Data report - PAGE21 WP3, Milestone 22

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1 - Introduction

The vulnerability of permafrost carbon to decomposition and greenhouse gas production is a key factor for understanding the potential carbon feedback mechanisms to the atmosphere from thawing permafrost. Northern hemisphere permafrost carbon pools represent approximately 50% of the global subsurface organic carbon pool and are important to the understanding of the global carbon cycle and Arctic carbon climate feedback mechanism.

This data report is delivered to WP8 and the rest of the WP3 as part of the milestone MS22: "All laboratory experiments completed". The report is therefore a data report providing input to coming scientific reports on results and interpretation.

In this report we present data from more than 230 CO_2 incubation measurements of arctic soil samples from the six PAGE21 primary sites over a period of about 40 days. The samples have been collected from various depths from different landforms located in different contrasting region of the Arctic in collaboration with the other WP3 partner, University of Stockholm, Peter Kuhry and co-workers. Incubation experiments have been made at Center for Permafrost (CENPERM), at the Department of Geosciences and Natural Resource Management, University of Copenhagen. The dataset provides an overview of the potential soil CO_2 production and release after thawing and allow a further analysis of the driving parameters controlling arctic permafrost carbon dynamics. Selected samples have furthermore been subject to detailed investigations on temperature-sensitivity and anaerobic conditions.

Data has been analysed using MATLAB and the results presented here are the final calculated CO_2 production rates. The full data set, including all CO_2 concentrations and analytical code, are available on request.

2 - Field sites and notation

Samples from all six “primary sites” within PAGE21 have been analysed, including two primary sites from Svalbard.

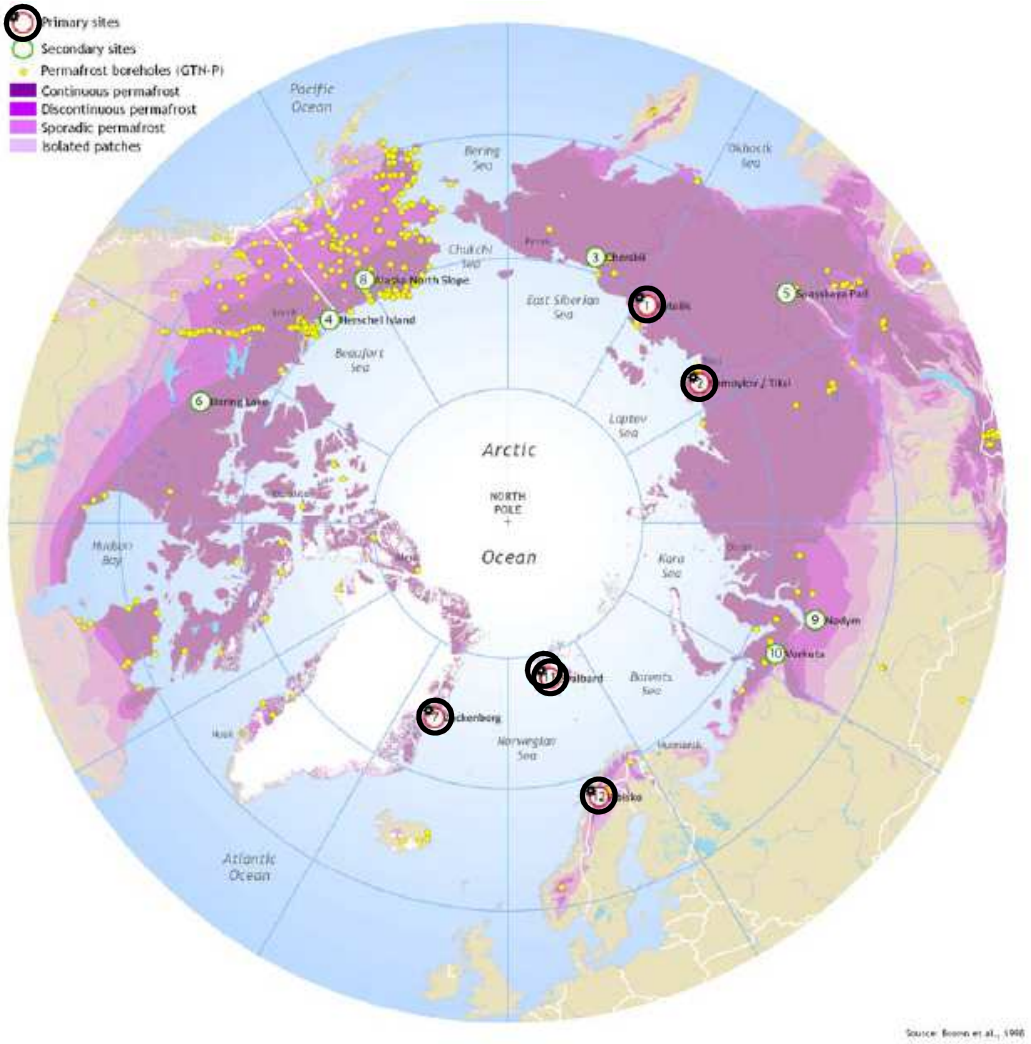

Figure 1. The official PAGE21 map showing the location of the six primary sites and marked with red and thick black circles.

For each of the six primary sites, sample locations and sample transects were selected prior to fieldwork to capture the most representative landforms for each of the investigated region. To ensure each precise site location had an equal probability for being sampled, coring points were selected along transects with a fixed

distance between sampling locations. In the field, the precise location of the coring site was selected randomly and recorded using a hand held GPS.

