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ORIGINAL PAPER

A cost-effective method for reducing soil disturbance-induced errors in static chamber measurement of wetland methane emissions

R. Scott Winton · Curtis J. Richardson

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Abstract Static chambers used for sampling methane (CH_4) in wetlands are highly sensitive to soil disturbance. Temporary compression around chambers during sampling can inflate the initial chamber CH_4 headspace concentration and/or lead to generation of non-linear, unreliable flux estimates that must be discarded. In this study, we tested an often-used rubber gasket (RG)-sealed static chamber against a water-filled gutter (WFG) seal design that could be set up and sampled from a distance of 2 m with a newly designed remote rod sampling system to reduce soil disturbance. Compared to conventional RG design, our remotely sampled static chambers reduced the chance of detecting inflated initial CH_4 concentrations (>3.6 ppm) from 66 to 6 % and nearly doubled the proportion of robust linear regressions ($r^2 > 0.9$) from 45 to 86 %. Importantly, the remote rod sampling system allows for more accurate and reliable CH_4 sampling without costly boardwalk construction. This paper presents results demonstrating that the remote rod sampling system combined with WFG static chambers improves CH_4 data reliability by reducing initial gas measurement variability due to chamber disturbance when tested on a mineral soil-restored wetland in Charles City County, Virginia, USA.

Keywords Gas flux · Greenhouse gas · Methane · Static chamber · Wetland

Introduction

Methane (CH_4) is the second most important long-lived greenhouse gas (GHG) after carbon dioxide (Myhre et al 2013), with wetlands representing the single most important source to the atmosphere (Bridgham et al 2006). As a result, the estimation of CH_4 flux has become an important component of many studies of wetland carbon biogeochemical cycling, with the non-flow-through-non-steady-state chamber method (Livingston and Hutchinson 1995) or "static chamber" method, most popular because of its simplicity and cost-effectiveness.

It is popular to use simple linear regression of chamber headspace concentrations as a function of time to estimate gas emissions' rate (Weishampel and Kolka 2008), but the act of repeated sampling from static chambers can cause soil disturbance and significant deviations from linearity (Forbrich et al 2010). Dissolved CH_4 within saturated or inundated wetland soil pores is particularly sensitive to pressure changes, which can lead to CH_4 ebullition (Strack et al 2005). Ebullition can occur in the absence of artificial

R. S. Winton (✉) · C. J. Richardson
Nicholas School of the Environment, Duke University Wetland Center, Duke University, Durham, NC 27708, USA
e-mail: scott.winton@duke.edu

disturbance, but disturbance-induced ebullition is typically the cause of non-linear (based on an r^2 threshold of 0.9) or unusable CH_4 flux data. Studies do not always report how much data are discarded due to soil disturbance, but anywhere from 45 % (Nahlik and Mitsch 2010) to 65 % (Morse et al 2012) of static chamber incubations can be affected. Loss of such a high proportion of data is not only resource inefficient but also poses the threat of introducing sampling errors and bias.

After observing persistent evidence that disturbance was affecting our CH_4 flux results using a rubber gasket (RG) static chamber (Weishampel and Kolka 2008), we redesigned our static chambers to incorporate a water-filled gutter (WFG) seal (Livingston and Hutchinson 1995; Wang et al 2006; Krauss and Whitbeck 2011), which allows setup and sampling to be conducted from a distance of 2 m using a remote rod sampling system (RRSS) so as to minimize wetland soil disturbance (Fig. 1). Here we compare reliability data from our WFG static chamber with RRSS compared against those of a more conventional RG static chamber. We also present reliability data testing whether the WFG seal alone or remote setup using the RRSS in conjunction with the WFG is necessary to improve reliability.

