1 Automation of soil flux chamber measurements: potentials

2 and pitfalls

3

- 4 C.-M. Görres^{1,2}, C. Kammann², and R. Ceulemans¹
- 5 [1]{Center of Excellence PLECO (Plant and Vegetation Ecology), University of Antwerp,
- 6 Universiteitsplein 1, 2610 Wilrijk, Belgium}
- 7 [2]{WG Global Change Research in Special Crops, Department of Soil Science and Plant
- 8 Nutrition, Hochschule Geisenheim University, Von-Lade-Str. 1, 65366 Geisenheim, Germany}
- 9 Correspondence to: C.-M. Görres (carolyn.goerres@hs-gm.de)

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

28

29

30

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 CO₂ 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 (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 datasets of the LI-8100A and the AGPS were 520 and 433 g CO₂ 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 CO₂ 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 CO₂ 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-8100 collars, 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.

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

28

29

30

31

32

1

2

3

4

5

6

7

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 m² 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 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 CO₂ fluxes. The latter is mainly due to a lack of available field gas analysers for CH₄ and

2 infrastructure and for intensive maintenance as compared to manual chamber measurements 3 have prevented the widespread application of automated systems. Therefore, only a few studies actually address the difficulties and challenges associated with running these systems 4 5 (Koskinen et al., 2014). 6 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 7 8 biases introduced by different chamber designs and sampling procedures have been quantified 9 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 10 Harvey, 2012). Several studies have compared the data quality of automated chamber systems 11 12 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 13 14 (Wang et al., 2013). However, different automated chamber systems have never been compared 15 in the field as has been done for eddy covariance flux systems (Janssens et al., 2000; Peltola et 16 al., 2013). 17 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 18 19 GHG flux measurements (de Klein and Harvey, 2012). Comparative analyses are important to 20 guarantee high quality data collection with these systems and a high comparability among 21 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 22 23 Flux System and the Automated Gas Sampling System AGPS. The LI-8100A is a fully 24 automated chamber system including multiplexer, gas analyser and flux calculation software. 25 The AGPS is a commercially available automated vial collector system in which each 26 automated chamber operates as an autonomous unit and the collected gas samples have to be 27 subsequently analysed by gas chromatography (Kitzler et al., 2006). For this study, the AGPS 28 has been equipped with a multiplexer and gas analysers for the first time, resembling a fully 29 automated custom-made chamber system with continuous gas analysis in the field. In parallel, 30 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 31 32 conditions within one vegetation period. In addition to the chamber measurements, CO₂

N₂O in the past (Venterea et al., 2008; Savage et al., 2014). The requirements for a larger

- 1 concentrations were monitored in the topsoil to give insights into the potential range of soil CO₂
- 2 fluxes at the site and to better understand the performance of the chambers under different soil
- 3 moisture conditions. The aim of this study was not to understand the processes driving soil
- 4 respiration or soil CO₂ efflux at the poplar plantation per se since these have already been
- 5 discussed amongst others by Verlinden et al. (2013) and Zenone et al. (2015). The results
- 6 presented here serve the comparison of the performance of the two chamber systems in
- 7 quantifying soil respiration fluxes under a wide range of different environmental conditions.

9

10

2 Materials and Methods

2.1 Field site and experimental design

- 11 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
- 13 2010 in a double-row planting system, i.e. the distance between two adjacent rows of poplar
- trees alternated between 0.75 m 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).
- 18 The part of the plantation in which this study took place was coppied for the second time in
- 19 March 2014. The poplar stems were cut manually about 10 cm above the soil surface. The
- 20 experimental set-up of the automated GHG flux monitoring inter-comparison campaign is
- shown in Fig. 1A and 1B. The measurement set-up consisted of eight automated chambers
- located only in wide rows due to their size (AGPS, UIT Umwelt- und Ingenieurtechnik GmbH
- 23 Dresden, Germany), eight automated chambers which were evenly distributed in narrow and
- 24 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,
- 26 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 sections 2.2 and 2.3
- below. The inter-comparison campaign took place from 15 April 2014 until 31 August 2014.
- 29 During this period the coppied poplar stools regrew to a height of about 3.40 m. Canopy
- 30 closure was achieved at the beginning of July 2014.

2.2 Automated soil flux chamber systems

1

7

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

28

29

30

31

- 2 The Greenhouse Gas Monitoring System AGPS (UIT Umwelt- und Ingenieurtechnik GmbH
- 3 Dresden, Germany) and the LI-8100A Automated Soil CO₂ Flux System (LI-COR Biosciences,
- 4 Lincoln, NE, USA) were both closed dynamic chamber systems with the chambers operating
- 5 in sequence. The technical specifications of the two chamber systems are displayed in Table 1,
- 6 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 x 1.21 m wide x 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⁻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 included in the set-up were the gas analysers. Here, we report CO₂ 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 Umwelt- und Ingenieurtechnik GmbH). 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
- 2 removed from inside the collars and around the chambers about every two weeks.
- 3 The AGPS sampling protocol consisted of the following steps: (i) 30 min tube heating, (ii) 5
- 4 min sampling of atmospheric air at 50 cm height outside the multiplexer for flushing the gas
- 5 analyser, (iii) 2 min purging of the tubes between the chamber and the multiplexer, (iv) 1 min
- 6 in which the chamber closed, the multiplexer pump automatically turned off during this time,
- 7 (v) 10 min measurement with 1 Hz gas sampling frequency, (vi) 1 min in which the chamber
- 8 opened (multiplexer pump turned off), (vii) step ii repeated for 11 min. Each chamber was
- 9 sampled every 4 hrs 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).
- 11 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 subzero temperatures. The chambers
- were moved by a non-flexible arm. The time needed to close a chamber was between 11 and
- 18 15 s during which the multiplexer pump did not turn off. Once closed, the chamber did not rest
- 19 directly on the collar rim, but on a metal plate surrounding the collar, leaving the collar
- 20 undisturbed. Tubing inlet and outlet were installed inside the chamber. Soil sensors were
- 21 installed the same way as for the AGPS. All measurement data were stored inside the analyser
- 22 control unit on a compact flash card which could be accessed and controlled remotely via the
- 23 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-
- 25 purge time. Each chamber was sampled every 2 hrs. 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

- 30 Each soil CO₂ concentration sampler consisted of a 16 mm thick, corrosion-resistant steel tube
- 31 with a 10 ml sampling cell (12 mm diameter) at its lower end. The length of the sampler

1 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 x 0.5 mm² 2 silicone disc to allow only the diffusion of gases between the soil and the cell. For sampling, 3 4 the steel tube contained two smaller tubes made from stainless steel needles (18G, inner 5 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-6 7 drilling a hole of the same diameter as the sampler to about 5 cm above the intended 8 measurement depth. The samplers were inserted into the hole and carefully pushed to the 9 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 10 11 cm depth. 12 Soil CO₂ was sampled about every two weeks between 10:00 and 14:00. A plastic syringe 13 containing 10 ml N₂ and 50 ppm C₂H₄ was connected via a two-way valve to one of the small 14 tubes inside the sampler. An empty 10 ml glass syringe (SIGMA-ALDRICH, Diegem, 15 Belgium) and a 12 ml pre-evacuated exetainer (Labco Ltd, Lampeter, UK) were connected to 16 the other tube via a three-way valve. The N₂/C₂H₄ mixture was injected into the diffusion cell 17 flushing the 10 ml soil gas sample via the second tube into the glass syringe. The glass syringe 18 was then emptied into the exetainer. At last, 12 ml N₂ were injected into the exetainer to create an overpressure needed for the subsequent gas analysis. The concentration of C₂H₄ recovered 19 20 in the collected sample was used to calculate the dilution of the original sample which occurred while replacing it with N₂ in the sampling cell, and to correct the measured CO₂ concentration 21 accordingly. The correction was performed with the assumption that there was full equilibrium 22 23 between the diffusion cell and the inlet and outlet tube. During the sampling, diffusive loss of 24 C₂H₄ 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 N₂ to remove 25 remaining traces of C₂H₄. For more details on the sampler design and the C₂H₄ correction see 26 27 Petersen (2014). 28 The gas samples were analysed on a Bruker Custom Greenhouse Analyser (Bruker Daltonik 29 GmbH, Bremen, Germany) equipped with a thermal conductivity detector (TCD) for the 30 analysis of CO₂ and C₂H₄. 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 31 32 ml min⁻¹ was used as reference flow. Total flow was 60 ml min⁻¹. Concentrations were

