

# Duration of extraction determines CO<sub>2</sub> and CH<sub>4</sub> emissions from an actively extracted peatland in eastern Québec, Canada

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**Abstract.** Draining and extracting peat alters the conditions that control CO<sub>2</sub> and CH<sub>4</sub> emissions. Carbon (C) emissions from  
15 peatlands undergoing horticultural peat extraction are not well constrained due to a lack of measurements. We determine the  
effect that production duration (years of extraction) has on the CO<sub>2</sub> and CH<sub>4</sub> emissions from an actively extracted peatland  
over three years of measurements (2018-2020). We studied five sectors identified by the year when extraction began (1987,  
2007, 2010, 2013, 2016). Greater average CO<sub>2</sub> and CH<sub>4</sub> emissions were measured from the drainage ditches (CO<sub>2</sub>: 2.05 ± 0.12  
g C m<sup>-2</sup> d<sup>-1</sup>; CH<sub>4</sub>: 72.0 ± 18.0 mg C m<sup>-2</sup> d<sup>-1</sup>) compared to the field surface (CO<sub>2</sub>: 0.9 ± 0.06 g C m<sup>-2</sup> d<sup>-1</sup>; CH<sub>4</sub>: 9.2 ± 4.0 mg C m<sup>-2</sup>  
20 d<sup>-1</sup>) regardless of sector. For peat fields, CO<sub>2</sub> fluxes were highest in the youngest sector, which opened in 2016 (1.5 ± 0.2 g  
C m<sup>-2</sup> d<sup>-1</sup>). The four older sectors all had similar mean CO<sub>2</sub> fluxes (~0.65 g C m<sup>-2</sup> d<sup>-1</sup>) that were statistically different from the  
mean 2016 CO<sub>2</sub> flux. A spatial effect on CO<sub>2</sub> fluxes was observed solely within the 2016 sector, where CO<sub>2</sub> emissions were  
highest from the centre of the peat field and declined towards the drainage ditches. These observations occur due to operators'  
surface contouring to facilitate drainage. The domed shape and subsequent peat removal resulted in a difference in surface  
25 peat age hence different humification and lability. <sup>14</sup>C dating confirmed that the peat contained within the 2016 sector was  
younger than peat within the 2007 sector and that peat age is younger toward the centre of the field in both sectors. Humification  
indices derived from mid-infrared spectrometry (MIRS) (1630/1090 cm<sup>-1</sup>) indicated that peat humification increases with  
increasing years of extraction. Laboratory incubation experiments showed that CO<sub>2</sub> production potentials of surface peat

samples from the 2016 sector increased toward the centre of the field and were greater than for samples taken from the 1987 and 2007 sectors. Our results indicate that peatlands under extraction are a net source of C where emissions are high in the first few years after opening a field for extraction and then decline to about half the initial value and remain at this level for several decades, and the ditches remain a 2 to 3 times greater source than the fields, but represent < 3.5% of the total area of a field.

## **1 Introduction**

### **1.1 CO<sub>2</sub> and CH<sub>4</sub> Production in Natural Peatlands**

Peatlands are important carbon (C) sequestering ecosystems containing one-third of global soil C stores (Limpens et al., 2008; Yu, 2012). Generally, peatlands are sinks of carbon dioxide (CO<sub>2</sub>) and sources of methane (CH<sub>4</sub>), although this can vary interannually and is dependent on environmental conditions (Bubier et al., 1993; 2005; Moore et al., 1990). Carbon is removed from the atmosphere in the form of CO<sub>2</sub> by surface vegetation via photosynthesis, which is then stored in peat soils as incompletely decomposed organic matter (Strack et al., 2008). Carbon dioxide is released from peatlands as a by-product of plant (autotrophic) and soil (heterotrophic) respiration, otherwise known as ecosystem respiration (ER). Respiration is, among other factors, dependent on labile C, soil temperature and moisture content (Strack et al., 2008).

Organisms in the soil break down complex molecules into low-molecular-weight substances, which are oxidized into CO<sub>2</sub> (Killham, 1994). Litter decomposition rates decrease over time because the remaining material becomes increasingly difficult for microbes to break down (Strack et al., 2008). The decomposition rate is influenced by the quantity and quality of peat and environmental conditions, including peat moisture, temperature, acidity, and the availability of alternative electron acceptors for organic matter oxidation (Killham, 1994). Carbon dioxide production rates indicate biogeochemical quality (e.g., nutrient content, humification, etc.) because they describe the rate at which microorganisms decompose organic matter. High-quality peat contains large amounts of labile C available to decompose (e.g., carbohydrates, proteins, amino acids), leading to greater rates of CO<sub>2</sub> production (Schlesinger & Andrews, 2000; Wardle et al., 2004). Decomposition rates are greatest in the youngest peat and have been found to decrease with peat age (Hogg, 1992).

Controlled by peat water saturation and microbial activity, CH<sub>4</sub> emissions from peatlands are spatially and temporally variable (Moore et al., 1990; 1994; Roulet et al., 1997). Methane is produced in the anoxic layers of peatlands via methanogenesis (Lafleur, 2009; Yavitt and Seidmann-Zager, 2006) and can be consumed in the oxic peat layers through a process known as methanotrophy (Turetsky et al., 2014). The greater the oxic layer thickness, the greater the opportunity for CH<sub>4</sub> oxidation, typically occurring within 25 cm of the oxic-anoxic boundary (Segers, 1998). Methane produced in the peat is released into the atmosphere through diffusion, ebullition, or plant-mediated transport via root tissue (Holden, 2005; Rosenberry et al., 2003; Whalen, 2005).

## 1.2 Peatland Disturbance

In Canada, 34,000 ha of bog have been harvested which represents 0.03% of that country's bog-covered surface. Agriculture is the single largest disturbance, followed by forestry, mining, roads, and peat extraction; the latter represents <3% of Canadian peatland disturbances (Harris et al., 2022). Globally, there ~ 460 Mha of peatlands; 50 Mha have been disturbed (Leifled & Menichetti, 2018), mostly by agriculture. Peat extraction intrinsically alters the C exchange dynamics of a peatland. In preparation for extraction, a peatland is drained by cutting ditches to lower the water table (WT), and all vegetation is removed. When the surface peat is sufficiently dry, in Canada, vacuum harvesters begin to extract a thin layer of surface peat. The process of harvesting increases the density and decreases the porosity in the top 20 cm on the peat profile (Lai, 2021). Following the end of extraction activities, peatlands disturbed by vacuum harvesting in this manner are often unable to naturally revegetate and regain their original ecosystem functions due to shifts in peat hydrophysical properties (McCarter and Price, 2015) and because the viable seed bank is primarily removed during extraction (Waddington et al., 2009).

If left unrestored, drained peatlands act as large sources of C to the atmosphere (Hirashi et al., 2014; Joosten et al., 2002; McNeil & Waddington, 2003; Rankin et al., 2018; Smith et al., 2014; Waddington et al., 2002). Drainage lowers the WT, creating a thicker oxic layer of peat (Poulin et al., 2005). This results in greater respiration rates and increases the volume within which CH<sub>4</sub> oxidation can occur (Abdalla et al., 2016; Holden, 2005; Sundh et al., 2000; Turetsky et al., 2014). Therefore, while CO<sub>2</sub> emissions to the atmosphere rise, CH<sub>4</sub> emissions are decreased by an average of 84% (Abdalla et al., 2016). Methane emissions become localized in the former drainage ditches that can become new anoxic zones due to the saturated conditions,

warm temperatures, and large amounts of labile C normally found in the ditches (Rankin et al., 2018; Schrier-Uijil et al., 2010; Sundh et al., 2000; Waddington & Day, 2007; Waddington et al., 2009).

Carbon emissions and controls on C exchange from undisturbed peatlands have been well-documented and researched (e.g. Bubier et al., 1993; 2005; Kohler et al., 2011 Moore et al., 1990; Nilsson et al., 2008; Pelletier et al., 2007; 2011; Roulet et al., 2007; Strachan et al., 2016; Updegraff et al., 1995). Post-extracted, unrestored peatlands are persistent sources of C to the atmosphere (Rankin et al., 2018); however, restoration can successfully revert disturbed peatlands from net C sources to net C sinks as a result of increased vegetative uptake of CO<sub>2</sub> (Nugent et al., 2018; Strack & Zuback, 2013). Until recently, research has focused on understanding the impacts of disturbance on the gas exchange after the disturbance has ended or has been conducted on sites where extraction has been halted (Ahlholm and Silvola, 1990; Aslan-Sungur et al., 2016; Bergman et al., 1998; Nykanen et al., 1995; Oleszczuk et al., 2008; Sundh et al., 2000; Waddington and Price, 2000; Waddington et al., 2002; Wilson et al., 2015). To our knowledge, this is one of the first studies conducted in a drained peatland undergoing active vacuum extraction. Thus, little is currently known about how C emissions from vacuum-harvested peatlands are altered during the active extraction process. This study aims to quantify the CO<sub>2</sub> and CH<sub>4</sub> emissions from a peatland undergoing active extraction and to link this with peat quality and environmental factors to better understand how C exchange changes over time.

