

17th Stable Isotope Network Austria (SINA) Meeting: Program and abstracts

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Program

Friday, 8th November 2019 12:00 13:30 Registration & N	Welcome	
Session 1: Ecology and paleoecolo	ogy	Title
13:30 14:15 <i>Keynote:</i> Rober		Tracing seasonal mammoth migrations in central Europe during the last ice using Sr and O isotopes
14:15 14:30 Rebecca Hood-	Nowotny	Non-exchangeable δ^2H of Japanese beetle, a potential tool for protecting Californian agriculture
14:30 14:45 Johannes Ingris	sch	Drought and recovery effects on belowground respiration dynamics of recent carbon in managed and abandoned grassland
14:45 15:15 Coffee and Tea	Break	
Session 2: Novel		Title
approaches		
15:15 16:00 <i>Keynote:</i> Hagit <i>i</i> 16:00 16:15 Kathrin Rosenth		Carbonate ¹⁷ O _{excess} as a paleo-hydrology proxy Ten years of Elementar and IsoPrime: Pioneers in simultaneous CNS isotope ratio analysis
16:15 16:30 Tobias Kluge		Laser-spectroscopic analysis of CO ₂ clumped isotopes
16:30 17:00 Poster introduc	ctions	
17:00 19:00 Poster Session		
Starting at Dinner 19:00		
Saturday, 9th November 2019		
Session 3: Climate and paleoclima	ate	Title
9:00 9:15 Stacy Anne Caro	lin	Rainwater stable isotopes over the Iranian plateau
9:15 9:30 Petra Bajo		Holocene climate variability - an insight from Nova
9:30 9:45 Attila Demény		Grgosova Cave (Croatia) speleothems Paleotemperature reconstructions for the ~100-350 ka period inferred from D/H analyses of fluid inclusion water of speleothems
9:45 10:00 Tanguy Racine		Stable isotopes in cave-ice, a link to past precipitation?
10:00 10:30 Coffee and Tea E	Break	
Session 4: Soil,		Title
water, and air		Title
10:30 11:15 <i>Keynote:</i> Nicola	s Brüggemann	Coupled biotic-abiotic production processes and related stable isotope signature of nitrogen trace gases
11:15 11:30 Giorgio Höfer-Ö	Öllinger	2 years of stable isotope measurements of rivers and springs in southern Salzburg Basin (Golling, Kuchl, Hallein) Salzburg, Austria
11:30 11:45 Elemér László		Continental CH ₄ concentration and ¹³ C/ ¹² C isotope ratio measurements in the Carpathian Basin, Hegyhatsal:

comparison with European clean air sites

Session 5: Geology and geo-engineering

Title Zsófia Kovács

11:45 12:00 Calcium isotope record of the Late Triassic

12:00 12:15 Ronny Boch Application of stable isotopes in geotechnical settings –

Examples from current projects

Starting at 12:30 Lunch

Starting at 13:30 SINA General Assembly

Friday, 8th November 2019 (17:00 - 18:00)

Title Name

Oxygen isotope equilibration of phosphate in the soil /plant system – an ¹⁸O-PO₄ Watzinger, Andrea

organic fertilizer test

Meeran, Effect of summer drought on the coupling of photosynthesis and soil respiration

Kathiravan under current and future climate

Radolinski, Jesse Simulating bypass flow in a two water worlds context

Stable isotope studies (δ^2 H, δ^{18} O) of precipitation and soil water movement in Königer, Paul

forested ecosystems at Solling, Germany

Denitrification dominates drought and post-drought dynamics of nitrous oxide Harris, Eliza

emissions

Cseresznyés, Dóra Dawsonite from surface environment, Covasna (Romania): preliminary results

Soleimani, Mojgan

Speleothem-based isotopic variation during the Holocene period in southern Iran

Höfer-Öllinger, Stable isotopes of daily precipitation sampling: 3 years of observation at Wals and

Sonnblick Observatorium (Salzburg, Austria) Giorgio

Pfeiffer, Jan Stable isotope-based derivation of a conceptual hydrogeological model of the

Vögelsberg landslide (Tyrol, Austria)

Horváth, Balázs Honey water: an easy solution to detect sugar syrup addition?