- 1 quantified with reference to three calibration gases with an accuracy of 2 %: (i) 50 μl l⁻¹ C₂H₄
- 2 in N₂, (ii) 799 μl l⁻¹ CO₂ in synthetic air, and (iii) 5.04 % CO₂ in synthetic air.

2.4 Soil sampling

- 4 To assess the impact of the permanently installed chambers on soil properties which potentially
- 5 control soil CO₂ concentrations and flux rates, as well as to assess the comparability between
- 6 the flux measurement plots, soil samples were taken before and after the inter-comparison
- 7 campaign. In February 2014, undisturbed topsoil samples were taken along two transects (Fig.
- 8 1A). At each transect, three samples were taken per row type and per sampling depth. For soil
- 9 C and dissolved organic carbon (DOC), soil was sampled at 0-10 and 10-20 cm depth with an
- auger (~ 2 cm diameter). Separate samples were taken with stainless steel cylinders (100 cm³)
- 11 (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 0.5M K₂SO₄ for 1 hr. This
- suspension was filtered with Whatman filter paper (grade 42, ashless, 150 mm) and the filtered
- 16 liquid analysed for DOC with Continuous Flow Analysis (CFA) (San++ Automated Wet
- 17 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
- 20 of necessity the February 2014 samples had to be aggregated by row type and sampling depth
- 21 prior to the grinding. The steel cylinder samples were dried at 105 °C to constant weight for dry
- bulk density determination.
- 23 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
- 25 Significant Difference test were used to compare group means ($\alpha = 0.05$). Normality for each
- 26 group and homogeneity of variance of the groups were tested with the Shapiro-Wilk test and
- 27 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
- 29 exception was the chamber flux calculation for the AGPS (section 2.5) which had to be
- 30 conducted with R version 3.0.2 due to a package incompatibility.

2.5 Chamber flux calculation and quality control

1

32

2 For the AGPS, descriptive statistics and water-corrected CO₂ fluxes were calculated with a selfwritten R script incorporating the "gasfluxes" script (Roland Fuß, Institute of Agricultural 3 4 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 5 6 packages "zoo", "xts" and "xtsExtra" (Zeileis and Grothendieck, 2005; Ryan and Ulrich, 2014; 7 Weylandt, 2014). For each AGPS measurement, the flux was calculated with linear regression, 8 robust linear regression with a Huber-M estimator (RLM) (Huber, 1981), and a modified 9 Hutchinson-Mosier non-linear function (HMR) (Pedersen et al., 2010). This procedure was performed twice for each measurement – for a closure time of 4 min and 9 min, respectively. 10 Prior to each flux calculation, the first minute of the CO₂ concentration curves was discarded 11 (= deadband) to account for the time needed to establish steady headspace mixing as well as 12 13 any disturbances caused by the chamber placement at the beginning of the measurement 14 (Christiansen et al., 2011; Koskinen et al., 2014). For each flux calculation, the "gasfluxes" 15 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 16 17 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 18 cases, RLM was chosen as the best-fitting model. The fluxes calculated by linear regression 19 and RLM were the same, except that RLM was robust against outliers in the CO₂ concentration 20 curves. Fluxes were converted from $\mu l m^{-2} s^{-1}$ to $\mu mol m^{-2} s^{-1}$ using the ideal gas law (Parkin 21 22 and Venterea, 2010) with air temperature and pressure provided by the AGPS and the LI-23 8100A, respectively. 24 For the LI-8100A, water-corrected mass CO₂ fluxes and descriptive statistics were 25 automatically provided by the LI-8100 File Viewer Version 3.0.0 (LI-COR Biosciences). For 26 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 27 28 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 CO₂ concentration 29 curve were discarded before the flux calculation. 30 31 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 CO₂

(equivalent to a root mean square error threshold of 1.0 ppm CO₂, Görres et al., 2014), (iii) 1 2 decrease in headspace temperature during the closure time by more than 0.5 °C or increase by 3 more than 1.0 °C, (iv) difference in the atmospheric CO₂ concentration 5 cm above the collar 4 directly before the chamber closure and after a deadband of 1 min of less than 0.0 ppm (i.e. 5 decreasing CO₂ concentration), and (v) mean relative humidity (RH) inside the closed chamber higher than 100 %. The first criterion detected chamber measurements with large leaks, whereas 6 7 smaller leakages and other measurement disturbances could be detected by selecting an 8 appropriate noise level threshold in the second criterion. CO₂ flux measurements can be very 9 sensitive to changes in environmental conditions, thus criteria (iii) – (v) removed measurements for which the CO₂ concentration increase curve looked okay, but which might still have been 10 11 biased by changes in environmental conditions too large to guarantee continuous identical 12 diffusion conditions during chamber closure.

2.6 Soil diffusivity and gradient-based CO₂ flux calculation

13

24

25

26

27

28

14 Changes in topsoil CO₂ concentration dynamics for each collar and each soil gas sampling plot 15 throughout the inter-comparison campaign were approximated by calculating the effective soil 16 diffusion coefficient (D_s) which is the product of the CO₂ diffusion coefficient in free air (D_a) 17 and the gas tortuosity factor ξ . D_a was corrected for temperature and air pressure by

18
$$D_a = D_{a\theta} \left(\frac{T}{293.15}\right)^{1.75} \left(\frac{P}{101.3}\right)$$
 (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 mm² s⁻¹ (Jones, 1992). The empirical Millington-Quirk model was used for estimating ξ (Millington and Quirk, 1961):

23
$$\xi = \frac{(\phi - VWC)^{10/3}}{\phi^2}$$
 (2)

where VWC is the volumetric water content at 5 cm depth and ϕ the total porosity (m³ 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 %.

- 1 Additionally, soil CO₂ fluxes were calculated via Fick's first law of diffusion by multiplying
- 2 the CO₂ concentration gradients between 5 and 15 cm depth obtained from the soil gas sampling
- 3 plots with the respective D_s (Roland et al., 2015). Prior to the flux calculation, soil CO_2
- 4 concentrations in ppm were converted to μmol m⁻³ by multiplying them by the molar volume
- 5 of a gas at standard temperature and pressure (0.04462 μmol L⁻¹, Brummell and Siciliano,
- 6 2011). Soil temperature and soil moisture values at 5 cm depth were obtained from the nearest
- 7 chamber in the same row type.

