

## Cross-calibration functions for soil CO<sub>2</sub> efflux measurement systems

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**Abstract** – Different soil CO<sub>2</sub> efflux measurement systems and methodologies were used to estimate the annual soil respiration of different forest sites. To allow comparison between these annual values, this study aimed to cross-calibrate five soil CO<sub>2</sub> efflux ( $R_S$ ) closed dynamic chamber systems, and compare the in situ measurement methodologies. We first assessed the impact of the measurement methodology on  $R_S$  by studying the effects of three parameters: record duration, time lag before starting to record and the mode of chamber-soil contact (use of collars or insertion of the chambers into the soil). Secondly, we directly compared systems with identical methodology during field measurements on three forest sites. We observed a significant influence of the chamber-soil contact mode (no impact of the record duration and duration before starting to record). Measurements obtained by insertion led to significantly higher estimates of  $R_S$  than those obtained using collars (up to 28%). Our inter-comparison showed that deviations existing between in situ measurements performed with the different systems were partly systematic and could be corrected using simple linear equations. Measurements of pressure difference between the inside and the outside of soil chambers allowed explaining a part of the observed deviations between systems. Finally, we assessed the influence of the cross-calibration equations on annual respiration of two beech forest soils.

**cross calibration / forest ecosystem / measurement system / pressure effect / soil CO<sub>2</sub> efflux**

**Résumé** – Fonctions d'inter-calibration pour des systèmes de mesure du flux de CO<sub>2</sub> du sol. Différents systèmes et protocoles ont été utilisés pour estimer la respiration annuelle du sol de plusieurs sites forestiers. Afin de comparer ces valeurs annuelles, cette étude avait pour but d'inter-calibrer cinq systèmes de mesure du flux de CO<sub>2</sub> du sol ( $R_S$ ) à chambre fermée dynamique, et de comparer in situ les méthodes de mesure. Nous avons évalué dans un premier temps l'impact la méthodologie de mesure sur  $R_S$  à travers trois paramètres : la durée de mesure, le délai avant de commencer la mesure et le mode de contact chambre-sol (utilisation de colliers ou insertion de la chambre). Dans un second temps, nous avons comparé directement les systèmes au cours de trois campagnes de mesures sur le terrain dans trois sites forestiers. Nous avons trouvé une influence significative du mode de contact chambre-sol, mais pas d'impact des paramètres de mesure. Les mesures obtenues par insertion de la chambre donnent des estimations plus importantes de  $R_S$  que celles obtenues par utilisation de colliers (jusqu'à 28 %). Notre inter-comparaison a montré que des écarts entre des mesures faites avec les différents systèmes sont partiellement systématiques, et pourraient être corrigés par des équations linéaires simples. Les mesures de différences de pressions entre l'intérieur et l'extérieur de la chambre ont permis d'expliquer en partie les écarts observés. Finalement, l'influence des équations d'intercomparaison sur la respiration annuelle du sol de deux hêtraies est présentée.

**écosystème forestier / effet de pression / flux de CO<sub>2</sub> du sol / inter-calibration / système de mesure**

### 1. INTRODUCTION

After photosynthesis, soil CO<sub>2</sub> efflux ( $R_S$ ) is the second CO<sub>2</sub> forest flux of importance [20] and soil constitutes the major carbon reserve in terrestrial ecosystems [4]. Even small  $R_S$  responses to global climatic change can induce important variation in CO<sub>2</sub> atmospheric concentration [26]. Accurate measurements are required for improving the understanding of the soil respiration process and its modeling.

Different methods have been used for  $R_S$  measurements, such as static chamber systems (alkali solution, soda lime

[1]), closed or open dynamic chambers connected to infrared gas analyzers (IRGA) [18,21], eddy covariance measurements below the canopy [12] and soil CO<sub>2</sub> concentration gradient analysis [23]. The choice of the measurement system mostly depends on the specific spatial and temporal resolution requested, as underlined for chamber techniques by Savage and Davidson [24].

This study only includes closed dynamic chamber systems (CDC). Indeed, these systems are easy portable allowing a high number of measurement repetitions and therefore are able to integrate the intra-plot spatial variability. When frequent measurement campaigns are performed within a stand (every

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week or every two weeks), these systems are also able to capture the seasonal variation of  $R_S$  [5, 22]. They are most appropriate to estimate the annual soil respiration of plots and, thus, can be used for the comparison of annual respiration between different forest sites.

Five different CDC systems are usually employed to measure the  $R_S$  on the different sites taking part in our plot comparison project. The  $R_S$  data recorded with these systems cannot be directly compared since the inter-plot variability can be masked by significant systematic deviations already observed among the different closed dynamic systems in number of studies [9, 13], mainly due to differences in air circulation and pressure conditions in the chamber headspace. It had also been shown that (i) most of these deviations were linear and (ii) correction coefficients could be applied for more accurate comparisons of soil  $\text{CO}_2$  efflux values, revealing the necessity of cross-calibrations [10, 11, 19]. Unfortunately, each of these studies has been performed on one single site or using a calibration device in standardized conditions. In addition, the influence of site characteristics as soil moisture, soil type or texture on deviations between systems has not yet been taken into account [14, 19].