For each primary site and sample location sample ID's were assigned the following name structure: **SITE-CORE-TYPE**

SITE: 2 letters representing the site: Abisko (AB), Adventdalen (AD), Ny-Ålesund (NA), Kytalyk (KY), Kurungnakh, Lena Delta (KU), First terrace, Lena Delta (LF, lower flood plain), Samoylov island, Lena delta (SA) & Zackenberg (ZK).

CORE: structured as TX_Y with X being the transect number in the area and Y the core number within this transect.

TYPE: 2 letters representing the type of sample (Organic layer (OL), Active layer (AL) & Upper permafrost (UP, 10 cm below current maximum thawing depth).

Sites are described briefly below.

Adventdalen, Svalbard (**AD**)

Adventdalen is a 40km long glacial valley located in Central Spitsbergen, Svalbard. It is surrounded by ice caped mountain ranging in altitude from 1000 to 1100m. Typically, permafrost thickness is around 400m and active layer depth of between 40 and 100cm. In Adventdalen, more than 80 samples were collected on more than 30 sites amount 4 long transects. Transect 1 (**T1**) and transect 2 (**T2**) were done in the central vegetated part of the valley. Transect 3 (**T3**) is from a solifluction slope NW from T1 and T2. While transect 5 (**T5**) show the transition between a mountain plateau and the valley system.

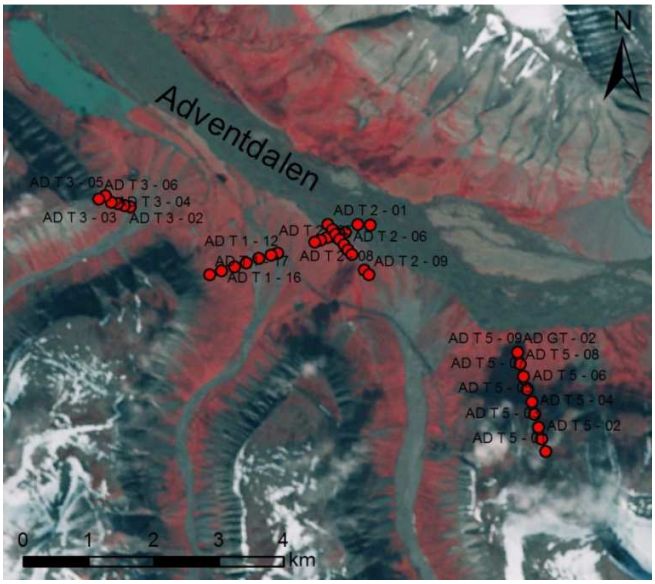

Figure 2. Sampling locations at "Adventdalen" primary site, Svalbard (AD)

Ny-Ålesund (**NA**)

Ny-Ålesund is a research town located on the coast of Kongsfjorden, North West of Spitbergen. The landforms are dominated by moraines deposits as well as raised beaches with very little vegetation and soil development. Transect 1 (**T1**) and transect 2 (**T2**) were selected in areas characterized by moraines and riverine deposits around the Bayelva river. Transect 3 (**T3**) and transect 4 (**T4**) were selected on Kvadehuka, a raised beaches complex characterized by very little vegetation.

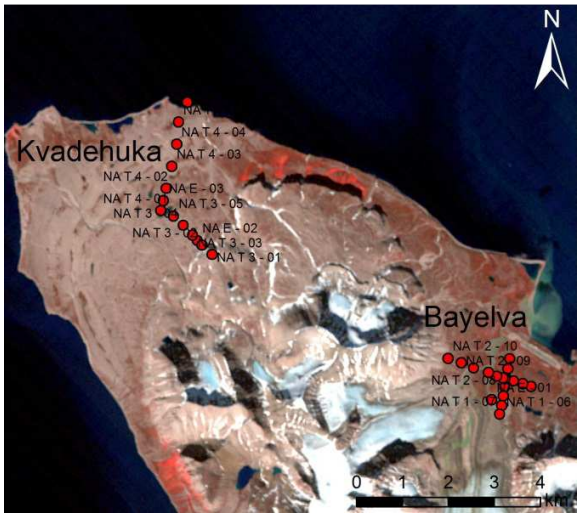

Figure 3. Sampling locations at “Ny-Ålesund” primary site, Svalbard (NA)

“Samoylov/Tiksi” locations in the Lena Delta

The Lena Delta is the largest delta in the Arctic located in Northwest Siberia at the Laptev Sea coast. It is characterized by a network of river channels and more than 1000 islands. The delta can be divided into 3 mains geomorphological terraces. The first terrace is the active floodplain and the youngest part of the delta. The second terrace, also of fluvio-deltaic origin, formed earlier between the Late Pleistocene and the Early Holocene. The third terrace is an erosional remnant of the Late Pleistocene plain characterized by giant ice-wedges systems formed within old Yedoma loess deposits.

First terrace (**LF, lower flood plain**)

In total, 4 transects (**T1**, **T2**, **T3** and **T4**) were done within the deposits of the first terrace. All the cores were retrieved from delta Islands in the surroundings of the Samoylov field station. Transects are crossing different terraces within the first terrace, with samples in the very active floodplain and in the older sporadically active domain influenced by decadal flood events.

Figure 4. Sampling locations at “Samoylov/Tiksi” primary site; *Kurungnakh Island and floodplain, Lena Delta (KU)*

Kurungnakh, Lena Delta (**KU**)

Kurungnakh is an island part of the third Pleistocene terrace. In total 4 transects (**T1**, **T2**, **T3** and **T4**) were studied as well as one cliff natural exposure (**EXP_1**). Most of the samples are taken from the elevated terrace but some of them were retrieved within at a drained thermokarst lake bottom (KU-T1_1 to KU-T1_3 and KU-T2_8 to KU-T2_10).

