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Porous tubing for use in monitoring soil CO_2 concentrations

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Abstract

The composition of the soil atmosphere is an indicator of biological processes, and soil CO_2 gradients have been used to estimate CO_2 efflux from the surface. Soil atmosphere samplers, constructed with gas-permeable materials, have been used to quantify soil CO_2 concentrations. The type of material used can influence the perceived real-time concentrations of CO_2 in the soil. Previous works have not directly compared different types of materials under the same conditions. The objective of this study was to determine the diffusion coefficient (D) and time of 95% equilibrium (t_{eq}) of CO_2 through several materials, and to evaluate the effect of long-term soil burial (183 days) on diffusion characteristics. Materials tested included silicone, expanded Teflon (ePTFE), and ultra high molecular weight polyethylene (PE) tubing. The D of each material was determined using a closed-loop system consisting of a CO_2-enriched (7800 ppm) chamber, a CO_2 analyzer and an inner tube (experimental tubing) placed inside the chamber. Air was re-circulated through the inner tube, and as CO_2 diffused from the chamber into the tubing, the analyzer recorded the increase in concentration. The silicone tubes had values of D ranging from 8.64 to $5.80 \times 10^{-6}\,cm^2\,s^{-1}$ with corresponding t_{eq} between 3.9 and 9.7 h. Diffusion coefficients of the ePTFE ($1.25 \times 10^{-4}\,cm^2\,s^{-1}$) and PE ($7.70 \times 10^{-4}\,cm^2\,s^{-1}$) materials were 2 orders of magnitude greater, with $t_{eq} < 6$ min. Exposure to the soil environment for 183 days did not visibly deteriorate the materials or significantly affect the D or t_{eq} values. Use of the ePTFE or PE materials, over the silicone materials, may allow for better characterization of dynamic CO_2 concentrations in the soil based on the greater D and lesser t_{eq} values of these materials.
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Keywords: Diffusion; Soil carbon dioxide concentrations; Expanded teflon tubing; Silicone tubing; Porous tubing; Polyethylene tubing

1. Introduction

Information on trace gas concentrations in soil can provide critical information to determine the sources and sinks of these biologically produced gases and the biological processes involved. Also, knowledge of CO_2 concentrations at discrete depths, coupled with the diffusivity of CO_2 in the soil, can allow for estimates of the flux of CO_2 within and from the soil. Numerous methods have been used to sample soil gases at discrete depths below the soil surface (Taylor and Abrahams, 1953; Burton and Beauchamp, 1994; Schmid et al., 2001; Risk et al., 2002; Maljanen et al., 2003). Recently, gas-permeable polymer tubing has been used to assist in the monitoring of soil gases and gases in water and animal manure. Some polymers that have been used include polypropylene (Gut et al., 1998, 2002; Schmid et al., 2001), silicone (Holter, 1990; Kramer and Conrad, 1993; Jacinthe and Dick, 1996) and porous Teflon (Parkin and Tiedje, 1984; Hirsch et al., 2004). Another gas-permeable polymer that has been used as a passive sampler equipped with a sorber for volatile organic compounds is polyethylene (PE) (Namiesnik et al., 2005). An advantage of using these three and other similar polymers is that they are permeable to gases but not to liquid water. Also, Gut et al. (1998) reported that after five months of being buried in soil that the diffusion coefficient of NO through the tubing wall was not affected when compared to initial tests.

Real-time determinations of soil CO_2 concentrations can be accomplished using state-of-the-art sensors that are buried at discrete depths in the soil (Hirano and Kim, 2003; Tang et al., 2003; Jassal et al., 2005). Although these sensors can operate under humid conditions (100% relative humidity, noncondensing), the sensing element of these sensors must be protected from liquid water. Thus, use of a

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membrane that is permeable to CO_2 and also covers and protects the sensor from liquid water is necessary when using these sensors for environmental research (Jassal et al., 2005). In such an application, knowledge of the diffusion properties of the material used is critical. In contrast to direct burial of sensors or gas wells, burial of lengths (m) of porous tubing in the soil (Gut et al., 1998) can provide information on soil gas concentrations and gradients at larger spatial scales. Soil gases that diffuse into the permeable tubing can then be circulated through aboveground analyzers and quantified. Irrespective of the method used to determine concentrations of CO_2 in the soil, using materials that allow for a timely equilibration of soil gases is essential. The purpose of this study is to provide detailed information on the diffusion of CO_2 through various tubing used for soil atmospheric research. Therefore, the objectives of this research are to: (i) evaluate the diffusion coefficient (D) and time of equilibrium (t_{eq}) of CO_2 through three silicone, one ePTFE, and four PE tubing under controlled, laboratory conditions; (ii) determine how long-term burial of these materials beneath the soil surface affects the D and t_{eq} of these materials, and (iii) provide discussion on the potential of using different tubing materials for use in sampling CO_2 concentrations beneath the soil surface.