In addition to the soil chamber type, the soil-chamber contact mode (i.e. inserted into soil or laid on a pre-inserted collar) is another methodological point that has been well discussed [11, 17]. Both of these methods present advantages and drawbacks. The direct insertion of a chamber into the soil potentially disturbs the litter-soil layer at a short-term (within 24 h) and does not allow multiple measurements in the same location, but it allows a large number of measurements. The use of collars is suspected to cut the litter and superficial soil fine root networks and thus to suppress a significant part of the root respiration [11, 25] whilst it avoids the short-term soil disturbance as the collar is inserted several days before measurements. Despite these considerations, both methods are still used.

In this study we aimed to compare five CDC systems and three measurement methodology parameters. The objective was to establish cross-calibration functions between the different systems and methodologies usually used by the team involved in this paper. We first tested the impact of measurement methodology on  $R_S$  measured, and focused on the soil-chamber contact mode. In a second step, we cross-calibrated these systems on three sites that mainly differ in their soil and humus type and tree-species compositions. Comparing systems and methodologies under different site conditions provided calibration functions, which can be used to correct the systematic divergences among the tested systems, also taking into account the influence of the site characteristics. In the final stage, annual soil respiration values of the forests studied were compared after the use of cross-calibration functions to erase the experimental set up impact.

## 2. MATERIALS AND METHODS

### 2.1. Sites

Our study was conducted in three forests sites. The Vielsalm forest (Belgium, 50° 18' N, 6° 00' E) and the Hesse forest (France, 48°

40' N, 7° 05' E) were described in Aubinet et al. [2] and in Granier et al. [6], respectively. Soil type in Vielsalm is classified as dry-stic cambisol (FAO classification) covered by a moder humus type. Soil type in Hesse is a stagnic luvisol covered by an oligo-mull humus type. The third site located in the Chaux forest (France, 47° 07' N, 05° 42' E) in a mixed deciduous stratified stand; the dominant species are oaks (*Quercus robur* L., *Quercus petraea* (Mattuschka) Liebl.), and several other deciduous species as *Carpinus betulus* L., *Fagus sylvatica* L., *Populus tremula* L. and *Betula verrucosa* Ehrh. are mainly in coppice. Mean annual temperature and precipitations averages 10.3 °C and 950 mm respectively. The soil is a gleyic luvisol with a meso-mull humus type.

### 2.2. Presentation of tested systems

There were five CDC systems involved in our cross-calibration experiment. The principle of the closed dynamic system is to calculate the soil  $\text{CO}_2$  efflux from the rate of increase of the  $\text{CO}_2$  concentration in a chamber that is hermetically in contact with a small area of soil [11]. The five systems were divided into two groups. The first group was made up of three systems based on the Licor company products (Licor, Lincoln, USA):

- the “Li-Gx” system (Faculté Universitaire des Sciences Agronomiques, Gembloux), described by Longdoz et al. [16] consisted of a Li-6252 (Licor, Lincoln, USA) IRGA connected to a homemade respiration chamber (185 mm height × 80 mm diameter) built following Norman et al. [18],
- the “Li-He” and “Li-62” systems (Unité Écologie et Écophysiologie Forestières, Nancy) both consisted of a Li-6252 IRGA connected with a Li 6000-9 chamber (Licor, Lincoln, USA).

For these three systems, the pump of the Li-6252 (flow rate 1.5 L min<sup>-1</sup>) provides air circulation inside the chamber and drives the air inside the chamber by a drilled ring that allowed air mixture and homogenization. The second group consisted of two systems based on the PP-systems company products (PP-Systems, Hutchin, UK):

- the “PP-Ch” system (Université de Franche-Comté, Besançon) consisted of a upgraded EGM-4 IRGA connected with a modified version of the SRC-1 chamber including a metal mesh in the lower part of the chamber,
- the “PP-Or” system (Université Paris-Sud XI, Orsay) consisted of a upgraded CIRAS-1 IRGA connected to the first version of the SRC-1 chamber that is not equipped with the metal mesh.

In these two last chambers, a vertical fan provided air mixture and homogenization inside the chamber. The flux data provided by all systems were compared to those from the “Li-62” that has been arbitrarily chosen as the common comparative system at each site.

### 2.3. Methodology parameters tested during the intercomparison

The methodology measurement usually used by each operator for the  $R_S$  survey of their forest plots (therefore called “particular protocol”) differs by (a) the time-lag between the placement of the chamber and the beginning of the record of the  $\text{CO}_2$  increase (defined as the “waiting time”), (b) the duration of this record and (c) the chamber-soil contact mode. These three modalities are set up by the operators and are not imposed by the system itself.

**Table I.** Description of the modalities used during the methodology comparison. Particular protocols corresponded to the protocols usually used by the teams for their studies of the temporal variations of soil respiration. For the soil-chamber contact mode, the chamber can be directly inserted in the soil (“Inserted”) or laid on collars (“Collar”).