László, Elemér Estimation of the natural variability of the tritium concentration of precipitation

Zhang, Shasha Climate and land use as important drivers of N dynamics at a continental scale in

Europe

Abstracts

Carbonate ¹⁷O_{excess} as a paleo-hydrology proxy

Affek H.P.*, Barkan E., Bergel S.J., Vieten R., Voarintsoa N.R.G.1

Carbonates are a common paleoclimate archive in both marine and land settings. Land carbonates record both temperatures and hydrological conditions. Paleo-temperatures derived from clumped isotopes in land carbonates are often combined with $\delta^{18}O$ to reconstruct parent water $\delta^{18}O$. However, the common interpretation of water $\delta^{18}O$ as rainfall amounts does not reflect the hydrological complexity of moisture sources and evaporation. These gaps can be filled by information from $^{17}O_{\text{excess}}$ in water, that depends on the conditions in which evaporation occurs, thus reflecting the moisture source in meteoric water or the extent of evaporation in a small water body (e.g., a lake). Water $^{17}O_{\text{excess}}$ can be reconstructed from three oxygen isotope analysis in the same carbonate in which Δ_{47} and $\delta^{18}O$ are measured.

We derive the fractionation factor $^{17}\alpha$ between CaCO₃ and water, using freshwater mollusk shells from well constrained spring environments, at a temperature range of 15-28°C. The spring water temperature and isotopic composition are constant year-round, avoiding uncertainty associated with seasonality.

The observed $^{18}\alpha$ and $^{17}\alpha$ between mollusk aragonite and parent water are used to calculate the fractionation slope θ (=ln $^{17}\alpha$ /ln $^{18}\alpha$), resulting in 0.5231±0.0003, with no discernible temperature dependence. This θ value is only slightly lower than the reference slope of 0.528, implying that $^{17}O_{excess}$ in carbonates relative to parent waters is only weakly temperature dependent. It is therefore a straightforward recorder of parent water $^{17}O_{excess}$. Speleothem carbonates may be a suitable archive for records of $^{17}O_{excess}$ in rainfall and therefore of moisture sources.

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Tracing seasonal mammoth migrations in central Europe during the last ice age using Sr and O isotopes

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Dental enamel is the most resistant tissue capable of quantitative retention of geochemical and isotopic record after animal's death. Despite the fact that in situ analyses of (bio)apatite appear analytically challenging and that enamel may still undergo post-mortem alterations, accurate Sr isotope composition can be recovered with high spatial resolution by laser ablation MC ICPMS (LA MC ICPMS). In this study, we combined spatially resolved Sr analyses of dental enamel with histologogical, trace element and oxygen isotopes investigation in order to decipher the migration patterns of wooly mammoth during the Last Glacial Maximum (LGM) in central Europe.

Histological studies of M3 molar tooth from the Spadzista site in southern Poland indicated about 11 years period of enamel formation in a single plate, while the whole tooth formed only about 1 year longer. Such relatively long time of enamel development provides an opportunity for investigating temporal changes in mammoths habits. Taking into account the applied analytical parameters and the estimated enamel daily extension rates, we achieved < 1 month time resolution of our LA MC ICPMS Sr isotopic composition measurements. Our analyses revealed a reproducible pattern of cyclic changes in ⁸⁷Sr/⁸⁶Sr ratios suggesting temporal changes of pastures located in two geologically distinct areas. The Sr record correlates with changes in O isotope composition supporting annual migration cycle of mammoths. Most of the year they resided in southern Poland where they were hunted by the nomads but for a shorter time periods they migrated to the second, still unidentified region. Commonly accepted scenario is that the mammoths migrated to the southern side of the Western Carpathians via the Moravian Gate. We verified this hypothesis by analysing wooly mammoth molars from Malé Leváre in Slovakia and from Langmannersdorf in Austria. In both cases Sr isotopic signatures showed cyclic variations in Sr composition analogous to those found in the Spadzista site. Identical ⁸⁷Sr/⁸⁶Sr values recorded in the sites in Austria and Slovakia indicate that mammoths from both locations used the same pastures and seasonally migrated between the same two regions separated by at least 100 km distance. Importantly, Sr isotopic signature recorded by mammoths from the southern locations are less radiogenic and do not show any resemblance with the Sr record found in the mammoths from the Spadzista site indicating that the two herds did not mix. Our study demonstrates that the mammoths mobility in central Europe during the Last Glacial Maximum was driven predominantly by seasonal weather changes but the migration pathways locally varied.