2. Methods and materials

2.1. Laboratory methods

The diffusion coefficient of CO_2 through the tubing wall (D) and time for equilibrium (t_{eq}) were determined for three different types of tubing: (1) silicone, (2) expanded Teflon (polytetrafluorethylene) (ePTFE), and (3) ultra high molecular weight polyethylene (PE). The silicone tubing that were evaluated were Tygon 3305 (silicone-1) and Versilic SPX-50 (silicone-2), which were manufactured from Saint Gobain Performance Plastics (Beaverton, MI), and a product that was distributed by US Plastics (silicone-3, manufacturer unknown) (Part #54038; United States Plastic Corporation Lima, OH) (Table 1). Although the silicone tubing had different wall thicknesses and curing processes, all three were translucent, flexible, had smooth surfaces, and could be pinched closed with finger strength.

The ePTFE tubing was manufactured by W.L. Gore and Assoc. (Product #062404-4; Elkton, MD) (Table 1). The ePTFE is very slippery and smooth in texture, flexible, and white in color. This tubing also can be pinched with finger strength but does take greater time to regain its shape compared to the silicone tubing. Neither the silicone or ePTFE products completely collapsed when buried to a depth of 15 cm below the soil surface.

The four PE tubes were rigid, smooth, white in color, and manufactured by GenPore (Reading, PA) (Table 1). PE-1 and PE-2 had pore sizes of 10 μm and OD and wall thicknesses of 25.4 and 1.7 mm and 21.6 and 4.5 mm, respectively. PE-3 and PE-4 had pore sizes of 35 μm and OD and wall thicknesses of 25.4 and 1.7 mm and 21.6 and 4.5 mm, respectively. Although the tubing structures were rigid, one could partially compress the thinner-walled tubing (PE-1 and P-3) with finger strength albeit not to complete closure.

The diffusion coefficient of CO_2 through the wall of each tubing product was determined using the procedures described in Jacinthe and Dick (1996) and Gut et al. (1998) with some modifications. The experimental setup consisted of an equilibration chamber made from an 8.8 and 10.2 cm ID and OD, respectively, acrylic tube that was fitted at each end with 10.2 cm rubber caps (QC-104; Fernco Inc., Davison, MI) (Fig. 1). Each rubber cap was center-punched to accept 1.27 cm barbed, bulkhead fittings (B521-21; MG Scientific, Pleasant Prairie, WI). The experimental tubing products were placed inside the equilibration chamber and secured to the bulkhead fittings directly or through the use of fitting adapters. Using a gas dilution system (Series 4040; Environics, Tolland, CT), a CO_2 concentration of approximately 7800 μmol mol^{-1}, a concentration of CO_2 observed at the 10 cm depth in a Harps loam soil (fine-loamy, mixed, superactive, mesic Typic Calciaquoll) near Ames, IA (unpublished data,

Table 1
Tubing specifications as defined by respective manufacturers

Designation	Dimensions, OD/wall (mm)	Pore size (μm)	Flexibility	Color	Manufacturer (product)	Approximate cost[a] US$ m^{-1}
Silicone-1	19.1/3.2	NA	Flexible	Translucent	Saint-Gobain (Tygon 3305)	19
Silicone-2	19.1/3.2	NA	Flexible	Translucent	Saint-Gobain (Versilic SPX-50)	24
Silicone-3	15.8/1.6	NA	Flexible	Translucent	USPlastics[b]	3
ePTFE	9.6/0.94	60% porous[c]	Flexible	White	W.L. Gore and Assoc.	49
PE-1[d]	25.4/1.7	10	Rigid	White	GenPore	28
PE-2[d]	21.6/4.5	10	Rigid	White	GenPore	33
PE-3[d]	25.4/1.7	35	Rigid	White	GenPore	28
PE-4[d]	21.6/4.5	35	Rigid	White	GenPore	33

[a]Prices reflect costs at the time of purchase and should only be used as a guideline.
[b]Purchased from USPlastics, manufacturer unknown.
[c]Manufacturer describes the tubing wall as being 60% porous.
[d]Ultra high molecular weight polyethylene.