	Particular protocols					Common Protocol
	“Li-62”	“Li-Gx”	“Li-He”	“PP-Ch”	“PP-Or”	
“Waiting time”	15 s / 15 ppm	10 s / 15 ppm	15 s	10 s	10 s	15 s
Recording	60 s	90 s / 30 ppm	60 s	76 s	76 s	76 s
Soil-Chamber contact	Collar	Collar	Inserted / Collar	Inserted / Collar	Collar	Collar

## 2.4. In situ comparisons

Four campaigns of soil respiration measurements took place at the Vielsalm (May 2003), Chaux (June 2004) and Hesse (mid and end of September 2003) forest sites. These campaigns, aiming at the comparison of methodologies and systems, were divided into two steps. The first step dealt with the impact of the different measurement methodologies on  $R_S$  values as related to the system characteristics and the operators. The second step comprised the system comparisons and cross-calibrations.

### 2.4.1. Step 1: Methodology comparisons

Table I shows the different parameters tested for each system. The “waiting time” varied from 10 to 15 s or the record started when  $\text{CO}_2$  concentration in the chamber was 15 ppm below the ambient concentration by using a soda lime scrub for the Licor systems.

The rate of  $\text{CO}_2$  concentration increase was recorded during a constant time that ranged from 60 s to 120 s or during an increase of 30 ppm or 50 ppm of the  $\text{CO}_2$  concentration in the chamber [11] (Tab. I). The impact of the “waiting time” and the record duration was tested at Vielsalm forest campaign for all systems except for the “PP-Or” system. For this purpose, measurements performed with the different particular protocols were compared to measurements obtained with a common protocol (Tab. I) employed by all systems. For this comparison,  $R_S$  was measured on 24 PVC collars (60 mm height, 119 mm diameter) inserted into the soil (15 mm depth) 2 weeks before measurements in order to avoid  $R_S$  measurements perturbations.

Two chamber-soil contact modes were used. The chamber can be either directly inserted in the soil or laid on collars pre-inserted in the soil. The chamber-soil contact mode was only assessed for both, “PP-Ch” and “Li-He” systems, because the three other systems were never used with the insertion mode (Tab. I). We compared two successive flux measurements, the first one performed with the chamber laid on a larger PVC collar (60 mm height, 119 mm diameter, a foam gasket ring provided an airtight seal between chambers and the collar), and the second one with the chamber inserted into the soil (15 mm depth) inside the collar. This test was realized at all sites, with 7 to 8 collars per site. The collars were inserted into the soil 2 weeks before measurements in order to avoid  $R_S$  measurement perturbations.

### 2.4.2. Step 2: Cross calibration

The four campaigns for system comparison and cross-calibration were performed in the Vielsalm (May 2003) forest with “Li-62”, “Li-He”, “Li-Gx” and “PP-Ch”, on 12 PVC collars. We performed campaigns in the Hesse forest with “Li-62” and “Li-He” on 23 PVC collars (mid-September 2003), and with “Li-62”, “Li-He”, “Li-Gx” and

“PP-Ch” on 23 collars (end of September 2003). The fourth campaign was performed in the Chaux forest (June 2004) with “Li-62”, “Li-He”, “PP-Ch” and “PP-Or” on 29 PVC collars. The collars were inserted in the forest soil 2 weeks before measurements. A foam gasket ring provided an airtight seal between chambers and the collar. For each system, a  $R_S$  value corresponded to the mean of three measurements on the same collar, the measurements on a same collar being alternated with those made on other collars.

Before the Vielsalm and Chaux campaigns, the pressure difference between the chamber headspace and the atmosphere (PDC) was verified for each system with a FCO42 (Furness Controls Ltd, Bexhill, UK). Each PDC measurement was performed with chambers laid on a collar that was hermetically sealed with a PVC disc. A PDC, induced by a leak in the air circulation circuit, is known to cause extensive measurement errors on  $R_S$  by pressure pumping or blocking [3, 16]. PDC was not checked before the Hesse campaign. Because no significant variation have been observed between the values obtained at Vielsalm and Chaux, the PDC impact can be considered as constant for all the campaign (see Results).

## 2.5. Impact of cross-calibration on annual soil $\text{CO}_2$ efflux

Soil respiration was monitored within the Hesse forest in 2003 and 2004 in three plots every 2–3 weeks on 36 collars in each plots with “Li-He”. Soil temperature at  $-10$  cm ( $T_S$ ) and volumetric soil water content of the 0–6 cm layer ( $\theta_{V\text{Surf}}$ ) were measured simultaneously with  $R_S$  by using homemade copper-constantan thermocouples (Faculty of Agronomy of Gembloux, Belgium) and a capacitive ML2x ThetaProbe (Delta-T Device, Cambridge, UK), respectively. In Vielsalm forest,  $R_S$  was monitored between August 1997 and August 1998 with “Li-Gx” in 29 collars inserted within a beech dominated patch (see [16] for more details). For the Hesse data, an empirical model was fitted to either measured or corrected  $R_S$  values:

$$R_S = R_{S10} Q_{10}^{(T_S-10)/10} e^{-e^{(a-b^* \theta_{\text{Surf}})}} \quad (1)$$