Figure 5. Sampling locations at “Samoylov/Tiksi” primary site; *Kurungnakh Island, Lena Delta (KU)*

Samoylov island, Lena Delta (**SA**)

The Samoylov island is part of the recent first alluvial terrace deposits. One core was retrieved and analyzed from it in 2009, prior to the important field campaign that took place in 2013. The studied site was selected among ice wedge carbon rich deposits.

Abisko, Sweden (**AB**)

Abisko is the only site located within the discontinuous permafrost zone. One single 2m long core was retrieved from a palsa site in Storedalen, a well-known preserved permafrost area surrounded by shallow lakes.

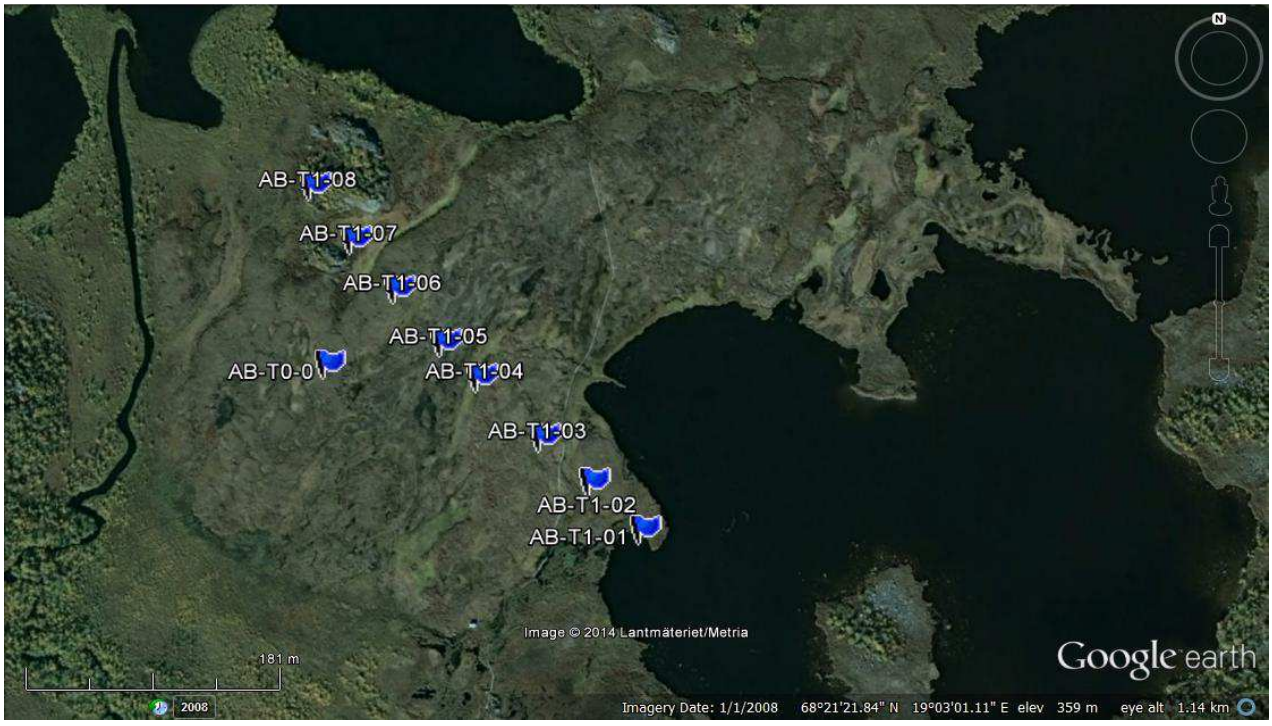

Figure 6. Sampling locations locations at Abisko, Sweden (AB).

Zackenberg, Greenland (**ZK**)

Zackenberg is located in the coast of Northwest Greenland, 30km southeast of the ice front and 30km northwest of the outer coast. The area is characterized by U-shaped glacial valleys opening to the Southeast. The area is covered in Quaternary glacial sediments and landforms like moraine ridges, till, relict delta-system and is currently dominated by periglacial processes. Nearly continuous vegetation cover is present in the Zackenberg lowlands with various types of arctic species. The samples from the area are from carbon rich landforms as fen, palsa, wetlands, as well as less carbon rich as grasslands.

Figure 7. Sampling locations at Zackenberg, Greenland (ZK).

Kytalyk (**KY**)

The Kytalyk research station (70° 49' 45''N; 147° 29' 39''E) is located in the zone of continuous permafrost. Landforms at Kytalyk include river floodplain, dry tundra on "ice complex" hills and plateaus and dry/wet tundra on drained thaw lake bottoms. Maximum active layer thaw depths are typically within 20-40 cm from the ground surface. All soil incubation CO_2 production rates for the Kytalyk primary site were performed at the research station, since a valid soil sample export permit could not be produced in due time for completion of the field work. Hence, reported rates will only cover the initial CO_2 production rates after pre-incubation.

Figure 8. Sampling location at “Kytalyk” primary site (KY).