## 90 **2 Materials and Methods**

### **2.1 Site Description**

*In situ* fluxes of CO<sub>2</sub> and CH<sub>4</sub> were measured at an active horticultural peat production site approximately five km southeast of Rivière-du-Loup, QC (47°47'26"N, 69°31'02"W). The site was initially a treed ombrotrophic bog system, with depths of peat in excess of 4.5 m (Anrep, 1914). Physical and chemical properties of the peatland are provided in Table S1. The location was prepared for peat extraction in 1985, using standard industry methods resulting in partially drained peat devoid of vegetation. The bare peat is sectioned into individual "fields" 500 m in length and 30 m in width via drainage ditches. These individual fields are combined into "sectors" classified by the year peat extraction began (Figure 1). In eastern Canadian peat production sites, each field is domed; the elevation of the middle of the field is highest and slopes down toward the drainage ditches to assist in precipitation drainage. A gravel service road approximately 1 km in length runs down the middle of the

100 site, separating the peatland into two halves. Large piles of loose peat and wood debris removed from the surface of the fields are stored on either side of this main road, between the gravel and the beginning of the individual fields. The storage piles are continuously moved and resized to transport the peat to a handling facility or to form new peat piles to prevent overheating and combustion. The site has been in operation for 36 years and was undergoing active extraction at the time of this study. Large machinery such as tractors and vacuum harvesters frequently drove over the surface of the fields during the measurement  
105 period. Measurements were taken from fifteen fields, each with an area of 1.5 ha. Peat harvesting normally occurs from June through September when the harrowed peat is dry enough; therefore, it is dependent on the spring temperature and summer rainfall patterns. Measurements were taken over three years in August 2018, June through August 2019, and July through September 2020.

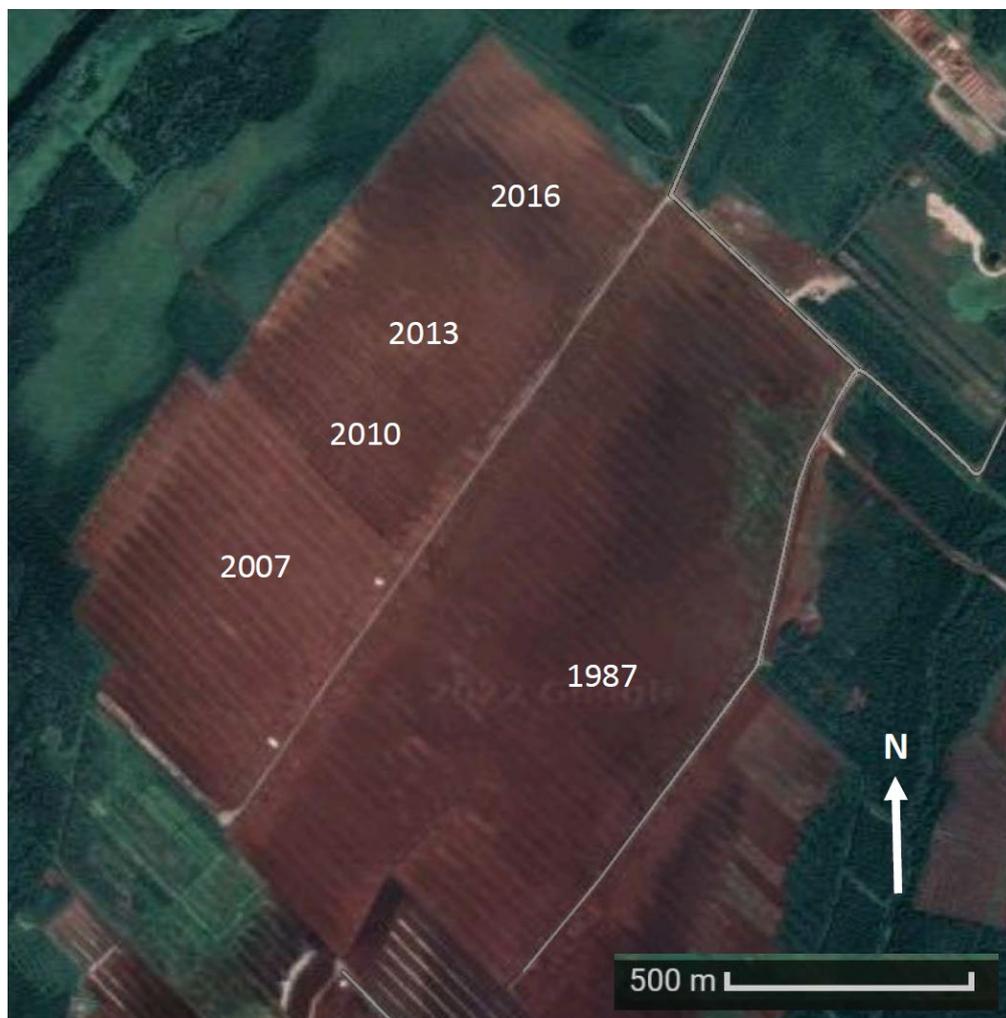
The climate of the study area is cool-temperate with a mean annual temperature of 3.5 °C and mean precipitation of  
110 963.6 mm (1981 – 2010 averages for St. Arsene, QC (47° 57' 00" N, 69° 23' 00" W) the closest weather station with 30 years of records (Environment and Climate Change Canada, 2021)). The mean temperatures for June, July, and August, are 14.9, 17.6, and 16.7 °C, respectively, and the corresponding mean monthly precipitation totals are 92.6, 95.0, and 94.2 mm, respectively.

The volumetric soil moisture (VSM), water table depth (WTD) and peat temperatures at the 2007 site have been  
115 summarized by Lai (2022). The VSM in the top 3 cm harrowed layer was ~ 10%, but was > 70% below 0.25 m. The WTD was highest in June (0.15 m) and dropped through the summer to 0.7 m in late August/September. The WTD then rose towards the surface in the autumn in response to rain, and dropped again over the winter. Peat temperatures followed a typical annual pattern; during most of the summer they were around 20 °C at 0.05 m and dropped to 0 °C in mid-winter.

## 2.2 Chamber Measurements

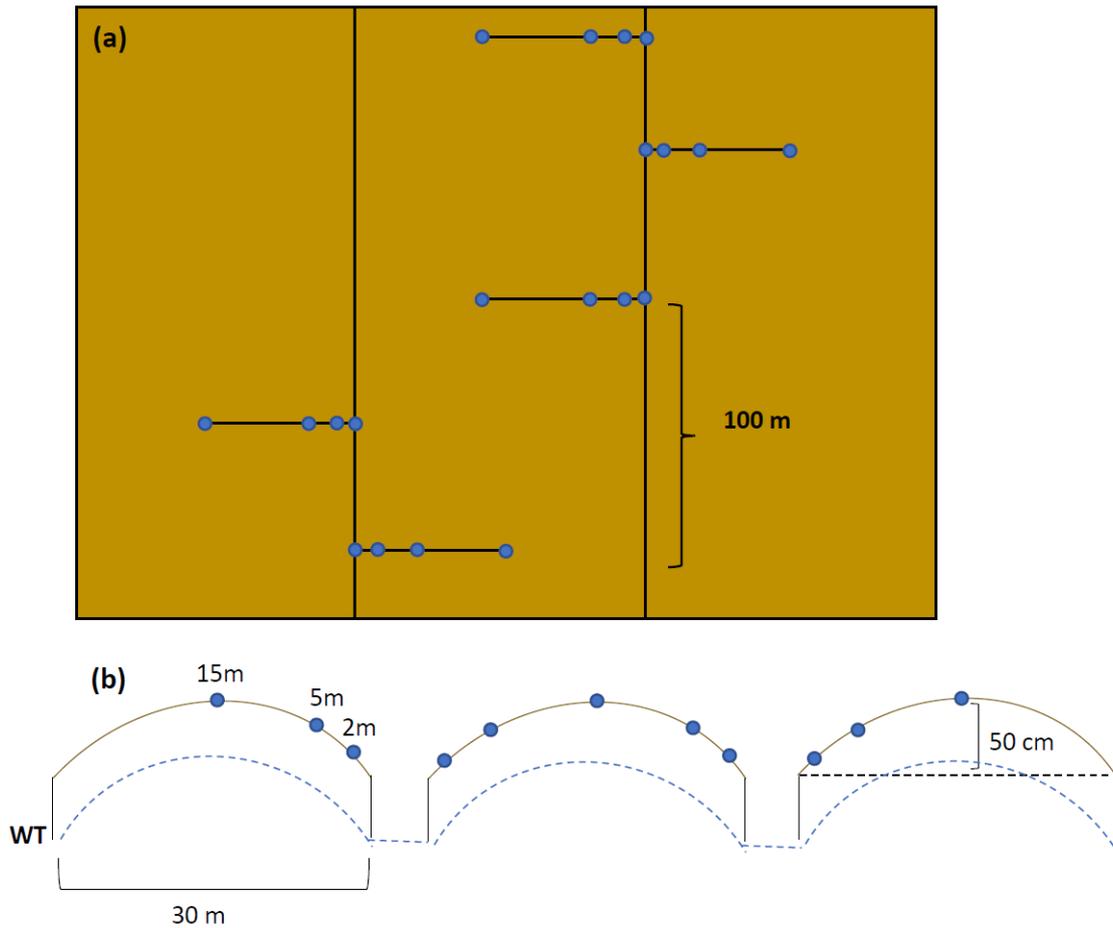
120 The closed chamber method (discussed in detail in Rankin et al. 2018) was used to measure fluxes of CO<sub>2</sub> and CH<sub>4</sub> from the peat surface. Fluxes of CO<sub>2</sub> and CH<sub>4</sub> were measured from five different sectors at this site, representing extraction beginning in 1987, 2007, 2010, 2013, and 2016 (Figure 1). Within these sectors, random measurements were taken from five transects 50 m apart perpendicular to the lateral drainage ditches, alternating across three consecutive fields (Figure 2a). Each transect contained four measurement locations: 0 (representing in the ditch itself), and 2, 5, and 15 m (field centre) away from

125 the drainage ditch, thus capturing spatial variability in the fluxes across the field (Figure 2b). To optimize coordination of  
sampling with the field operations, the 1987 (oldest) sector age was under-sampled relative to the other four sectors in 2020  
because our previous measurements indicated that this sector had CO<sub>2</sub> and CH<sub>4</sub> flux values similar to other fields, except the  
most recently open field (2016).