where  $R_{S10}$  is the soil  $\text{CO}_2$  efflux at  $10^\circ\text{C}$ ,  $Q_{10}$  the temperature sensitivity of soil respiration and  $a$  and  $b$  are the parameters for the Gompertz function [9]. For the Vielsalm data, an Arrhenius-type function was fitted to data:

$$R_S = R_{S10} e^{\left\{ \frac{E_a(T_S-283.2)}{283.2RT_S} \right\}} \quad (2)$$

$$\text{with } E_a = \frac{E_{a0} 10^3 T_S}{T_S - T_0}$$

where  $R_{S10}$  is the soil  $\text{CO}_2$  efflux at  $10^\circ\text{C}$ ,  $R$  is the universal gas constant ( $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$ ),  $T_S$  is the soil temperature ( $K$ ),  $T_0$  is a reference temperature and  $E_{a0}$  a reference activation energy ( $\text{J mol}^{-1}$ ).

Following Lloyd and Taylor [15], the parameters  $T_0$  and  $E_{a0}$  were fixed to 227.13 K and 12970 J mol<sup>-1</sup>, respectively [16]. An influence of soil water content was not taken into account, as it had not been observed. We simulated daily  $R_S$  values by applying equations (1) or (2) with continuous measurements of  $T_S$  (Hesse and Vielsalm) and  $\theta_{Vsurf}$  (Hesse). Then these daily values were cumulated for 2003 and 2004 (Hesse) and 1997–1998 (Vielsalm) providing annual soil CO<sub>2</sub> efflux ( $R_{SA}$ ). At Hesse site, equation (1) was fitted to  $R_S$ ,  $T_S$  and  $\theta_{Vsurf}$  datasets measured on 3 plots (HesseA, HesseB and HesseC), leading to one  $R_{SA}$  value per plot. Relationships deduced from cross-calibrations among systems were used to assess the impact of systems on  $R_{SA}$  (see Results). For the Hesse dataset, we converted each value measured by the “Li-He” system into a corrected value that represented  $R_S$  as it would be if measurements were performed with either the “Li-62”, “Li-Gx” or “PP-Ch” systems. For the Vielsalm dataset, we converted each value measured by the “Li-Gx” system into a corrected value that represented  $R_S$  as it would be if measurements were performed with either the “Li-62”, “Li-He” or “PP-Ch” systems. Depending on the site and the measuring system, the corrected values were either fitted to equation (1) or (2), and corrected annual soil respiration ( $cR_{SA}$ ) was obtained following the same procedures as presented above.

## 2.6. Statistical analysis

Linear regressions (Statview 5.0, SAS Institut Inc., N.C., USA) were used to cross-compare data from each system to the “Li-62” system used as the comparative system. Comparisons tests of the mean between the systems, measurement methodologies and campaign were performed by two-way ANOVA tests and post-hoc Fisher’s Protected Least Significant Difference tests. Least square non-linear regression analyses were performed to fit empirical models to  $R_S$  data (Statgraphics Plus 4.1).

## 3. RESULTS

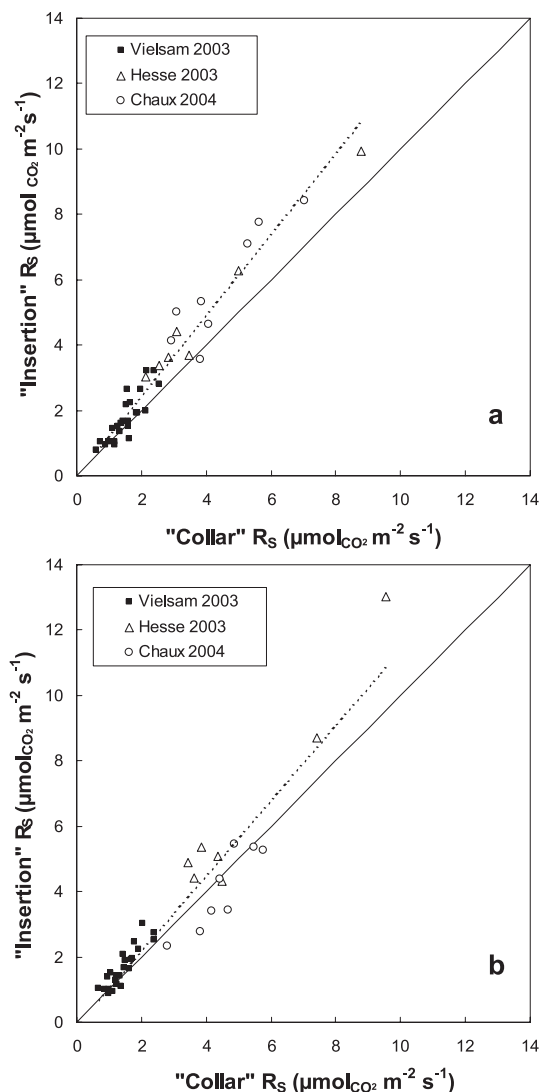
### 3.1. Pressure difference between the chamber headspace and the atmosphere (PDC)

Measurements of PDC in the center of the chambers gave values lower than  $0.05 \pm 0.01$  Pa (mean  $\pm$  standard deviation) in the “Li-62”, “Li-Gx” and “Li-He” systems. PDC values reached  $0.01 \pm 0.001$  Pa in “PP-Ch” and  $0.92 \pm 0.35$  Pa in “PP-Or”.