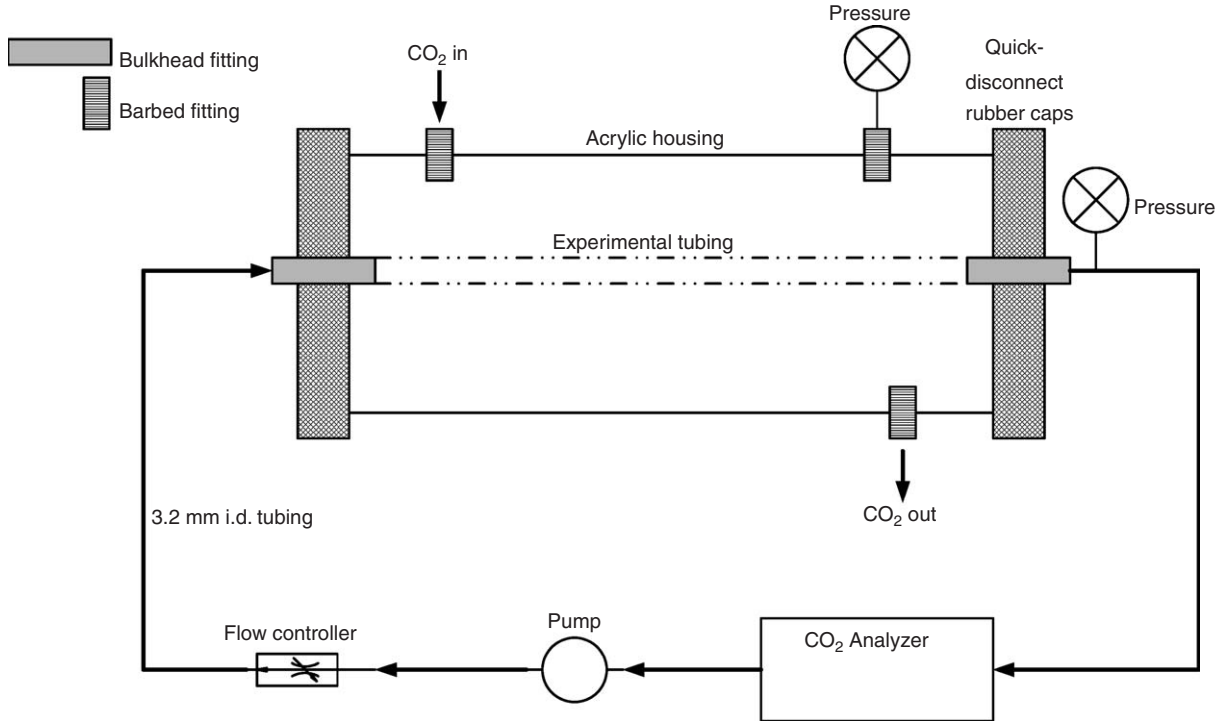

Fig. 1. Schematic drawing of the supplies and application design used to determine the diffusion coefficient of CO_2 through the walls of tubing.

2005), was allowed to flow freely into and out of the chamber (about 1.0 l min^{-1}) creating a constant concentration of CO_2 inside the chamber. The gas inside the experimental tubing, initially at ambient levels (370 μmol mol^{-1}) was circulated at about 0.5 l min^{-1} in a closed-loop using 3.2-mm-ID tubing that was resistant to CO_2 diffusion (Bev-A-Line IV; Thermoplastic Processes, Stirling, NJ) and a pump (NMP830-KNDC; KNF Neuberger, Trenton, NJ). The flow rate of the pump was adjusted using a rheostat and monitored using a flow meter (C-32045-18; Cole Parmer, Vernon Hills, IL). The flow rates through the tubing and chamber were adjusted accordingly so that the pressure differential between the chamber and tubing was less than 25 Pa measured with a pressure transducer (26410R1WB2DT1C; Setra Systems Inc., Boxborough, MA). The CO_2 that diffused from the chamber and into the tubing was determined and recorded every minute using a solid-state CO_2 sensor with a range of 1–10,000 μmol mol^{-1} (GMT222; Vaisala, Woburn, MA) and datalogger (21X; Campbell Scientific, Logan, UT), respectively. All diffusion measurements were conducted at 24 ± 0.5 °C.