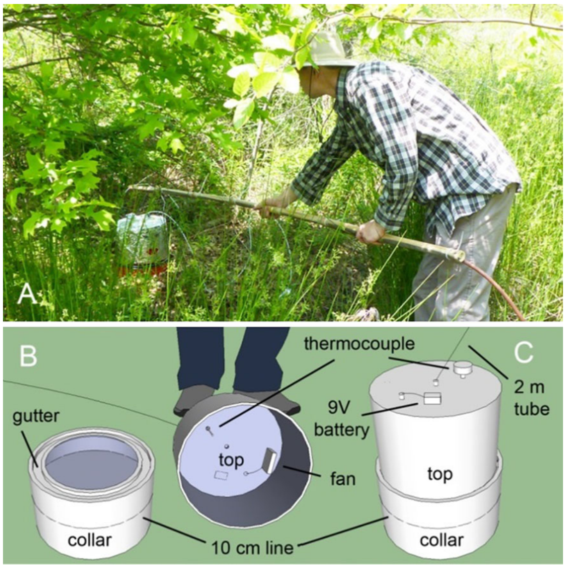

Fig. 1 Illustrations of the water-filled gutter static chamber design that allows the use of remote rod sampling system (RRSS): Photograph of chamber being deployed using RRSS in the Charles City Wetland in Charles City County, Virginia (*a*); schematic of chamber disassembled to reveal water-fillable gutter on rim of collar that creates an air tight seal, internal fan to mix headspace air and thermocouple to monitor internal chamber temperature (*b*); schematic of chamber assembled (*c*)

Our findings may help improve field effort efficiency and eliminate boardwalk construction costs for those planning to utilize static chambers for estimation of CH_4 gas flux from wetlands.

Methods

The study took place within the 20.8-hectare Charles City Wetland Mitigation Site, which is located in Charles City County, Virginia, USA, and owned by the Virginia Department of Transportation as part of its compensatory mitigation program (Bailey et al. 2007). Precipitation is the dominant hydrologic input and the CCW may hold up to 0.5 m of standing water during cooler months (Bailey et al 2007). The soil is mapped as a complex of Chickahominy (fine, mixed, semiactive, thermic Typic Endoaquults) and Newflat (fine, mixed, subactive, thermic Aeric Endoaquults) (Bergschneider 2005). Site history is complex, with one unsuccessful restoration attempt for this formerly forested wetland being followed by an experimental addition of organic matter amendments to improve tree growth. For more detail on site characteristics, see Bruland and Richardson (2004), Bergschneider (2005), and Bailey et al. (2007).

In late summer 2011, we imbedded a 20-cm-diameter PVC collar 10–15 cm into the soil in each of 20 plots (4.6 by 3.1 m) for RG static chamber trace gas sampling. We set up the RG static chambers by placing a PVC cap with a rubber gasket over beveled collar tops by hand. Mean assembled RG chamber height was approximately 13.5 cm corresponding to a volume of >4 L. We sampled gas using RG static chambers from 20 collars in September and October 2011 and from 27 collars in February 2012 for a total of 67 sets of gas measurements.

In spring of 2012, we imbedded 45-cm-diameter plastic collars affixed with water-fillable gutters to a depth of 10–15 cm in the same 20 plots for WFG static chamber trace gas sampling using RRSS. Both WFG and RG chamber caps are equipped with an internal computer fan powered by a 9-volt battery to circulate chamber headspace as recommended by Christiansen et al (2011) and a thermocouple allowing for internal chamber temperature (T) to be recorded during each sample extraction. We coated the WFG caps with reflective aluminum foil to minimize solar warming as recommended by the US Department of Agriculture (Parkin and Venterea 2010).

We set up and sampled WFG static chambers from a distance of approximately 2 m using our RRSS in which we fill the gutter with water using a 2-m polyethylene tube and place the plastic top using a 2-m rod. We then extracted air samples using a plastic syringe via the 2-m, 1-mm-inner diameter plastic tube following flushing to mix tube air with chamber headspace (total tube volume is 1.6 mL—approximately 0.01 % of headspace volume). The mean height of assembled WFG chamber was 23 cm with headspace volume ranging from approximately 10 L during periods of high water to 16 L during dry periods. We sampled CH_4 gas using WFG static chambers from 20 collars in May, July, September (twice), and November 2012 and February 2013 for a total of 120 sets of gas measurements. During one of the two sampling dates in September 2012, we sampled 10 of the 20 WFG static chambers without using the RRSS for setup. Instead we simply walked up to fill the gutter and placed the chamber top by hand (the traditional method, required when using RG) to test for disturbance during WFG setup without RRSS.

