

# Soil Respiration Measurements: A Comparison between Soda Lime Technique and LI-8100A Automated Soil CO<sub>2</sub> Flux System

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## ABSTRACT

Soil respiration (Rs) is one of the major pools in the global carbon cycle. However, there are uncertainties in quantifying Rs rates, partly because of the differences in methods of quantifying this pool. This study compares two widely used methods for measurement of Rs rates, the static closed chamber (SCC) (soda lime) and the automated dynamic closed chamber (DCC) (LI-8100A automated soil CO<sub>2</sub> flux system). Soda lime (SL) results were compared against LI-8100A automated soil CO<sub>2</sub> flux system with the use of continuous long-term chamber (LTC) measurement and survey chamber (SC) techniques. Effects of soil temperature and soil moisture on Rs rates were also assessed. Results showed that Rs rates measured with SL are in good agreement with the LTC ( $R_2 = 0.78$ ) and SC ( $R_2 = 0.59$ ) techniques. The SL technique, however, underestimated Rs by about 23% compared with SC ( $p < 0.001$ ,  $n = 574$ ) and about 20% to 29% compared with LTC ( $p < 0.001$ ,  $n = 48$ ). Rs rates and soil temperature demonstrated strong relationship ( $R_2 = 0.56, 0.64, 0.71$ ), but not with the soil moisture ( $R_2 = 0.38$ ). The soil temperature provides a lot of information about Rs rates; hence, a useful variable in Rs modelling. The derived discrepancy values between the two methods of Rs measurements are useful information that could be used to adjust the values of Rs rates measured with the SL technique.

**Keywords:** soil respiration, carbon balance, dynamic chambers, soil temperature, soil moisture, long-term chamber, survey chamber.

## I. INTRODUCTION

Understanding the processes involved in sequestration and CO<sub>2</sub> emissions is critical in estimating carbon balances. In terrestrial ecosystem, carbon (C) balances are determined as a difference between the amount of stored C (i.e. sequestration) and released C via respiration process. Green plants sequestered C through photosynthesis process, in which product is stored in the above- and belowground biomass of plants and soil organic carbon (SOC). The rates of sequestration and CO<sub>2</sub> emission in soils depend on soil texture and structure, rainfall, temperature, farming system and soil management [1, 2].

Some researchers estimated that about half of the amount of C fixed by photosynthesis is lost through respiration pathways, and much of this emission is sourced from the soil respiration (Rs) [1], constituting about 40 to 80% of the total ecosystem respiration [3, 4, 5, 6]. However, the Rs is one of the main sources of uncertainty in the carbon balance estimates, not only because of the large spatial and temporal variation of Rs rates, but also due to differences in methods of Rs measurements.

Common methods to measure Rs rates include the static closed chamber (SCC), using soda lime (SL) CO<sub>2</sub> absorbent; and dynamic closed chambers (DCC) [7] using soil respiration machines. These methods of Rs measurements are based on the principle that soil CO<sub>2</sub> diffuses from soil due to concentration gradients [8], and in most cases the soil CO<sub>2</sub> concentration is higher than the atmosphere due to the combined respiratory activities from roots (autothropic) and decomposers (heterothropic) [3]. Some authors pointed out that the soil stores about more than two to three times higher than in terrestrial biomass [6, 9, 10, 11, 12, 13]; hence, CO<sub>2</sub> diffuses from soil into the atmosphere. Due to the large contribution of Rs to the global C cycle, quantifying the rate of its emissions with accuracy is critical for insuring reliable estimates of global C balances.

The traditional method of quantifying Rs rates is the soda lime (SL) technique, which is a non-flow-through steady-state chamber system. This technique has been used by many researchers for more than 30 years [14]. It involves the utilization of sodium hydroxide (NaOH) and calcium hydroxide (Ca(OH)<sub>2</sub>) as absorbents of CO<sub>2</sub> emissions [15, 16] from the soil, which are confined within a closed chamber inserted into the soil. In the SL technique, Rs rates are determined based on initial and final weights of soda lime. Because the SL method is inexpensive and easy to employ in the field, it has

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attracted the attention and preference of many researchers, especially those who are interested in soil respiration studies requiring simultaneous monitoring of Rs over large areas and involving large numbers of replicates [4, 16, 17].

In contrast, the DCC method is a system in which air is recirculated between the chamber and an infrared gas analyzer (IRGA), and estimates Rs rates based on rise of internal CO<sub>2</sub> concentration [14, 18]. Among the newest type of closed chamber machines is the *LI-8100A automated soil CO<sub>2</sub> flux system (LI-COR, Lincoln, NE)* or *LI-8100A*, for brevity. The said apparatus employs an automated system of Rs measurement where CO<sub>2</sub> concentration is determined by the Infrared Gas Analyzer (IRGA) [4]. The Rs rates are calculated automatically by a computer based on the rise of internal CO<sub>2</sub> concentration in the chamber [14, 18]. The LI-8100A also allows the unattended and continuous measurement of Rs rates in the field. Furthermore, the it's design has helped in resolving issues encountered with the use previous DCC machines. Such issues include the inadequate mixing of air due to the reduced air exchange between the soil and atmosphere [19]; the modification of the microclimate within the closed chamber due to the exclusion of precipitation and increased temperature inside the chamber [7]; and difference between the amount of pressure inside and outside of a chamber [20].

As a soil respiration machine, the LI-8100A prevents the alteration of the soil microclimate (which is the result of the closure of the chamber during measurements) since its chamber moves automatically from the measurement area when the measurement is not in progress, allowing precipitation in the sampling area and prevents temperature modification within the chamber. The chamber is also equipped with a pressure vent at the top, a feature absent in other DCC machines. The pressure vent ensures the pressure inside the chamber to track the pressure at the soil surface outside the chamber, in both calm and windy conditions. Errors in Rs measurements are minimized when the difference between the pressure inside and outside of the chamber is low and the wind speed inside the chamber matches the ambient wind conditions [20]. Xue et al [21] showed that the new vent design virtually eliminated the occurrence of artifactual negative chamber pressure excursions under windy conditions.