### 3.2. Impact of the different measurement methodologies (Step 1)

Measurements obtained with the methodologies usually used by the different teams (particular protocol) did not differ significantly (PLSD,  $p > 0.05$ ) from those obtained with the common measurement methodology (common protocol, Tab. I). Thus, during system comparison (step 2), each system measured  $R_S$  with its particular protocol without introducing methodological divergences.

A significant impact of the chamber-soil contact mode (use of “collar” or “insertion” in the soil) was found for the two



**Figure 1.** Comparison between  $R_S$  measured with the chamber laid on collars (“Collar”  $R_S$ ) and  $R_S$  measured with the chamber inserted in the soil (“Insertion”  $R_S$ ) for “Li-He” (a) and “PP-Ch” (b). The solid line is the 1:1 and the dashed line shows the general linear regression on all values. Regression equations are (a) [“Insertion”  $R_S$ ] =  $1.28 \times$  [“Collar”  $R_S$ ] ( $R^2 = 0.95$ ;  $n = 39$ ); (b) [“Insertion”  $R_S$ ] =  $1.15 \times$  [“Collar”  $R_S$ ] -  $0.13$  ( $R^2 = 0.91$ ;  $n = 39$ ).

tested systems (“Li-He” and “PP-Ch”). Figure 1a shows a significant linear relationship between “collar” and “insertion” values for “Li-He”, when the data from the different campaigns were regrouped. The “insertion” values gave fluxes 28% higher than “collar” values. A similar result was observed for “PP-Ch” with an increase due to insertion amounting to between 2% and 13% (Fig. 1b) when “collar” values ranged from  $1 \mu\text{mol}_{\text{CO}_2} \text{m}^{-2} \text{s}^{-1}$  to  $6 \mu\text{mol}_{\text{CO}_2} \text{m}^{-2} \text{s}^{-1}$  (range usually measured, data not shown). However, when considering the campaign separately, the “PP-Ch” system showed a decrease of about -11% when passing from “collar” to “insertion” during the Chaux campaign. This decrease has been verified for this site during other campaigns (data not shown).

**Table II.** Mean soil respiration efflux ( $R_S$ , in  $\mu\text{mol}_{\text{CO}_2} \text{m}^{-2} \text{s}^{-1}$ ) of the five tested systems for the three campaigns ( $n = 12$ ,  $n = 46$  and  $n = 30$  for Vielsalm, Hesse and Chaux sites respectively) and mean soil temperature ( $T_s$ , in  $^{\circ}\text{C}$ ) during the measurement duration. Values in brackets are the corresponding standard errors. During the Hesse Campaign each collar was measured twice.

Site	$R_S$ “Li-62” “Li62”	$R_S$ “Li-Gx”	$R_S$ “Li-He”	$R_S$ “PP-Ch”	$R_S$ “PP-Or”	$R_S$ Mean	$T_s$
Vielsalm	1.54 (0.18)	1.50 (0.17)	1.59 (0.17)	1.55 (0.15)	–	1.53 (0.08)	7.7 (0.07)
Hesse	1.82 (0.08)	2.17 (0.15)	1.80 (0.09)	2.37 (0.13)	–	1.98 (0.06)	11.8 (0.20)
Chaux	3.93 (0.18)	–	3.87 (0.17)	4.68 (0.27)	5.21 (0.40)	4.42 (0.14)	16.0 (0.07)

**Table III.** Linear regression parameters between  $R_S$  values of the relationship:  $R_{SX} = A \times R_{SLi-62} + B$ , where  $R_{SX}$  and  $R_{SLi-62}$  are the  $R_S$  value of the “X” system and the  $R_S$  value given by “Li-62”, respectively. The regression analysis was performed on the pooled data from the three campaigns. Each point represents the mean of the three replicates made on each collar. For “Li-He”, additional data from 23 other collars were added. Each parameter was significant for  $p = 0.05$  level (NS indicates non-significant parameter in the regression analysis).

Parameters	“Li-Gx”	“Li-He”	“PP-Ch”	“PP-Or”
$A$	1.03	0.96	1.21	1.84
$B$	0.15	NS	NS	–2.05
$R^2$	0.58	0.94	0.92	0.69
$n$	53	110	83	29

### 3.3. System cross calibration (Step 2)

Table II summarizes the mean  $R_S$  values measured with the different systems for each campaign. At the Vielsalm site, mean  $R_S$  values did not differ among systems. At the Hesse site, the “PP-Ch” system measured significantly higher mean  $R_S$  values ( $p < 0.05$ ) than the other systems. At the Chaux site, a significant difference of mean  $R_S$  values was found ( $p < 0.05$ ) among systems, but the “Li-62” and “Li-He” systems did not record significantly different mean  $R_S$  values. When considering the variability among sites, the “Li-Gx” and “PP-Ch” systems recorded mean  $R_S$  values that significantly differed among the three sites (Fisher’s PLSD,  $p < 0.05$ ) whereas both, “Li-62” and “Li-He” systems, recorded a significantly higher mean  $R_S$  value ( $p < 0.0001$ ) at Chaux compared to the other sites. Significant differences among mean values of  $R_S$  calculated with all the systems were also observed among sites ( $p < 0.0001$ ).