After setup of both RG and WFG static chambers, we immediately extracted a 50-mL headspace sample via a plastic syringe and deposited it into a mylar gas-tight sample bag. We recorded ambient air temperature (T), internal chamber T, soil T at 5 cm depth for initial and subsequent samples taken approximately 5, 15, and 30 min following chamber setup. Total extracted sample volume (200 mL) was never more than 5 % of total headspace volume and acts to counterbalance the pressure buildup from emitted gases over 30 min of incubation. We transported gas bags to the Duke University Wetland Center laboratory and analyzed within one week of sampling on a Varian 450 Gas Chromatograph (GC) equipped with a flame ionization detector. We analyzed duplicates of all samples, and when duplicate values differed by <10 %, we used the mean for flux calculations.

In addition to CH_4, we simultaneously measured carbon dioxide (CO_2) concentrations using a methanizer in our GC, allowing us to test for static chamber sampling effects on CO_2. Since CO_2 is approximately 50 times more soluble in water, it is far less susceptible to disturbance-induced ebullition effects and did not manifest obvious performance differences between RG and WFG chambers with or without the RRSS system. Therefore, we limit the scope of this paper to the effects we observed on CH_4 reliability. CH_4 flux estimates at the CCW (described in detail in Winton and Richardson 2015) range from below detection limits to as high as approximately 5 mg m^{-2} h^{-1}.

We consider two performance metrics in our evaluation of chamber reliability: (1) the initial headspace CH_4 concentration (C_{initial}) as sampled immediately after chamber setup, with higher values indicative of more soil disturbance during chamber setup, and (2) r^2 of the calculated regression line.

We separately analyzed data from one of our two sampling dates in September 2012 when 10–20 plots were sampled without using the RRSS for WFG static chamber setup, to evaluate the importance of the RRSS in improving data reliability over WFG static chambers setup by hand.

To test for differences in mean C_{initial} between RG ($n = 67$) and WFG with RRSS ($n = 100$) static chamber methods, and between WFG with and without the RRSS, we use Welch's t test on data log transformed to better meet the assumption of a normal distribution. To test for differences in mean r^2 between RG and WFG with RRSS methods, we excluded 54 "no flux" regressions (Pedersen et al 2010) that showed insignificant deviations from ambient CH_4 concentration over the four sample points since relatively minor concentration differences stemming from experimental or analytical error often produced a low r^2 value unrelated to chamber or soil disturbance during setup or sampling. We used the non-parametric Mann–Whitney U test on rank-transformed r^2 absolute values from the remaining linear regressions since the distributions of r^2 absolute values do not adequately meet the normal distribution parameter required by t tests.

Since we sampled from WFG chambers with the RRSS subsequent to our sampling from RG chambers (rather than a side-by-side comparison), it is necessary to investigate whether site conditions can explain differences in performance results. We consider water level and chamber T as potentially important site condition variables because each of them has potential to influence the pressure on interstitial soil gases and ebullition. To evaluate the relative importance of site conditions on CH_4 data quality, we compared the mean and standard deviation of standing water level and T between static chamber methods. We compared chamber T recorded between methods using a Welch's *t* test. Conventional statistical tests are not appropriate for water level, which contains excessive zeros (indicating water below the soil surface), so we converted the water level data to a binary factor indicating the presence/absence of ponded water. We tested whether the presence/absence of ponded water or chamber design had a more important impact of log-transformed t_0 CH_4 concentration using a 2-way analysis of variance (ANOVA) and Tukey's honest significant difference test.

Results

Incubations from selected plots (sampled 21 October 2011 using RG; 26 September 2012 using WFG with RRSS) illustrate the differences in data reliability typically observed between the two sampling methods. RG static chambers often produced $C_{initial}$ values that were inflated one to three orders of magnitude above ambient CH_4 concentration with non-linear CH_4 accumulation over time (Fig. 2a). In contrast, WFG static chambers sampled using the RRSS regularly produced circum-ambient $C_{initial}$ values and linear slopes (Fig. 2b).

The use of RG static chambers produced a $C_{initial}$ greater than 3.6 ppm (double ambient CH_4 concentration) in 44 out of 67 (66 %) incubations and a $C_{initial}$ greater than 18 ppm (10 times ambient CH_4 concentration) in 30 out of 67 (45 %) incubations compared to 6 out of 100 (6 %) and 0 out of 100 (0 %) when using WFG static chambers with RRSS (see Fig. 3a). Welch's *t* test measured significantly higher mean $C_{initial}$ values ($p < 0.0001$) from RG compared to WFG static chambers with RRSS.