In spite of how the LI-8100A significantly improved the accuracy of Rs measurements, the cost of this machinery is prohibitive, particularly for studies which require large numbers of samples for spatial integration [4]. Given how the cost of acquiring the LI-8100 restricts it's utilization especially in research projects with small budgets; the SL technique, being comparatively inexpensive, remains a good alternative measurement method. It is, however, of equal importance to note how the accuracy of data gleaned via the SL technique has been questioned [17], especially in comparison with the new machinery in dynamic closed chamber methods of measuring Rs rates [22].

Many researchers who have comparatively analyzed the different approaches to the measurement of soil respiration rates reported inaccuracies in the SL technique [e.g. 17, 22] but these researchers have made these comparisons using old DCC machinery with many limitations [19] which have likewise been found to be sources of errors in the estimation of Rs rates. As previously mentioned, the invention of the LI-8100A system with its advanced and sophisticated features has effectively diminished issues regarding the accuracy of Rs estimates gleaned via DCC machinery. Being the state-of-the-art DCC equipment available, it is therefore important to examine and determine the discrepancy in Rs rates between SL and LI-8100A especially if the outcome of such examination are to be used as basis/bases for the improvement of the reliability of SL technique.

Unfortunately, the information about the amount of discrepancy between these two methods remains a gap. The objectives of this study were to compare the two chamber methods (soda lime and LI-8100A automated soil CO<sub>2</sub> flux system) for estimating Rs, and to evaluate the effects of temperature and soil moisture on Rs rates. The authors hypothesized that there are no significant differences in Rs rates between soda lime and LI-8100A techniques. The derived values in this study could be used to adjust the Rs rates values measured with SL technique

## II. METHODS AND MATERIALS

### Site description

This study was conducted at Tully (42° 47' 30" N and 76° 07' 30" W) and Lafayette (48° 52' 42" N and 76° 06' 45" W), Central New York. Both sites have humid continental climate with cold winters and warm summers. Average annual rainfall of about 600 mm rainfall is evenly distributed across the growing season [23, 24, 25, 26], the monthly mean temperature is about 7.9°C, and precipitation is about 847 mm (www.ncdc.noaa.gov). The soil texture in both sites is silt loam [23, 24, 25, 27]. The soil at Tully is well-drained while Lafayette is dominated by well- to moderately well-drained soils, with imperfectly drained patches in low lying topography. To increase the scope and validity of the study, the authors have examined ten experimental plots: eight of these plots were located in Tully and two in Lafayette. Prior to this study, the Tully and Lafayette sites received a variety of treatments (e.g, organic amendments, fertilizer trials, irrigation, tillage, weed control). To avoid treatment effects from previous experiments, measurement plots were established within buffer zones which received no treatments from previous experiments. The willow crops were established from cuttings, spaced 1.5 m between two double rows and 0.6 m within double rows; for single rows, spacing was 1.0 m between two single rows, and 0.6 m within rows [23]. During early spring of 2010, the four willow fields, representing the four ages (i.e 6,

13, 15, 20), were harvested. Half of the newly cut willow fields was allowed to coppice and regrow (continuous production plots) while the other half was terminated by chipping the stool and roots, and then incorporated into the soil (tear-out plots). These sites, together with two additional willows sites (treated with cover crop and without cover crop) planted in 2010, were used as measurement plots in this study.

### Experimental Design

We used a split plot design in this study, with age as whole plot factor and treatment as split-plot factor. All four willow fields (5, 12, 14, and 19-yr. old) were harvested in early spring 2010; half of each field was allowed to coppice and regrow for the entire duration of the study (continuous production (CP)) while the other half was terminated by application of herbicide (Glyphosate at 3 to 5 kg ai ha<sup>-1</sup>) the following spring to re-growing coppices before crushing and mixing the entire stool and roots system into about 20-cm depth (tear-out (TO)) using a Fecon truck mulcher (FTX 350) mounted on a 350 hp tractor. Each treatment was replicated four times in all fields.

### Materials and Equipment

Closed chambers with passive trapping of respired CO<sub>2</sub> in alkali (soda-lime) were used to trap the CO<sub>2</sub> emitted from soil. The researchers used a granular (3 mm dia x 3-12 mm long) sodasorb CO<sub>2</sub> absorbent (W.R. Grace & Company, Chicago, IL)

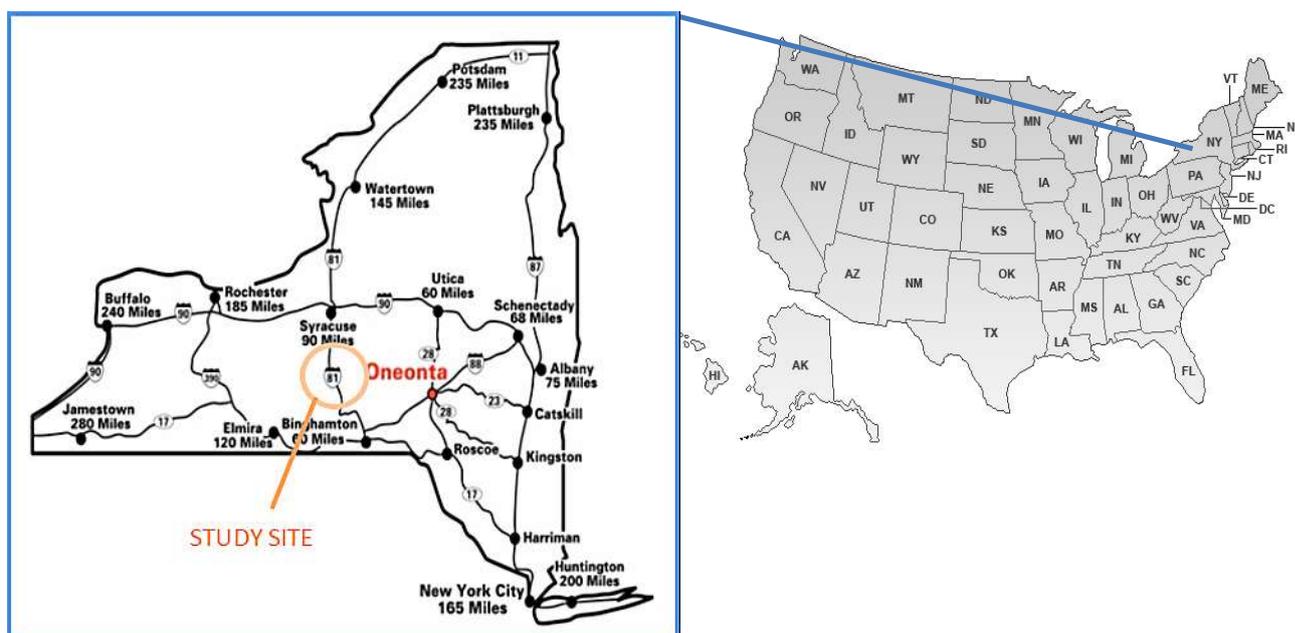


Figure 1. Location of the study sites in Tully and Lafayette, New York State, USA.

