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## BIASES OF CHAMBER METHODS FOR MEASURING SOIL CO<sub>2</sub> EFFLUX DEMONSTRATED WITH A LABORATORY APPARATUS

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Investigators have historically measured soil CO<sub>2</sub> efflux as an indicator of soil microbial and root activity and more recently in calculations of carbon budgets. The most common methods estimate CO<sub>2</sub> efflux by placing a chamber over the soil surface and quantifying the amount of CO<sub>2</sub> entering the chamber per unit area of soil per unit time. Schlesinger (1977), Anderson (1982), Rolston (1986a), Raich and Nadelhoffer (1989), and Nakayama (1990) have reviewed various chamber methods. No single method is established as a standard (Anderson 1982, Nakayama 1990, Norman et al. 1992), partly because methods are not compared to known effluxes (Nakayama 1990). Past comparisons have only shown a method to be higher or lower than another method.

This study compared the responses of two commonly used chamber methods to known effluxes from the surface of a simulated soil. Our known effluxes are based on calculations using Fick's law of diffusion. The two methods we tested were a static-chamber method with soda lime as a CO<sub>2</sub> absorbent and a dynamic-chamber method consisting of an infrared gas analyzer in a closed air-circulation loop. Because the absorption rate of alkali materials used in static chambers is thought to be a source of bias (Freijer and Bouten 1991, Nakadai et al. 1993), we were also interested in how the soda-lime absorbent affected the headspace CO<sub>2</sub> concentration of the static chambers.

### Methods

The apparatus for testing the two methods (Fig. 1) consisted of a CO<sub>2</sub> generator, a diffusion box, and a diaphragm pump to circulate air between the two. Carbon dioxide was generated in a flask of 0.5 mol/L HCl

solution by continuous additions of 0.3 mol/L NaHCO<sub>3</sub> solution at a controlled rate ranging from 0 to 30 mL/h. The diffusion box, constructed of 0.5-cm-thick Plexiglas, had inside dimensions of 75 × 75 × 40 cm. Air with CO<sub>2</sub> from the generator was introduced into the bottom half of the box (foot-space) through a plenum and mixed with two fans. Above the foot-space, an 18-cm-thick layer of polyurethane foam provided a porous medium through which the CO<sub>2</sub> diffused. We used a water manometer to test for overpressure of the foot-space; none was detected. Laboratory air temperatures ranged from 21° to 29°C and relative humidity ranged from 21 to 55%.

Four different CO<sub>2</sub> efflux rates ranging from 0 to 0.77 g·m<sup>-2</sup>·h<sup>-1</sup> were achieved in trials lasting 24 h. This range of effluxes is similar to those reported for soils globally (Raich and Schlesinger 1992). The efflux of CO<sub>2</sub> ( $J$ ) from the surface of the foam was calculated based on Fick's law of diffusion:

$$J = -D \frac{dC}{dz},$$

where  $D$  is the diffusivity of CO<sub>2</sub> in the foam and  $dC/dz$  is the CO<sub>2</sub> concentration gradient through the foam. The diffusivity of the foam was determined at 25°C to be  $0.099 \pm 0.002$  cm<sup>2</sup>/s (mean  $\pm$  1 SE,  $n = 3$ ), using methods described by Rolston (1986b). The CO<sub>2</sub> gradient was calculated as the difference in CO<sub>2</sub> concentration between the top and bottom surfaces of the foam divided by the foam thickness.  $D$  was corrected for minor variations in air temperature by using the equation:  $D = 0.083(T/273K)^2$ , where 0.083 cm<sup>2</sup>/s is the foam diffusivity adjusted to 273K and  $T$  is air temperature in kelvins.

Air samples were collected with a 0.5-mL syringe at the top foam surface and through a septum in the foot-space, and the CO<sub>2</sub> concentrations were determined by gas chromatography (GC-8A fitted with a Porapak Q column and a thermal conductivity detector [Shimadzu, Kyoto, Japan]). These samples were taken at  $\approx$  30- to 60-min intervals. Throughout the trials, the foot-space CO<sub>2</sub> concentration was regulated by minor adjustments to the flow rate of NaHCO<sub>3</sub> solution.

The *static-chamber method*, based on Edwards (1982), estimated CO<sub>2</sub> entering the chamber by the mass increase of the soda-lime absorbent. Three polyvinyl chloride (PVC) chambers 21 cm in diameter by 20 cm in height were used in each trial. Soda lime (60 g, 1.7–3.4 mm granules) was contained in tins 8 cm in diameter by 5 cm in height and set directly on the foam surface inside a collar. Five-centimetre-tall collars made of the same PVC material as the chambers were inserted into the foam to a depth of 2.5 cm. Static chambers were affixed to the collars and sealed with duct tape. We used three blanks in each trial to account for