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Holocene climate variability- an insight from Nova Grgosova Cave (Croatia) speleothems

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In the recent decades speleothems have been increasingly used in studies of past climate variability. This is not surprising considering their advantages compared to other palaeoclimate archives.

Here we present a speleothem record from Nova Grgosova Cave, Croatia. The cave is located in the northwestern part of the country and was artificially opened in 2004. Monitoring of the cave's microclimate has revealed stable temperatures and relative humidity throughout the year. Stable isotope analyses of composite drip-water samples collected from three sites at monthly intervals show that the rainfall δ^{18} O signal is attenuated but the average drip-water values are ~0.7% more negative than the weighted mean annual value of the rainfall covering the same time period. This information, together with a slight negative water balance during the summer months based on long-term climatological data collected at the neighboring climate station, implies that the colder part of the year is the main infiltration season.

Four speleothems were retrieved from the cave, each of which was cut in the half and polished. Powder samples were subsampled for $\delta^{18}O$ and $\delta^{13}C$ analyses. Solid and powder samples were also taken for dating by the U-Th method. The results reveal that the whole record spans the time period from 10.5 ka BP to recent. Excellent correspondence in $\delta^{18}O$ profiles amongst the four stalagmites has allowed placing them onto a common depth/age scale by using an intra-site correlation age modelling (ISCAM) approach to produce a composite record. In this presentation, we discuss these results and place them into the wider context of Holocene climate variability by comparing them to previously published palaeoclimate records.

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Application of stable isotopes in geotechnical settings – Examples from current projects

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The intended functions of constructional components in (geo)technical settings often suffer from the formation of unwanted carbonate and accessory mineral precipitates. Widespread examples are chemical-sedimentary deposits (scaling) clogging deep wells, thermal water pipelines and heat exchangers in the course of hydrogeothermal energy production or impaired water flows in drainages of railway and highway tunnels.

In all of these cases disturbed natural (hydro)chemical equilibria (e.g. in the aquifer), as well as technically altered physicochemical gradients and substrate materials result in the occurrence and variable growth dynamics of unwanted scale deposits. Multi-parameter (proxy) approaches and environmental monitoring of the geotechnical infrastructure and spatiotemporally evolving precipitates target an advanced process understanding of changing fluid-solid interaction and envisage tailor-made (site-specific) countermeasures and optimization. In this context, traditional stable isotope tracers (e.g. H, C, O) and new isotope systems (e.g. clumped isotopes) are applied to fluid and solid phases based on elaborated sampling and monitoring strategies in the field (in-situ) and experimental laboratory. Stable O (δ^{18} O) and clumped isotopes (Δ_{47}) are recognized as sensitive tracers of natural and operational temperature changes and for fluid provenance tracing. Stable C and O isotopes further reflect variable outgassing of CO2 and H2O (steam) intimately coupled to variable CaCO₃ precipitation rates based on the aqueous carbonate chemical (dis)equilibrium, e.g. separated two-phase (water & gas) fluid flows in geothermal pipelines. Considering different types and locations of tunnel drainages, these stable isotope ratios capture information on varying CO2 outgassing versus CO₂ absorption from the (tunnel) atmosphere, evaporation towards complete desiccation, and possible microbial influences affecting the carbonate mineralogy, compact to porous fabrics and the scale material consistency.

Results and new insights from current research and consulting projects involving carbonate dominated scale materials from geothermal installations in Hungary and Germany, as well as from Austrian railway and highway tunnels will be presented.