consisting of NaOH and Ca(OH)<sub>2</sub> with about 20% absorbed water (**Figure 2a**), which is a requirement for an effective chemical absorption of CO<sub>2</sub> to form Na<sub>2</sub>CO<sub>3</sub> and CaCO<sub>3</sub> [16]. The SL contains ethyl violet indicator which turns from white to violet as absorbent is depleted or upon moisture loss.

In the DCC system, a LI-8100A automated soil CO<sub>2</sub> flux system, consisting of LI-8100, multiplexer (LI-8150), long-term chamber (LI-8100-104), survey chamber (LI-8100A-102), was employed. The LI-8100A was equipped with an auxiliary sensor (LI-8100-663) for soil temperature and soil moisture measurements. The LI-8100A also controlled the survey and long-term measurement chambers and multiplexer, and housed the infrared gas analyzer (IRGA) that measured the change in CO<sub>2</sub> and H<sub>2</sub>O concentration in the soil chamber. The system was equipped with a Wi-Fi (“wireless infidelity”) enabled device, which stored the measured data in the flash memory (**Figure 2b**).

## Measurements Procedure

### Soda Lime Method

Cylindrical plastic containers, with dimensions 19 cm dia. x 19 cm ht. and covered lid (5678 cm<sup>3</sup>) were used as chambers of SL during the measurements of CO<sub>2</sub> emissions in the field. The bottom of the container was opened with a knife and then inserted into the mineral soil at about a 3 cm depth. These chambers were installed in the field prior to the initiation of measurements and remained in place during the study period.

In the laboratory, a 10-g soda lime was weighed using a 200/0.1 mg precision digital weighing scale. The weighed soda lime was placed inside a cylindrical glass bottle (5 cm dia. X 5 cm ht.) with screw cap. Previous studies used various weights of soda lime: 6 g to assess spatial variability of forest Rs with chamber dimension 20 cm dia. x 8 cm tall [4], 60 g for 11,000 cm<sup>3</sup> internal volume of chamber [17], 40 g soda lime to assess soil CO<sub>2</sub> flux in bare and cropped soils [28]. In this study, however, initial trials revealed no significant differences in the amount of adsorbed CO<sub>2</sub> efflux using more than 10-, 20-, 30-, and 60-g of soda lime for 24-h measurements.

In the initiation of measurements in the field, the glass bottles with SL were opened before placing them inside the plastic chambers. Then, the plastic chambers were covered with plastic lids. The soil



Figure 2. Chemicals and equipment used in soil respiration measurements, consisting of (a) soda lime and (b) LI-8100A automated soil CO<sub>2</sub> flux system.

temperature and soil moisture inside the chamber were also measured simultaneously with the Rs rates using a soil temperature probe (Decagon Devices, Pullman, WA). The soil temperature inside the chamber was measured, at 30 minute intervals, using a soil temperature probe (HOBO data loggers, MA). The probe was inserted in a small hole in the chamber lid, which was sealed with a tape to prevent CO<sub>2</sub> leakage.

The soda lime inside the closed plastic chamber was allowed to absorb CO<sub>2</sub> for approximately 24 hours (i.e. complete day and night cycle) to avoid any diurnal influence on the CO<sub>2</sub> estimate [22]. The carbon dioxide adsorbed by the SL in each glass bottle was determined as the difference between pre- and post-incubation weights of SL, less the weight gain found in blank sample. Weight gains were multiplied by 1.69 [18] to correct the weight loss due to the release of water during the chemical bonding of CO<sub>2</sub> to SL. The carbonate formation, as reflected in the weight gain of granules, was converted to μmol m<sup>-2</sup> s<sup>-1</sup>.

### LI-8100A Soil CO<sub>2</sub> Flux System

Fully automated soil CO<sub>2</sub> flux measurement systems were employed: LI8100 (analyzer control unit), Multiplexer (LI8150), Survey Chamber (LI8100-103), and Long-Term Chamber (LI8100-104) (LI-COR Biosciences, Lincoln, NE). To capture temporal and spatial variations of  $R_s$  rates across different sites and seasons, two measurement techniques of  $R_s$  were used: SC that allows rapid measurements at different points on the ground to capture spatial variation, and LTC measurement that allows unattended continuous  $R_s$  rate measurements over a long period of time. Measurements were conducted for four months during the late summer and early fall, from August to November.

For the LTC, four chambers were used: two chambers for each treatment, which were randomly placed within double and within two double rows. Because five willow fields were measured with only one set of LTC, LI-8100, and LI-8150, the machinery was moved every four days, from one field to another. Observations were made at 30 minute intervals for four consecutive days at each site. The measurements were carried out side-by-side with SL technique for 24 hours. Four LTC were used to measure the  $R_s$  rates at 30-minute intervals. These chambers were connected to the multiplexer and positioned within and between two double rows of the continuous production and tear-out plots. To ensure a good representative sample of air during the measurement, a 10 second dead band (which starts at the moment the chamber closes completely and until steady mixing of air), was observed.

Measurement duration was 120 seconds to reduce the impact of chamber closure on soil environment [8].