3 - Sampling procedure

At each site, vegetation and landforms were described according to the standard protocol. All fieldwork were conducted during the summer where the active layer was melted allowing to be dug out for easier upper soil description and sampling. The volume of each sample was calculated on the field depending on the sampling technique in order to obtain an accurate density after weighing. Incubation measurements are very time and resources consuming. For each sites, we selected representative samples of the different soil horizons. This technique offers an important number of investigated sites within a region with very little loss on precision. We acquired mostly 3 different types of samples:

- **OL**: sample from the Organic Layer. It is typically the upper part of the soil, containing dead vegetation. The fresh vegetation was removed manually.
- **AL**: sample from the Active Layer. It is located between the bottom of the organic layer and the permafrost table. The sample is representative of the entire active layer. If within the active layer 2 contrasting soils horizons were described, then 2 active layer samples from each horizon were retrieved (**AL**).
- **UP**: sample from the Upper Permafrost. Sampled 5 to 10cm below the permafrost table to unsure an undisturbed permafrost sample.

For a number of sites, coring to depths deeper than 20-30 cm was not possible, usually due to the presence of high amounts of stones. Hence, only the upper part of the soil was retrieved.

Non permafrost samples (OL and AL) were stored at -15 °C the same day of retrieval. Permafrost samples were kept frozen using thermal boxes after retrieval, and stored frozen in the same conditions. They were afterwards shipped frozen to CENPERM and stored around -18 °C. All the samples were then prepared for incubation in a freeze room in order to better control the start of the incubation.

Laboratory analyses

To ensure representative mass reduction of the primary sample for the incubation vials, samples were gently crushed in their frozen state using steel mortar. The frozen soil material was split and sieved until all the particles diameter was could pass through an 8mm sieve. The sieved sediment was divided into identical

replicates using a rotary riffle splitter where between 3 and 5g of sediment was transferred into the 20mL incubation vials.

For each sample, one replicate vial was used to calculate the water, carbon and nitrogen content:

- Water content was calculated using the difference between wet weight and weight after freeze drying. The results are expressed as % of the mass. Using the water content and the Wet Bulk Density calculated on the field we extracted the Dry Bulk Density of each samples expressed in g cm^{-3}.
- Carbon and nitrogen were measured using a LECO TruSpec Carbon Nitrogen Determinator.

One replicate vial was kept for the incubation measurements. The incubation were conducted under oxic conditions so the vials were closed using plastic foil allowing gas exchange but no water exchange. To ensure maximum aeration of the soil promoting oxic conditions, vials were drained until field capacity soil water content upon thawing during the pre-incubation process.

A separate batch of replicate vials were prepared for incubation under anoxic and water saturated soil moisture conditions, by keeping the original soil moisture content unaltered and flushing the headspace with oxygen-free argon (Ar 5.0) while closed with a butyl-rubber septa and aluminium crimp cap. The anoxic vials were pre-incubated under dark conditions for 90 days at a constant temperature of 5 °C. Prior to the first measurement, the headspace CO_2 concentration was reset by flushing with argon.

Incubation experiments

The potential release of CO_2 from soils following decomposition of soil organic matter has traditionally been measured by gas chromatography by which the different constituents of a gas mixture is separated and individually quantified. In gas chromatography, the mobile phase is an inert and pure carrier gas which transports the injected analyte to the stationary phase. This stationary phase is typically a microscopic layer of polymer on an inert solid support inside a glass or metal column, on which the individual components of the analyte is adsorbed and eluted at the retention time of the compound. While offering measurements of high precision and accuracy, gas chromatograph data requires ultra-pure carrier and make-up gases for operation, needs frequent calibration and maintenance and considerable training to operate.

The alternative to gas chromatography is the use of near-infrared gas analysers which measures gas concentrations by determining the adsorption of an emitted infrared light-source through a certain air sample. Initially tested to facilitate incubation experiments and gas measurements directly under field conditions beyond access to compressed inert carrier gas, we have used a LI-840A absolute, non-dispersive, infrared (NDIR) gas analyser based on a single interchangeable optical path, and a dual wavelength, infrared detection system (Figure 9).

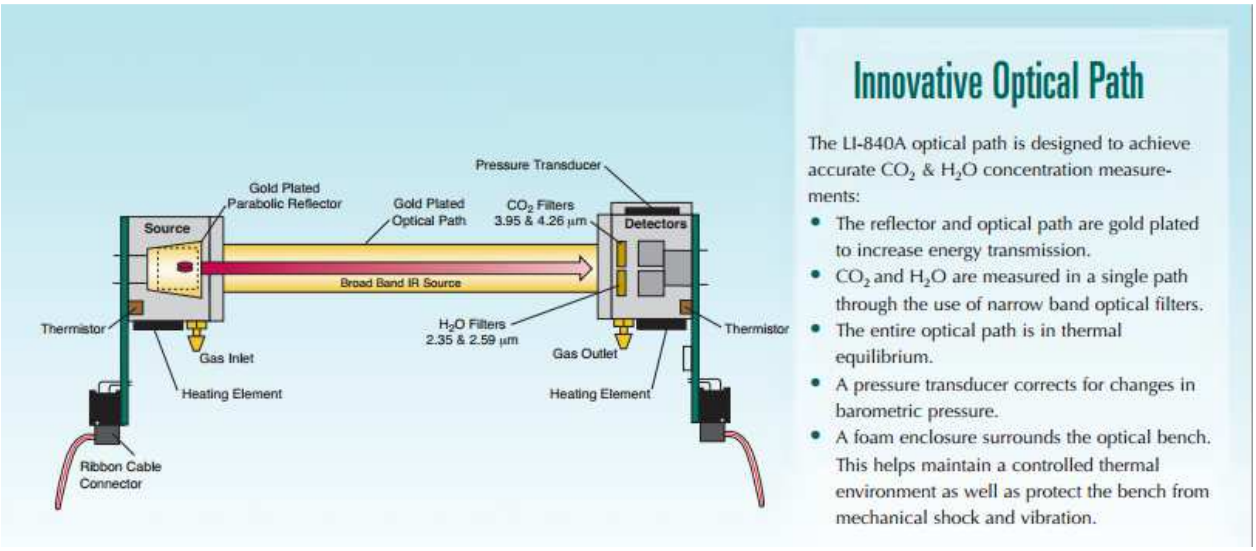

Figure 9. Illustration of LI-840A optical path and CO_2 detection design.