2.7 Comparison of the CO₂ flux datasets

- 9 The AGPS and LI-8100A soil CO₂ fluxes were directly compared for four different
- environmental conditions, namely (i) daytime, constant atmospheric CO₂ concentration, (ii)
- daytime, fluctuating atmospheric CO₂ concentration, (iii) nighttime, constant atmospheric CO₂
- 12 concentration, and (iv) nighttime, fluctuating atmospheric CO₂ concentration. Daytime and
- 13 nighttime fluxes were separated based on local sunrise and sunset times. Atmospheric CO₂
- 14 concentration was considered as constant when the CO₂ concentration measured at 50 cm height
- above the soil surface had a standard deviation < 1.0 ppm (3 min measurements). Constant
- ambient CO₂ 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
- datasets. Firstly, average CO₂ flux rates for each automated chamber system and their respective
- 20 95 % confidence intervals were estimated using generalized additive models (GAM) with a
- 21 nonlinear smooth (thin plate regression spline) for time and with random smooths (factor
- smooth interactions) over time for each chamber (R packages 'itsadug' and 'mgev', function
- 23 'bam') (Wood, 2006; van Rij et al., 2015). Autocorrelation was accounted for by including an
- 24 AR1 model. Input data were the unfiltered and filtered CO₂ fluxes, respectively, averaged by
- 25 day and by chamber. Secondly, the chamber flux datasets were quantitatively compared by
- using the common approach of modelling soil respiration (SR) according to Lloyd and Taylor
- 27 (1994) (eq. 11):

28 SR =
$$R_{I\theta} \exp E_{\theta} \left(\frac{1}{56.02} - \frac{1}{T - 227.13} \right)$$
 (3)

- where $R_{I\theta}$ is the respiration rate at 10 °C (µmol CO₂ m⁻² s⁻¹), E_o the temperature sensitivity
- 30 coefficient (K), and T the soil temperature at 5 cm depth (K). SR was modelled separately for
- and each combination of chamber system and row type. Each model was also fitted once with the

unfiltered and the filtered dataset, respectively, using nonlinear regression (R function 'nls'). Input data were the single measured CO₂ fluxes. A part of the chamber measurements were excluded from the modelling due to missing soil temperature values (sensor malfunctions). Cumulated CO₂ 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±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 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. A, supplementary material). 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 CO₂ 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 1 2 throughout the monitoring time. They were on average slightly higher in the narrow rows than 3 in the wide rows (Fig. 2A and 2B). This variability was driven by several heavy rain events 4 resulting in sharp soil moisture increases (Fig. 2C). From July 2014 onwards standing water 5 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 6 7 types. 8 The AGPS collars received more direct sunlight than the LI-8100A collars, resulting in higher 9 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 10 surrounding the chamber decreased with increasing collar area. The average daily soil 11 temperature difference between the AGPS and the LI-8100A was generally less than 1 °C when 12 the fraction of shading by the vegetation was homogenous throughout the study site. However, 13 14 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 15 until the middle of July also coincided with the warmest and the driest monitoring period. 16 Canopy closure above the AGPS collars was reached about a week later in comparison to the 17 LI-8100A chambers because the larger structure of the AGPS chambers hindered the growing 18 poplar stems from leaning towards each other (see Fig. B in the supplementary material for the 19 20 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 21 22 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 23 24 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 25 measurements, respectively, fulfilling quality control criterion 3. 26 27 Variability in air and soil temperature decreased after the canopy closure at the beginning of 28 July, but the opposite was true for the atmospheric CO₂ concentration measured 50 cm above 29 the soil surface. Constant atmospheric CO₂ concentrations at that height were only observed 30 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

31

- 1 more than 1.0 ppm prior to the chamber closure. Atmospheric CO₂ concentrations measured 5
- 2 cm above the collars at the time of chamber closure matched well between the AGPS and the
- 3 LI-8100A most of the time (Fig. 2E). However, the AGPS recorded a number of atmospheric
- 4 CO₂ concentrations at chamber closure above 500 ppm which were not observed by the LI-
- 5 8100A. Two-thirds of the AGPS CO₂ values above 500 ppm were measured during nighttime.

3.2 Technical reliability of the two chamber systems

- 7 During the inter-comparison campaign, the LI-8100A conducted 12874 chamber measurements
- 8 (wide rows: 6253, narrow rows: 6621) of which only 1 measurement had to be discarded due
- 9 to technical problems with the chamber closing mechanism. Overall, the LI-8100A showed a
- 10 high robustness despite having previously operated continuously in the poplar plantation for
- 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
- 16 (n=111). Chamber malfunctions were all caused by the steel cables which moved the AGPS
- 17 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.
- 20 Two main issues prevented the AGPS from operating continuously in the field. First, the air
- 21 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
- 23 the collars would have been equipped with air filters the same way as LI-8100A chambers are.
- 24 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
- 26 than 2 hr. 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.
- 29 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
- 31 %) and 328 (7 %) measurements provided a dataset at 1 Hz frequency, respectively. The median

- 1 number of data points for the short and long flux calculation period (i.e. chamber closure time
- 2 excl. 1 min deadband period) was 167 and 328, respectively. During the first half of June 2014,
- 3 the number of data points per measurement even dropped below 50 and 100, respectively.

3.3 Flux quality

4

31

5 In total 23 % of the LI-8100A CO₂ flux measurements were discarded, mainly because of 6 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 7 daytime and nighttime dataset based on headspace temperature changes once the canopy was 8 9 closed. With respect to row type, headspace temperature problems were more often encountered 10 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-11 8100A concentration curves were best-fitted exponentially, especially under fluctuating 12 13 atmospheric CO₂ concentrations. For the AGPS dataset, a higher proportion of the concentration curves were best-fitted linearly 14 15 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 16 17 total, 71 and 94 % of the AGPS fluxes using the short and the long flux calculation period, 18 respectively, did not pass the quality control (Table 2). No correlation was found between the 19 amount of discarded data and the tubing length of the chambers. For the unfiltered dataset, the fluxes calculated for 9 min closure time were 0.21±0.50 µmol m⁻² s⁻¹ (average±STD) lower 20 than the fluxes calculated for a 4 min closure time. In the filtered dataset, this was reduced to 21 0.18±0.18 µmol m⁻² s⁻¹. Due to the low data quality for the long flux calculation period, only 22 the fluxes calculated for the 4 min closure time were considered in the remaining result sections 23 24 below. For the 4 min closure time, 2882 flux measurements had a $SSN_{Lin} > 1.0$ ppm. This included 25 26 almost all data from the first half of June where we had the severe gas analyser logging problem. 27 The SSN_{Lin} criterion also already filtered out 60 % and 79 % of the flux measurements with 28 headspace temperature problems and with a decrease of the CO₂ headspace concentration 29 during the deadband period, respectively. The latter criterion filtered out most of the flux 30 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

- 1 median pre-closure atmospheric CO₂ concentration at 5 cm height above the collar of 600 ppm,
- 2 whereas measurements passing this criterion had a median pre-closure atmospheric CO₂
- 3 concentration of 433 ppm.