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135 **Figure 1: Location of measured sectors within the study site. Image modified from Google Maps (Imagery ©2022 CNES/Aribus, Imagery©2022 CNES/Airbus, Landsat, Copernicus, Maxar Technologies, Map data© 2022) (<https://www.google.ca/maps/@47.7910172,-69.5156644,3144m/data=!3m1!1e3!5m1!1e4>), accessed July 6, 2022). The field labels for the year the sectors were opened for peat extraction. As the first C measurements began in 2018, the 2016, 2013, 2010, 2007 and 1987 sectors represent 2, 5, 8, 11, and 31 years respectively.**



140 **Figure 2: (a) Sampling transects and (b) measurement locations within transects with an estimated elevation increase at the field**  
**145 centre. The field contouring results in about 50 cm difference in surface peat elevation between the centre of the field and the edge**  
**of the field.**

Collars could not be left in place between measurements because we were measuring from active peat extraction fields that had harvest machinery driving on them. Therefore, at each measurement location on the peat field, a metal collar was inserted approximately 5 cm into the surface of the field. An opaque aluminum chamber (64 x 64 cm) was then placed on top of the collar. Air was cycled between the chamber and a trace gas analyzer. Measurements each summer were made from June through September at varying intervals depending on weather and industry operations. In 2018, a PP Systems EGM-4 IRGA was used. In the first two weeks of measurements in 2019, a Los Gatos Research Ultraportable Greenhouse Gas Analyzer was

used and in the remainder of 2019 and in 2020, a LI-COR Biosciences LI-7810 Trace Gas Analyzer was used. A one-way ANOVA ( $\alpha = 0.05$ ) was conducted between the fluxes from the different analyzers for the 2016 sector 2 m position and 2007 sector 15 m position. There were no significant differences between the means of the fluxes measured with the three analyzers from the 2016 ( $p = 0.552$ ,  $F_{2,85} = 0.599$ ) or 2007 ( $p = 0.06$ ,  $F_{2,87} = 2.848$ ) sectors. A measurement lasted four minutes, after which the chamber was lifted for a minimum of 30 seconds to allow the  $\text{CO}_2$  and  $\text{CH}_4$  to return to ambient concentrations. The measurements taken in the drainage ditches required a different chamber because the ditches were too narrow to accommodate the field chamber. The ditch chamber was cylindrical (35 cm in height, 27 cm diameter) and was composed of translucent plastic covered in opaque reflective tape. The same measurement procedure was followed for the ditch measurements. A battery-powered fan was installed on the interior of each of the field and ditch chambers to ensure adequate air mixing during measurements. The chamber and collar were removed from the field after each measurement was completed and moved between measurement locations.

The interior height of the field chamber above the peat surface, including the collar, was measured at all four corners at each sampling location. The height of the ditch chamber, including the collar, was measured at three different points around the perimeter. Peat volumetric water content (%VWC) was measured at three separate locations at each measurement location using a CSI Hydrosense II soil moisture sensor inserted from 0 to -10 cm. For each measurement, peat temperature was taken at depths of 2, 5, 10, 15, and 20 cm below the surface to attain a temperature profile at each measurement location.

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### 2.2.1 Data Analysis and Chamber Flux Calculation

Trace gas flux ( $F$ ) in  $\text{mg m}^{-2} \text{d}^{-1}$  was determined as the change in concentration over time using the equation

$$F = \frac{f_x \left( \frac{V_c}{R(273+T_a)} \right) n \cdot t}{S} \quad (1)$$

where  $f_x$  is the rate ( $\text{ppmv min}^{-1}$ ),  $V_c$  is the chamber volume ( $\text{m}^3$ ),  $R$  is the ideal gas constant ( $0.0821 \text{ L atm K}^{-1} \text{ mol}^{-1}$ ),  $T_a$  is the air temperature ( $^{\circ}\text{C}$ ) inside the chamber,  $n$  is the molecular mass of each gas ( $\text{CO}_2 = 0.044 \text{ kg mol}^{-1}$ ;  $\text{CH}_4 = 0.016 \text{ kg mol}^{-1}$ ),  $S$  is the surface area of the collar ( $\text{m}^2$ ), and  $t$  is the number of minutes in a day (1440 minutes). Change in concentration over time for both  $\text{CO}_2$  and  $\text{CH}_4$  were plotted for each measurement location, and the flux was kept if a linear increase or decrease

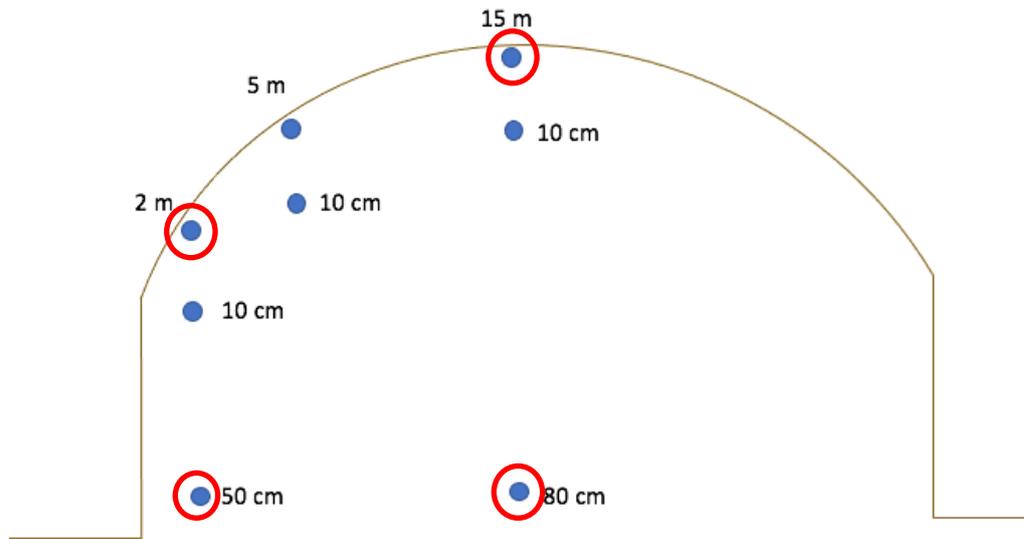
was observed to ensure that low values were not disproportionately discarded. In 2018, 49% of CO<sub>2</sub> and 55% of CH<sub>4</sub> measurements were rejected. In 2019, 21.8% of CO<sub>2</sub> and 26% of CH<sub>4</sub> measurements were rejected, and in 2020, 11.6% of CO<sub>2</sub> and 37.6% of CH<sub>4</sub> measurements were rejected.

All statistical analyses were performed in the R software package (R Core Team, 2021), and figures were produced using the R package ggplot2 (Wickham, 2016). A one-way ANOVA of CO<sub>2</sub> and CH<sub>4</sub> fluxes between the field surface and drainage ditches was performed, and a two-way ANOVA between sector age and measurement position was performed, excluding drainage ditch measurements, with  $\alpha = 0.05$ . An interaction test was conducted to determine the relationship between sector age and measurement position and a Tukey post-hoc test was conducted to show the specific interactions. Linear regressions were performed between surface VWC, temperature measurements, and CO<sub>2</sub>/CH<sub>4</sub> flux.

## **2.3 Peat Incubation**

### **2.3.1 Field sampling**

In order to investigate differences in peat substrate quality among sectors and field positions, incubations were performed with peat samples taken from the 1987, 2007, and 2016 sectors, spanning continuous extraction of ~3 to 30 years. Samples were taken at the second chamber measurement transect from each of the three-sector ages (Figure 2a). Within each transect, approximately 1 kg of peat was obtained at 2, 5, and 15 m away from the drainage ditches both from the surface and from a depth of 10 cm. Additional samples were taken from a depth of 50 cm, at a distance of 2 m from the ditch and from a depth of 80 cm, 15 m away from the ditch (Figure 3). The 50 and 80 cm positions were estimated to be parallel at depth, based on an elevation difference of approximately 50 cm resulting from the field doming. Samples were kept in sealed plastic bags during transport from the field and frozen upon arrival at the lab. Four samples (Figure 3) were taken from both the 2007 and 2016 sectors for <sup>14</sup>C dating performed by a 3MV accelerator mass spectrometer (AMS) at the AEL AMS Laboratory at the University of Ottawa. After physical and chemical pre-treatments, the samples were combusted, producing CO<sub>2</sub> for graphitization (<https://ams.uottawa.ca/analytical-methods-radiocarbon-laboratory/>). Carbon dating was calibrated using the OxCal .4.4 (Bronk Ramsey, 2009) and IntCal 2020 (Reimer et al., 2020) curves.