The system offers high absolute and relative stability with low zero and span drift, high accuracy and precision due to automatic temperature and pressure compensation and automatic corrections of CO_2 concentrations for band broadening due to the presence of water vapour. It has a <1ppm signal noise at 370 ppm for CO_2, a certified CO_2 measurements range of 0-20.000 ppm and an operation temperature from -20 to + 45 C, making it ideal for both field and laboratory measurements.

Analytical system for CO_2 concentration measurements

The central part of the analytical system consists of a LI-840A CO_2/H_2O Gas analyser (LICOR® Biosciences) which inlet and outlet are connected in a closed loop to a brush-less diaphragm pump (flow rate: 500 mL minute^{-1}),

two 3-ways flushing valves and an injection vial with a replaceable butyl rubber septa through which a gas sample can be injected to the closed loop (Figure 10, see APPENDIX – LI-840A specifications).

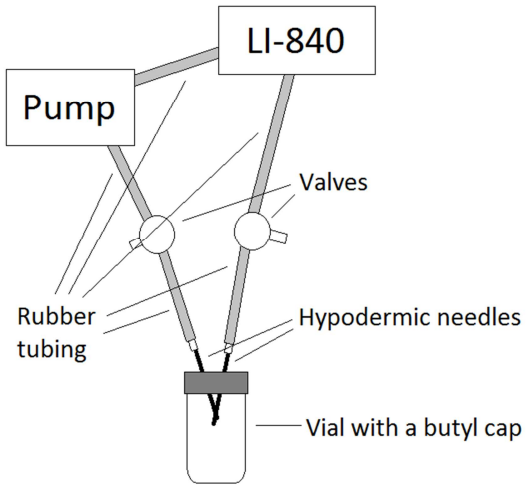

Figure 10. Conceptual figure of the closed loop system.

Concentration determination of the injected gas sample is calculated based on a modified mass balance equation:

$$C_2 = (C_3*V_3 - C_1*V_1)/V_2 \qquad \text{(eq.1)}$$

Where

C_1: Measured CO_2 concentration in the closed loop before the sample is injected (ppm).

C_2: CO_2 concentration of the injected sample (ppm).

C_3: CO_2 concentration in the closed loop after the sample is injected (ppm).

V_1: Volume of the system before injection (ml).

V_2: Volume of the injected sample (ml).

V_3: Volume of the system after injection (ml).

Prior to each injection and between measurements the internal air volume of the analytical system (40 ml) is ventilated with ambient atmospheric air for approximately 30 seconds until system concentration is stable (<0.5 ppm above or below mean value).

System calibration

The analytical system was calibrated with certified standard gases (MikroLab, Århus, Denmark) in the concentration range 0, 500; 1.000; 2.000; 5.000 & 10.000 ppm CO_2 (n=20 for each level).

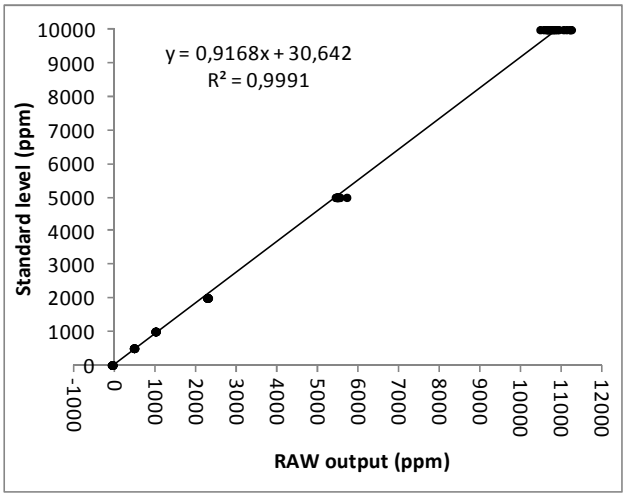

Figure 11. System calibration curve. X-axis show calculated RAW output values (ppm - equal to C2) and Y-axis show standard gas calibration level.

Resulting show a linear response in the output to increasing concentration levels within the calibration range with a determination coefficient (R^2) of 0.9991 (Figure 11).

Stabilization period following injection

Stable gas concentrations in the closed loop before and after sample injection are a prerequisite to achieving both accurate and precise CO_2 measurements. Tests show that stable gas concentrations in the closed loop are established after the first 30 seconds following the closure of system ventilation valves or sample injection.

After this stabilization period a concentration independent complete mixture of the injected gas volume in the total system volume is achieved (Figure 12).