- 4 The SSN_{Lin} criterion would have also detected 83 % of the negative CO₂ fluxes. Negative CO₂
- 5 fluxes were clearly associated with severely leaking chambers. Insufficient airtight sealing was
- 6 also probably a problem for a part of the fluxes with a high SSN_{Lin}. In contrast to the LI-8100A
- 7 chambers, the AGPS chambers had no mechanism which additionally pressed them onto the
- 8 collar once the sealing and the collar rim came in contact. The AGPS chamber and the collar
- 9 had to be perfectly aligned to achieve an airtight sealing which was challenging and required
- 10 regular re-adjustments of the collars throughout the monitoring. However, the noise in the
- 11 AGPS flux dataset was large regardless of the environmental conditions, and the noise was
- lower at constant atmospheric CO₂ 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 CO₂ flux datasets

- Until the beginning of July 2014, CO₂ concentrations at 5 and 15 cm depths in the narrow rows
- 16 were 4702±762 ppm (average±SE, n=16) and 12565±2145 ppm (n=15), respectively, and in
- the wide rows 6664 ± 1108 ppm (n=14) and 12251 ± 1512 ppm (n=15), respectively. Afterwards,
- 18 CO₂ concentrations increased at 5 and 15 cm depth in the narrows rows to on average
- 19 11797±2365 ppm (n=20) and 27071±3615 ppm (n=19), respectively. In the wide rows, CO₂ at
- 5 cm depth reached the same concentrations as in the narrow rows, whereas at 15 cm depth, it
- 21 increased even further (38008±4574 ppm, n=19). The increasing steepness of the soil CO₂
- 22 concentration gradient was probably partly the result of CO₂ accumulation in the soil due to the
- 23 reduced air-filled porosity as the magnitude of the surface CO₂ fluxes measured with the
- chambers did not increase strongly during this period (Fig. 4). Contrastingly, the CO₂ fluxes
- based on the flux gradient method were unrealistically high in July and August 2014 (wide row:
- $8.9\pm1.5 \mu \text{mol m}^{-2} \text{ s}^{-1}$, n=19; narrow row: $10.8\pm1.5 \mu \text{mol m}^{-2} \text{ s}^{-1}$, n=17), whereas prior to the
- 27 rewetting, they were in the same range as the chamber CO₂ fluxes (wide row: 2.6±0.5 µmol m⁻¹
- 28 ² s⁻¹, n=12; narrow row: 3.9±0.6 μmol m⁻² 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
- 30 CO₂ concentration profiles and soil diffusion coefficients during high soil moisture conditions.
- 31 Short-term fluctuations in the soil CO₂ 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

1 2014 because the soil CO₂ concentration samplings were performed at least three days after

2 such events, except for the last sampling.

Daily average CO₂ fluxes estimated from the unfiltered flux datasets did not differ significantly 3 4 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). 5 6 With regard to the single unfiltered CO₂ flux measurements, the dataset from the AGPS showed higher flux variability throughout the inter-comparison campaign than the unfiltered CO₂ flux 7 8 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 9 only by the AGPS during the open canopy phase (Fig. 5). In contrast to the AGPS, filtering 10 mainly thinned out the LI-8100A dataset of the open canopy phase since the biggest problem 11 for these chambers was overheating. LI-8100A chambers installed in the wide rows were more 12 13 subjected to this problem than those in the narrow rows (Fig. C, supplementary material). The 14 fit of the GAM for the LI-8100A dataset was only slightly changed by the filtering whereas the 15 fit for the AGPS changed significantly. This led to a distinct separation of the two GAM curves for the daily CO₂ fluxes (Fig. 5, top panel) with the AGPS flux estimates being constantly lower 16 in comparison to the LI-8100A. However, both chamber systems still seemed to be able to 17 18 capture the same temporal flux dynamics although the AGPS model curve was slightly shifted to the left in comparison to the LI-8100A model curve. 19 20 Modelling CO₂ fluxes along the soil temperature gradient revealed discrepancies between the 21 datasets of the AGPS and the LI-8100A similar to those visualized by the GAM. Filtering the LI-8100A dataset changed only slightly the model fits of the Lloyd and Taylor model and had 22 no significant effect on the subsequently calculated SR balances (Table 3, Fig. 6). The opposite 23 24 was observed for the AGPS dataset. The regression parameters based on the AGPS dataset were 25 lower than the ones based on the LI-8100A dataset from the wide rows (Table 3); the 26 discrepancies between the regression lines increased with increasing soil temperature and the 27 data filtering (Fig. 6). Similar SR balances and R₁₀ values were only obtained by the two 28 chamber systems during the closed canopy phase. Regardless of chamber type, row type and 29 environmental conditions, the filtering led to a decrease in the SR balance estimates, but it also 30 improved the model fit (see Fig. D in the supplementary material for the distribution of the residuals). The SSN_{Lin} and the headspace temperature criteria filtered out fluxes mainly above 31 1.5 µmol CO₂ m⁻² s⁻¹. Only criterion (iv) mainly removed positive fluxes below 1.5 µmol CO₂ 32

- 1 m⁻² s⁻¹, but this criterion was not applicable to the LI-8100A chambers since these chambers
- 2 did not provide an undisturbed pre-closure atmospheric CO₂ concentration.
- 3 The tendency that the absolute differences in the CO₂ flux rates between the two datasets varied
- 4 throughout the monitoring period was also visible when looking just at the measurement periods
- 5 with the highest data quality (Fig. 7). During the open canopy phase, the AGPS flux rates were
- 6 $0.31\pm0.03 \,\mu\text{mol CO}_2\,\text{m}^{-2}\,\text{s}^{-1}$ (average \pm SE, n=47) lower than the ones obtained by the LI-8100A,
- 7 whereas during the closed canopy phase, the opposite was observed with the AGPS flux rates
- 8 being $0.08\pm0.06~\mu mol~CO_2~m^{-2}~s^{-1}~(n=15)$ higher.

10

24

4 Discussion

- 11 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
- 14 Klein and Harvey, 2012). These biases have been quantified for different chamber types under
- 15 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
- 19 chosen automated chamber systems in a wide range of environmental conditions within a short
- 20 period of time. The following sections are going to address real alterations of the field SR
- 21 introduced by the presence of the chamber systems, by measurement artefacts caused by
- 22 environmental conditions that affect chamber performance, and by biases introduced by the
- 23 subsequent data analysis.

4.1 Effect of chambers on environmental conditions

- 25 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
- 27 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
- 29 height of the chambers restricts the height to which vegetation can be allowed to grow inside
- 30 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 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 root 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 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

1

2

3

4

5

6

7

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

28

29

30

31

32

- 1 the field and it makes it easy to relocate the chambers. However, the contribution of any type
- 2 of collar edge effect to the total SR flux increases with an increasing collar perimeter to collar
- 3 area ratio, and is thus more of a problem for smaller chambers.

4.2 Effect of environmental conditions on chamber performance

5 Collars have the purpose to help provide an air-tight system during chamber measurements by 6 (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 7 8 soil during chamber deployment (Hutchinson and Livingston, 2001). Chamber leakages can 9 lead to negligible or significant flux underestimation depending on the environmental 10 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 11 12 increasing air-filled porosity (Hutchinson and Livingston, 2001; Heinemeyer and McNamara, 2011; Creelman et al., 2013). The increase in soil CO₂ concentrations during the closed canopy 13 14 phase in the poplar plantation which was not accompanied by a change of magnitude in the 15 chamber CO₂ 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 16 thus diffusivity (Turcu et al., 2005; Hashimoto and Komatsu, 2006). The application of the flux 17 18 gradient method has been shown to be problematic in soils which are near water saturation 19 because of the difficulties to estimate low soil diffusion coefficients with high certainty (Maier 20 and Schack-Kirchner, 2014). The AGPS was more prone to lateral diffusive soil gas losses than 21 the LI-8100A due to its shallower collar insertion depth. Thus, lateral soil gas diffusion losses 22 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 23 24 comparison to the closed canopy phase with its higher soil moisture conditions. 25 Flux underestimation caused by leakages in the aboveground seal was certainly also an issue 26 for the AGPS as could be seen from the high maintenance needs necessary to keep the seal 27 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 28 29 the poplar plantation, especially regardless of the wind protection. This is a good indicator that 30 the chambers had no issue with the airtightness of the rubber sealing. Under windy conditions, 31 one can expect to see more noise in the CO₂ concentration curves obtained during chamber 32 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
- 2 highest during calm conditions. Additionally, high SSN_{Lin} values were often associated with
- 3 higher fluxes (>1.5 μ mol CO₂ m⁻² s⁻¹). These are indicators that the sample air flow rate between
- 4 the multiplexer and the chambers was not high enough to sufficiently mix the chamber
- 5 headspace during the measurements (Liu and Si, 2009; Christiansen et al., 2011). Insufficient
- 6 headspace mixing leads also to flux underestimation (Liu and Si, 2009; Christiansen et al.,
- 7 2011).