A point-in-time sampling of  $R_s$  was also conducted using SC, which was connected to the IRGA found inside the LI-8100A. Across the ten willows fields, 120 soil collars were used to obtain  $R_s$  rates' samples. The measurements were carried out every two weeks from May to November 2010, between 09.00 and 18.00 h within the same day, and were conducted side by side with SL measurements. A dead band of 10 seconds and a measurement length of 120 seconds were observed in sampling the  $R_s$  rates. Soil temperature and soil moisture were also measured with  $R_s$ . For the automated chamber system, soil temperature and soil moisture probes were employed (Decagon Devices, Pullman, WA), which were attached to the LI-8100-663 [29].

### Comparison between Soda Lime and LI-8100A System

A direct comparison between soda-lime and LI-8100A automated soil CO<sub>2</sub> flux system was made by measuring the mean daily  $R_s$  rates in paired chambers, placed directly adjacent to each other. Chambers were pushed about 3 cm below the mineral soil to obtain an effective seal against leakage while minimizing root disturbance. One chamber was sampled with SL while the other with LI-8100A automated soil CO<sub>2</sub> flux system. Chamber pairs were located within and between the two double rows of willow crop. Two comparisons were made: between SL and LTC measurements and between SL and SC measurements (**Figure 3**)



Figure 3. Measurements of soil respiration in paired chambers, using soda lime technique and LI-8100A soil CO<sub>2</sub> flux system.

## Statistics

The relationship of  $R_s$  measured with soda lime and LI-8100A was examined using Pearson's product moment correlation (SAS Statistical package 9.1). Regression analysis was used to determine the relationship between SL technique and LTC method and between SL and SC method. A paired t-test was used to test the null hypothesis of no significant differences in  $R_s$  rates between these two techniques. In all analyses, a probability level of  $P \leq 0.05$  was considered significant.

## III. RESULTS AND DISCUSSION

During four months duration of measurements,  $R_s$  rates ranged from 0 to 4.8- $\mu\text{mol m}^{-2} \text{ s}^{-1}$  for the SL technique, and from 0.3 to 7.3- $\mu\text{mol m}^{-2} \text{ s}^{-1}$  for the LTC measurements. The SL technique underestimated  $R_s$  by about 29% compared with the LTC (i.e. 1.12 vs 1.59). Coefficient of variation of the  $R_s$  rates ranged from 14 to 37% for the LTC, and from 20 to 50% for the SL technique (data not shown). Across the ten different measurement plots, mean  $R_s$  rates were significantly different between SL and LTC ( $p = 0.0168$ ,  $n = 92$ ). Estimated  $R_s$  for the LTC was approximately 1.3 times greater than the SL technique ( $P < 0.0001$ ). The SL technique and LTC showed moderately strong relationship ( $r_2 = 0.78$ ) (Figure 4).

The average daily  $R_s$  rates measured with SL method ranged from 0 to 7.0- $\mu\text{mol m}^{-2} \text{ s}^{-1}$ . In contrast, the mean daily  $R_s$  rates for the SC method ranged from 0.3 to 8.0- $\mu\text{mol m}^{-2} \text{ s}^{-1}$ . Across space and time, the mean  $R_s$  rates measured with the SL technique was about 23% lower than the SC method, with mean difference of about  $0.98 \pm 0.78 \mu\text{mol m}^{-2} \text{ s}^{-1}$ . The SL technique also showed high variability in  $R_s$  rates ( $CV = 33\%$ ) than the SC method ( $CV = 21\%$ ). The  $R_s$  rates measured with SL method were significantly different from  $R_s$  rates measured with SC method ( $P < 0.0001$ ,  $n = 1148$ ). The relationship between the SL technique and SC method is slightly strong ( $r_2 = 0.59$ ) (Figure 5).

SC and LTC measurements were designed to reduce the error of  $R_s$  estimates resulting from spatial and temporal variations of  $R_s$  rates. Thus, the  $R_s$  rates measured with SC and LTC techniques (LI-8100A for brevity) were averaged, and then compared it with the  $R_s$  rates measured with the SL technique. During the four months (Aug-Nov) measurements, mean  $R_s$  results from SL (1.7- 2.3- $\mu\text{mol s}^{-1} \text{ m}^{-2}$ ) was lower compared with  $R_s$  rates from LI8100A (1.7-3.0 -  $\mu\text{mol s}^{-1} \text{ m}^{-2}$ ). On average across the eight different plots, SL underestimated 20% of  $R_s$  rates compared with the LTC. The underestimation was higher (ca 29%) in warmer month (Aug) than in colder months (Sep-Nov). At low  $R_s$  rates (i.e.  $< 1 \mu\text{mol s}^{-1} \text{ m}^{-2}$ ), the difference in  $R_s$  rates between SL and

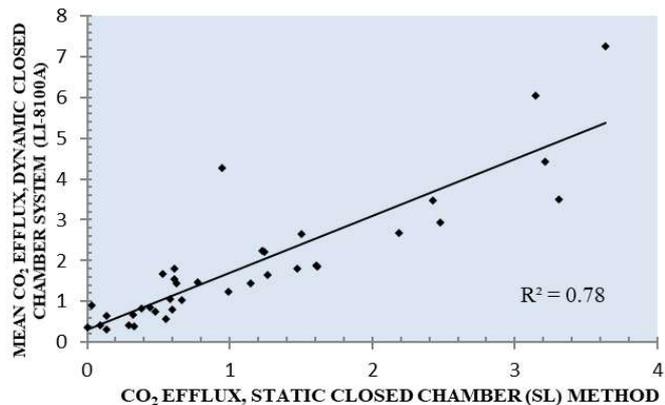


Figure 4. Mean rates of  $R_s$  ( $\mu\text{mol m}^{-2} \text{ s}^{-1}$ ) measured continuously with dynamic long-term chamber and soda lime technique in side-by-side position of chambers across the 10 different *Salix x dasyclados* sites. Each point represents an average  $R_s$  rates ( $n=48$ ) measured continuously a 24-hour period. The solid line shows the fitted regression ( $r_2=0.78$ ).