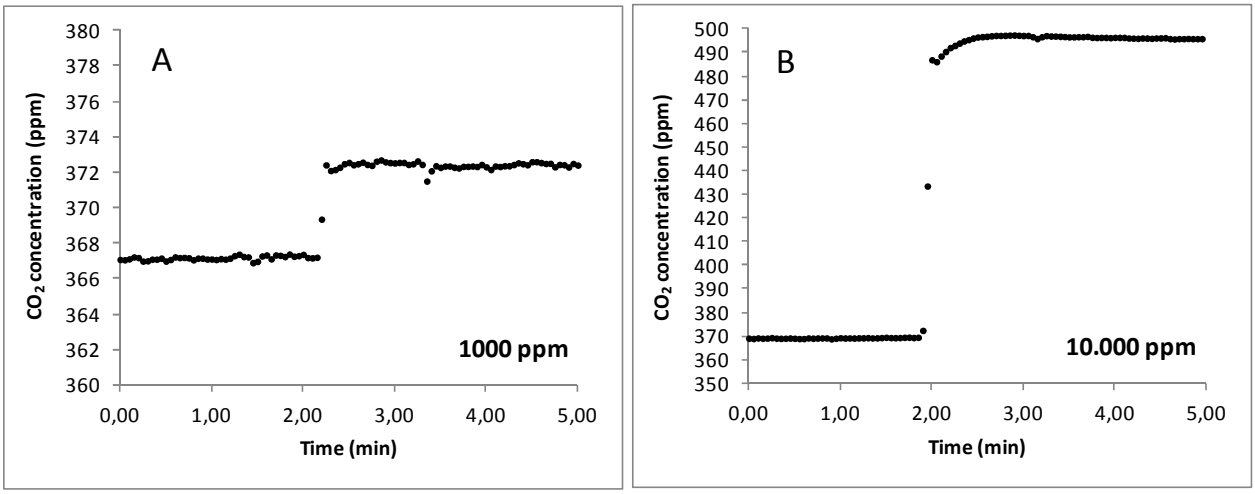

Figure 12. CO_2 concentration in closed loop before, during and after injection of gas standards of (A) 1.000 ppm CO_2 and (B) 10.000 ppm CO_2.

Injection of gas with a CO_2 concentration of 10.000 ppm results in system CO_2 concentrations in the concentration range around 500 ppm (Figure 12), which is within the operation range of 0-20.000 ppm.

Calculated of CO_2 production rates

The measured development in headspace CO_2 concentration over time was calculated into CO_2 production rates per gram dry weight or gram carbon using the modified ideal gas law equation:

$$n = \frac{P \times V}{R \times T} \times C \qquad \text{(eq.2)}$$

where *n* is CO_2 concentration (µmol), *P* is system pressure (atm), V is headspace volume (L), R is the gas constant (0.0831 L atm^{-1}/mol K^{-1}), *T* is incubation temperature (Kelvin) and *C* is the headspace CO_2 concentration (µmol mol^{-1}).

Temperature sensitivity incubations

The temperature dependence on CO_2 production rates in permafrost soil was determined by incubating identical soil sample replicates in a stable temperature gradient using an insulated solid aluminium thermoblock of 1.85 m length. Incubation temperatures ranged from -12 °C to 12 °C in uniform temperature increments with an average temperature difference between samples of approximately 0.75 °C. The temperature of each incubation interval was measured using either type-T or pt100 thermocouples and logged on a microcontroller (Campbell Scientific CR1000) every 10 minutes. Standard deviation at each temperature interval was less than 0.1 °C over the entire duration of the incubation experiment. The Q_{10} temperature coefficient was calculated on basis of an exponential fit to the incubation data with temperature as independent variable and CO_2 production rate as dependent variable using the following equation,

$$Q_{10} = \left(\frac{R_2}{R_1}\right)^{10/(T_2 - T_1)} \qquad \text{(eq.3)}$$

where R_1/R_2 are the CO_2 production rates at temperature T_1/T_2.

4 - Results

Time dependent CO_2 production rates

Results of the incubation experiment are summarized for the different samples at the different location and grouped according to the total organic carbon content (Figure 13). The replication method was tested by incubating 3 replicates of the same sample.

The rate of CO_2 release after 40 days show that the OL samples had the highest rates and rates from active layer and top permafrost are similar. No significant correlations were noted between CO_2 production rates and the carbon content.

