Basis for Comparisons of Soil CO$_2$ Respiration Test Procedures

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McGOWEN et al. (2018) recently reported a gas chromatograph (GC) method for soil CO$_2$ respiration, claiming advantages in time, labor, and precision compared with Solvita (Woods End Laboratories), a commercial soil respiration method. Herein we address core ideas that we find insufficiently supported and outline gaps in their description of methods.

When two methods are compared, circular reasoning is invoked, and therefore it is ideal to have additional method comparisons. We applaud the fact that the authors agreed and provided our team the same set of refrigerated soils from which their conclusions were drawn. We performed several respiration tests, including Solvita and three differing infrared gas analyzers (IRGA).

Two methods may be considered equivalent when the r^2 is high, slope approaches 1.0, and the intercept is close to zero. The authors demonstrated significant correlation between GC and Solvita ($r^2 = 0.73$). However, the slope was very low (0.226); thus, their GC method measured CO$_2$ significantly lower than Solvita. We found that their GC method also gave much lower results compared with IRGA methods. Moreover, when we compared a LiCor 7000 CO$_2$ IRGA to Solvita, we obtained a slope of 1.05 at a zero intercept ($r^2 = 0.887$)—essentially, a perfect correspondence. We corroborated Solvita's accuracy by the new IRTH method, embedding an infrared sensor inside a Solvita jar during the test, resulting in $r^2 = 0.92$ and a slope of 0.945. Incidentally, this confirms earlier work by Chitraker et al. (2006) showing a linear range of detection for Solvita compared with calibration gases from 0 to 3% CO$_2$ ($r^2 = 0.99$). In Table 1, we show several of the comparisons we made with the respective r^2 values and slopes (intercepts adjusted to zero).

We question the time advantage claimed for GC over Solvita. Both methods require similar setup and incubation. The authors report GC requires 8 min per test (McGowen et al., 2018). The Solvita digital reader enables four samples per minute, including labor. Recognizing GC autosamplers as generally labor saving, it is still difficult to comprehend how the GC method will ever allow the high-throughput per day for which Solvita is designed.

We ran all our trials in similar-sized 475-mL jars, which is very common for IRGA soil methods. We obtained the best data with the 475-mL jars as compared to the 265-mL jars commonly used for Solvita. Increasing jar size would address the concerns raised in the original paper about oversaturation of Solvita at higher values (presumably >3% CO$_2$). Larger jars dilute the CO$_2$ concentration to be within an acceptable range. This is likely to eliminate or minimize the need for dilution and sample rerun with the Solvita method, which may have been part of the authors' calculation regarding time disadvantages.

We had difficulty understanding the authors' reported free air space in GC vials, an important number since CO$_2$ mass is calculated via the Ideal Gas Law. Normally, airspace is jar volume minus soil volume corrected for particle density. The authors refer to headspace as 0.015 L (vials were 0.020 L), suggesting that their 5-g soil sample equated to 5 mL, which would skew calculations.

The authors indicated difficulty rewetting soils for respiration, which may explain differences comparing Solvita results. Presently, 50% water-filled pore space is favored over a blanket addition of water. Brinton and Burger (2015)

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Abbreviations: GC, gas chromatograph; IRGA, infrared gas analyzer.

Table 1. Soil respiration methodological comparisons conducted using Oklahoma soils from McGowen et al. (2018).

Method 1 (*y*)†	Method 2 (*x*)†	r_2	Slope
OKSU-GC	UME IR	0.7847	0.505
OKSU-GC	WE-CZ	0.6661	0.485
OKSU-GC	WE-SOL	0.8126	0.538
OKSU-GC	OKSU-SOL	0.7331	0.226
OKSU-GC	WE-IRTH	0.7123	0.562
OKSU-SOL	WE-SOL	0.5790	2.291
OKSU-SOL	WE-CZ	−0.2630	1.990
UME IR	WE-CZ	0.7506	0.953
UME IR	WE-SOL	0.8866	1.047
WE-CZ	WE-SOL	0.8559	1.054
WE-IRTH	WE-SOL	0.9214	0.945
WE-IRTH	UME IR	0.8815	0.926

† CZ, COZIR IR cell; GC, gas chromatograph; IR, LiCor 7000 IRGA; IRTH, embedded IR cell; OKSU, Oklahoma State University data; SOL, Solvita; UME, University of Maine data; WE, Woods End Laboratory data.

reported the need for this new wetting protocol to avoid respiration suppression caused by the Haney method (Haney and Haney, 2010), which was discontinued in 2016 (Solvita, 2016).

A factor that may influence the amount of CO_2 determined after dried soil is rewetted is the ratio of headspace volume to soil in jars or tubes used. We are skeptical that micro-methods can be made reliable for soil respiration. We speculate that the miniaturized GC method is problematic, possibly because the small quantity of soil is difficult to moisten properly or because aerobic respiratory suppression occurs. Using our own data, we calculated that with the authors' jar configuration, CO_2 concentrations as high as 7.75% of headspace may have resulted, whereas the GC was calibrated only to 40,270 $\mu L\ L^{-1}\ CO_2$. A semiautomatic respiration method has been reported using a IR CO_2 alarm detector limited to 0 to 2% CO_2 (Haney et al., 2018),

which presumably must be dealt with similarly by increasing jar size, reducing soil quantity, or recalibrating beyond the design specifications for the instrument with potential loss of resolution. Therefore, issues of accuracy related to calibration and design limits of instrumentation are a common feature for all methods of measuring soil respiration.

We commend Oklahoma State University for the choice of soils in this study representing a wide range of respiratory activity, ideal for method comparisons, and for offering insight into soil health. The authors' team selected nine regions with paired comparisons of tilled and no-till farms. Our ANOVA analysis showed that all methods differentiated no-till versus conventional at a high degree of statistical significance. This implies that all these soil respiration tests do illuminate real differences in soil health. The path to method improvement will undoubtedly be never-ending; however, we should not lose sight of the enduring importance of distinguishing management impacts on soil health.

References

Brinton, W., and B. Burger. 2015. Solvita for soil respiration: Influence of various soil factors. Paper presented at: Synergy in science: Partnering for solutions. ASA, CSSA, and SSSA 2015 Annual Meetings, Minneapolis, MN. 15–18 November.

Chitraker, S., C.J. Bern, and D.S. Shresta. 2006. Quantifying corn deterioration due to fungal growth by use of CO_2-sensitive gel. Appl. Eng. Agric. 22:81–86. doi:10.13031/2013.20174

Haney, R.L., and E.B. Haney. 2010. Simple and rapid laboratory method for rewetting dry soil for incubations. Commun. Soil Sci. Plant Anal. 41:1493–1501. doi:10.1080/00103624.2010.482171

Haney, R.L., E.B. Haney, M.J. White, and D.R. Smith. 2018. Soil CO_2 response to organic and amino acids. Appl. Soil Ecol. 125:297–300. doi:10.1016/j.apsoil.2017.12.016

McGowen, E.B., S. Sharma, S. Deng, H. Zhang, and J.G. Warren. 2018. An automated laboratory method for measuring CO_2 emissions from soils. Agric. Environ. Lett. 3:180008. doi:10.2134/ael2018.02.0008

Solvita. 2016. Soil CO_2 burst official method version 2016.1. Woods End Laboratories, Mt. Vernon, ME.