200 **Figure 3: Sampling locations (2 m, 5 m and 15 m from ditches) and depths (cm) from the 1987, 2007, and 2016 sectors. Those with red circles were also sampled for  $^{14}\text{C}$  dating at the 2007 and 2016 sectors. Horizontal lines indicate approximate ditch surface with peat extending below these points.**

### 2.3.2 Incubation Experiment Methodology and Analysis

205 Incubations were performed in 250 mL mason jars fit with air-tight lids and a short plastic tube fixed with a stopcock valve, sealed with epoxy. Nine replicates and an additional blank were used for each sampling position. 30 g of peat was weighed and placed into each jar after woody debris were removed. To keep moisture conditions between the samples similar, 30 mL of distilled water was added to the jars and mixed with the peat to create a slurry. This is not meant to represent field conditions but to obtain rates reflecting potential, standardized decomposability/respiration rates. The estimated VWC of the slurries was 80 – 90%. The height and diameter of peat in the jar was recorded to calculate the headspace volume for each sample. Jars were kept at a constant temperature of 23 °C.

210 5 mL of the headspace from the jars were sampled at 0, 6, 12, 24, 48, and 72 hours. After the initial 72-hour samples were taken, the lids were removed, the jars were left open for 12 hours, resealed, and sampling was repeated for an additional 72 hours. This was done to account for increased respiration rates that may have occurred during the first sampling period

from cellular rupture after the samples were thawed. 5 mL of ambient air was backfilled into each jar after each sample was taken.

215 The concentrations of the gas samples were analyzed using two gas chromatographs (GC) (Shimadzu 2014 GHG GC & SRI 8610 C GHG GC). The carrier gas was N<sub>2</sub>, the SRI column temperature was 70 °C and the flame ionization detector (FID) was at 110 °C. Three standards of 5000 ppm CO<sub>2</sub> and 5 ppm CH<sub>4</sub> were run through the GC before injecting the 5 mL gas samples at each sampling interval. Gas samples were analyzed within six hours of withdrawal from the jars. Samples were consistently run on the same GC throughout the course of the experiment. Gas concentrations from fifteen samples of ambient  
220 air were run on both GCs and were compared every 72 hours throughout the course of the experiment to cross calibrate between the readings of the two machines. The Shimadzu 2014 GHG GC had an average ( $\pm$  SD) ambient CO<sub>2</sub> reading of 609.2 ppm ( $\pm$  152.0) and the SRI 8610 C GHG GC had an average ambient CO<sub>2</sub> reading of 589.5 ( $\pm$  132.6) ppm. CO<sub>2</sub> and CH<sub>4</sub> concentrations were corrected for dilution from backfilling of ambient air and for variation in ambient concentrations of CO<sub>2</sub> and CH<sub>4</sub> using the blank measurements. CO<sub>2</sub> and CH<sub>4</sub> production were calculated as a change in concentration over time. 10% of data was  
225 discarded after quality control, where values with  $r^2 < 0.8$  were rejected. A three-way ANOVA was used to determine the variance of means between sector age, position, and depth.

## 2.4 Peat Quality Analysis

Samples were prepared for Fourier transform mid-infrared (FT-MIR) spectrometry corresponding to the sampling  
230 locations used in the peat incubations. All samples were oven-dried at 60 °C for 48 hours and ground into a fine powder using a mortar and pestle, after which they were run through a 50  $\mu$ m mesh sieve. Once prepared, the samples were analyzed using FTIR spectroscopy (Agilent Cary 660 FTIR spectrometer; 32 scans per spectrum, 2 cm<sup>-1</sup> resolution). To this end, a mixture of approximately 2 mg sample and 200 mg potassium bromide (KBr) (FTIR grade, Sigma Aldrich, St. Louis, MO, USA) were pressed. A KBr background spectrum was subtracted from the raw absorbance spectra. Finally, the spectra were baseline  
235 corrected (Beleites and Sergio, 2020: <https://github.com/cbeleites/hyperSpec>) using the R package *ir* (Teickner, 2020: <https://github.com/henningte/ir>) and further processed with the R package ‘*irpeat*’ (Teickner & Hodgkins, 2021). A Humification index (HI) was computed as ratio of the absorbances at  $\sim$ 1650 cm<sup>-1</sup> (indicative of lignins and other aromatics)

and  $\sim 1090 \text{ cm}^{-1}$  (indicative of polysaccharides representing the labile fraction), as described in detail in Broder et al. (2012). Larger ratios ( $1650/1090 \text{ cm}^{-1}$ ) indicate a greater degree of humification, assuming a residual enrichment of refractory moieties and preferential degradation of more labile fractions (Broder et al. (2012)).

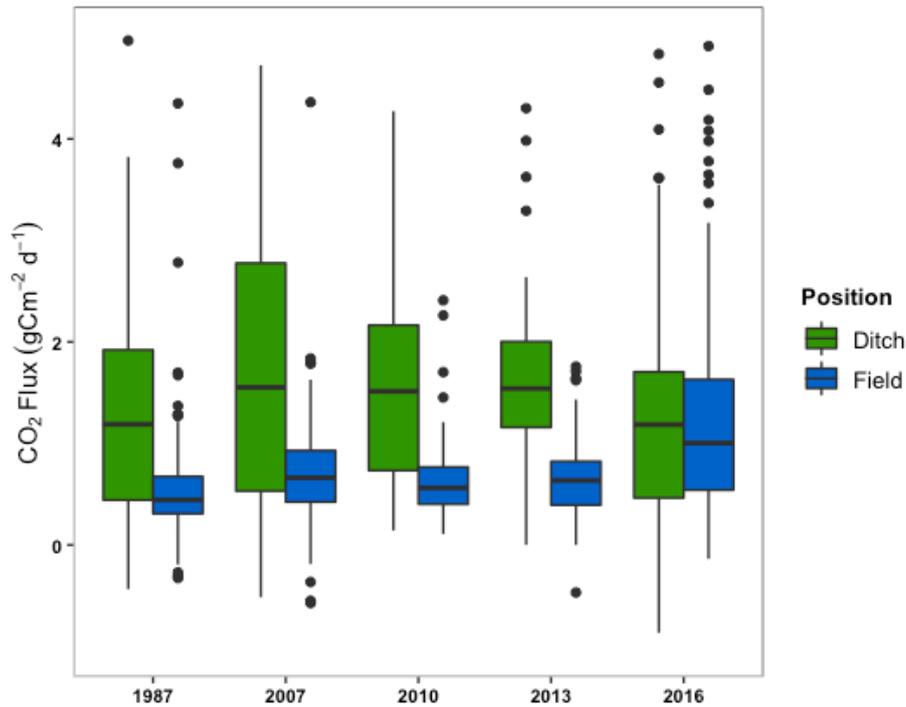
### 3. Results

#### 3.1 CO<sub>2</sub> Fluxes

The average ( $\pm$  SD) CO<sub>2</sub> flux from all sectors, field locations and ditches combined was  $1.2 (\pm 2.1) \text{ g C m}^{-2} \text{ d}^{-1}$  (see also Table S2). The mean CO<sub>2</sub> flux from all fields combining all sector ages and excluding the drainage ditch measurements was  $0.9 (\pm 1.6) \text{ g C m}^{-2} \text{ d}^{-1}$ . The mean CO<sub>2</sub> flux from the drainage ditches across all sectors was  $2.05 (\pm 2.2) \text{ g C m}^{-2} \text{ d}^{-1}$ . A significant difference was present ( $F_{1,1272} = 79.47, p < 0.001$ ) between the CO<sub>2</sub> emissions from the drainage ditches and the field surface.