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Coupled biotic-abiotic production processes and related stable isotope signatures of nitrogen trace gases

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Most N trace gas studies usually differentiate between nitrification, nitrifier denitrification, "classical" denitrification and fungal denitrification as source processes of N₂O on the basis of stable isotopic information (bulk $\delta^{15}N$, ^{15}N site preference, $\delta^{18}O$), but mostly neglect chemical source processes of N₂O, such as reactions of NO₂ with reduced metal cations, reactions of NO₂ with soil organic matter (SOM), the comproportionation reaction between NO₂⁻ and NH₂OH, and the oxidation of NH₂OH by transition metal ions. However, disentangling microbiological from purely chemical N₂O production is impeded by the fact that the chemically formed N₂O is either isotopically undiscernible from N₂O produced during nitrification, or shows an intermediate ¹⁵N site preference between that of N₂O from nitrification/fungal denitrification on the one hand, and nitrifier and classical denitrification on the other hand. Results from experiments with live and sterilized soil samples, with artificial soil mixtures and with phenolic lignin decomposition model compounds demonstrate the potential contribution of these abiotic processes to soil N trace gas emissions, but also the limitation of the information gained from the ¹⁵N site preference in the N₂O produced. This suggests that new avenues towards unambiguous N2O source partitioning are needed due to the limited explanatory power of the stable isotopic information. Furthermore, an outlook is given which role coupled bioticabiotic production processes could play in N₂ formation in soils and if the isotopic signature of this chemically produced N₂ could be used to differentiate it from biologically produced N₂.

Rainwater stable isotopes over the Iranian plateau

Stacy Carolin¹*, William Roberts², Morteza Talebian³, Richard Walker⁴, Chris Day⁴, Vasile Ersek², Gideon Henderson⁴

Rapid population growth in the arid and semi-arid regions of West Asia has placed many towns and cities on the brink of water deficiency. Accurate climate projections will assist in the development of water resource management practices needed for these areas. Iran is covered by several orographic features, which complicate efforts to model and predict future climate change. Further, Iran sits at the edge of multiple large-scale climate systems, setting the stage for substantial climate shifts in the region in response to alterations in atmospheric system dynamics. Indeed, some studies have drawn connections between the development and collapse of ancient civilizations in the Middle East and abrupt shifts in climate using the limited climate records available.

Records of rainwater isotopes assist in verifying and improving atmospheric circulation models. Understanding the large-scale mechanisms forcing rainwater oxygen isotopic variations is also an important step in interpreting paleoclimate oxygen isotope records constructed from multithousand-year-old geological samples. A large observation network of stable hydrogen and oxygen isotopes in rainwater (GNIP) was initiated by the International Atomic Energy Agency in 1958 across the globe. GNIP today involves over 1,000 meteorological stations in more than 125 countries, but certain regions, particularly Western Asia, remain greatly under-sampled.

Through a strong partnership with the Geological Survey of Iran (GSI), a year's worth of rainwater samples were collected on an event timescale at three sites: Tehran, Mashhad, and Kerman. The water samples' oxygen and deuterium isotopic ratios were analysed in Oxford University's Stable Isotope Lab. The dataset reveals preliminary information on the spatial pattern of rainwater isotopes over the Iranian plateau. I compare the results to the available Tehran monthly-resolution dataset in GNIP's database (1960-1970), and to isotope-equipped global model simulations of modern climate. I also present northern Iran speleothem oxygen isotopes coupled with global climate model precipitation-evaporation simulations. The data strongly suggests moisture loss and rainwater isotope depletion along a west to east moisture trajectory path across northern Iran 100-70 thousand years ago.

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Dawsonite from surface environment, Covasna (Romania): preliminary results

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Dawsonite is an indicator mineral of CO_2 flooding in the geological reservoir, that is the reason why it is important to know the formation parameters of this mineral. In case of a CCS (Carbon Capture and Storage) project, the safest CO_2 storage mode is when the CO_2 is trapped in the mineral phase. The final results of CO_2 flooding in a geological reservoir is that new carbonate minerals may be formed, for example dawsonite [NaAlCO₃(OH)₂]. Most papers dealing with natural CO_2 reservoirs report the presence of dawsonite and the published formation environment is restricted to the depth deeper than 200-2000 m. The formation parameters of dawsonite (temperature, pressure) and the behavior of the stable isotope fractionation – e.g. in the CO_2 -dawsonite, H_2O -dawsonite system – is yet unknown.