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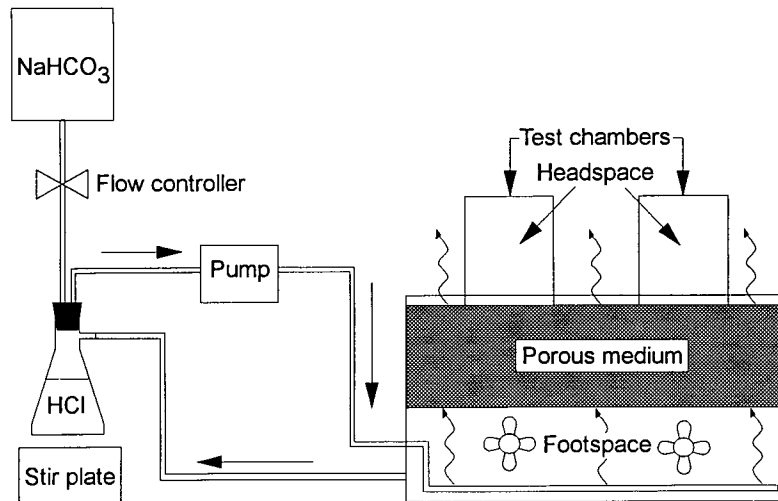


FIG. 1. Laboratory apparatus for testing chamber methods to measure soil CO<sub>2</sub> efflux.

mass change from handling of the soda lime. The headspace concentrations of CO<sub>2</sub> in the static chambers were also monitored during a trial by taking 5–8 syringe samples through a rubber septum in the top of each of the chambers.

The *dynamic-chamber method*, based on Norman et al. (1992), used an infrared gas analyzer (LI-6200, LICOR, Lincoln, Nebraska, USA) to monitor changes in the CO<sub>2</sub> concentration of air circulating to and from the dynamic chamber. Our dynamic chamber was of the same dimensions and materials as our static chambers. Before each sampling the chamber was allowed to equilibrate with the ambient air by resting on its side. After equilibration the chamber was affixed to a collar, also the same as the static chambers, and sealed with a closed-cell foam-rubber gasket. The rate of CO<sub>2</sub> concentration buildup was then measured for 78 s. Three locations on the foam surface were sampled  $\approx$  8 times over the course of each trial except during the highest efflux when two locations were sampled.

#### Results and Discussion

Both the static- and dynamic-chamber methods exhibited biases when compared to the calculated efflux based on Fick's law of diffusion. The static-chamber method greatly overestimated the zero efflux, overestimated the two intermediate CO<sub>2</sub> effluxes of 0.12 and 0.24 g·m<sup>-2</sup>·h<sup>-1</sup> by  $\approx$  25%, and underestimated the highest CO<sub>2</sub> efflux of 0.77 g·m<sup>-2</sup>·h<sup>-1</sup> by 57%. The dynamic-chamber method consistently underestimated all effluxes above zero by 15% (Fig. 2).

The average headspace CO<sub>2</sub> concentrations of the static chambers differed from the ambient air by -180, -60, +15, and +450  $\mu$ mol/mol during the CO<sub>2</sub> efflux trials of 0, 0.12, 0.24, and 0.77 g·m<sup>-2</sup>·h<sup>-1</sup>, respec-

tively. The three greatest differences were statistically significant (ANOVA,  $\alpha = 0.05$ ). No significant differences were found among headspace concentrations within a trial. The headspace CO<sub>2</sub> concentration of the dynamic chamber changed on average from the ambient air by +36  $\mu$ mol/mol in the 0.77 g·m<sup>-2</sup>·h<sup>-1</sup> CO<sub>2</sub> efflux trial and was proportionally less for the other trials.

Our study demonstrates that both overestimates and underestimates of CO<sub>2</sub> efflux result from the use of a

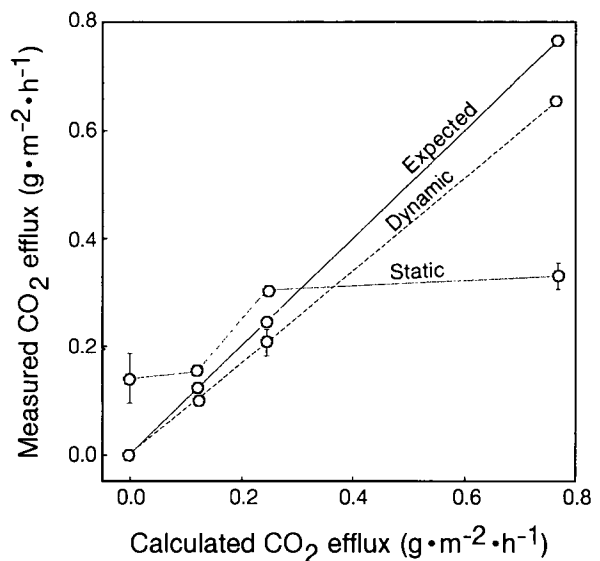


FIG. 2. CO<sub>2</sub> efflux measured by chamber method compared with CO<sub>2</sub> efflux calculated by Fick's law, with  $\pm$  1 SE for chamber method estimates (some SEs obscured by data points).

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