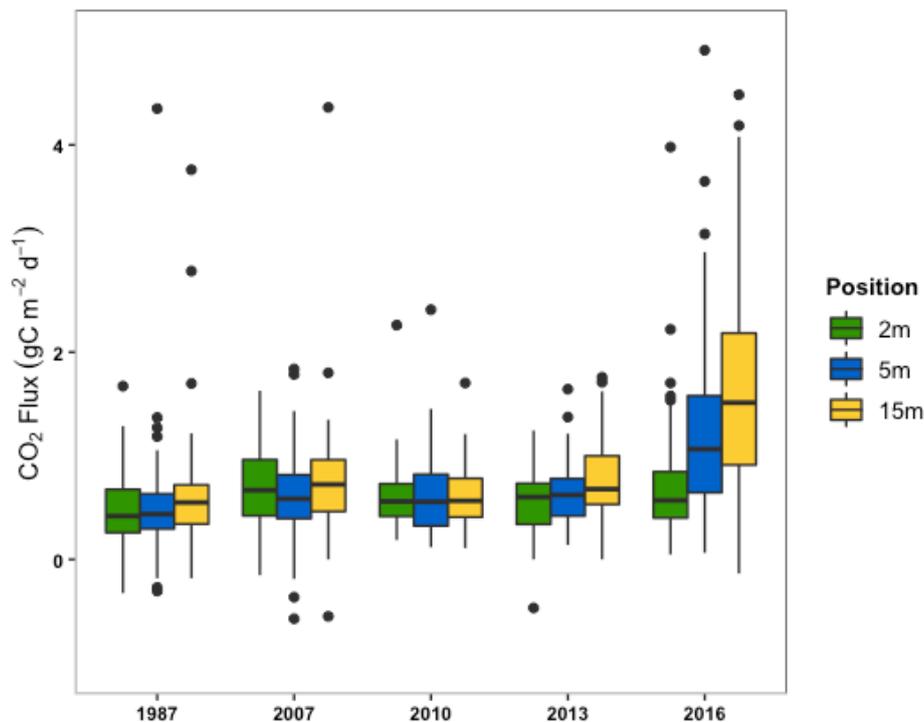
The base of the drainage ditches was closer to the WT than the surface of the fields and, as a result, was frequently saturated. The C cycling dynamics within the ditches are different than those at the surface of the field and thus, the results from the drainage ditches will not be directly compared to those from the field surface.

Measurements of CO<sub>2</sub> flux from the sectors ranged from  $37.1 \text{ g C m}^{-2} \text{ d}^{-1}$  to  $-0.3 \text{ g C m}^{-2} \text{ d}^{-1}$ . A single value of  $-36.5 \text{ g C m}^{-2} \text{ d}^{-1}$  was deemed to be an outlier and removed from the 1987 sector flux data. A two-way ANOVA between sector age and measurement position was performed and the outcomes for sector age and measurement position, as well as any interactions, will be discussed separately below. The two-way ANOVA showed that the 2016 sector had significantly greater CO<sub>2</sub> emissions than all other sectors ( $F_{4,942} = 12.80, p < 0.05$ ) (Figure 4; Table S1). The 1987, 2007, 2010, and 2013 sectors exhibited similar fluxes over time, with no significant difference between their means, although the 2010 and 2013 sectors were only measured in 2020.



260 **Figure 4: The box and whisker plots (median, upper and lower quartiles, and outliers) of CO<sub>2</sub> fluxes from the drainage ditch and the three field locations combined (2, 5 and 15 m) by the year a sector was opened.**

When measurements are averaged by field position (2, 5, and 15 m away from the drainage ditches) across all five sectors, the mean CO<sub>2</sub> fluxes ( $\pm$  SD) were 0.7 ( $\pm$  0.7), 0.9 ( $\pm$  1.0), and 1.2 ( $\pm$  2.4) g C m<sup>-2</sup> d<sup>-1</sup>, respectively (Figure 5). A  
 265 statistically significantly different mean CO<sub>2</sub> flux from the 15 m position compared to both the 2 m and the 5 m positions ( $F_{2,942}$  = 6.90,  $p < 0.05$ ) was found.



270 **Figure 5: The box and whisker plots of CO<sub>2</sub> flux by sector and measurement location from the edge of the ditches in a field (i.e. 2, 5 and 15 m).**

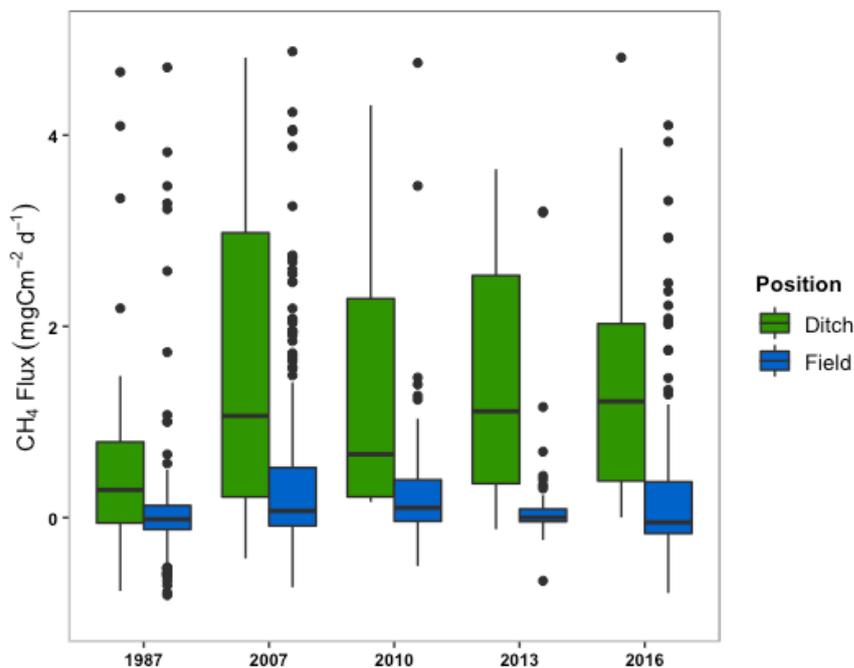
CO<sub>2</sub> emissions at different distances from the ditches differed for different age sectors ( $F_{8,942} = 3.41, p < 0.001$ ). The mean CO<sub>2</sub> emissions from the 15 m position in the 2016 sector significantly differed from every other sampling position and sector. Within the 2016 sector, the means of the CO<sub>2</sub> emissions from the 15 m position were statistically different from those of the 2 m position ( $F_{8,942} = 2.22, p < 0.001$ ). No statistical difference emerged between the means of 2016 15 m and 2016 5 m positions. Within the 2016 sector, a difference was only found between the middle and edge of the fields. No differences were noted within or between the other four sectors. Across the whole data set, correlations between VWC ( $r = -0.2, p < 0.001$ ) or soil temperature ( $r = 0.19; p = 0.037$ ) and CO<sub>2</sub> flux were significant but only described a small amount of the variation.

275

### 3.2 CH<sub>4</sub> Fluxes

280 Variation in CH<sub>4</sub> emissions was much greater than that of CO<sub>2</sub> between the field and drainage ditches. The mean CH<sub>4</sub> flux ( $\pm$  SD) from the drainage ditches in all sectors was 84.2 ( $\pm$  325.4) mg C m<sup>-2</sup> d<sup>-1</sup> (see also Table S2). The mean CH<sub>4</sub> flux

( $\pm$  SD) from the total field surface was  $9.2 (\pm 103.0) \text{ mg C m}^{-2} \text{ d}^{-1}$ . Drainage ditches were much larger sources of  $\text{CH}_4$  to the atmosphere than the exposed peat at the field surface (Figure 6). A high standard error was present in both the field and the drainage ditch measurements, although the drainage ditches showed more variation. The mean  $\text{CH}_4$  emissions from the  
285 drainage ditches were statistically greater than that of the fields ( $F_{1,905} = 15.6, p < 0.001$ ).

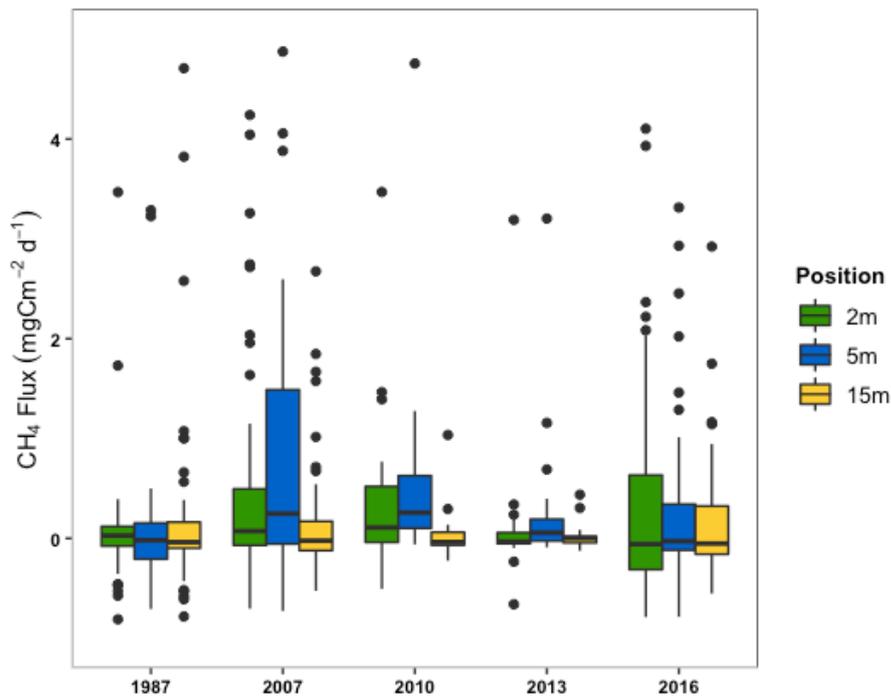


**Figure 6: The box and whisker plots of the  $\text{CH}_4$  fluxes from drainage ditches and all locations from field surface according to the age of the sector.**

290 There were no significant differences in mean  $\text{CH}_4$  among sectors from fields or from ditches (Table S1). The maximum  $\text{CH}_4$  flux from the fields and ditches were  $2518.5$  and  $2737.8 \text{ mg C m}^{-2} \text{ d}^{-1}$ , respectively, and the minimum fluxes were  $-74.7$  and  $-5.8 \text{ mg C m}^{-2} \text{ d}^{-1}$ , respectively. A single value of  $10822 \text{ mg C m}^{-2} \text{ d}^{-1}$  was deemed an outlier and removed from the 2016 drainage ditch flux data.

