 1.5 µmol CO$_2$ m$^{-2}$ 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$_2$ concentration.

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 are going to address real alterations of the field SR introduced by the presence of the chamber systems, by measurement artefacts caused by environmental conditions that affect chamber performance, and by biases introduced by the subsequent data analysis.

4.1 Effect of chambers on environmental conditions

The impact of the automated chamber systems on the environmental conditions 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 air-tight 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 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 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). Secondly, 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 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 can 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 (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 rot mats along the interior collar walls. Root respiration is an important component of the total SR flux (Vargas et al., 2011; Heinemeyer et al., 2011). The development of rot 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 to help provide an air-tight 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 by (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$_2$ concentrations during the closed canopy phase in the poplar plantation which was not accompanied by a change of magnitude in the chamber CO$_2$ 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 to estimate 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 aboveground 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 dataset. The
LI-8100A flux dataset 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$_2$ concentration curves obtained during chamber closure if the chamber seal is not perfectly airtight (Bain et al., 2005). However, the AGPS dataset had a high noise level throughout the entire inter-comparison campaign, and it was highest during calm conditions. Additionally, high SSN$_{Lin}$ values were often associated with higher fluxes (>1.5 µmol CO$_2$ m$^{-2}$ 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 dataset 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$_2$ 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 dataset, 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 dataset are to a large extent caused by CO₂ 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 dataset 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 presents the biggest challenge for the processing of automated chamber datasets. In a study on N₂O 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 if 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 deadband has been introduced in the flux calculation proce-
dure, 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 ad-
vanced 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 standardized procedures available for checking
the quality of chamber flux datasets 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 RMSE seems to be such a promising cri-
terion (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 reso-
lution of the flux dataset and thus change the conclusions of field measurements. In
the present study, this was very obvious for the AGPS dataset. 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 dataset should be the last step of
any flux quality control protocol as it can give valuable insights into the performance of
the chambers and potential measurement artefacts, but it also offers 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$_2$ diffusing out of the soil is not transported away anymore from the emission site, but starts to accumulate on the soil surface leading to a very steep CO$_2$ gradient between 0 and 100 cm above the soil surface (Schneider et al., 2009; Lai et al., 2012). However, the accumulation of CO$_2$ on the soil surface leads to a decreasing CO$_2$ 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$_2$ into the chamber during chamber placement when the CO$_2$ layer directly above the soil surface is broken up, and secondly to an increasing soil-atmosphere CO$_2$ 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 if 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$_2$ 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, on empirical methods to correct biased flux measurements or on 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 datasets 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 CO₂ gradient. A gentle chamber movement during the closing procedure reduces the risk of breaking up that stable atmospheric layer and to mix it with overlaying atmospheric layers which have lower CO₂ 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 CO₂ 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 CO₂ headspace concentration during the 1 min deadband. The open AGPS chamber which was not flushed before the closure and which was parked about 10 cm above the soil surface, probably had a lower CO₂ 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 CO₂ headspace concentration. This dilution is equivalent to the initial CO₂ flush into the chamber observed by Koskinen et al. (2014) who measured the pre-closure CO₂ 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 CO₂ 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 CO₂ concentration because no undisturbed pre-closure CO₂ 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 if part of the LI-8100A nighttime measurements at high ambient CO₂ 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, 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 above-ground 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 seems 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 standardized protocols exist for checking the quality of automated chamber flux datasets. We propose to include a comparison of the unfiltered and filtered dataset 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 CO₂ 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 which 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.