Figure 2 shows the linear relationship between the  $R_S$  values of different systems and those of “Li-62”. The values for slopes (Tab. III) revealed very low deviations for “Li-He” and “Li-Gx”. However the relationship between “Li-62” and “Li-Gx” measurements presented a larger variability than with “Li-He”, in addition with a significant intercept from the regression analysis found for “Li-Gx”. Considering the same system characteristics, values given by “Li-He” were very close to those of “Li-62”. Figure 2 shows a higher deviation for “PP-Ch” and “PP-Or”. For “PP-Ch”, a constant discrepancy of about 20% was found. According to the higher mean  $R_S$  value recorded by “PP-Or” at the Chaux site, the slope revealed a high deviation from “Li-62” (Tab. III).

### 3.4. Annual soil $\text{CO}_2$ efflux

Actual annual estimates of  $R_S$  calculated directly from measurements ( $R_{SA}$ ) exhibited pronounced spatial variability among plots and sites (Tab. IV), even if  $R_{SA}$  values were not determined for the same time periods in both sites. Annual estimates of  $R_S$  from corrected values ( $cR_{SA}$ ) for the “Li-62” were very close to annual estimates from  $R_S$  values measured in Hesse A, Hesse B and Hesse C plots. Deviations for a given “X” system were calculated as: Deviation “X” =  $(A_X - A_{Li-62})/A_X \times 100$ , where  $A$  is the actual or corrected annual soil respiration of the “X” system and  $A_{Li-62}$  refers to the corrected annual soil respiration for the “Li-62” system. Deviations for the “Li-He” were very low (between  $-0.6\%$  and  $-0.15\%$ , Tab. IV) despite the different site characteristics. Deviations for the “Li-Gx” were higher (between  $+8.2\%$  and  $+21\%$ ). Deviations for the “PP-Ch” were relatively constant (around  $+19\%$ ) but higher than for the “Li-He”.

## 4. DISCUSSION

### 4.1. PDC

Preliminary PDC measurements showed that there were no major pressure differences in the tested systems except for the “PP-Or” system. The higher PDC value obtained for “PP-Or” (overpressure of  $0.92 \pm 0.351$  Pa) was probably due to the air mixing by a fan placed inside the chamber. Following the PDC influence on  $R_S$  found by Longdoz et al. [16] for the Vielsalm forest soil, the impact of “PP-Or” overpressure leads to a blockage of the  $R_S$  flux and corresponds to a  $R_S$  underestimation of 69%. However a higher  $R_S$  values is measured with “PP-Or” compared to those of “Li-62”. This discrepancy could be attributed to the presence of a possible negative PDC (leading to a  $R_S$  overestimation) measured between the atmosphere and the points located near the collar walls. Another explanation would be an excessive turbulence within the SRC-1 chamber due to the fan [7]. This action is prevented in “PP-Ch” by the addition of the metal mesh at the bottom of the chamber. Our results confirmed that the addition of a metal mesh in the SRC-1 chamber was a benefit. “PP-Ch” and Licor-based systems did not induce any pressure pumping or blocking effects, and gave a good confidence in the air tightness of these systems. As a consequence, the comparisons of measurement methodologies and systems were realized for these four systems without any biases coming from a pressure problem. Indeed, if closed dynamic chamber techniques are

unable to reproduce wind conditions prevailing in the forest floor and boundary layer conditions inside the chamber [17], it seems that the unmodified SRC-1 configuration implies artificial and unrealistic conditions. In previous tests, measurements of mean wind speed inside the chamber gave  $0.9 \text{ m s}^{-1}$  [13] and  $0.13 \text{ m s}^{-1}$  (unpublished data) for the unmodified and modified version of the SRC-1, respectively. This large difference in wind speed and the PDC could explain the  $R_S$  values divergence between “PP-Or” and the other systems. Such deviations between systems using the first version of SRC-1 and Li 6000-9 chambers have already been reported [10, 13, 18].

#### 4.2. Chamber-soil contact mode

The chamber-soil contact mode has a significant impact on the  $R_S$  measurements. Three hypotheses could explain the general higher  $R_S$  values obtained for the “insertion” mode. First, a transient change of diffusion conditions of the  $\text{CO}_2$  in the litter and the upper mineral soil layers might occur when inserting the chamber. For the soil types dealt with in this study, the insertion would have perturbed the soil aggregates and leaf litter structure increasing the vertical diffusivity coefficient, thus, inducing a rapid release of upper soil  $\text{CO}_2$ . Second, the collar placement could affect  $R_S$  measurements over the long-term due to the cutting of fine roots [8, 11]. Wang et al. [24] showed that a reduction in  $R_S$  values for a larch forest occurs when measurements were performed at least 12 h after collar installation. Third, the distance between the soil and the drilled ring (“Li-He”) or the fan (“PP-Ch”) insuring air mixing inside the chambers, differs between the “insertion” and “collar” situation. Since this distance was lower without collars, the thinner boundary layer could have lead to higher  $R_S$  values. This argumentation shows also that both chamber-soil contact modes have advantages and disadvantages and none of them could be considered a reference method.