Combining the sectors and stratifying data by measurement position, the average ( $\pm$  SD)  $\text{CH}_4$  fluxes from the 2, 5,  
295 and 15 m positions on the fields were  $13.4 (\pm 167.8)$ ,  $8.5 (\pm 45.9)$ , and  $5.3 (\pm 33.4) \text{ mg C m}^{-2} \text{ d}^{-1}$ , respectively. Lower  $\text{CH}_4$  emissions were seen mid-field, but there were no statistically significant differences between the means of the three field

measurement positions (Figure 7). Across the whole data set, relationships between VWC ( $r = -0.077$ ,  $p < 0.001$ ) or temperature ( $r = 0.084$ ,  $p = 0.033$ ) and  $\text{CH}_4$  flux were significant but described only a small part of the variation.



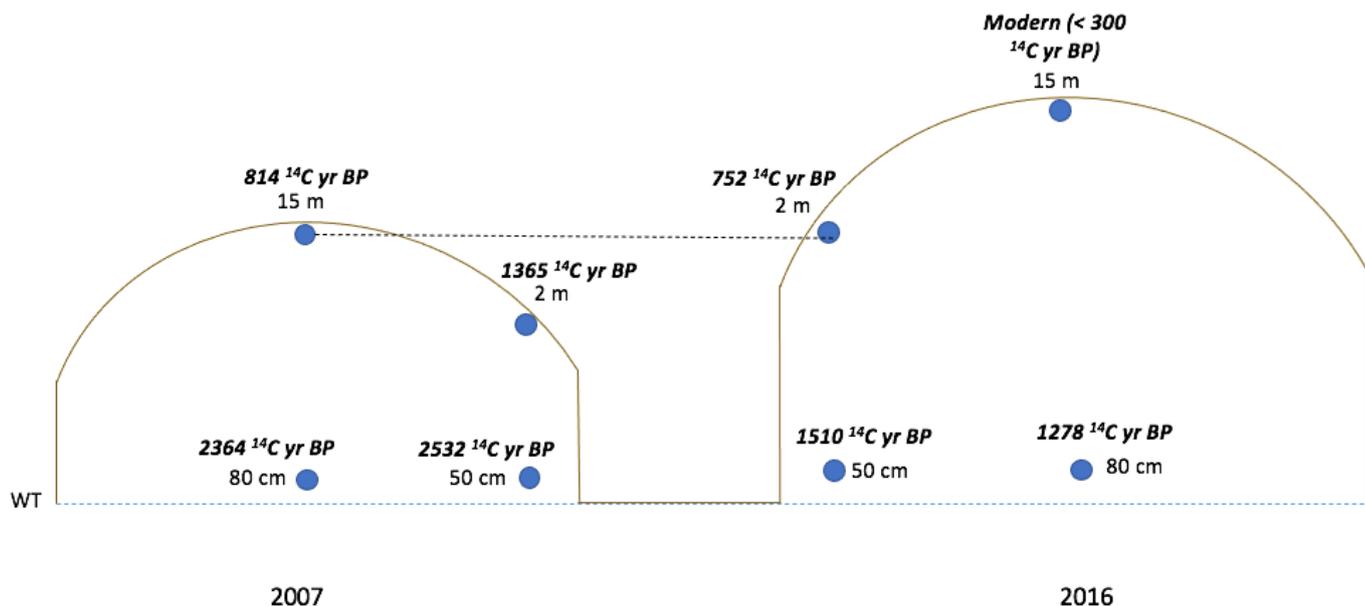
300

**Figure 7: Box and whisker plots of the  $\text{CH}_4$  flux by measurement distance from the ditch within each age sector.**

### 3.3 Peat Age and Quality

The  $^{14}\text{C}$  dating results of peat samples from the 2007 and 2016 sectors revealed distinct differences in peat age across and within sectors (Figure 8). The elevation difference across the field introduced an age difference between the peat closest to the drainage ditches and the peat at the center. The results indicated that peat age ( $\Delta^{14}\text{C}$ ) decreased toward the centre of the field, with elevation, in both the 2007 ( $-163.46 \pm 3.27\%$  and  $-104.10 \pm 3.54\%$  for 2 and 15 m, respectively) and 2016 ( $-94.06 \pm 3.56\%$  and  $30.03 \pm 4.00\%$  for 2 and 15 m, respectively) sectors. Mid-field, at a depth of 80 cm from the surface, the age difference was also apparent between sectors ( $-276.62 \pm 2.88\%$  and  $-154.39 \pm 3.29\%$  from the 2007 and 2016 sectors, respectively) (Figure 8).

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**Figure 8: Incubation sampling locations with respective <sup>14</sup>C ages, all depths approximate. The horizontal line is drawn to show that these elevations are approximately equal.**

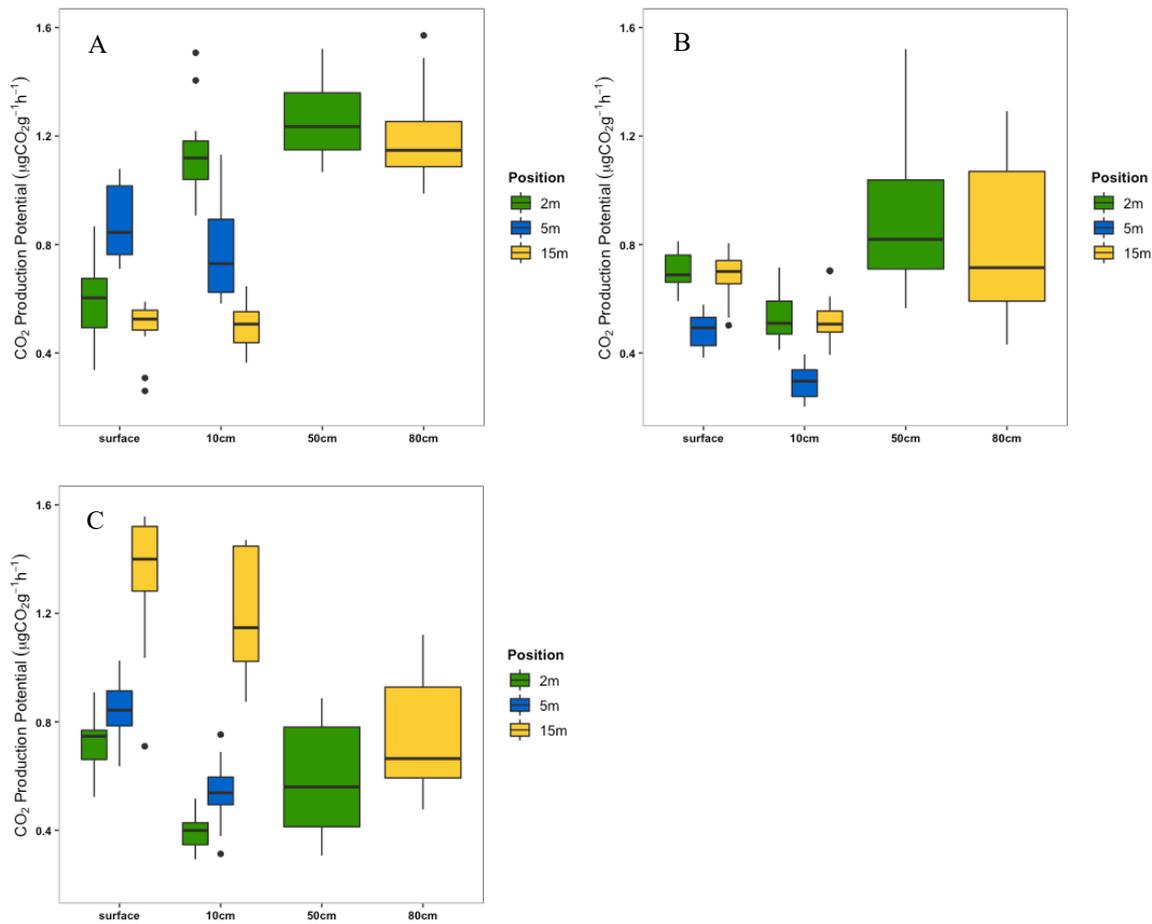
315 FT-MIR analysis results indicated that the humification degree increased with years of extraction. Assessing samples from the surface and 10 cm depths, the sector average HI ( $\pm$  SD) were 1.05 ( $\pm$  0.08), 0.82 ( $\pm$  0.08), and 0.70 ( $\pm$  0.09) from the 1987, 2007, and 2016 sectors, respectively. The 50 and 80 cm samples were excluded from this analysis because the deep samples were older and more humified than the surface peat as a result of their depth in the profile, however, they were included in the <sup>14</sup>C dating. A one-way ANOVA demonstrated that there was a significant difference between the average HI from the 320 1987 and 2007 sectors ( $F_{(2,21)} = 26.73$ ,  $p < 0.001$ ) and from the 1987 and 2016 sectors ( $F_{(2,21)} = 26.73$ ,  $p < 0.001$ ).