- 8 Chamber design-induced flux estimation errors can be reduced by shortening the chamber
- 9 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
- 12 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 CO₂ saturation effects.

4.3 Effect of data processing on flux rates

- 18 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
- 21 curves obtained by non-steady-state chambers are always nonlinear. However, whether
- 22 nonlinearity can be detected with sufficient statistical significance depends on the length of the
- 23 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
- 25 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).
- 29 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
- 31 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

2 caused by CO₂ flux underestimation of the AGPS.

3 Over the last years, several advanced nonlinear flux models based on diffusion theory have 4 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 5 6 (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 7 8 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 9 diffusion than other advanced nonlinear flux models, but the model also constantly showed the 10 highest flux underestimation across a wide range of environmental conditions (Venterea, 2013). 11 However, the study was based on model simulations with only five sampling points during the 12 chamber closure time. It still has to be tested if this underestimation also prevails when fitting 13 14 the HMR model with high temporal frequency data. A general problem of all nonlinear models 15 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 16 17 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 18 flux calculation method cannot be ruled out for both automated chamber systems, but these 19 biases can only be accurately quantified under laboratory conditions (Pihlatie et al., 2013) or 20 21 with advanced model simulations (Creelman et al., 2013). 22 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 23 24 flux datasets like they are in the eddy covariance community (Vargas et al., 2011). The 25 challenge is to find criteria which are able to identify faulty measurements of different origins, 26 but at the same time not to discard large amounts of good flux measurements as well. The RMSE seems to be such a promising criterion (Christiansen et al., 2011; Jassal et al., 2012; 27 28 Görres et al., 2014). Since the susceptibility of the chamber methodology to certain 29 measurement artefacts changes with environmental conditions, any filtering can lead to a bias 30 in the temporal resolution 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-31 32 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
- 2 canopy phase. However, the amount of data collectible with the LI-8100A was so high that
- 3 even a discard of a quarter of the data did not alter the modelled SR balance significantly.
- 4 Comparing the unfiltered and filtered dataset should be the last step of any flux quality control
- 5 protocol as it can give valuable insights into the performance of the chambers and potential
- 6 measurement artefacts, but it also offers a way to check the quality of the filter protocol itself.

4.4 Nighttime chamber measurements

- 8 Obtaining reliable nighttime SR fluxes is one of the biggest remaining methodological
- 9 challenges. During nighttime, atmospheric turbulences tend to calm down. Consequently, CO₂
- 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 CO₂ gradient between 0 and 100 cm
- above the soil surface (Schneider et al., 2009; Lai et al., 2012). However, the accumulation of
- 13 CO₂ on the soil surface leads to a decreasing CO₂ gradient between the soil pore space and the
- 14 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 CO₂
- into the chamber during chamber placement when the CO₂ layer directly above the soil surface
- is broken up, and secondly to an increasing soil-atmosphere CO₂ gradient (Schneider et al.,
- 19 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
- 21 with chambers under calm conditions.
- 22 This is a serious problem since nighttime chamber measurements have been used to assess the
- 23 measurement bias of the eddy covariance method which systematically underestimates CO₂
- 24 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
- 26 time, on empirical methods to correct biased flux measurements or on the use of daytime
- 27 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
- 29 they fulfil certain design criteria regarding chamber height, direction of chamber movement,
- 30 chamber closing speed, and sample inlet position. The combination of a low chamber height
- 31 (<20 cm) and a mainly horizontal movement of the chamber from its parking position to the
- 32 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

2526

27

28

29

30

31

32

1

2

3

4

5

6

7

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

5 Recommendations for automation of soil respiration measurements

concentrations have been overestimated.

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

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

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 tradeoff 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
- 2 have the potential to solve this issue if certain design criteria are considered. Thus besides
- 3 providing high temporal frequency flux data, automated chamber systems would offer another
- 4 possibility to greatly improve our understanding of soil GHG fluxes.

6 Acknowledgements

- 7 The authors thank Nicola Arriga, Joris Cools, Fred Kockelbergh, Kristof Mouton, Jan Segers,
- 8 UIT, Stefan Vanbeveren, and Marc Wellens for their technical assistance at the field site. We
- 9 also acknowledge Gerald Moser (Justus Liebig University Giessen, Germany) who analyzed
- 10 the soil CO₂ 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
- 12 (# 233366) POPFULL under the EC 7th Framework Programme (FP7/2007-2013), the
- 13 Flemish Hercules Foundation as Infrastructure contract # ZW09-06, and the Methusalem
- 14 Program of the Flemish Government.

15

16

References

- Ambus, P., Skiba, U., Drewer, J., Jones, S. K., Carter, M. S., Albert, K. R., and Sutton, M. A.:
- 18 Development of an accumulation-based system for cost-effective chamber measurements of
- 19 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
- 22 chambers, Agr Forest Meteorol, 131, 225–232, 2005.
- 23 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,
- 27 2015.
- 28 Breuer, L., Papen, H., and Butterbach-Bahl, K.: N₂O emission from tropical forest soils of
- 29 Australia, J. Geophys. Res, 105, 26353–26367, 2000.

- 1 Broeckx, L. S., Verlinden, M. S., and Ceulemans, R.: Establishment and two-year growth of a
- 2 bio-energy plantation with fast-growing Populus trees in Flanders (Belgium): Effects of
- 3 genotype and former land use, Biomass Bioenerg, 42, 151–163, 2012.
- 4 Brummell, M. E. and Siciliano, S. D.: Measurement of carbon dioxide, methane, nitrous oxide,
- 5 and water potential in soil ecosystems, Method Enzymol, 496, 115–137, 2011.
- 6 Carbone, M. S. and Vargas, R.: Automated soil respiration measurements: new information,
- 7 opportunities and challenges, New Phytol, 177, 295–297, 2008.
- 8 Christiansen, J., Korhonen, J. J., Juszczak, R., Giebels, M., and Pihlatie, M.: Assessing the
- 9 effects of chamber placement, manual sampling and headspace mixing on CH₄ fluxes in a
- laboratory experiment, Plant Soil, 343, 171-185, 2011.
- 11 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, 2014.
- 13 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. (eds.): Nitrous oxide chamber methodology guidelines, Ministry
- 18 for Primary Industries, New Zealand, online available at:
- 19 <u>http://www.globalresearchalliance.org/research/livestock/activities/nitrous-oxide-chamber-</u>
- 20 methodology-guidelines/, 2012.
- Denmead, O. T.: Chamber systems for measuring nitrous oxide emission from soils in the field,
- 22 Soil Sci Soc Am J, 43, 89–95, 1979.
- FAO: Agriculture, forestry and other land use emissions by sources and removals by sinks:
- 24 1990 2011 analysis. FAO Statistics Division Working Paper Series, Rome, 2014.
- Forbrich, I., Kutzbach, L., Hormann, A., and Wilmking, M.: A comparison of linear and
- 26 exponential regression for estimating diffusive CH₄ fluxes by closed-chambers in peatlands,
- 27 Soil Biol Biochem, 42, 507–515, 2010.
- Giltrap, D. L., Li, C., and Saggar, S.: DNDC: A process-based model of greenhouse gas fluxes
- 29 from agricultural soils, Agr Ecosyst Environ, 136, 292–300, 2010.