In opposition to all other observations, the “PP-Ch” system during the Chaux campaign (Fig. 1b) was the only one giving lower values for “collar” than for “insertion”. This has been confirmed by other campaigns at the same site (data not shown). A possible cause is that the “insertion” of the SRC-1 chamber might not have trenched the broadleaf litter layer at Chaux, but only have compressed it. Then air tightness between the chamber’s edge and the soil-litter interface may not have been sufficient, thus, leading to  $\text{CO}_2$  leaks and an underestimation of the fluxes.

The highly significant linear relationships between “collar” and “insertion”  $R_S$  values (Figs. 1a and 1b) suggest that scaling coefficients could be used when “collar” and “insertion” data have to be compared. The difference in the relationship parameters between the two systems tested shows that the scaling coefficient is dependent on the system. Further experiments are needed to recommend coefficients specific for each site (soil type).

#### 4.3. Cross-calibration

The  $R_S$  values measured during the cross-calibration experiments were in good agreement with the range observed during

a seasonal evolution survey performed on each site (data not shown). Differences in mean  $R_S$  values among sites could be partially explained by the influence of soil temperature variation among sites and campaigns, especially for the high  $R_S$  during the Chaux campaign where the soil was exceptionally warm (Tab. II).

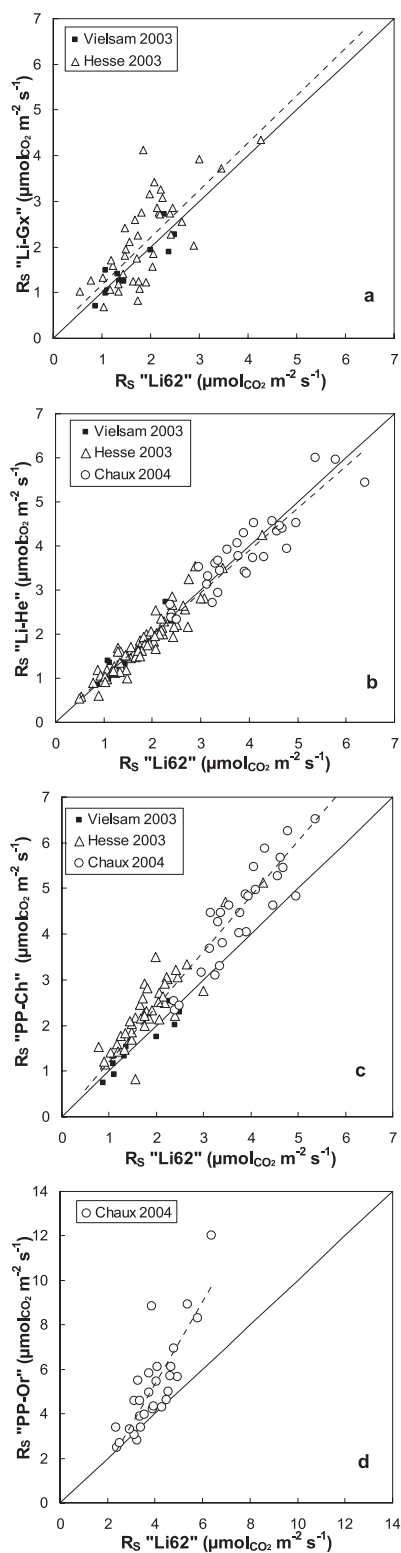
The linear regression presented in the Figure 2 shows that systematic deviations existed between in situ measurements performed with different systems. However these deviations could be corrected with a linear equation, even though the accuracy of corrections depends on the similarity of measurements performed by the different systems, and varies according to the  $R^2$  values.

Logically, when the same system constituted with same material are considered (“Li-62” and “Li-He”)  $R_S$  values are very close in all campaigns. The lower  $R^2$  of the relationship between measurements of “Li-62” and “Li-Gx” may be due to higher measurement variability as a consequence of differences in the foam gaskets assuring the airtight seal of the chambers of these two systems. This may induce a lack of air tightness in one of the two systems when the chambers are placed on a not perfectly horizontal collar’s edge. Consequently an over- or underestimation could be the result, depending on the impact importance of the small PDC or/and potential  $\text{CO}_2$  leak.