### 3.4 Production Potential

#### 3.4.1 CO<sub>2</sub>

Carbon dioxide production potentials ranged from 0.50 – 1.39, 0.28 – 0.88, and 0.40 – 1.36  $\mu\text{g CO}_2 \text{ g}^{-1} \text{ h}^{-1}$  from the 1987, 2007, and 2016 sectors, respectively. Carbon dioxide production from the 1987 sector samples did not appear to follow any

325 clear pattern or trend due to the position on the field or depth (Figure 9A). However, statistically, significant differences can be seen among depths of the surface and 10 cm from the 2 and 5 m positions and the 5 and 15 m positions. Carbon dioxide production within the 2007 sector also did not appear to follow a trend or pattern (Figure 9B). Notable statistically significant differences within the 2007 sector emerged at a depth of 10 cm between the 5 and 15 m positions. The 2016 sector samples, however, exhibited a clear increase in CO<sub>2</sub> production with increasing distance from the drainage ditches at the surface and 10 cm depths (Figure 9C). From these depths, the 2 and 15 m positions and the 5 and 15 m positions were statistically different (F<sub>6,366</sub> = 19.5, p < 0.001). From all three sectors, CO<sub>2</sub> production potentials were similar between the 50 and 80 cm depths, although the absolute values varied between the sectors.



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**Figure 9: CO<sub>2</sub> production potentials of all samples from the A) 1987, B) 2007, and C) 2016 sectors**

### 3.4.2 CH<sub>4</sub>

340 As expected, given the oxic conditions of the incubations, no incubations showed a consistent increase in CH<sub>4</sub> concentration for the experiment, and all CH<sub>4</sub> r<sup>2</sup> values were < 0.8.

## 4.0 Discussion

The net ecosystem exchange (NEE) of undisturbed peatlands ranges between -10 to -60 g C m<sup>-2</sup> yr<sup>-1</sup> (Koehler et al., 2010; Roulet et al., 2007; Sagerfors et al., 2008), whereas our study site is a net source of C to the atmosphere, similar to values  
345 reported from disturbed and post-extraction, unrestored peatlands. Aslan-Sungur et al. (2016) reported CO<sub>2</sub> fluxes of 246, 244, and 663 g C m<sup>-2</sup> yr<sup>-1</sup> in 2011, 2012, and 2013 respectively, from a peatland site, drained for mining and agricultural use. Salm et al. (2012) report net CO<sub>2</sub> emissions of 480 g C m<sup>-2</sup> yr<sup>-1</sup> for extracted peatlands in Estonia. Rankin et al. (2018) reported annual CO<sub>2</sub> emissions of 173 - 259 g C m<sup>-2</sup> yr<sup>-1</sup> from a 20-year post-extracted, unrestored peatland. For our study site, using  
350 the mean daily emission of 0.7 g C m<sup>-2</sup> for six months and ~0.5 g C m<sup>-2</sup> d<sup>-1</sup> for the six coldest months would yield an estimate of 200-250 g C m<sup>-2</sup> yr<sup>-1</sup>, which is in line with previous results (Alm et al., 2007; Aslan-Sungur et al., 2016; Nykanen et al., 1995; Wilson et al., 2015).

A greater respiration rate from the drainage ditches is consistent with findings from previous studies at post-extraction (Waddington et al., 2010) and unrestored (Rankin et al., 2018) peatland sites. The CO<sub>2</sub> emissions from the drainage ditches alone are similar in magnitude to emissions from natural peatlands (~12 ± 21 g C m<sup>-2</sup> yr<sup>-1</sup> (Abdalla et al., 2016)).

355 Our measured CH<sub>4</sub> fluxes correspond to published values from other drained peatland sites (Manning et al., 2019; Waddington et al., 1996). Korkiakoski et al. (2020) reported that a drained peatland site in Finland even became a CH<sub>4</sub> sink over the growing season, measuring atmospheric emissions following precipitation events. Although our site is not a net sink, some uptake of CH<sub>4</sub> by the field surface was measured consistently over all three years of study from all sector ages. We likely have under-sampled gas fluxes after rainfall events, which may influence temporal variation in CH<sub>4</sub> emissions.

## 360 4.1 Environmental Variables

Peat fibre content, indicated by visual analysis and industry specification, does not appear to influence respiration rates greatly. According to industry quality classifications, the 1987 sector contains the most fibric peat, while the 2007 through 2016 sectors do not vary significantly in fibre content (Dr. P-O. Jean, Premier Tech, Pers. Comm). Thus, this physical characteristic of the peat structure does not predict variation in CO<sub>2</sub> production within fields or between sectors. Temperature is widely documented to be a driver of CO<sub>2</sub> production (Blodau, 2002; Holden, 2005; Moore & Dalva, 1993; Yavitt et al., 1997); however, surface temperature exerts little influence over our measured CO<sub>2</sub> flux ( $r = 0.19$ ). It is possible that other drivers, such as substrate quality, may have a larger impact on CO<sub>2</sub> emissions. Surface VWC also does not appear to have an influence on CO<sub>2</sub> flux ( $r = -0.2$ ), possibly due to increased respiration rates in the deeper aerated peat that would offset a decline in CO<sub>2</sub> production from desiccation (Dimitrov et al., 2010; Marwanto & Agus, 2014; Waddington et al., 2002). Average VWC in the upper 10 cm is lower than values documented from other disturbed peatlands (Manning et al., 2019; Waddington et al., 2002), but values from actively extracted peatland sites are difficult to find in the literature.

## 4.2 Chamber Measurement CO<sub>2</sub> Fluxes

An expected result was the overarching observation that the most recently opened 2016 sector had greater CO<sub>2</sub> emissions than the older sectors. It can be best explained by the relative age and thus the degree of decomposition of the surface peat. Since this sector was most recently opened, aerated peat in the upper profile is younger than sites where peat extraction has occurred for many years, exposing older peat present deeper in the profile. The range of water table depths across a field from beside the ditch to the centre of the field in the summer are 0.2 to 0.5 and 0.3 to 0.8 m, respectively. The peat water content was between 40 to 50% at 0.1 m below the surface and > 70 to 80% below 0.3 m depth (Lai 2022). Further, the elevation gradient that results from the practice of contouring the field surface exposes older peat deeper in the soil profile to the surface near the ditches, leaving younger peat in the middle of the fields (Figure 8).

As peat extraction continues, older, more humified peat with less nutrients are now the surface peat (Croft et al., 2001; Glatzel et al., 2004). The decrease in CO<sub>2</sub> production could also be a result of an accumulation of inhibitory compounds such as lignins, phenolics, or humic substances, that hinder extracellular enzyme activity (Hogg et al., 1992). This is supported by the HI (1650/1090 cm<sup>-1</sup>), which shows an increase in humification with time since extraction began. In addition, the reduced

385 water contents due to drainage most likely lead to secondary decomposition of the peat in the top 0.3 to 0.5 m of peat in the field undergoing extraction (Biester et al., 2014). In an incubation study of peat samples from 2- and 7-year post-extraction peatlands, Waddington et al. (2001) concluded that CO<sub>2</sub> production did not change from peat below a depth of 35 cm. The authors did not find a significant difference in CO<sub>2</sub> production between the young and old cutover sites and argue that peat age strongly influences CO<sub>2</sub> production more than gas transport through peat layers (Waddington et al., 2001).

390 Spatial variation within fields further illustrates the effect of peat age on respiration. The site average CO<sub>2</sub> flux is similar to average values from post-extraction, unrestored sites while the 2016 sector 15 m position emits more CO<sub>2</sub> than is recorded in the literature for post-extraction, unrestored sites (Rankin et al., 2018; Strack & Zuback, 2013). The 2016 sector displays a clear linear increase in CO<sub>2</sub> flux with increasing distance from the ditch, but this effect declines and plateaus in the older sectors. Spatial variation in CO<sub>2</sub> emissions is not apparent in the older extracted sectors because the surface peat is older, and the respiration rates are correspondingly lower. The peat at the 2 m position in the 2016 sector has a similar <sup>14</sup>C age to the middle of the 2007 sector (Figure 8) and also displays a similar mean CO<sub>2</sub> flux to the 2007 15 m position (0.57 and 0.72 g C m<sup>-2</sup> d<sup>-1</sup> for 2016 and 2007, respectively).

Previous studies have compared trace gas production from natural and cutover peatlands (Croft et al., 2001; Glatzel et al., 2004; Waddington et al., 2001; Waddington et al., 2002; Waddington & Price, 2000); however, none have compared the spatial variation in respiration rates between peat age based on depth and production year at an extracted peatland. Our results indicate that in the first few years of peat extraction, the residual labile C contained in the surface peat encourages C mineralization and high levels of CO<sub>2</sub> production. This is clearly observed in the high emissions from the center of the youngest field, opened two years prior to our initial measurements. An obvious decline in respiration within the 2016 sector is demonstrated over a horizontal distance of 10 m (Figure 5), with an estimated 15 cm difference in depth and an age difference of ~ 450 years, driven by the easily available C consumed by microbes. Over approximately three to four years, based on measurements from the sector that opened in 2013, respiration rates plateau. Peat quality appears to decline with extraction length, as indicated by the HI (1650/1090 cm<sup>-1</sup>). Soil moisture and temperature, typical drivers of CO<sub>2</sub> production, have relatively less influence on respiration rate, further indicating peat age (i.e., peat quality due to secondary decomposition) and corresponding lability to be the primary control.