- 1 Görres, C.-M., Kutzbach, L., and Elsgaard, L.: Comparative modeling of annual CO₂ flux of
- 2 temperate peat soils under permanent grassland management, Agr Ecosyst Environ, 186, 64–
- 3 76, 2014.
- 4 Hashimoto, S., and Komatsu, H.: Relationships between soil CO₂ concentration and CO₂
- 5 production, temperature, water content, and gas diffusivity: implications for field studies
- 6 through sensitivity analyses, J For Res, 11, 41–50, 2006.
- Heinemeyer, A., Di Bene, C., Lloyd, A. R., Tortorella, D., Baxter, R., Huntley, B., Gelsomino,
- 8 A., and Ineson, P.: Soil respiration: implications of the plant-soil continuum and respiration
- 9 chamber collar-insertion depth on measurement and modelling of soil CO₂ efflux rates in three
- 10 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,
- 13 2011.
- Hopkins, F., Gonzalez-Meler, M. A., Flower, C. E., Lynch, D. J., Czimczik, C., Tang, J., and
- 15 Subke, J.-A.: Ecosystem-level controls on root-rhizosphere respiration. New Phytol, 199, 339–
- 16 351, 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 CO₂
- efflux: an in situ comparison of four techniques, Tree Physiol, 20, 23–32, 2000.
- Jassal, R., Black, A., Novak, M., Morgenstern, K., Nesic, Z., and Gaumont-Guay, D.:
- 23 Relationship between soil CO₂ concentrations and forest-floor CO₂ effluxes, Agr Forest
- 24 Meteorol, 130, 176–192, 2005.
- Jassal, R. S., Black, T. A., Nesic, Z., and Gaumont-Guay, D.: Using automated non-steady-state
- 26 chamber systems for making continuous long-term measurements of soil CO₂ efflux in forest
- ecosystems, Agr Forest Meteorol, 161, 57–65, 2012.
- 28 Jones, H. G.: Plants and microclimate: A quantitative approach to environmental plant
- 29 physiology, Cambridge University Press, Cambridge, UK, 1992.

- 1 Kitzler, B., Zechmeister-Boltenstern, S., Holtermann, C., Skiba, U., and Butterbach-Bahl, K.:
- 2 Nitrogen oxides emission from two beech forests subjected to different nitrogen loads,
- 3 Biogeosciences, 3, 293–310, 2006.
- 4 Koskinen, M., Minkkinen, K., Ojanen, P., Kämäräinen, M., Laurila, T., and Lohila, A.:
- 5 Measurements of CO₂ exchange with an automated chamber system throughout the year:
- 6 challenges in measuring night-time respiration on porous peat soil, Biogeosciences, 11, 347–
- 7 363, 2014.
- 8 Kutzbach, L., Schneider, J., Sachs, T., Giebels, M., Nykänen, H., Shurpali, N. J., Martikainen,
- 9 P. J., Alm, J., and Wilmking, M.: CO₂ flux determination by closed-chamber methods can be
- seriously biased by inappropriate application of linear regression, Biogeosciences, 4, 1005-
- 11 1025, 2007.
- 12 Lai, D. Y. F., Roulet, N. T., Humphreys, E. R., Moore, T. R., and Dalva, M.: The effect of
- 13 atmospheric turbulence and chamber deployment period on autochamber CO₂ and CH₄ flux
- measurements in an ombotrophic peatland, Biogeosciences, 9, 3305–3322, 2012.
- Levy, P. E., Gray, A., Leeson, S. R., Gaiawyn, J., Kelly, M. P. C., Cooper, M. D. A., Dinsmore,
- 16 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.
- 18 LI-COR Biosciences: LI-8100A Automated Soil CO₂ Flux System & LI-8150 Multiplexer
- 19 Instruction Manual, online available at: http://envsupport.licor.com/docs/LI-
- 20 8100A Manual.pdf, 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.
- 23 Lloyd, J. and Taylor, J. A.: On the temperature dependence of soil respiration, Funct Ecol, 8,
- 24 315–323, 1994.
- 25 Maier, M. and Schack-Kirchner, H.: Using the gradient method to determine soil gas flux: A
- 26 review, Agr Forest Meteorol, 192–193, 78–95, 2014.
- 27 Millington, R. J. and Quirk, J. P.: Permeability of porous solids, Trans. Faraday Soc., 57, 1200–
- 28 1207, 1961.
- Olander, L. P., Wollenberg, E., Tubiello, F. N., and Herold, M.: Synthesis and Review:
- 30 Advancing agricultural greenhouse gas quantification, Environ Res Lett, 9, 75003, 2014.

- 1 Parkin, T., and Venterea, R.: Chapter 3. Chamber-based trace gas flux measurements, in: Follet,
- 2 R. (ed.): Sampling Protocols, pp. 3-1 to 3-39, online available at:
- 3 www.ars.usda.gov/research/GRACEnet, 2010.
- 4 Pedersen, A. R., Petersen, S. O., and Schelde, K.: A comprehensive approach to soil-
- 5 atmosphere trace-gas flux estimation with static chambers, Eur J Soil Sci, 61, 888–902, 2010.
- 6 Peltola, O., Mammarella, I., Haapanala, S., Burba, G., and Vesala, T.: Field intercomparison of
- 7 four methane gas analyzers suitable for eddy covariance flux measurements, Biogeosciences,
- 8 10, 3749–3765, 2013.
- 9 Petersen, S.O.: Diffusion probe for gas sampling in undisturbed soil, Eur J Soil Sci, 65, 663-
- 10 671, 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.
- 14 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.:
- 17 Comparison of static chambers to measure CH₄ emissions from soils, Agr Forest Meteorol,
- 18 171–172, 124–136, 2013.
- 19 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 CO₂ efflux, Agr Forest
- 23 Meteorol, 123, 159–176, 2004.
- Qu, W., Bogena, 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.
- 26 R Core Team: R: A language and environment for statistical computing. R Foundation for
- 27 Statistical Computing, Vienna, Austria, 2014.
- 28 Roland, M., Vicca, S., Bahn, M., Ladreiter-Knauss, T., Schmitt, M., and Janssens, I. A.:
- 29 Importance of nondiffusive transport for soil CO₂ efflux in a temperate mountain grassland, J.
- 30 Geophys. Res. Biogeosci., 120, 502–512, 2015.

- 1 Rühlmann, J., Körschens, M., and Graefe, J.: A new approach to calculate the particle density
- 2 of soils considering properties of the soil organic matter and the mineral matrix, Geoderma,
- 3 130, 272–283, 2006.
- 4 Ryan, J. A., and Ulrich, J. M.: xts: eXtensible Time Series: R package version 0.9-7, online
- 5 available at: http://CRAN.R-project.org/package=xts, 2014.
- 6 Savage, K., Phillips, R., and Davidson, E.: High temporal frequency measurements of
- 7 greenhouse gas emissions from soils, Biogeosciences, 11, 2709–2720, 2014.
- 8 Schipper, L. A., Hobbs, J. K., Rutledge, S., and Arcus, V. L.: Thermodynamic theory explains
- 9 the temperature optima of soil microbial processes and high Q10 values at low temperatures,
- 10 Glob Change Biol, 20, 3578–3586, 2014.
- 11 Schneider, J., Kutzbach, L., Schulz, S., and Wilmking, M.: Overestimation of CO₂ respiration
- 12 fluxes by the closed chamber method in low-turbulence nighttime conditions, J. Geophys. Res.,
- 13 114, G03005, 2009.
- 14 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,
- 16 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.
- 19 Vargas, R., Carbone, M., Reichstein, M., and Baldocchi, D.: Frontiers and challenges in soil
- 20 respiration research: from measurements to model-data integration, Biogeochemistry, 102, 1–
- 21 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., Baker, J. M.: Effects of soil physical nonuniformity on chamber-based gas flux
- 25 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 CO₂ efflux in a bioenergy
- 29 plantation with fast-growing *Populus* trees influence of former land use, inter-row spacing
- 30 and genotype, Plant Soil, 369, 631–644, 2013.