Three replications of the silicone and ePTFE tubing and single tubes of each of the PE (Table 1) were exposed to laboratory conditions (25 °C, 20% relative humidity) with restricted light for 183 days. Also, three replications of silicone-1, silicone-2, silicone-3, and the ePTFE tubing were buried in soil (Harps loam) to a depth of 15 cm in a recently tilled field of corn (*Zea mays*) on 7 October 2004 to determine the affect of the soil environment on D and t_{eq}. The ends of the tubing were sealed prior to burial to prevent water and soil from entering the tubing. On 4 April 2005 (183 days later) the tubing was removed and D and t_{eq} were determined in the laboratory as above.

2.2. Data analysis

Following the methods of Jacinthe and Dick (1996), the collected data were fit to a first order diffusion model expressed as

$$\ln(C_h - C_i) = -kt_{eq} + \ln(q_0), \quad (1)$$

where C_h and C_i are the concentration of CO_2 in the chamber and the tubing at time t, respectively; q_0 is the CO_2 concentration difference across the tubing wall ($C_h - C_i$) at time $t = 0$; and k is the first order rate constant (min^{-1}) determined from a plot of $\ln(C_h - C_i)$ vs time (min).

The D ($cm^2\ s^{-1}$) for each tubing was determined using methods outlined by Jacinthe and Dick (1996) and was expressed as

$$D = \frac{kVL}{A}, \quad (2)$$

where V is the internal volume of the tubing (cm^3); L is the wall thickness of the tubing (cm); and A is the diffusing surface area of the tubing (cm^2). Using the collected information, the time required for 95% equilibration (t_{eq}) of the inner membrane CO_2 with the chamber CO_2 was determined from a modification of the first order diffusion model by solving (Eq. 1) for t and substituting $0.05q_0$ for $C_h - C_i$ (Jacinthe and Dick, 1996). Although a slight

pressure differential existed between the inside and outside of the tubing, this differential was not considered in the calculation of D.

Experimental factors that were statistically tested, using the Fisher LSD and Student's t test, were the types of membranes (silicone and ePTFE) and exposure to laboratory and soil environments (silicone and ePTFE), respectively.

3. Results

Determinations of k ($r^2 \geqslant 0.99$) were achievable in less than 60 min for all silicone tubing and less than 15 min for the ePTFE and PE tubing (Table 2). Burial did not appear to have affected values of k for the four different materials tested. PE-1 and PE-3 had slighter higher values of k (0.57 and 0.55 min^{-1}, respectively) than did PE-2 and PE-4 (0.45 and 0.41 min^{-1}, respectively), which may be explained by the thinner wall thicknesses of PE-1 and PE-3.

3.1. Diffusion coefficients

Plots of $\ln(C_h - C_i)$ vs t for both laboratory and field treated tubing have been provided in Fig. 2 for silicone-3 (A) and ePTFE (B). Plots of the $\ln(C_h - C_i)$ vs t for the remaining silicone and PE materials were very similar to A and B, respectively. The D of CO_2 through the wall of the ePTFE was significantly greater ($P = 0.05$) than for the three silicone membranes (Table 2). This result was not surprising based on the manufacturer's 60% porosity rating for the ePTFE (Table 1). Although only single samples of the different PE tubes were tested, the values of D were very similar to that of the ePTFE tubing. However, both of the thinner walled PE tubes (PE-1 and PE-3) had values of D greater than the thicker walled PE tubes (PE-2 and PE-4) and the ePTFE.

Values of D between laboratory and field-treated tubing were nearly identical and no significant differences ($P = 0.05$) in D were observed for any of the materials tested (Table 2). The field-exposed tubing was carefully inspected after removal from the soil. Neither the inside or