### 410 4.3 Peat Substrate Age and Decomposability

The CO<sub>2</sub> production potential from the incubation experiments mirrors those observed in our field chamber fluxes, suggesting that peat samples from the field site behave similarly under field conditions when experimentally controlling for moisture and temperature. In high-latitude peatlands, deeper peat has been shown to be more resistant to decomposition than more recently formed peat (Hogg et al., 1992). Supporting our field measurement results, the CO<sub>2</sub> production potential of  
415 incubated peat from the 2016 sector increases with distance from the ditch and decreasing peat age in the top layers of peat (surface and 10 cm depths; Figure 9C). Increased CO<sub>2</sub> production potential with younger peat supports the conclusion that the 2016 sector contains C in the substrate that is more readily available for decomposition. This is not observed from the older sectors that have undergone extraction for longer durations of time, also in accordance with our field measurements.

Decreased CO<sub>2</sub> production potential from older, lower quality peat is consistent with what has previously been  
420 presented in the literature for temperate peatlands. Research has shown that CO<sub>2</sub> production potential declines with depth (Bridgham & Richardson, 1992; Waddington et al., 2001), helping to explain the consistent behaviour of the deep peat samples from the 2007 and 2016 sectors. This was an expected outcome as these samples were parallel to each other at depth and had similar (within ~100 years) <sup>14</sup>C ages (Figure 9). Research has shown that intra- and inter-community CO<sub>2</sub> production potential from well-humified peat does not vary significantly (Bridgham & Richardson, 1992). McKenzie et al. (1998) reported that  
425 CO<sub>2</sub> and CH<sub>4</sub> production potential declined with depth from different locations at two flooded peatland sites, which they attribute to differences in peat quality as a result of age.

Moreover, previous studies found decreased CO<sub>2</sub> production potential from peat in extracted peatlands compared to natural and restored sites (Croft et al., 2001; Glatzel et al., 2004). Glatzel et al. (2004) observed lower rates of respiration from surface peat at a production site, compared to natural and restored sites, finding the degree of humification to be an important  
430 control on CO<sub>2</sub> production. Waddington et al. (2001) also found that CO<sub>2</sub> production was lower in block-cut sites compared to a natural peatland and that the most active CO<sub>2</sub> production was in the surface layers. Croft et al. (2001) found lower microbial biomass in a vacuum-harvested production site, leading to lower CO<sub>2</sub> production, and found that microbial populations increased following restoration.

#### 4.4 Chamber CH<sub>4</sub> Fluxes

435 A measured difference in CH<sub>4</sub> fluxes between the field surface and drainage ditches was an expected outcome, as this has been demonstrated in many previous studies (Manning et al., 2019; Minkinen et al., 1997; Minkinen & Laine, 2006; Rankin et al., 2018; Schrier-Uijl et al., 2010; Strack & Zuback, 2013; Sundh et al., 2000; Waddington & Day, 2007). The drainage ditches act as localized anoxic zones that exhibit ideal moisture and temperature conditions for CH<sub>4</sub> production. Greater CH<sub>4</sub> fluxes, particularly if standing water is present, from the drainage ditches, could be explained by microbial  
440 breakdown of dissolved organic carbon (DOC) or the lateral transport of dissolved CH<sub>4</sub> produced in the anoxic peat field layers (Billett & Moore, 2008; Teh et al., 2011; Cory et al., 2014; Logue et al., 2016).

Drainage has been documented to decrease CH<sub>4</sub> emissions (Abdalla et al., 2016; Basiliko et al., 2007; Korkiakoski et al., 2020; Waddington & Price, 2000), but soil moisture is widely accepted to have a significant influence on CH<sub>4</sub> emissions (e.g., Abdalla et al., 2016; Basiliko et al., 2007; Manning et al., 2019; Moore & Dalva, 1993; Moore & Roulet, 1993). VWC  
445 measurements likely do not correlate well with CH<sub>4</sub> flux as they were taken in the surface peat that is disconnected from the moisture profile as a result of harrowing.

WTD measurements taken from June 2019 through August 2020 by Lai (2022) indicate that during the summer months, the WT decreases toward the edge of the field, measuring approximately 0.6 m from the surface at a distance of 1 m from the ditches, due to the drainage of water into the base of the drainage ditches. Between June and October, at a distance  
450 of ~ 13.5 m away from the drainage ditches, the WT remained at ~ 0.8 m below the surface, except after a few large rain events. The peat water content above the water table in the field centre ranged from ~50% at 0.1 m depth below the surface to >70 to 80% at 0.3 m depth. These measurements indicate little difference in the potential oxidation path length from the field edge to the centre, further supporting our assertion that peat age is the primary control over CO<sub>2</sub> production.

Vegetation removal also plays a role in the decline of CH<sub>4</sub> transmission to the atmosphere compared to natural or  
455 restored sites. The absence of vegetation removes the input of labile C to the anoxic layer usually facilitated by sedge roots in natural peatlands (Joabsson et al., 1999; Dorodnikov et al., 2011) and the transport of CH<sub>4</sub> to the surface via vascular plants ceases (Korkiakoski et al., 2020).

Our results indicate that the field surfaces from all sectors are not significant sources of CH<sub>4</sub> to the atmosphere. At the same time, the drainage ditches produce almost seven times more CH<sub>4</sub> on average (9.2 and 72.0 mg C m<sup>-2</sup> d<sup>-1</sup> for the field and drainage ditches, respectively) during the warm seasons; ditches are frozen for five-six months of the year. Ultimately, no other significant trends or correlating variables were found to explain variation in our measured CH<sub>4</sub> fluxes.

## 5. Summary and Conclusion

We were able to determine that peatlands undergoing active peat extraction are net sources of C to the atmosphere, with average CO<sub>2</sub> and CH<sub>4</sub> flux values similar to those of post-extraction, unrestored peatland sites. The newly open sectors are a greater source of CO<sub>2</sub> to the atmosphere for the first few years, but then the emissions become independent of the duration of extraction. This suggests that two different emission factors, one for newly opened, and one for older sectors might be appropriate. The spatial-age effect across the domed fields, where CO<sub>2</sub> emissions increase with increasing distance from the drainage ditches, also declines and plateaus. CH<sub>4</sub> emissions do not appear to exhibit a clear spatial or temporal pattern between sector ages or measurement positions, although lower CH<sub>4</sub> fluxes are observed from the centre of the peat fields. The drainage ditches are sources of CH<sub>4</sub> to the atmosphere, while the field surfaces have very low CH<sub>4</sub> emission. In the fields of this study, the ditch spacing was every 30 m, so the ratio of the ditch to field surface area was ~30:1. The CO<sub>2</sub> from the field and ditches was not substantially different, the two surfaces contribute roughly proportional to their area in a sector (e.g. fields and ditches contributed 97% and 3%, respectively to the overall flux from a sector). Conversely, the CH<sub>4</sub> was significantly greater from the ditches than the fields, so the ditches emitted disproportionately more CH<sub>4</sub> than their relative area (field emitted 79% and ditches 21% of the overall CH<sub>4</sub> flux from a sector). Laboratory incubations did not show measurable CH<sub>4</sub> production from the peat samples, at an estimated 80-90 % moisture content. Under constant moisture and temperature conditions, the CO<sub>2</sub> production potential of peat from the 1987, 2007, and 2016 sector ages displayed the same behaviour as CO<sub>2</sub> emissions under field conditions. CO<sub>2</sub> production potential increased with distance from the ditch from the top peat layers in the youngest 2016 sector, but this pattern was not displayed from the older 1987 or 2007 sectors. CO<sub>2</sub> production potential of peat samples at depths of 50 and 80 cm was similar between all three sector ages, although a significant amount of variation was observed

between the sector age. Peat age, which appears to be a good indicator of quality difference, was determined to be the primary driver of CO<sub>2</sub> production across the field width and sectors.

The quantification of C emissions from these sites allows for more accurate estimates of peat production's overall impact on atmospheric C accumulation. The results of this study provide the industry with scalable numbers of CO<sub>2</sub> and CH<sub>4</sub> emission to determine potential mitigation tactics and move forward with the continued sustainable and responsible management of this resource.

### **Author Contributions**

LC was responsible for the collection of the field and laboratory data, analysis and the initial draft preparation; IBS was the primary supervisor of LC, reviewed and edited the manuscript and contributed to the methodological design; MS was co-supervisor of LC, reviewed and edited the manuscript and contributed to the methodological design; NTR reviewed and edited the manuscript, contributed to the methodological design and is the PI on the project that funded the study. KHK and HT contributed to laboratory analyses and FT-MIR data evaluation and reviewed and edited the manuscript.

### **Competing Interests**

The authors declare that they have no conflict of interest.

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