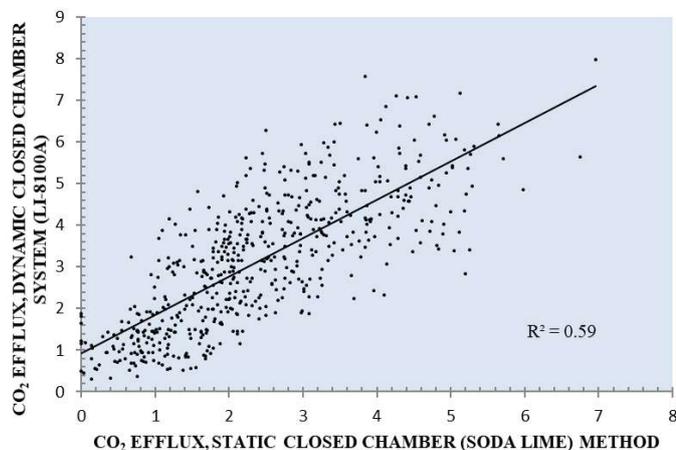


Figure 5. Combined  $R_s$  rates ( $\mu\text{mol m}^{-2} \text{ s}^{-1}$ ) measured with soda lime and survey chamber across the ten different *Salix x dasyclados* sites. Each point represents a paired comparison ( $n = 574$ ), measured from June to December 2010. The solid line represents the fitted regression ( $r_2=0.59$ ).

LI8100 was small (Table 1). Among the different plots, the continuous production (CP) plots displayed a generally higher mean Rs rates than the tear-out (TO) plots. Plots that were covered with cover crops (CC) also showed higher mean Rs rates than those plots without cover crop (NCC). Rs rates decline steadily in fall season, as the soil temperature continued to drop.

Rs rates showed strong relationship with soil temperature, but not with soil moisture. Soil temperature explained about 56% of Rs rates variations for the LTC ( $R^2 = 0.56$ ), 71% for the SC ( $R^2 = 0.71$ ), and 64% for the SL ( $R^2 = 0.64$ ) technique (Figure 6). The Rs rates approached to  $<1 \mu\text{mol m}^{-2} \text{s}^{-1}$  when the soil temperature dropped below  $5^\circ\text{C}$ . Diurnal temperature variation was low (5 to 20%) during the warmer months (May-Oct), and was high (40 to 60%) in colder month (November). The mean daily soil temperature ranged from 1 to  $38^\circ\text{C}$  during the entire period of measurements. In contrast, Rs rates and soil moisture content (MC) showed a very weak relationship ( $R^2 = 0.34$ ) (data not shown).

### Comparison between soda lime and LI-8100A System

The SL technique and LI-8100A system showed a linear relationship, which suggests a good agreement between these two methods. This corroborates the report of Rochette et al. [15] in which a linear relationship between the

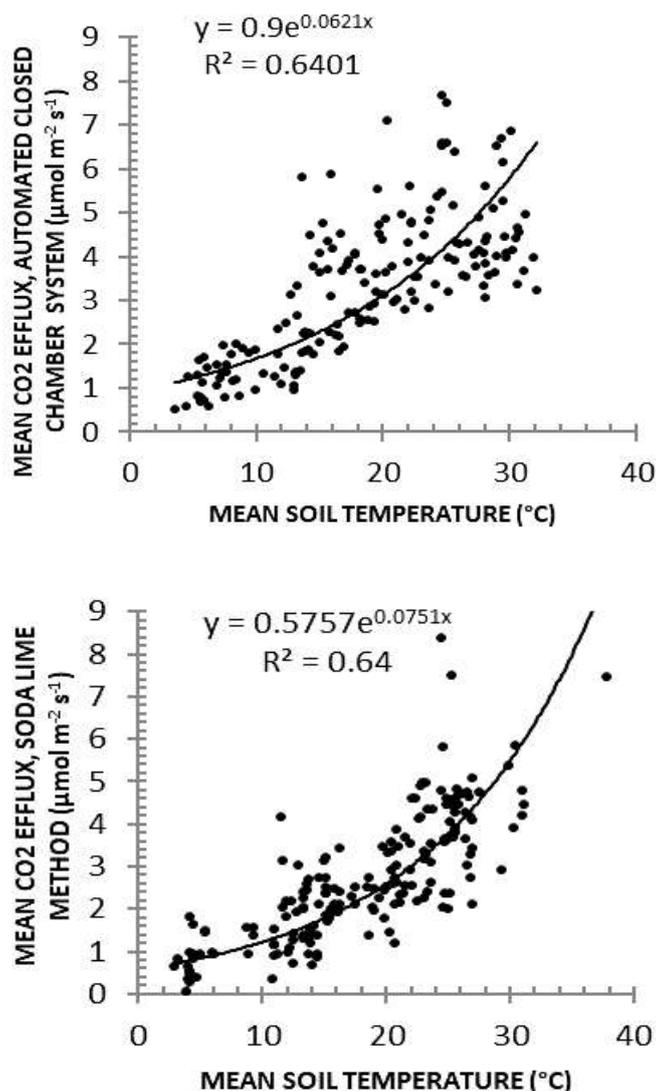


Figure 6. Mean Rs rates and soil temperature (5 cm depth) measured simultaneously with a long-term chamber and a static chamber (soda-lime). Long-term chamber measured Rs and soil temperature continuously over the entire duration of study while survey chamber and static chamber on a bi-weekly intervals. All points shown represent average values of eight sampling points (chambers) in each site placed side-by-side to each other.

Table 1. Different soil CO<sub>2</sub> efflux rates (mean  $\pm$  SE) results across the different willow plots, ranging in age from 0- to 20-year old. LI8100 represents the combined results of the survey and long-term chamber soil CO<sub>2</sub> efflux measurements, which was measured simultaneously with soda lime (SL) over 24 hours. Prior to measurements, the measurement plots received treatments: cover crop (CC) and without cover crop (NCC) for 0-age and tear-out (TO) and continuous production (CP) for the 6, 16, and 20-year old willow fields.