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References


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

<table>
<thead>
<tr>
<th></th>
<th>Greenhouse Gas Monitoring System AGPS</th>
<th>LI-8100A Automated Soil CO₂ Flux System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chamber</td>
<td>200 cm L × 80 cm W × 50 cm H</td>
<td>48 cm L × 38 cm W × 33 cm H</td>
</tr>
<tr>
<td>Headspace volume</td>
<td>25000 cm³, square</td>
<td>4076 cm³, round</td>
</tr>
<tr>
<td>Material</td>
<td>stainless steel frame with white FOREX box</td>
<td>white coated stainless steel</td>
</tr>
<tr>
<td>Sealing</td>
<td>1 continuous hollow and soft PVC strip per chamber side, 1 cm thick, transparent</td>
<td>1 neoprene gasket plus 1 neoprene collar gasket, black</td>
</tr>
<tr>
<td>Vent</td>
<td>20 cm long tube on the outside, 1 cm I.D.</td>
<td>special vent design</td>
</tr>
<tr>
<td>Fan</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>Collar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dimensions</td>
<td>48 cm × 48 cm²/58 cm × 58 cm³</td>
<td>20.3 cm I.D./21.3 cm O.D.</td>
</tr>
<tr>
<td>Enclosed soil area</td>
<td>2304 cm²</td>
<td>318 cm²</td>
</tr>
<tr>
<td>Insertion depth</td>
<td>~3 cm</td>
<td>~7 cm</td>
</tr>
<tr>
<td>Offset³</td>
<td>2.1 ± 0.7 cm</td>
<td>4.1 ± 1.1 cm</td>
</tr>
<tr>
<td>Material</td>
<td>stainless steel</td>
<td>PVC, green</td>
</tr>
<tr>
<td>Tubing⁹</td>
<td>Length</td>
<td></td>
</tr>
<tr>
<td></td>
<td>11–25 m</td>
<td>15 m</td>
</tr>
<tr>
<td></td>
<td>6.0 mm I.D.</td>
<td>3.2 mm I.D.</td>
</tr>
<tr>
<td></td>
<td>PTFE, protected inside a black plastic tube</td>
<td>Bev-a-line, protected inside a black plastic tube</td>
</tr>
<tr>
<td>Flow rate</td>
<td>3.0–3.2 lpm⁹/0.4–0.5 lpm⁹</td>
<td>2.4–2.9 lpm⁹/1.7 lpm⁹</td>
</tr>
<tr>
<td>Multiplexer pump</td>
<td>diaphragm</td>
<td>diaphragm</td>
</tr>
<tr>
<td>Gas analyser</td>
<td>Principle</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Off-Axis Integrated Cavity Output Spectroscopy</td>
<td>Non-dispersive Infrared</td>
</tr>
<tr>
<td>Measurement range</td>
<td>200–4000 ppm⁶, 7000–70 000 ppm⁶</td>
<td>0–20 000 ppm⁶, 0–40 mmol mol⁻¹</td>
</tr>
<tr>
<td>Uncertainty</td>
<td>total uncertainty: &lt; 0.25 % of reading⁶/⁷</td>
<td>accuracy: 1.5 % of reading⁶/⁷</td>
</tr>
<tr>
<td></td>
<td>precision⁶ CO₂: 150 ppb</td>
<td>RMS noise CO₂: &lt; 1 ppm⁶</td>
</tr>
<tr>
<td></td>
<td>precision⁶ H₂O: 100 ppm</td>
<td>RMS noise H₂O: &lt; 0.01 mmol mol⁻¹</td>
</tr>
<tr>
<td>Total gas volume</td>
<td>30 294–33 719 cm³</td>
<td>5372–6294 cm³</td>
</tr>
</tbody>
</table>
Table 1. Continued.

<table>
<thead>
<tr>
<th>Operational range</th>
<th>Chamber</th>
<th>&gt;2 °C, RH: non-condensing&lt;sup&gt;a&lt;/sup&gt;</th>
<th>–20 to 45 °C, 0 to 95 % RH (non-condensing)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas analyser</td>
<td>0 to 45 °C, &lt;98 % RH (non-condensing)</td>
<td>–20 to 45 °C, 0 to 95 % RH (non-condensing)</td>
<td></td>
</tr>
<tr>
<td><strong>Accessories</strong></td>
<td><strong>Air temperature</strong></td>
<td>Easytemp TMR31, Pt100 A SPADE&lt;sup&gt;b&lt;/sup&gt;, DS18B20 digital thermometer, accuracy ±0.5 °C</td>
<td>thermistor, accuracy ±0.5 °C</td>
</tr>
<tr>
<td></td>
<td><strong>Soil temperature</strong></td>
<td>SPADE&lt;sup&gt;c&lt;/sup&gt;, DS18B20 digital thermometer, accuracy ±0.5 °C</td>
<td>thermistor, accuracy ±1.0 °C</td>
</tr>
<tr>
<td></td>
<td><strong>Soil moisture</strong></td>
<td>SPADE&lt;sup&gt;c&lt;/sup&gt;, ring oscillator, relative accuracy ±4 %</td>
<td>Decagon ECH&lt;sub&gt;2&lt;/sub&gt;O model EC-5, ±3 % VWC, most mineral soils</td>
</tr>
<tr>
<td></td>
<td><strong>Air pressure</strong></td>
<td>not implemented</td>
<td>1.5 % accuracy</td>
</tr>
<tr>
<td><strong>Power requirement</strong></td>
<td>max. 2000 W</td>
<td>max. 60 W</td>
<td></td>
</tr>
</tbody>
</table>