- 1 Wang, K., Liu, C., Zheng, X., Pihlatie, M., Li, B., Haapanala, S., Vesala, T., Liu, H., Wang, Y.,
- 2 Liu, G., and Hu, F.: Comparison between eddy covariance and automatic chamber techniques
- 3 for measuring net ecosystem exchange of carbon dioxide in cotton and wheat fields,
- 4 Biogeosciences, 10, 6865–6877, 2013.
- 5 Weylandt, M. R.: xtsExtra: Supplementary Functionality for xts: R package version 0.0-1/r824,
- 6 online available at: http://R-Forge.R-project.org/projects/xts/, 2014.
- Wood, S. N.: Generalized Additive Models: An Introduction with R, Chapman and Hall/CRC,
- 8 2006.

- 9 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 CO₂ flux chamber and the ambient air, J.
- 11 Geophys. Res., 111, D08S10, 2006.
- 12 Zeileis, A. and Grothendieck, G.: zoo: S3 infrastructure for regular and irregular time series, J
- 13 Stat Softw, 14, 1–27, 2005.
- Zenone, T., Zona, D., Gelfand, I., Gielen, B., Camino-Serrano, M., and Ceulemans, R.: CO₂
- uptake is offset by CH₄ and N₂O emissions in a poplar short rotation coppice, Glob Change
- 16 Biol Bioenerg, doi: 10.1111/gcbb.12269, 2015.
- 27 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, 2014.

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

		Greenhouse Gas Monitoring System AGPS	LI-8100A Automated Soil CO ₂ Flux System
Chamber	Dimensionsa	200 cm L x 80 cm W x 50 cm H	48 cm L x 38 cm W x 33 cm H
	Headspace volume	25000 cm ³ , square	4076 cm ³ , 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	20 cm long tube on the outside, 1 cm I.D. ^b	special vent design ^c
	Fan	no	no
Collar	Dimensions	48 cm x 48 cm ^d /58 cm x 58 cm ^e	20.3 cm I.D./21.3 cm O.D.
	Enclosed soil area	2304 cm^2	318 cm^2
	Insertion depth	~ 3 cm	~ 7 cm
	Offset ^f	2.1±0.7 cm	4.1±1.1 cm
	Material	stainless steel	PVC, green
Tubingg	Length	11 – 25 m	15 m
8	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 ^g /0.4-0.5 lpm ^h	2.4-2.9 lpm ^g /1.7 lpm ^h
Multiplexer pump		diaphragm	diaphragm
Gas analyser	Principle	Off-Axis Integrated Cavity Output Spectroscopy	Non-dispersive Infrared
	Measurement range	200-4000 ppm ⁱ , 7000-70000 ppm ^j	0-20000 ppm ⁱ , 0-40 mmol/mol ^j
	Uncertainty	total uncertainty: <0.25 % of reading ^{i,j,m}	accuracy: 1.5 % of reading ^{i,j}
		precision ⁿ CO ₂ : 150 ppb	RMS noise CO ₂ : <1 ppm ^k
		precision ⁿ H ₂ O: 100 ppm	RMS noise H ₂ O: <0.01 mmol/mol ¹
Total gas volume		30294-33719 cm ³	5372-6294 cm ³
Operational range	Chamber	>2 °C, RH: non-condensing°	-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	SPADE ^p , DS18B20 digital thermometer, accuracy ±0.5 °C	thermistor, accuracy ±1.0 °C
	Soil moisture	SPADE ^p , ring oscillator, relative accuracy ±4 %	Decagon ECH ₂ O model EC-5, ±3% VWC, most mineral soils
	Air pressure	not implemented	1.5 % accuracy
Power requirement		max. 2000 W	max. 60 W

 $^{^{\}rm a}$ the entire supporting structure, not only the chamber itself, $^{\rm b}$ according to Parkin and Venterea (2010); unlike the LI-8100A vent, this one was not specifically tested, $^{\rm c}$ Xu et al. (2006) $^{\rm d}$ internal, $^{\rm e}$ rim included, $^{\rm f}$ collar height above the soil surface, $^{\rm g}$ chamber to multiplexer, $^{\rm h}$ multiplexer to gas analyser, $^{\rm i}$ CO₂, $^{\rm j}$ H₂O, $^{\rm k}$ at 370 ppm with 1 s signal averaging, $^{\rm n}$ without calibration, $^{\rm n}$ 1-sigma, 5 s signal averaging, $^{\rm o}$ 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, ^p 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. ID: inner diameter.

1 Table 2. Number of discarded CO₂ fluxes after each filter step for the two automated chamber systems (LIN = linear fit, EXP = exponential fit, SSNLin = normalized sum of squares of 2 3 residuals for linear fit, $\Delta Tair =$ change in air temperature inside the closed chamber during the 4 closure time, ΔCO_2 = difference in the atmospheric CO_2 concentration 5 cm above the collar directly before chamber closure and after a deadband of 1 min, RH = relative humidity, NA = 5 information not available for that chamber system). Datasets were grouped by time of the day 6 7 and stability of the atmospheric CO₂ concentration at 50 cm above the soil surface. Day and 8 night were based on sun rise and sunset times. Atmospheric CO₂ concentration was considered 9 as constant when the standard deviation for a 3-min measurement prior to the chamber closures 10 was ≤ 1.0 ppm. The AGPS total closure time was 10 min. Fluxes were once calculated for the 11 first 4 min of the closure time (left of the vertical line) and once for 9 min closure time (right 12 of the vertical line), each with a 1 min deadband.

	Tot	tal	Day (co	nstant)	Day (fluc	tuating)	Night (co	onstant)	Night (fluctuating)		
	LIN	EXP	LIN	EXP	LIN	EXP	LIN	EXP	LIN	EXP	
AGPS											
Unfiltereda	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	
$SSN_{Lin} > 1.0 ppm$	1717 3510	1165 719	321 699	276 223	858 1583 577 29		86 215	58 46	452 1013	254 152	
-0.5 $<$ $\Delta T_{air} >$ 1.0 $^{\circ}C$	146 192	138 3	29 14	29 0	44 33	44 33 36 2		2 0	73 130	71 1	
$\Delta CO_2 < 0.0 \text{ ppm}$	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	
$SSN_{Lin} > 1.0 ppm$	201	191	66	31	68	69	13	21	54	70	
$-0.5 < \Delta T_{air} > 1.0 ^{\circ}C$	1328	1102	533	263	663	386	11	24	121	429	
$\Delta CO_2 < 0.0 \text{ ppm}$	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	

^a 21 measurements discarded prior to the filtering because of missing air temperature measurements for the flux calculation

13

14

Table 3. Number of measurements (N), 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_0 , 15.04 – 31.08.2014), the open canopy phase (OC, 15.04 – 30.06.2014) and the closed canopy phase (CC, 01.07 – 31.08.2014), 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	1	V	E_{θ}			R_{I0}			RSE			Average cSR		
			Е	OC	Е	OC	CC	Е	OC	CC	Е	OC	CC	E	OC	CC
						(K)		(µm	$(\mu mol~CO_2~m^{2}~s^{1})$		(µmo	$(\mu mol\ CO_2\ m^{-2}\ s^{-1})$			$(g CO_2 m^{-2})$	
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)

