

# Moisture and Temperature Dependence of $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ of $\text{CO}_2$ from Heterotrophic Soil Respiration

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## I. Background

Understanding the role of terrestrial ecosystems in governing the isotopic composition of atmospheric  $\text{CO}_2$  requires knowledge of environmental controls of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  of  $\text{CO}_2$  in biosphere-atmosphere  $\text{CO}_2$  exchange. Heterotrophic respiration contributes approximately half of the total respiratory backflow of  $\text{CO}_2$  from terrestrial ecosystems to the atmosphere, but too little is known about environmental controls of C and O isotopic signatures of  $\text{CO}_2$  released from soils to be able to reliably quantify the contribution of different  $\text{CO}_2$  flux components (photosynthesis, above- and belowground autotrophic as well as heterotrophic respiration) to net ecosystem  $\text{CO}_2$  exchange.

## II. Experiment

The dependencies of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  of  $\text{CO}_2$  from heterotrophic soil respiration on soil temperature and soil moisture were investigated. Samples of the organic layer (O) and humified A horizon ( $A_h$ ) of a Norway spruce (*Picea abies* (L.) Karst) forest (Höglwald close to Augsburg, Germany) were incubated at different temperature and moisture levels in gas-tight incubation tubes (exetainers), followed by isotope-ratio mass spectrometer (IRMS) analysis of the  $\text{CO}_2$  formed during heterotrophic soil respiration. The increase of  $\text{CO}_2$  mixing ratios during the incubations, monitored by IRMS at different sampling times, allowed for calculation of the  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  of the respiratory source of the  $\text{CO}_2$  by the Keeling plot methodology.

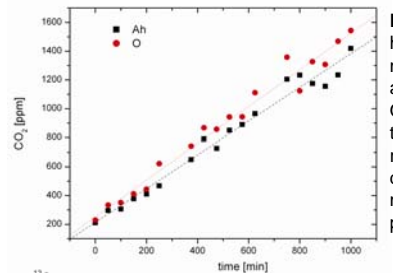
## III. Method

Samples of the O layer below the needle litter and of the  $A_h$  horizon were taken on the 10<sup>th</sup> of May 2007 and 30<sup>th</sup> of August 2007, taken back to the lab on the same day, and kept refrigerated at 5°C until incubation and analysis. Preparation of refrigerated soil samples started two days prior to analytical determination of the  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  of soil-respired  $\text{CO}_2$ . First, samples were sieved at 4 mm in order to homogenate the sample material and to exclude fine roots. Subsequently, the sieved samples of O and  $A_h$  horizon were air-dried for one night. Then, some g of soil material, depending on the temperature and humidity treatment, were placed into 12 mL exetainers (Labco Ltd., UK). For each temperature and moisture level and each horizon 18 replicates were analyzed. The specific amount of water required to achieve the target soil moisture level was added to each exetainer. To avoid drying of samples, the exetainers were sealed with parafilm. Samples were then incubated for one night at the respective temperature, which was controlled with a custom-made sample tray connected to a water bath. The parafilm was removed and the samples were aerated for another 1–2 hours before sealing the exetainers with the appropriate septum screw caps prior to IRMS analysis with a gas bench coupled to a continuous-flow mass spectrometer (DeltaPlusXP, ThermoFisher, Bremen, Germany).

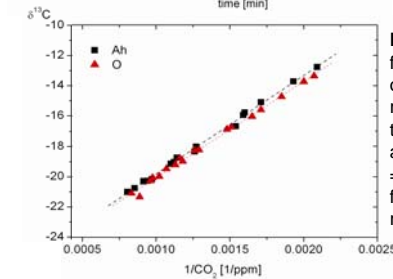


## IV. Results

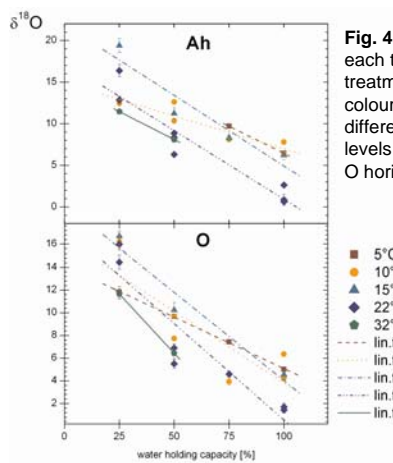
Values for  $\delta^{13}\text{C}$  are given in ‰ vs. PDB, values for  $\delta^{18}\text{O}$  in ‰ vs. SMOW; MWHC = maximum water holding capacity.