The use of RG static chambers produced r^2 greater than 0.9 for just 29 out of 64 (45 %) non-"no flux" linear regressions compared to 42 out of 49 (86 %) for those of the WFG static chamber with RRSS (see Fig. 3b). Mann–Whitney U test found significantly lower mean r^2 values ($p < 0.0001$) from RG compared to WFG static chambers with RRSS.

To determine if environmental site conditions found in 2011 versus 2012 can explain the differences in performance results between the RG compared to WFG static chambers with RRSS, we compared water tables and soil temperatures. We found that chamber T was similar during RG and WFG static chamber sampling both years, with means (±standard deviation) of 21.6 (±3.3) and 20.5 (±7.0) degrees C, respectively. A Welch's *t* test found the difference in mean chamber T to not be significant ($p = 0.18$). Water level data suggest that conditions may have been wetter during RG static chamber sampling compared to WFG, with mean (±standard deviation) standing water of 1.8 cm (±2.8) and 0.8 cm (±1.7) cm, respectively. Ponded water was present for 32 of 67 (48 %) RG chamber incubations compared to just 30 out of 100 (30 %) of WFG chamber incubations. However, a 2-way ANOVA and Tukey's honest significant difference test found that differences in $C_{initial}$ were related to sampling method ($p = 0.0001$) rather than the presence/absence of ponded water ($p = 0.86$).

We tested whether it was necessary to use our RRSS with the WFG by sampling half the WFG chambers without the RRSS in September 2012. In these 10 incubations, the RRSS system never produced $C_{initial}$ values greater than 3.6 ppm. When we did not use the RRSS, in contrast, all $C_{initial}$ values were greater than 3.6 ppm with 5 out of 10 being greater than 18 ppm (see Fig. 4). The Welch's *t* test found that the RRSS produced significantly lower ($p < 0.001$) mean $C_{initial}$ values. When we used the RRSS, 9 out of 9 incubations that did not meet "no flux" criteria produced r^2 values greater than 0.9 compared to just 7 out of 10 when we did not use the RRSS for setup. While, the Mann–Whitney U test for a difference in mean r^2 values did not produce sufficient evidence to reject the null, the comparison shows that more samples will be lost when the RRSS system is not used.

Discussion

In studies of CH_4 flux from wetlands, static chamber design and setup can have a huge influence on data reliability and therefore on the efficiency of data collection effort. The use of a RG-style static chamber (Weishampel and Kolka 2008) or any design that requires a close approach during chamber setup and sampling has the potential to cause soil compression resulting in high initial CH_4 values. While disturbance may be imperceptible to a field observer, our data suggest it can artificially inflate $C_{initial}$ and/or reduce