The higher  $R_S$  values given by “PP-Ch” compared to those of “Li-62” could be explained by a thicker boundary layer resistance in the Li 6000-9 chamber compared to the SRC-1 modified chamber. In spite of a higher wind speed in the Li 6000-9 ( $0.4 \text{ m s}^{-1}$  [12]) and the presence of a grid mesh in the SRC-1, the position of the drilled ring in the Li 6000-9 chamber might allow airflow to move more parallel to the soil surface compared to the more vertically directed airflow induced by the fan placed in the SRC-1 chamber. Therefore a thicker boundary layer might be more easily induced in the Li 6000-9 chamber. This hypothesis, while explaining the cause of the divergence, does not allow a conclusion on which system offers measuring conditions closer to the natural situation. The slope of the linear regression for the comparison between “PP-Ch” and “Li-62” (1.21, Fig. 2c and Tab. III) is in the range of the results presented by Pumpanen et al. [19] for equivalent systems and performed on a calibration tank (slopes ranged from 1.16 for coarse sand to 1.33 for wet fine sand). Finally, as explained in the first section of this discussion, the PDC problem could explain the large deviation between  $R_S$  measurements of “Li-62” and those of “PP-Or” (Fig. 2d and Tab. III).

#### 4.4. Impact of corrections on annual soil respiration

$R_{SA}$  values calculated directly from measurements exhibited pronounced spatial variability among plots and sites, even when data were cross-calibrated to obtain corrected flux (Tab. IV). However the impact of this cross-calibration on the spatial variability was important. For example, the actual  $R_{SA}$  of Vielsalm that is the higher one became one of the lowest after the cross-calibration for “Li-62”, (Tab. IV). Beyond this spatial variability, the results showed that, logically, the two



**Figure 2.** Comparison of  $R_S$  efflux “Li-Gx” (a), “Li-He” (b), “PP-Ch” (c) and “PP-Or” (d) with “Li-62”. The dashed line represents the linear regression from the overall data set. Equations and main parameters of the regressions are also presented. For analysis conveniences, for “Li-He”, we gathered together data from the two campaigns performed at Hesse (see Materials and Methods).

**Table IV.** In the upper panel: mean annual soil  $\text{CO}_2$  efflux (in  $\text{g}_C \text{m}^{-2}$ ) for 3 plots at Hesse and 1 plot at Vielsalm. Bold values correspond to the annual means that were directly calculated from measurements ( $R_{SA}$  in the text). For other values, the measurements have been transformed (using cross-calibration equations of Tab. III) to simulate the corrected annual mean ( ${}_cR_{SA}$  in the text) that would be obtained with the system listed in the first column. In the lower panel: deviations were calculated as: Deviation “X” =  $(A_X - \text{Li-62})/A_X \times 100$ , with  $A_X$  the actual or corrected annual soil respiration of the “X” system, Li-62 referring to the corrected annual soil respiration for the “Li-62” system.

System	Hesse A		Hesse B		Hesse C		Vielsalm
	2003	2004	2003	2004	2003	2004	1997–1998
Li-62 ( $\text{g}_C \text{m}^{-2}$ )	608	552	700	631	813	742	685
Li-He ( $\text{g}_C \text{m}^{-2}$ )	<b>599</b>	<b>544</b>	<b>696</b>	<b>627</b>	<b>808</b>	<b>737</b>	683
Li-Gx ( $\text{g}_C \text{m}^{-2}$ )	687	638	774	714	885	823	<b>867</b>
PP-Ch ( $\text{g}_C \text{m}^{-2}$ )	747	683	862	782	996	913	828
Deviation “Li-He” (%)	-1.5	-1.4	-0.6	-0.6	-0.6	-0.6	-0.2
Deviation “Li-Gx” (%)	11.4	13.5	9.5	11.7	8.2	9.8	21.1
Deviation “PP-Ch” (%)	18.6	19.2	18.8	19.3	18.4	18.8	17.3

identical systems (“Li-62” and “Li-He”) gave very close annual soil respiration an all plots. The values obtained from the “Li-Gx” system were slightly higher, due to the particularities of this system (air circulation, foam gasket). The difference with the “Li-62” is able to partly mask the natural inter-plot variability, especially when considering Vielsalm forest. Finally, the comparison “PP-Ch” with “Li-62” showed clearly that the discrepancies between two different materials do not allow inter-plot comparison without cross-calibration functions.

### 5. CONCLUSION

We confirmed that the unmodified SRC-1 chamber induced system specific deviations, but confidence in the measured values was improved by including a grid mesh in the chamber (“PP-Ch” system) as proposed by the manufacturer. The discussion on the possible causes of differences among systems and the choice of soil contact mode revealed that properties such as soil texture, soil-litter interface porosity and chamber design influenced  $R_S$  values, with a strong dependence on the study site.

Our study showed that systematic deviations exist among in situ measurements performed with different systems; however these deviations are in the range of the already published results. Deviations were explainable and could be corrected with simple linear equations. Thus,  $R_S$  values obtained with different systems for different study sites can be used to compare soil respiration effluxes after corrections using cross-calibration results. Otherwise, difference of annual soil respiration between sites could be hold against (partly when the compared systems were built one the same model or completely when the compared systems came from different

manufactories) the deviations among the systems used. These deviations could also affect our estimation of the forest annual carbon sequestration because soil respiration data could be used for the NEE correction procedure and deviations are of the same order that the NEE uncertainties.

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