In this study, we investigated a dawsonite-bearing rock sample which was collected on the surface, in the Hankó creek from Covasna, Transylvania. The area is characterized by lot of CO_2 mofettas/surface emanations. Dawsonites generally appear as white fibrous minerals on the surface and between sandstone clasts. Besides dawsonite, calcite, ankerite, siderite, realgar, SiO_2 (quartz and chalcedony) and anatase were also observed. Stable H-C-O isotope compositions have been determined on the carbonate and hydroxil content of dawsonite in order determine the origin of parent fluids. The isotopic compositions indicate a dominantly magmatic origin for the ascending CO_2 that was mixed with meteoric waters infiltrating the sandstones.

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Paleotemperature reconstructions for the ~100-350 ka period inferred from D/H analyses of fluid inclusion water of speleothems

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A large stalagmite called Zeppelin (ZEP) was studied in the Baradla Cave, northeastern Hungary, whose base is a flowstone and was drilled at three locations. U-Th ages indicate that the flowstone cover the period of ~300 to 150 ka and partly overlap with the ABA flowstone (Abaliget cave southern Hungary, Koltai et al., 2017), together representing the stages of MIS9 to MIS5.

C isotope compositions are generally low in interglacials and high in glacials in the region's speleothems due to changing biological soil activity, but the MIS6 glacial is characterized by low δ^{13} C values both in the ABA and the ZEP flowstones, indicating cold and wet consitions. Sr contents measured in the ZEP-1 core support this assumption.

The ZEP-1, ABA-1 and ABA-2 cores were analysed for D/H ratios of fluid inclusion-hosted water. On the base of H isotope differences from present day drip water compositions, and using a δ D-temperature relationship of 5 ‰/°C (estimated from multidecadal GNIP data), paleotemperatures were calculated from the inclusion compositions. The MIS9 and MIS5 interglacials yielded similar temperatures to the Holocene, while the MIS7c-a period is characterized by less warm interglacial phases (~8 °C). The MIS6 end phase was either very cold, or the cold climate was associated with elevated amount of low δ D winter precipitation. The later assumption would be in agreement with the C isotope compositions and indicate a cold and wet MIS6 period.

Clumped isotope analyses were conducted as – on the basis of literature data – flowstones are expected to reflect paleotemperature, and as the Zeppelin flowstone covers large climatic changes. However, even the ~10 °C variations are obscured by kinetic fractionation processes.

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Denitrification dominates drought and post-drought dynamics of nitrous oxide emissions

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Nitrous oxide is a powerful greenhouse gas whose atmospheric growth rate has increased rapidly over the past decade. Causes of N_2O emission dynamics from terrestrial ecosystems and their responses to climate change remain poorly understood. We coupled high-resolution isotopic measurements (Picarro G5131i) with automated chambers to monitor N_2O production pathways throughout a precipitation manipulation experiment. We found that microbial functional gene abundances and soil moisture were the main drivers of variability in baseline emissions and pathways. After fertilization and, surprisingly, under drought, denitrification dominated grassland N_2O production and dynamics, contributing 70% of emissions. Drought responses of N_2O emissions were supported by a clear reversible enrichment in nitrogen-bearing organic matter compared to oxygen on the surface of soil microaggregates, shown using NanoSIMS analyses. We observed hysteresis for both total N_2O flux and denitrification contribution during rewetting, which were higher than control and drought for the same soil moisture range. These results illustrate that the magnitude of feedbacks between climate change and N_2O emission pathways is sufficient to account for increases in atmospheric growth rate in the past decade. We expect these effects will be amplified in the coming decades as climatic extremes are expected to increase in severity.

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Non-exchangeable δ^2 H of Japanese beetle, a potential tool for protecting Californian agriculture.