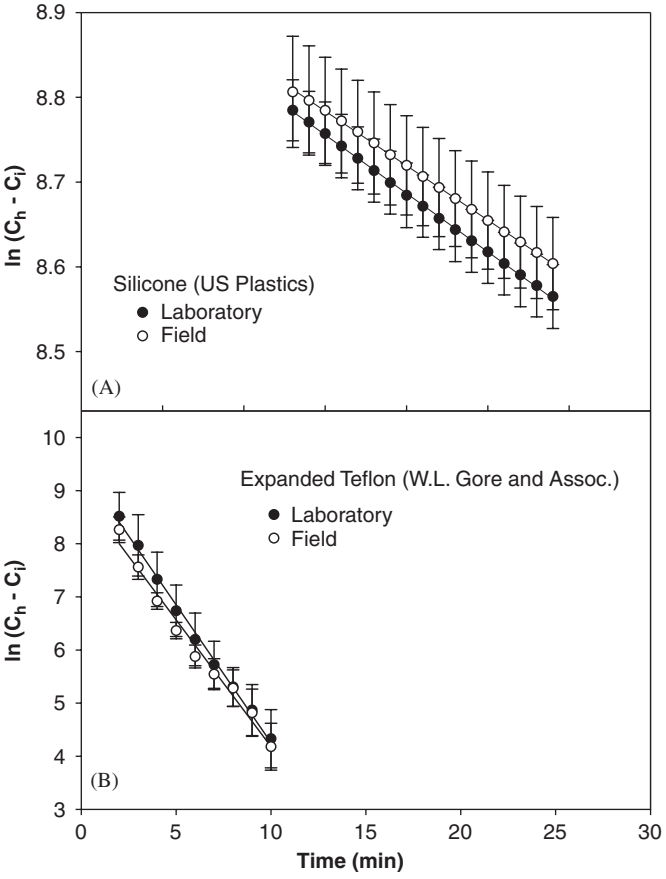

Fig. 2. Data fit to first order diffusion model for a silicone (A) and expanded Teflon tubing (B). Error bars represent standard deviations.

Table 2
First order rate constant (k), coefficient of determination (r^2) of the determined values of k, diffusion coefficient (D), and time for 95% equilibrium (t_{eq}) for various tubing products that were new or field-exposed

Designation	New				Field-exposed			
	k (min^{-1})	r^2	D ($cm^2\ s^{-1}$)	t_{eq}[a]	k (min^{-1})	r^2	D ($cm^2\ s^{-1}$)	t_{eq}[a]
Silicone-1	6.03×10^{-3}	0.99	6.88×10^{-6} (4.3) ab[b]	8.3 h	6.29×10^{-3}	0.99	7.10×10^{-6} (2.0)	7.9 h
Silicone-2	5.17×10^{-3}	0.99	5.80×10^{-6} (3.5) a	9.7 h	5.50×10^{-3}	0.99	6.22×10^{-6} (3.5)	9.1 h
Silicone-3	0.01	0.99	8.60×10^{-6} (4.0) b	4.1 h	0.01	0.99	8.64×10^{-6} (4.4)	3.9 h
ePTFE	0.51	0.99	1.23×10^{-4} (2.4) c	5.9 min	0.51	0.99	1.25×10^{-6}(8.1)	5.9 min
PE-1[c]	0.57	0.99	7.70×10^{-4}	5.3 min				
PE-2[c]	0.45	0.99	6.27×10^{-4}	6.7 min				
PE-3[c]	0.55	0.99	7.20×10^{-4}	5.5 min				
PE-4[c]	0.41	0.99	5.57×10^{-4}	7.3 min				

All values represent the average of three replications unless noted. Numbers in parentheses represent the coefficient of variability.
[a]Time for 95% equilibration.
[b]Means in column followed by the same letter are not significantly different at $P = 0.05$.
[c]Measurements were performed on a single sample.

outside of the tubing exhibited damage/deformation due to prolonged exposure in the soil.

3.2. Equilibration

The time for 95% equilibration (t_{eq}) was greatest with the three silicone materials. Silicone-1 and silicone-2, both of which had wall thicknesses of 3.2 mm, had t_{eq} values of 7.9 and 9.7 h, respectively (Table 2). In contrast, silicone-3, which had a wall thickness of 1.6 mm, had a t_{eq} of about 4.0 h. Values for t_{eq} for the ePTFE were 5.9 min and for the PE materials ranged from 5.3 min with the thin-walled tubing (PE-1, wall = 1.7 mm) and up to 7.3 min with the thicker-walled tubing (PE-4, wall = 4.5 mm).