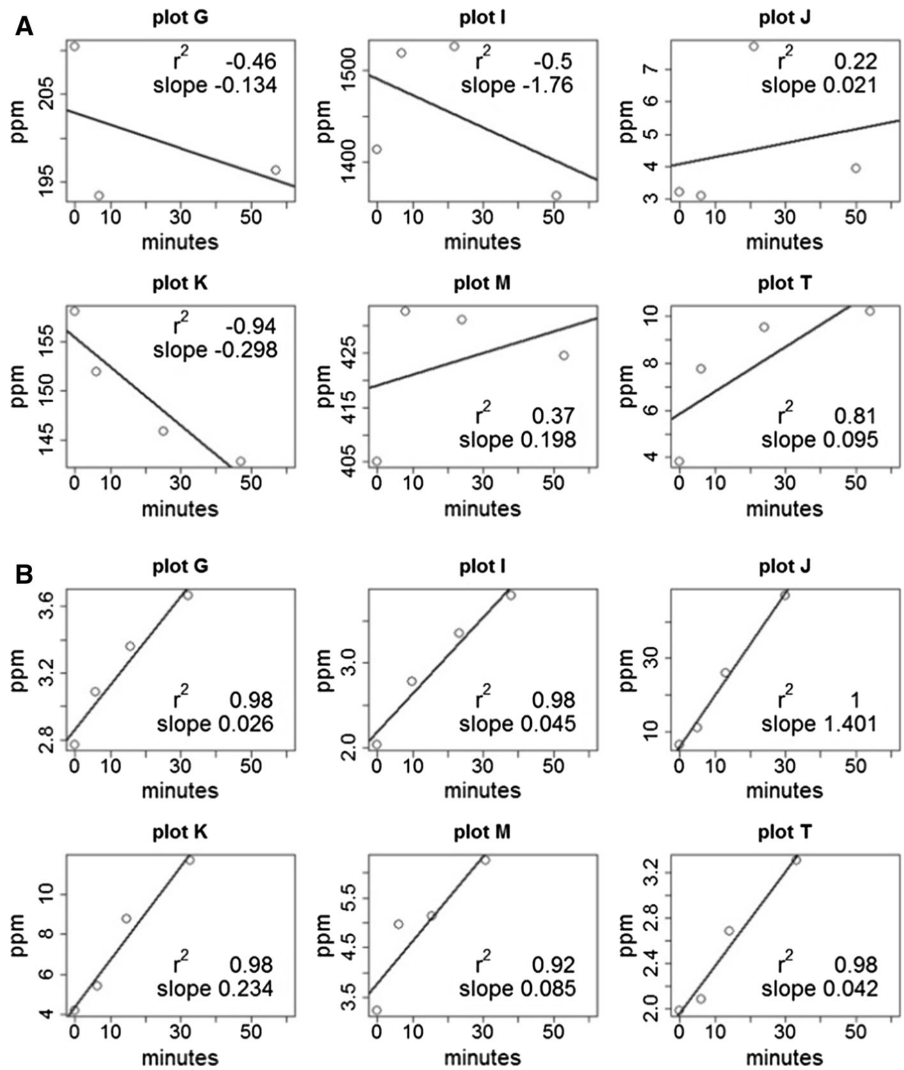

Fig. 2 Examples of gas data illustrating unsuccessful use of a rubber gasket static chamber design on 21 October 2011(**a**) and successful use of water-filled-gutter static chamber design with remote rod sampling system on 26 September 2012 (**b**)

linear regression r^2 such that a large portion of flux estimates must be discarded, thus reducing statistical power and introducing a potential source of bias.

We found that a WFG static chamber design used in conjunction with our newly developed RRSS produced more reliable and readily-useable CH_4 concentration data for flux estimates compared to other wetland studies that have reported static chamber CH_4 data loss (Nahlik and Mitsch 2010; Morse et al 2012). Elevated $C_{initial}$ values, which we frequently detected when not using the RRSS, indicate that CH_4 has been purged from soil pores into the chamber headspace during setup. While this could be related to pushing on the PVC cap of RG static chambers to form a good seal, the results of our test of the RRSS for WFG static chamber setup suggest that elevated $C_{initial}$ can be caused by merely standing close to the chamber collars with either RG or WFG designs. Elevated $C_{initial}$ on its own may not necessarily lead to a decrease in r^2, but repeated approaches to the chamber during each of the four sampling time points, as we did before implementing the RRSS, significantly compromised regression linearity, thus leading to a rejection of a large number of measurements.

For decades, researchers have discussed optimal chamber design for achieving precision and accuracy in the estimation of gas flux from soils (Hutchinson and Mosier 1981; Anthony et al 1995; Conen and Smith 2000; Forbrich et al 2010). The data we present here demonstrate that disturbance of wetland soil