<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> 1-sigma, 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).
Table 2. Number of discarded CO₂ fluxes after each filter step for the two automated chamber systems (SSN Lin = normalized sum of squares of residuals for linear fit, ΔTair = change in air temperature inside the closed chamber during the closure time, ΔCO₂ = difference in the atmospheric CO₂ concentration 5 cm above the collar directly before chamber closure and after a deadband of 1 min, RH = relative humidity, NA = information not available for that chamber system). Datasets were grouped by time of the day and stability of the atmospheric CO₂ concentration at 50 cm above the soil surface. Day and night were based on sun rise and sunset times. Atmospheric CO₂ concentration was considered as constant when the standard deviation for a 3 min measurement prior to the chamber closures was ≤ 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.

<table>
<thead>
<tr>
<th></th>
<th>AGPS</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
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<tbody>
<tr>
<td></td>
<td></td>
<td>Total</td>
<td>Day (constant)</td>
<td>Day (fluctuating)</td>
<td>Night (constant)</td>
<td>Night (fluctuating)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>LIN</td>
<td>EXP</td>
<td>LIN</td>
<td>EXP</td>
<td>LIN</td>
<td>EXP</td>
<td>LIN</td>
<td>EXP</td>
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<tr>
<td>Unfiltered</td>
<td>2806</td>
<td>4140</td>
<td>2105</td>
<td>771</td>
<td>580</td>
<td>824</td>
<td>492</td>
<td>248</td>
<td></td>
</tr>
<tr>
<td>Negative fluxes</td>
<td>120</td>
<td>197</td>
<td>311</td>
<td>150</td>
<td>491</td>
<td>157</td>
<td>171</td>
<td>119</td>
<td></td>
</tr>
<tr>
<td>SSN Lin &gt; 1.0 ppm</td>
<td>1717</td>
<td>3510</td>
<td>1165</td>
<td>719</td>
<td>321</td>
<td>699</td>
<td>276</td>
<td>223</td>
<td></td>
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<tr>
<td>−0.5 &lt; ΔTair &gt; 1.0°C</td>
<td>1461</td>
<td>192</td>
<td>138</td>
<td>13</td>
<td>29</td>
<td>14</td>
<td>29</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>− ΔCO₂ &lt; 0.0 ppm</td>
<td>88</td>
<td>153</td>
<td>58</td>
<td>15</td>
<td>8</td>
<td>1</td>
<td>30</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>RH &gt; 100 %</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>Filtered</td>
<td>735</td>
<td>288</td>
<td>713</td>
<td>114</td>
<td>173</td>
<td>151</td>
<td>162</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>248</td>
<td>116</td>
<td>238</td>
<td>11</td>
<td>79</td>
<td>13</td>
<td></td>
</tr>
</tbody>
</table>

|                  | LI-8100A |                  |                  |                  |                  |                  |                  |                  |                  |
|                  |          |                  |                  |                  |                  |                  |                  |                  |                  |
| Unfiltered       | 5640     | 7233             | 1376            | 888              | 2781            | 3233            | 313             | 437             |
| Negative fluxes  | 0        | 1                | 0               | 0                | 0               | 0               | 0               | 0                |
| SSN Lin > 1.0 ppm| 201      | 191              | 61              | 31               | 68              | 69              | 13              | 21              |
| −0.5 < ΔTair > 1.0°C | 1328 | 1102              | 533             | 263              | 663             | 386             | 11              | 24              |
| ΔCO₂ < 0.0 ppm   | NA       | NA               | NA              | NA               | NA              | NA              | NA              | NA               |
| RH > 100 %       | 74       | 61               | 14              | 8                | 37              | 28              | 14              | 10              |
| Filtered         | 4037     | 5878             | 763             | 586              | 2013            | 2749            | 275             | 382             | 986             | 2161 |

*21 measurements discarded prior to the filtering because of missing air temperature measurements for the flux calculation.
Table 3. Regression parameters ($E_0$ = temperature sensitivity coefficient; $R_{10}$ = soil respiration rate at 10°C soil temperature at 5 cm depth) and residual standard errors (RSE) 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–30 June 2014) and the open canopy phase (OC, 15 April–31 August 2014), respectively. The standard errors for the regression parameters and the 5% confidence intervals for the average CSR, respectively, are shown in brackets.