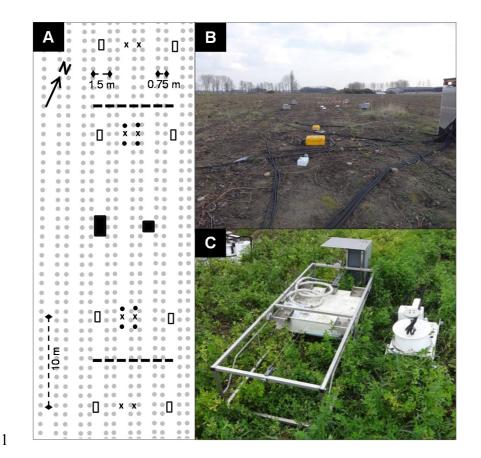


Figure 1. Schematic drawing of the field site (A), view of the southern half of the field site on 25 March 2014 shortly after the harvest (B), and size comparison of an AGPS chamber (left, chamber open) and a LI-8100A chamber (right, chamber closed) (C). In A, the big black-filled rectangle shows the location of the housing for the 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.

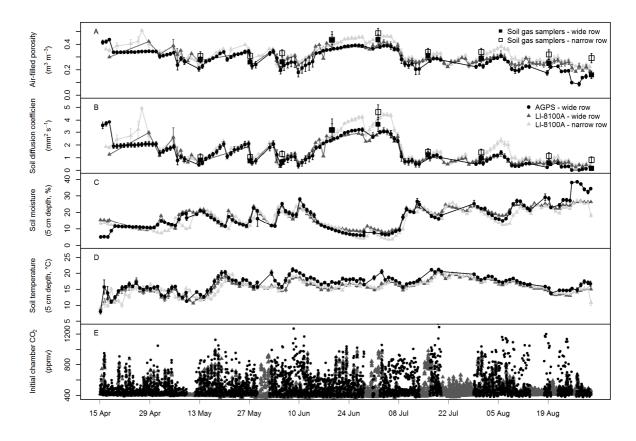
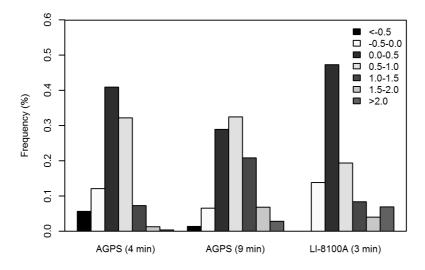


Figure 2. Comparison of different environmental variables measured by the AGPS and the LI-8100A before each chamber closure. Panel A – D show daily averages with their respective standard deviations. For air-filled porosity and the soil diffusion coefficient only measurements have been included for which 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 CO_2 concentration (panel E) which is equivalent to the CO_2 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.



2 Figure 3. Air temperature change inside the chambers during the closure time. For the AGPS,

- 3 temperature change is shown for the first 4 min of the closure time and for 9 min closure time.
- 4 The LI-8100A had a closure time of 3 min.

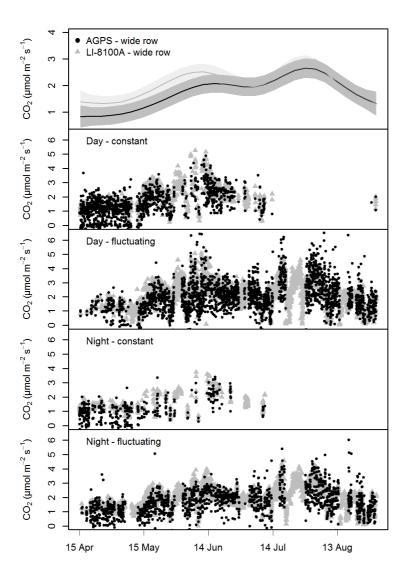
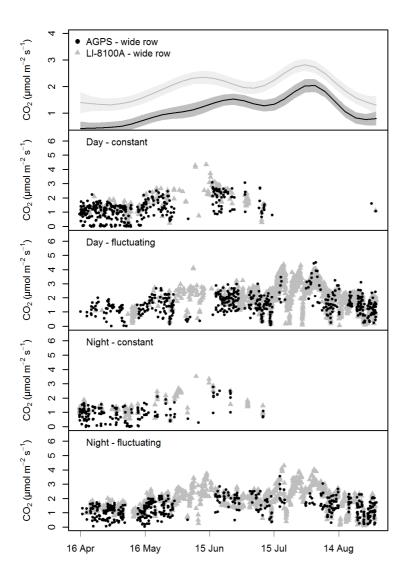


Figure 4. Unfiltered chamber CO_2 flux datasets for the entire monitoring period (wide rows only). The top panel shows the average daily CO_2 flux and its respective 95 % confidence interval for each chamber system estimated using generalized additive models (GAM) (deviance explained: AGPS 64.9 % (n=896), LI-8100A 58.6 % (n=527). In the other panels, the single measured CO_2 fluxes over time were grouped by time of the day and stability of the atmospheric CO_2 concentration at 50 cm above the soil surface. The datasets were divided into day and night based on sun rise and sunset times. Atmospheric CO_2 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 CO_2 fluxes were calculated from the first 4 min of the closure time (including 1-min deadband).



2 Figure 5. Filtered chamber CO₂ flux datasets for the entire monitoring period (wide rows only).

- 3 Modelling and grouping of the data is the same as in Fig. 4 (deviance explained for the GAM:
- 4 AGPS 71.1 % (n=582), LI-8100A 57.0 % (n=526)).

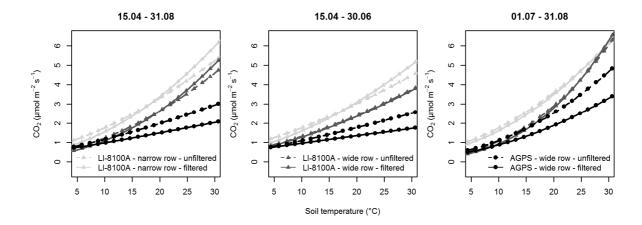


Figure 6: The Lloyd and Taylor model fitted with the different CO_2 flux datasets for the entire monitoring period (15.04 – 31.08.2014), and separately for the open and closed canopy phase (15.04 – 30.06.2014 and 01.07 – 31.08.2014 respectively) using soil temperature at 5 cm depth.

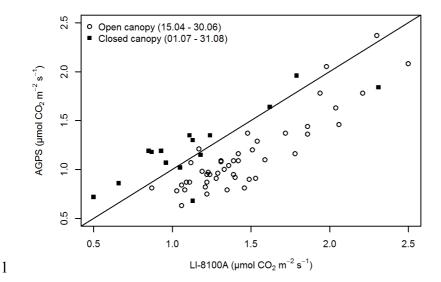


Figure 7. Direct comparison of average CO_2 fluxes obtained with the automated chamber systems LI-8100A and AGPS. AGPS fluxes were averaged for each complete measurement cycle which consisted of eight chambers run in sequence within a 4 hour window. Only those 4 hour windows were included in the figure where at least five of the eight chambers passed the quality control protocol. Filtered LI-8100A fluxes were averaged for the matching 4 hour windows (n=4-8). Standards errors varied between 0.08 and 0.37 μ mol CO_2 m⁻² s⁻¹ for the AGPS, and between 0.03 and 0.34 μ mol CO_2 m⁻² s⁻¹ for the LI-8100A, respectively.