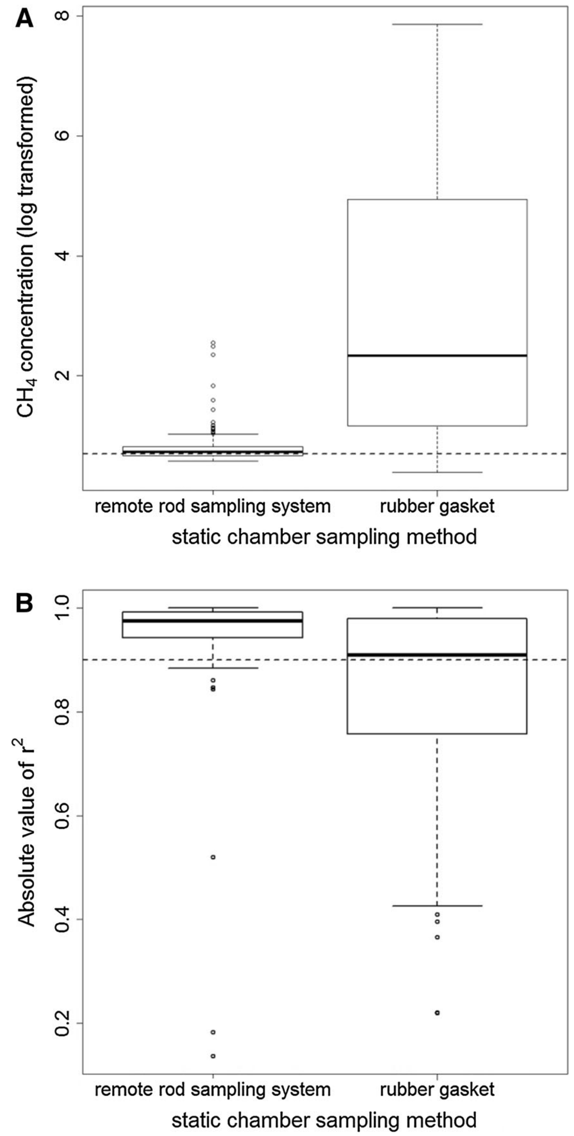

Fig. 3 Tukey boxplots of log-transformed initial CH_4 concentrations in chamber headspace (**a**) and r^2 value of linear regression line used to estimate CH_4 flux (**b**) from 67 incubations using the rubber gasket static chamber design taken from September 2011 through February 2012, and 110 incubations using the water-filled gutter with remote rod sampling system method taken from May 2012 through January 2013. *Dashed lines* mark ambient CH_4 concentration (**a**) and r^2 threshold of 0.9 (**b**)

around static chambers can be effectively avoided by employing a WFG that allows for chambers to be set up and gas to be sampled using a RRSS to prevent soil disturbance. We suggest that disturbance is far more likely to impair data reliability than other potential

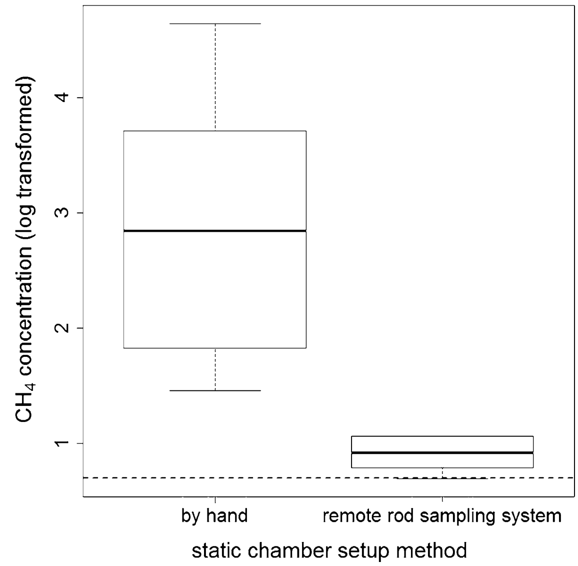

Fig. 4 Tukey boxplot of log-transformed initial CH_4 concentration in chamber headspace from 10 incubations using remote rod sampling system to setup the chamber cap to minimize disturbance and from 1110 incubations for which the chamber cap setup by hand. *Dashed line* marks ambient CH_4 concentration

sources of error endemic to the static chamber method, such as slight variations in internal chamber pressure due to extracted sample volume.

Chambers measured continuously using laser off-axis integrated cavity output spectroscopy (Mastepanov et al 2008), automated flux chambers (Scott et al 1999), and eddy covariance towers (Kormann et al 2001) are viable alternative methods for estimating methane flux from wetlands while avoiding soil disturbance, but they can increase costs by two or three orders of magnitude and the latter may not be appropriate for small footprint applications. The manual static chamber method remains popular because of its efficiency and cost-effectiveness, yet in a wetland setting a standard practice to avoid soil disturbance during sampling is to construct boardwalks, which can be costly, labor intensive, and logistically impractical. Our newly developed RRSS circumvents the need for boardwalks.

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Compliance with Ethical Standards

Conflict of Interest We declare no conflict of interest.

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