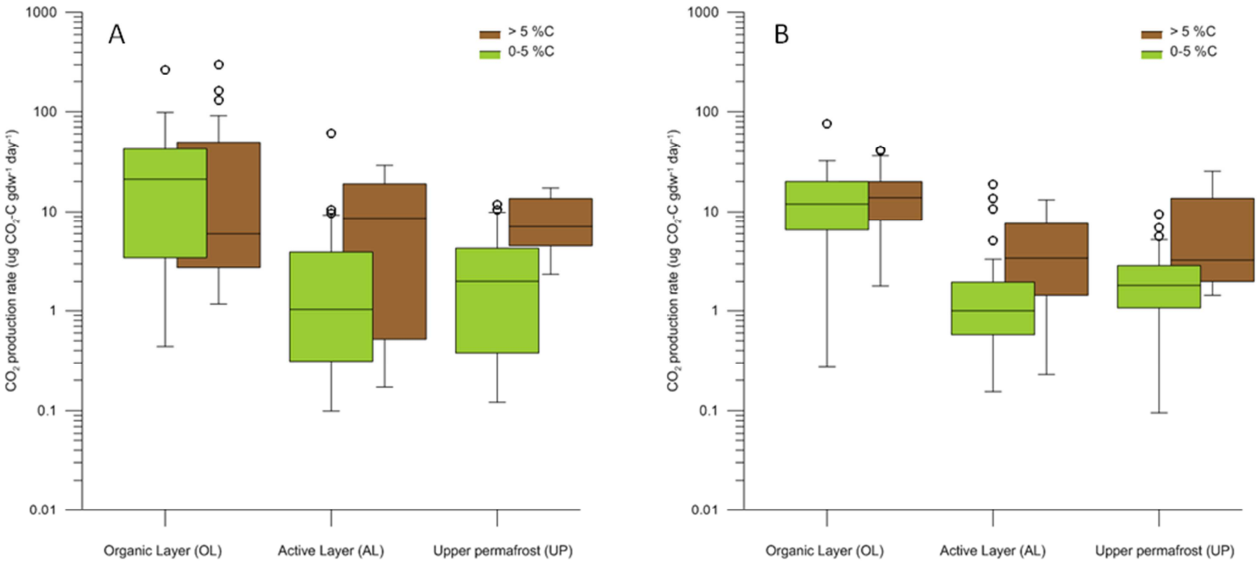

Figure 13. Observed CO_2 production rates according to TYPE: A) Initial CO_2 production rates (t0); B) CO_2 production rates after 40 days incubation at 5 °C. Results are grouped according to a total organic carbon content (%C) threshold of 5%.

Temperature-dependent CO_2 production

Selected samples from PAGE21 sites have been incubated in an insulated solid aluminium thermoblock of 1.85 m length to assess the temperature-dependency of CO_2 productions. Results are based on incubated soil (equivalent to 2-3 g dry soil) in a stable (±0.3°C) temperature gradient (Figure 14).

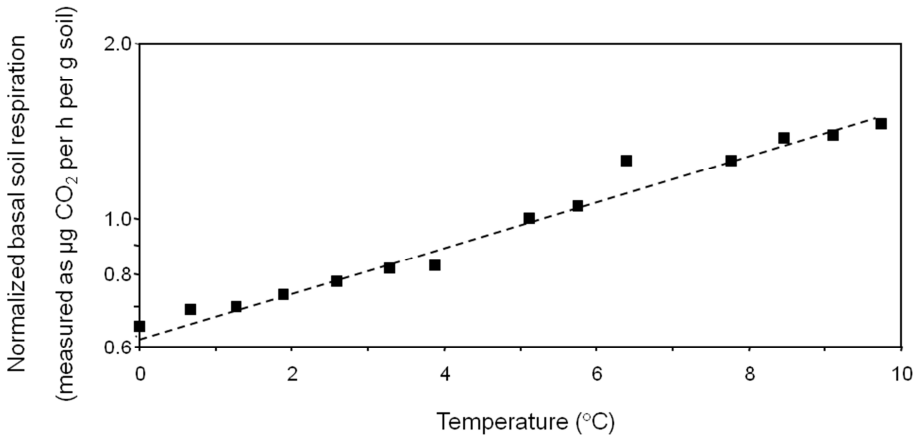

Figure 14. Temperature-dependent CO_2 production based on permafrost samples from Zackenberg measured three times during the incubation experiment. Data are normalized to 5 °C and an exponential fit to data is shown as a dashed line (y = 0.6233e0.0903x, R^2 = 0.9825, p< 0.05) indicating a Q_{10}-value of 2.47.

Moisture and O_2 dependent CO_2 production rates

Selected non-drained permafrost samples were incubated under anoxic waterlogged environmental conditions. Results are summarized in Figure 15 showing an overall rate decrease of active layer samples.

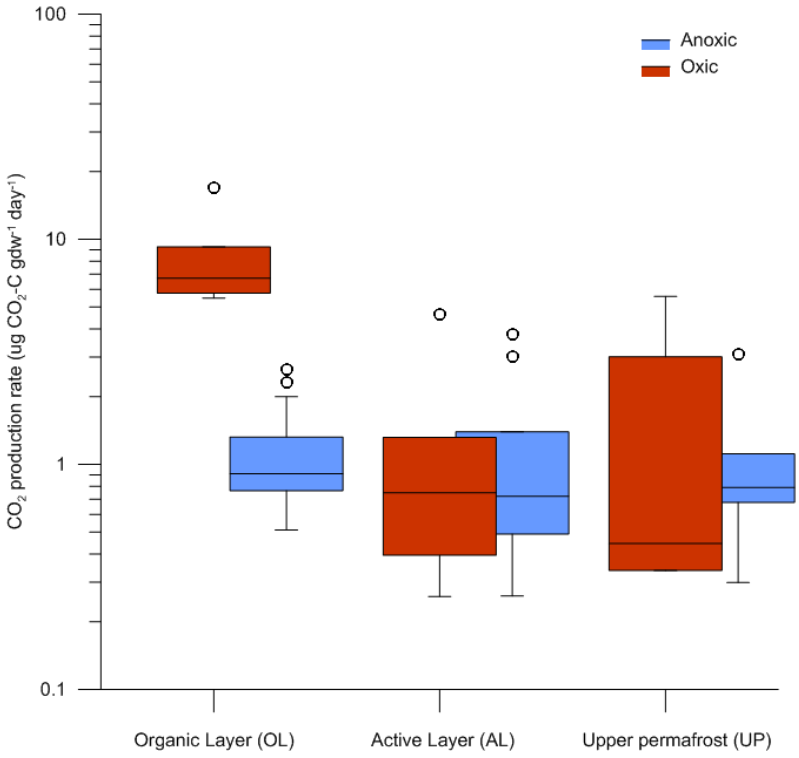

Figure 15. Observed CO_2 production based on intact permafrost samples (ambient) and after anoxic conditions, Adventdalen.