4. Discussion

With the assistance of permeable tubing, the concentration profiles of gases beneath the soil surface can be evaluated. The choice of permeable tubing may be a function of the gases of interest, timescale of the experiment, and available resources. The evaluation of the tubing in this paper clearly provides evidence that CO_2 can move into the inner-tube volume from an outside source. The polymer used to construct the tubing and also the permeability of that polymer will dictate diffusion of CO_2 across the tubing wall. Although the three silicone tubing materials evaluated here share the same molecular makeup and thus have similar values of D, the curing process (i.e. platinum-cured vs peroxide-cured) can affect the overall porosity and movement of CO_2 through the tubing wall. Similar results were observed with the GenPore tubing, where a significant relationship between D, pore size, and wall thickness was observed.

The D of CO_2 through the silicone tubing used by Holter (1990) was determined by Jacinthe and Dick (1996) to be $1.8 \times 10^{-5}\,cm^2\,s^{-1}$. This value is greater than the values reported for CO_2 here (Table 2). Holter (1990) used a static method to measure the atmospheric composition of dung pats and thus, differences in our methodologies (dynamic vs static, temperature) may account for these discrepancies. The D of N_2O was also determined by Jacinthe and Dick (1996) from regular silicone tubing and reinforced silicone tubing. At 22 °C, the D of N_2O through the tubing wall of the regular and reinforced tubing was 1.4×10^{-5} and $8.8 \times 10^{-6}\,cm^2\,s^{-1}$, respectively, which was slightly greater than the values of D for CO_2 reported here. In free air, the values of D for CO_2 and N_2O are both $0.139\,cm^2\,s^{-1}$ (Sommerfeld et al., 1993) and, unless preferential sorption of either compound occurs in the tubing, the values of D through the tubing should be similar.

Sampling frequency can also be an important consideration when selecting permeable tubing. Diurnal fluctuations of CO_2 in the soil are well documented and monitoring these changes requires the use of permeable tubing that can equilibrate quickly with a changing environment. Both the ePTFE and PE tubing allow for fast equilibration times (<10 min) with surrounding gases whereas the silicone tubing required times of 4 h or greater. Temperature can also affect the diffusion rates and thus equilibration times of gases as noted by Jacinthe and Dick (1996). The results of their experiment indicate that as the temperature of the tubing is decreased the equilibration time is increased. Although not investigated here, one can predict that the tubing investigated here would behave similarly to the tubing used by Jacinthe and Dick (1996) in their N_2O experiments. Since an objective of taking gas samples from soil might be to look at concentration profiles, then using tubing having fast equilibration times would lessen the temperature-induced changes in diffusion coefficients of the tubing on soil gas samples taken at time t. For example, when using fast-equilibrating tubing, gas samples taken at shallow depths during early afternoon when soil temperatures are reaching maximum values can confidently be compared to deeper depth samples where the soil temperature is cooler. In contrast, using silicone tubing that may not have a fast equilibration time may tend to promote a lag in gas concentration at cooler temperatures compared to warmer temperatures and thus not allow for comparable gas concentration data.

Exposure of membranes to the soil environment may influence the diffusion properties of the materials and resulting gas concentration information. Kramer and Conrad (1993) determined, through scanning electron microscopy, that biofilms were present on a silicone membrane after brief exposure (1 day) to a paddy soil. These authors concluded that biogenic gases produced from these biofilms might influence the concentrations of biogases in the soil atmosphere samplers. The integrity of the membranes, as a result from exposure to micro- and macro-fauna and rodents, should also be considered. Another consideration is environment in which these materials are used. The force needed to "pinch" the ePTFE tubing was determined to be about $16\,g\,mm^{-2}$ and thus, may not be suitable for deep burial or rocky soils. However, the PE membranes evaluated in this study were very rigid and could be used in most soil atmospheric studies.

Finally, costs of the tubing may be a factor when designing an experiment to measure soil–gas concentrations. The tubing that allowed for the fastest equilibration times, of the materials studied, were also the most expensive. The ePTFE tubing was 49 US$ m^{-1} and the two GenPore sizes were 28 and 33 US$ m^{-1} (Table 1). In contrast, the USPlastics silicone tubing was only 3 US$ m^{-1} and the Tygon 3305 and Versilic SPX-50 tubing were 19 and 24 US$ m^{-1}, respectively. Thus, with time sensitive applications, such as assessment of diurnal changes in soil–gas concentrations, use of the more expensive tubing would be justified. However, in applications where fast response time is not critical, use of the lesser expensive tubing may be justified.

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Reference to a trade or company name is for specific information only and does not imply approval or recommendation of the company or product by the USDA to the exclusion of others that may be suitable.

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