	Soil CO <sub>2</sub> Efflux ( $\mu\text{mol s}^{-1} \text{m}^{-2}$ )							
	0-year old				6-year old			
	CP-NCC		CP-CC		TO		CP	
	SL	LI8100	SL	LI8100	SL	LI8100	SL	LI8100
Aug	2.7 $\pm$ 0.3	2.8 $\pm$ 0.1	3.0 $\pm$ 0.3	3.5 $\pm$ 0.2	2.9 $\pm$ 0.2	3.8 $\pm$ 0.2	2.2 $\pm$ 0.1	3.9 $\pm$ 0.2
Sep	1.9 $\pm$ 0.1	2.0 $\pm$ 0.1	2.5 $\pm$ 0.2	2.4 $\pm$ 0.1	2.3 $\pm$ 0.3	2.5 $\pm$ 0.1	2.6 $\pm$ 0.2	3.3 $\pm$ 0.2
Oct	1.2 $\pm$ 0.1	1.2 $\pm$ 0.1	1.5 $\pm$ 0.3	1.5 $\pm$ 0.1	0.9 $\pm$ 0.1	1.4 $\pm$ 0.1	1.5 $\pm$ 0.2	1.9 $\pm$ 0.1
Nov	1.1 $\pm$ 0.1	0.7 $\pm$ 0.1	1.3 $\pm$ 0.2	1.2 $\pm$ 0.1	0.6 $\pm$ 0.1	0.7 $\pm$ 0.1	0.7 $\pm$ 0.1	1.2 $\pm$ 0.1
Mean	1.7 $\pm$ 0.2	1.7 $\pm$ 0.1	2.1 $\pm$ 0.3	2.2 $\pm$ 0.1	1.7 $\pm$ 0.2	2.1 $\pm$ 0.1	1.8 $\pm$ 0.1	2.6 $\pm$ 0.1
	16-year old				20-year old			
	TO		CP		TO		CP	
	SL	LI8100	SL	LI8100	SL	LI8100	SL	LI8100
Aug	3.7 $\pm$ 0.3	3.5 $\pm$ 0.2	2.6 $\pm$ 0.2	4.8 $\pm$ 0.2	3.6 $\pm$ 0.4	4.4 $\pm$ 0.2	1.6 $\pm$ 0.1	3.4 $\pm$ 0.1
Sep	2.7 $\pm$ 0.2	2.2 $\pm$ 0.1	2.8 $\pm$ 0.1	3.8 $\pm$ 0.1	2.6 $\pm$ 0.4	3.4 $\pm$ 0.2	1.8 $\pm$ 0.2	2.9 $\pm$ 0.1
Oct	1.3 $\pm$ 0.1	1.3 $\pm$ 0.1	1.5 $\pm$ 0.1	2.2 $\pm$ 0.1	2.0 $\pm$ 0.3	1.7 $\pm$ 0.1	1.9 $\pm$ 0.3	2.1 $\pm$ 0.1
Nov	0.4 $\pm$ 0.1	0.6 $\pm$ 0.1	0.5 $\pm$ 0.1	1.0 $\pm$ 0.1	1.1 $\pm$ 0.1	1.2 $\pm$ 0.1	0.7 $\pm$ 0.1	1.4 $\pm$ 0.1
Mean	2.0 $\pm$ 0.2	1.9 $\pm$ 0.1	1.9 $\pm$ 0.1	3.0 $\pm$ 0.1	2.3 $\pm$ 0.3	2.7 $\pm$ 0.2	1.5 $\pm$ 0.2	2.5 $\pm$ 0.1

SL method and DCC methods was observed. In contrast, some authors reported an exponential relationship between SL technique and DCC. But, these studies employed a point-in-time sampling measurements (survey) using SRC-1 and EGM-1 soil respiration machines [22, 30]. Point-in-time measurements had been observed to incur large error due to the unpredictability of diurnal and seasonal  $R_s$  patterns [7, 18, 31].

The SL and LTC also showed a moderately strong relationship ( $r_2 = 78$ ). This is somewhat inconsistent with some previous reports in which poor agreement of  $R_s$  rates between SCC and DCC was observed, with the SCC (soda-lime) tended to underestimate or overestimate  $CO_2$  efflux [22, 30, 31, 32]. One of the probable reasons for this inconsistency is the machinery employed in the measurement of  $R_s$  rates. Previous DCC machines have lower data resolutions compared with the LI-8100A system. As pointed out earlier, old DCC machines cannot perform continuous unattended  $R_s$  measurements and highly susceptible to error due to microclimate modification resulting from exclusion of rainfall and increased temperature within the chamber, and pressure gradients [7, 19, 20]. The ability of the LI-8100A to perform unattended  $R_s$  measurements in the field insures that the temporal variations of  $R_s$  rates are captured in the measurements. The chamber also moves automatically away from the measurement points when not doing measurements, hence, allows precipitation in the sampling area and prevents temperature modification within the chamber. Furthermore, the LI-8100A's chamber has pressure vents, which stabilizes the inside and outside air pressures; thus, minimizes the error brought about by the imbalance of air pressure inside the chamber and ambient environment [32]. These features are not present in old DCC machines.

The result also revealed that the SL technique underestimated the  $R_s$  rates by about 20 to 29% compared with the LTC, and about 23% compared with the SC measurement. This difference is within the range of values reported by Pumparen et al [32] in which the SCC method was found to underestimate  $R_s$  rates by about 23–31% compared with the Li-Cor-6400-09 (automated chamber system). The underestimation was attributed to the altered

diffusion gradient in static chamber that slowed down the diffusion of  $CO_2$  from soil into the chamber. In another study, a higher discrepancy between SL and DCC methods was observed by about 40% [30]. Grogan [14] also reported that the SL technique overestimated fluxes at efflux rates  $\leq 1.6$  g  $CO_2$   $m^{-2}$   $d^{-1}$  and underestimated fluxes at efflux rates of 5.0 g  $CO_2$   $m^{-2}$   $d^{-1}$ . Furthermore, Jensen et al [22] also observed that the SL method gave an average 12% higher flux between rates below 100 mg  $CO_2$   $m^{-2}$   $h^{-1}$ , but much lower flux rates above 100 mg  $CO_2$   $m^{-2}$   $h^{-1}$ . In the current study, however,  $R_s$  rates of  $\leq 1.6$  g  $CO_2$   $m^{-2}$   $d^{-1}$  were not observed during the measurement period. In winter season, the lowest observed  $R_s$  rates was about 2.0 g  $m^{-2}$   $d^{-1}$  for the automated chamber and about 2.3 g  $m^{-2}$   $d^{-1}$  for the SL method.