Field incubation results

Field incubations were made at Russian field sites due to the uncertainty of getting frozen samples exported to University of Copenhagen. Samples were subsequently received from Lena delta but not from Kytalyk. Thus. Field incubation (Figure 16) from Kytalyk are the only rates available, whereas field incubation experiments from Lena delta are now far more well described based on laboratory incubations. However, the general conclusions from comparing field and laboratory incubations is that field results can be used as a fair proxy for detailed laboratory incubations.

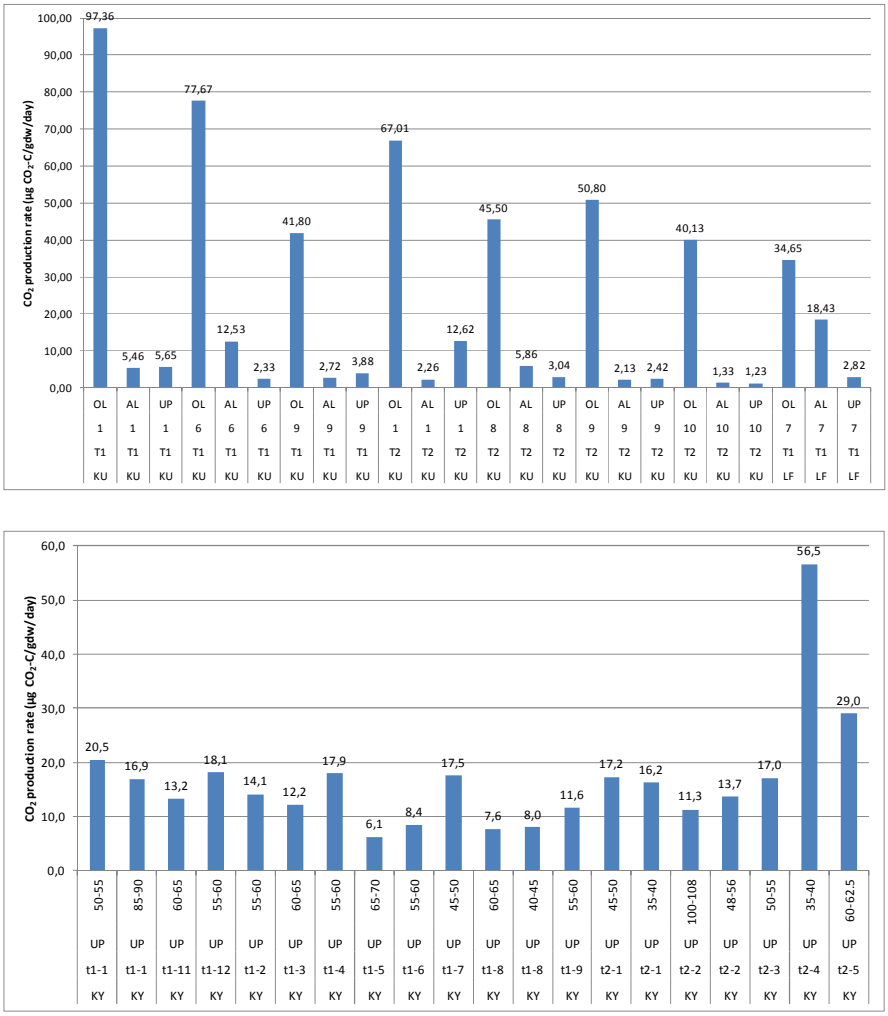

Figure 16. CO_2 production rates based on intact permafrost samples incubated in the field from Samoylov and Kytalik

APPENDIX – LI-840A specifications

CO_2

Measurement Range:	0-20,000 ppm
Accuracy:	<1% of reading
Calibration Drift	
Zero Drift[1]:	<0.15 ppm/°C
Span Drift[2]:	<0.03%/°C
Total Drift[3] at 370 ppm:	<0.4 ppm/°C
RMS Noise at 370 ppm with	
1 sec signal filtering:	<1 ppm

H_2O

Measurement Range:	0-60 ppt
Accuracy:	<1.5% of reading
Calibration Drift	
Drift[1] at 0 ppt:	<0.003 ppt/°C
Span Drift[2] at 10ppt:	<0.03%/°C
Total Drift[3] at 10 ppt:	<0.009 ppt/°C
RMS Noise at 370 ppm with	
1 sec signal filtering:	<0.07 ppt
Sensitivity to CO_2:	<0.0001ppt H_2O/ppm CO_2

[1] Zero Drift is the change with temperature at 0 concentration.
[2] Span Drift is the residual error after re-zeroing following a temperature change.
[3] Total Drift is the change with temperature without re-zeroing or re-spanning.

Measurement Principle:	Non-Dispersive Infrared
Traceability:	Traceable gases to WMO standards from 0 to 3,000 ppm. Traceable gases to EPA protocol gases from 3,000 to 20,000 ppm
Pressure Compensation Range:	15 kPa – 115 kPa
Maximum Gas Flow Rate:	1 liter/min
Output Signals:	Two Analog Voltage (0-2.5V or 0-5V) and Two Current (4-20mA) Digital: TTL (0-5V) or Open Collector
DAC Resolution:	14-bits across user-specified range
Source Life:	18,000 Hours (~2 years continuous use)
Power Requirements:	Input Voltage 12-30 VDC; 1.2A @ 12V (14W) maximum during warmup with heaters on; 0.3A @ 12V (3.6W) average after warmup with heaters on
Operating Temperature Range:	–20 to 45°C
Relative Humidity Range:	0 to 95% RH, Non-Condensing
Dimensions:	8.75" x 6" x 3" (22.23 x 15.25 x 7.62 cm)
Weight:	2.2 lbs. (1 kg)

* Specifications subject to change without notice.