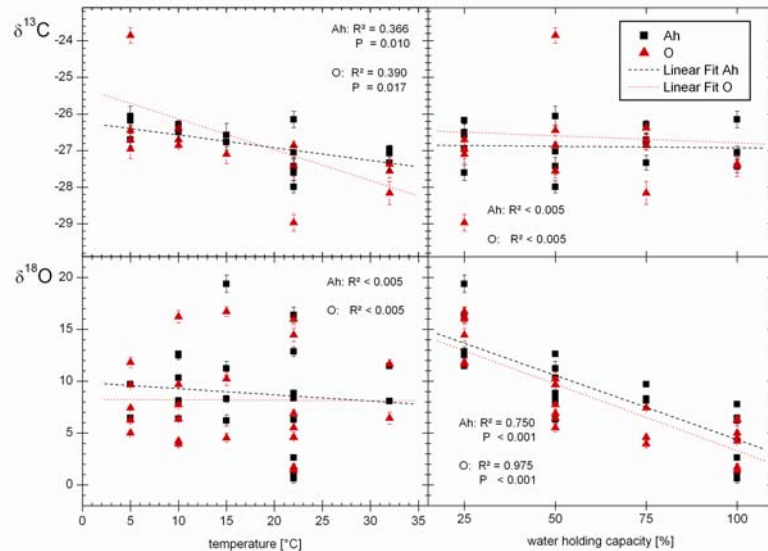
**Fig. 1.** Increase in headspace  $\text{CO}_2$  mixing ratios above soil from the O layer and from the  $A_h$  horizon, respectively, during a 20 h measurement period.



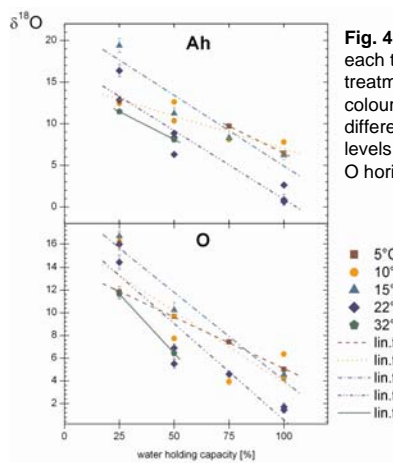
**Fig. 2.** Keeling plots for the determination of  $\delta^{13}\text{C}$  of soil-respired  $\text{CO}_2$ ; treatment at 10°C and 50% MWHC ( $R^2 = 0.966$  and  $0.998$  for  $A_h$  and O, respectively).



**Fig. 4.**  $\delta^{18}\text{O}$  values for each temperature treatment (differently coloured symbols) at different soil moisture levels for  $A_h$  (top) and O horizon (bottom).



**Fig. 3.**  $\delta^{13}\text{C}$  (top panels) and  $\delta^{18}\text{O}$  (bottom panels) at different temperature (left panels) and soil moisture levels (right panels), expressed as % of MWHC, for soil samples of the organic layer and  $A_h$  horizon. Significant linear relationships between  $\delta^{13}\text{C}$  and temperature as well as between  $\delta^{18}\text{O}$  and water holding capacity were found.



**Fig. 5.** Relationship between  $\delta^{13}\text{C}$  of soil-respired  $\text{CO}_2$  of  $A_h$  horizon (top panels) and organic layer (bottom panels) with  $\delta^{13}\text{C}$  of soil carbon (left panels) and soil C/N ratios (right panels). Red lines display best linear fit (blue encircled data point omitted); including all data results in a linear fit indicated by the blue line.

## V. Summary

The  $\delta^{13}\text{C}$  of the  $\text{CO}_2$  respired from root-free organic layer and  $A_h$  horizon did not show any significant relation to soil water content. In contrast,  $\delta^{13}\text{C}$  had a negative significant linear relationship with temperature. Data fluctuated around an average  $\delta^{13}\text{C}$  of  $-25.6\text{‰}$  for the  $A_h$  horizon and  $-26.4\text{‰}$  for the organic layer, reflecting the fact that the organic layer was more depleted in  $^{13}\text{C}$  than the  $A_h$  horizon. Likewise, the  $\text{CO}_2$  from the organic layer was more depleted in  $^{18}\text{O}$  than the  $\text{CO}_2$  from the  $A_h$  horizon. In contrast to the  $\delta^{13}\text{C}$  results, no significant relationship of  $\delta^{18}\text{O}$  to temperature could be observed. However, a highly significant decrease in  $\delta^{18}\text{O}$  of respiratory  $\text{CO}_2$  from both soil layers with increasing soil moisture was found, decreasing from an average of  $+14\text{‰}$  vs. SMOW at 25% MWHC to  $+4\text{‰}$  at 100% MWHC.