#### **Relationship between $R_s$ rates and soil temperature and soil moisture**

The soil temperature explained about 56 to 71% of the variation of  $R_s$  rates. Similar observations were reported in other studies [20, 33, 34], in which soil temperature's changes explained the large variation of  $R_s$  rates. In the study of Arevalo et al [35] in a poplar plantation, it was reported that about 88 to 94% of the observed variability of  $R_s$  rates is attributable to soil temperature. In another study [36], it was found that the soil temperature explained about 73 to 88% of the variability of  $R_s$  rates. Also, it was observed that soil temperature increased  $R_s$  rates exponentially. A similar observation was also reported by Liang et al. [20] in which the  $R_s$  rates increased exponentially with increased soil temperature.

The soil moisture and  $R_s$  rates showed a very weak agreement. Some researchers reported similar observations [20, 36, 37]. Jannsen et al. [4] mentioned that in the absence of drought stress, soil temperature will exert a dominant control in  $R_s$  rates. In this study, the soil temperature masked the influence of soil moisture on  $R_s$  rates because of favorable soil moisture conditions during the observation period. Decreased  $R_s$  rates were observed when the soil is flooded with water, which could be explained by saturated conditions of the soil. When the soil is in saturated conditions, the displacement of soil air in macropores by water

limits the diffusion of oxygen, which is the vital substrate for both soil microbes and root respirations [34]. In this study, increased Rs rates were observed when the soil moisture ranged from 0.4 to 0.6 cm<sup>3</sup> cm<sup>-3</sup>, which is considered as a favorable soil moisture conditions. Grant & Roche [38] reported that an increased Rs rates is a common pattern when the soil moisture contents ranged from 0.6- to 0.7-cm<sup>3</sup> cm<sup>-3</sup>. Rs rates decreased when the soil moisture fell <0.2-cm<sup>3</sup> cm<sup>-3</sup> or rose above 0.9-cm<sup>3</sup> cm<sup>-3</sup>.

#### IV. CONCLUSIONS

The SL technique underestimated the CO<sub>2</sub> efflux by about 23% compared with the SC method. Similarly, the SL technique also underestimated Rs rates by about 20% to 29% with reference to the Rs rates measured with the LTC. These findings suggest that the Rs rates measured with SL technique could be adjusted by about 20 to 30% more to make it comparable with the LI-8100A system. The soil temperature strongly influenced Rs rates, suggesting that this variable provides a lot of information about Rs rates, and therefore an important component in Rs models. The soil moisture provides little information about Rs rates during favorable soil moisture conditions. But, under soil moisture stress conditions, soil moisture may affect Rs rates, as reported by some authors [4, 38]. However, in this study the absence of soil moisture stress conditions during the observation period precluded us to support the foregoing observations with evidence.

#### ACKNOWLEDGMENTS

This research was financially supported by the USDA CSREES. We also acknowledge Rebecca Allmond, Eric Fabio, Philip Castellano, and Ken Burns, Jacob Bakowski, Tyler Harvey, Gabe Kellman, Jason Maurer, and Ryan Newby – for their invaluable help in the data collection.

#### REFERENCES

- [1] Scott-Denton L.E., Rosentiel T.N., Monson R.K. 2006. Differential controls by climate and substrate over the heterotrophic and rhizospheric components of soil respiration. *Global Change Biology* 12:205-216.
- [2] Lal R. 2004. Soil carbon sequestration impacts on global climate change and food security. *Science* 304, 1623-1637.
- [3] Clark D.A., Brown S., Kicklighter D.W., Chmaber J.Q., Thomlinson J.R., Ni J. 2001. Measuring the net primary production in forests: concepts and field methods. *Ecological Applications* 11(2): 356-370.
- [4] Jannsens I.A., Ceulemans R. 1998. Spatial variability in forest Rs assessed with a calibrated soda lime technique. *Ecology Letters* 1, 95-98.
- [5] Law B.E., Ryan M.G., Anthoni P.M. 1999. Seasonal and annual respiration of ponderosa pine ecosystem. *Global Change Biology* 5: 169-182
- [6] Jassal R., Black A., Novak M., Morgenstern K., Nestic Z., Gaumont-Guay D. 2005. Relationship between soil CO<sub>2</sub> concentrations and forest-floor CO<sub>2</sub> effluxes. *Agricultural and Forest Meteorology* 130, 176-192.
- [7] Naishen L., Inoue G., Fujinuma Y. 2003. A multichannel automated chamber system for continuous measurement of forest Rs. *Tree Physiology* 23, 825-832.
- [8] LI-COR. 2004. LI-8100 Instruction Manual, LI-8100 automated soil CO<sub>2</sub> flux system. LI-COR, Inc, Lincoln, NE, USA 68504.
- [9] Post W.M., Emmanuel W.R., Zinker P.J., Stangenberger A.G. 1982. Soil carbon pools and world life zones. *Nature* 298: 156-159
- [10] Bruce J.P, Frome M., Haites E., Janzen H., Lal R., Paustin K. 1999. Carbon sequestration in soils. *Journal of Soil and Water Conservation* 54:1 382.
- [11] Trumbore S. 2000. Age of organic matter and soil respiration: radiocarbon constraints on belowground C dynamics. *Ecological Applications* 10(2):399-411.
- [12] Ahn M.A., Zimmerman A.R., Comerford N.B., Sickman J.O., Grunwald S. 2009. Carbon mineralization and labile carbon pools in the sandy soils of a North Florida watershed. *Ecosystems* 12:672-685.