<table>
<thead>
<tr>
<th>Chamber Row type</th>
<th>Filtered</th>
<th>AGPS Wide</th>
<th>OC E0</th>
<th>OC R10</th>
<th>OC RSE</th>
<th>CC E0</th>
<th>CC R10</th>
<th>CC RSE</th>
<th>OC CC Avg CSR</th>
<th>OC CC Avg CSR</th>
<th>OC CC Avg CSR</th>
<th>OC CC Avg CSR</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>(K)</td>
<td>(µmol CO$_2$ m$^{-2}$ s$^{-1}$)</td>
<td>(µmol CO$_2$ m$^{-2}$ s$^{-1}$)</td>
<td>(g CO$_2$ m$^{-2}$ s$^{-1}$)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Narrow</td>
<td>Yes</td>
<td>198</td>
<td>0.70</td>
<td>177</td>
<td>0.03</td>
<td>307</td>
<td>0.03</td>
<td>0.03</td>
<td>0.07</td>
<td>0.83</td>
<td>1.10</td>
<td>1.10</td>
</tr>
<tr>
<td>Wide</td>
<td>No</td>
<td>117</td>
<td>0.07</td>
<td>110</td>
<td>0.02</td>
<td>110</td>
<td>0.02</td>
<td>0.02</td>
<td>0.07</td>
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<td>0.80</td>
<td>0.80</td>
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<tr>
<td>AGPS Wide No</td>
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<td>0.70</td>
<td>177</td>
<td>0.03</td>
<td>307</td>
<td>0.03</td>
<td>0.03</td>
<td>0.07</td>
<td>0.83</td>
<td>1.07</td>
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<tr>
<td>Chamber Row type</td>
<td>Filtered</td>
<td>AGPS Wide</td>
<td>OC E0</td>
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<td>OC RSE</td>
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<td>CC RSE</td>
<td>OC CC Avg CSR</td>
<td>OC CC Avg CSR</td>
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<td>(K)</td>
<td>(µmol CO$_2$ m$^{-2}$ s$^{-1}$)</td>
<td>(µmol CO$_2$ m$^{-2}$ s$^{-1}$)</td>
<td>(g CO$_2$ m$^{-2}$ s$^{-1}$)</td>
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<td>177</td>
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<td>307</td>
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<td>0.03</td>
<td>0.07</td>
<td>0.83</td>
<td>1.07</td>
<td>1.07</td>
</tr>
</tbody>
</table>

Note: The standard errors for the regression parameters and the 5% confidence intervals for the average CSR, respectively, are shown in brackets.
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). (a) The big black-filled rectangle shows the location of the housing for the LosGatos analysers and the AGPS multiplexer, the small black-filled rectangle 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, and grey circles indicate the position of the poplars. The dashed black lines indicate the soil sampling transects.
Figure 2. Comparison of different environmental variables measured by the AGPS and the LI-8100A before each chamber closure. (a–d) Show daily averages with their respective standard deviations. For air-filled porosity and the soil diffusion coefficient only 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 CO₂ concentration (e) which is equivalent to the CO₂ concentration at time = 0 s of the flux measurement. 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.
Figure 4. Soil gradient based CO₂ fluxes and unfiltered chamber CO₂ flux datasets for the entire monitoring period (wide rows only). Carbon dioxide fluxes over time were grouped by time of the day and stability of the atmospheric CO₂ concentration at 50 cm above the soil surface. The datasets were divided into day and night based on sun rise and sunset times. Atmospheric CO₂ concentration was considered as constant when the standard deviation for a 3 min measurement prior to the chamber closures was ≤ 1.0 ppm. The AGPS CO₂ fluxes were calculated from the first 4 min of the closure time (including 1 min deadband).
Figure 5. Filtered chamber CO$_2$ flux datasets for the entire monitoring period (wide rows only). Grouping of the data is the same as in Fig. 4.
Figure 6. The Lloyd and Taylor model fitted with the different CO$_2$ flux datasets 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.