- [13] Adair E.C., Reich P.B., Hobbie S.E., Knops J.M.H. 2009. Interactive effects of time, CO<sub>2</sub>, N, and diversity on total belowground carbon allocation and ecosystem carbon storage in a grassland community. *Ecosystems* 12:1037-1052.
- [14] Grogan P. 1998. CO<sub>2</sub> flux measurement using soda lime: correction for water formed during CO<sub>2</sub> adsorption. *Ecology* 74(5), 1467-1468.
- [15] Rochette P, Ellert B., Gregorich E.G., Desjardins R.L., Pattey E., Lessard R., Johnson B.G. 1997. Description of a dynamic closed chamber for measuring soil respiration and its comparison with other techniques. *Can J. Soil Sci.* 77: 195-203.
- [16] Keith H. & Wong S.C. 2006. Measurement of soil CO<sub>2</sub> efflux using soda lime absorption: both quantitative and reliable. *Soil Biology & Biochemistry* 38, 1121-1131.
- [17] Raich J.W., Bowden R.D., Steudler P.A. 1990. Comparison of two static chamber techniques for determining carbon dioxide efflux from forest soils. *Soil Sci Soc. Am. J.* 54:1754-1757.
- [18] Rayment M.B., Jarvis P.G. 1997. An improved open chamber system for measuring soil CO<sub>2</sub> effluxes in the field. *Journal of Geophysical Research* 102 D24, 28, 779-28,784.
- [19] Rayment M.B. 2000. Closed chamber system underestimate soil CO<sub>2</sub> effluxes. *European Journal of Soil Science* 51, 107-110.
- [20] Liang N, Nakadai T., Hirano T., Qu L., Koike T., Fujinuma Y., Inoue G. 2004. In situ comparison of four approaches to estimating soil CO<sub>2</sub> efflux in a northern larch (*Larix kaemferi* Sarg.) forest. *Agricultural and Forest Meteorology* 123, 97-117.
- [21] Xue L., Furtaw MD., Madsen RA., Garcia RL., Anderson DJ., McDermitt DK. 2006. On maintaining pressure equilibrium between a soil CO<sub>2</sub> flux chamber and the ambient air. *Journal of Geophysical Research* 111 D08 S10, 14.
- [22] Jensen L.S. Mueller T., Tate KR, Ross DJ, Magid J. 1996. Soil surface CO<sub>2</sub> flux as an index of soil respiration in situ: a comparison of two chamber methods. *Soil Biol. Biochem.* 28 (10/11), 1297-1306.
- [23] Abegbidi H.G., Volk T.A., White E.H., Abrahamson L.P., Briggs R.D., Beckelhaupt D.H. 2001. Biomass and nutrient removal by willow clones in experimental bioenergy plantations in New York State. *Biomass and Bioenergy* 20: 399-11.
- [24] Volk T.A. 2002. Alternative methods of site preparations and coppice management during the establishment of short-rotation woody crops. Ph. D Thesis. State University of New York-Environmental Science and Forestry, Syracuse, NY, USA.
- [25] Pacaldo RS, Volk TA, Briggs RD. 2014. Carbon sequestration in fine roots and foliage offsets soil CO<sub>2</sub> effluxes along a 19-year chronosequence of Shrub Willow Biomass Crops. *Bioenergy Research*. <http://dx.doi.org/10.1007/s12155-014-9416-x>
- [26] Aydin M, Pacaldo RS, Volk TA. 2018. Soil respiration in shrub willow (*Salix dasyclados*) biomass crops increased on the third year after removal. *International Journal of Global Warming* 15 (1). 54-66.
- [27] Hutton F.Z., Rice C.E. 1977. Soil Survey of Onondaga County, New York. USDA Soil Conser, Serv. In Cooperation with Cornell University Agric. Exp. Stn., Ithaca, NY 233 pp.
- [28] Monteith J.L., Szeicz G., Yabuki K. 1964. Crop photosynthesis and the flux of carbon dioxide below the canopy. *Journal of Applied Ecology* 1 (2) 321-337.
- [29] Pacaldo RS, Volk TA, Briggs RD, Abrahamson LA, Bevilacqua E, Fabio E. 2014. Soil CO<sub>2</sub> effluxes, spatial and temporal variability, and root respiration in shrub willow biomass crops (*Salix x dasyclados*) along a 21-year chronosequence as affected by continuous production and crop removal (tear-out) treatments. *Global Change Biology-Bioenergy*, 6:488-498
- [30] Pongracic S., Kirschbaum M.U.F., Raison R.J. 1997. Comparison of soda lime and infrared gas analysis techniques for in situ measurement of forest soil respiration. *Can J. For Res* 27(11), 1890-1895.
- [31] Mc Ginn S.M, Akinremi O.O, McLean H.D.J., Ellert B. 1998. An automated chamber system for measuring soil respiration. *Can J. Soil Sci* 78: 573-579.
- [32] Pumpanen J., Kolari P, Ilvesniemi H., Minkinen K., Vesala T., Niinistö S., Lohila A., Larmola T., Morero M., Pihlatie M., Janssens I., Yuste JC, Grünzweig SR, Subke J-A, Savage K., Kutsh W., Østregren G, Ziegler

- W., Anthoni P., Lindroth A., Hari P. 2004. Comparison of different chamber techniques for measuring soil CO<sub>2</sub> effluxes. *Agricultural and Forest Meteorology* 123, 159-176.
- [33] Fang C., Moncrieff J.B., Gholz H.L., Clark K.L. 1998. Soil CO<sub>2</sub> efflux and its spatial variation in a Florida slash pine plantation. *Plant and Soil* 205: 135-146.
- [34] Davidson E.A., Richardson A.D., Savage K.E., Hollinger D.Y. 2006b. A distinct seasonal pattern of the ratio of soil respiration to total ecosystem respiration in a spruce-dominated forest. *Global Change Biology* 12:230-239.
- [35] Arevalo C.B.M, Bhatti J.S, Chang S.X. Jassal R.S., Sidders D. 2010. Soil respiration in four different land use systems in north central Alberta, Canada. *Journal of Geophysical Research* 115.
- [36] Borken W., Savage K., Davidson E.A., Trumbore S.E. 2006. Effects of experimental drought on soil respiration and radiocarbon efflux from temperate forest soil. *Global Change Biology* 12: 177-193.
- [37] Longdoz B, Yernaux M., Aubinet M. 2000. Soil CO<sub>2</sub> efflux measurements in a mixed forest: impact of chamber disturbances, spatial variability and seasonal evolution. *Global Change Biology* 6, 907-917.
- [39] Grant R.F., Rochette P. 1994. Soil microbial respiration at different water potentials and temperatures: theory and mathematical modeling. *Soil Science Society of America Journal* 58: 1681-1690.