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Japanese Beetle (JB) *Popillia japonica* (Coleoptera: Scarabaeidae) represents a significant threat to California crop production and is among one of the world's worst plant pests due to its broad host range and its ability to defoliate through adult feeding, in addition to the damage it can cause to root systems through larval feeding.

Despite exhaustive exclusion efforts by Californian Department of Agriculture, JB are known to be accidentally introduced into California as interstate trade "hitch hikers" on commercial and private vehicles. Understanding the pathways of introduction and establishment based on stable isotope analysis would allow for implementation of better exclusion strategies, to prevent future infestations. In addition, CDFA would be able to determine if JB specimens collected within California are established populations or represent incipient infestations. These deductions are based on the isotopic signature of non-exchangeable hydrogen of the beetle, which captures the isotopic information in the precipitation water; an isotope signal which varies systematically across the US. In this research we intended to establish a stable isotope reference map for JB from target regions throughout the eastern United States and to determine whether these isotopic tools could be wielded against this problematic pest.

We collected beetles from a wide range of locations across the USA and ran the pre-incubated Japanese beetles' elytra in a temperature conversion elemental analysis (TCEA) in order to determine its $\delta^2 H$ value of non-exchangeable hydrogen. We mined the data for the $\delta^2 H$ of precipitation for all sites where the beetles were collected using the Online Isotopes in Precipitation Calculator (OIPC). We compare the average $\delta^2 H$ of precipitation between April and October, when beetles are active, with $\delta^2 H$ of the beetles.

Standard deviation of non-exchangeable δ^2H for beetle samples collected in the same area ranged from 5-6 % suggesting that differences of 10 % could be reliably distinguished.

Excluding the values from Colorado as they appeared not to fit the model (flagging them as possible incipient populations), regression analysis revealed there was a highly significant (p<0.001) and predictive (r^2 =0.510 relationship between) latitude and non-exchangeable hydrogen values of the beetle elytra in line with our predictions. Multiple regression improved predictability, when both elevation and longitude were taken into the model.

We used a random training set of 25% of the sample to get an initial beetle to precipitation (OPIC) specific correlation. The comparison between δ^2H of beetles and average of the local precipitation during the life cycle months (from April to October) showed a good correlation (R²= 0.724). That correlation was slightly better than when comparing δ^2H of beetles with δ^2H of mean annual precipitation (R²= 0.709). Using these tools, it should be possible to predict and validate specific entry pathways of JB into California.

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2 Years of Stable Isotopes Measurements of Rivers and Springs in Southern Salzburg Basin (Golling, Kuchl, Hallein) Salzburg, Austria

Giorgio Höfer-Öllinger^{1*}, Kathrin Müggenburg¹

Between September of 2017 and September of 2019, at the rivers Salzach, Alm, Spumbach, Taugl, Lammer, Torren, Fischbach, Schwarzenbach and Weißenbach and at the Schwarze Torren spring, a weekly monitoring campaign for delta ¹⁸O, ¹⁷O and ²H was carried out. Schwarzenbach corresponds to Golling Waterfall spring. The measurements were accompaigned by in situ parameters and sampling of important wells, including Taugl well, Vigaun well and Gamp 1 and 2 wells. As comparison, the runoff of the Königssee in Berchtesgaden, Germany, was measured, as well.

The very strong dataset of more than 1000 individual measurements is subject to ongoing profound interpretations. However, right now important statistics and very first interpretations can and shall be presented ad the SINA conference and be subject to discussion:

- The optained dataset shows a very clear image of instantaneous transport, depicted by proximal flow systems like Schwarze Torren and Fischbach. At these catchments, the main retardation factor is snow in winter season.
- During springtime, depleated water starts in melting period, mostly early to mid of April. Water from snow melting is substituted consecuently from May to July.
- Given a high density of data, the average D excess figures out as typical character of a catchment (values between 9 and 13), whereous the ¹⁷O excess, in average, almost everywhere plots around 0.
- Alpine lakes don't show evaporation effects.
- Salzach river shows an expected allogene catchment area without significant local contribution.
- Groundwater in Gamp wells, meanwhile, show an almost horizontal time series plot of all isotopes, depicting old water (≥ 5 years). There is no evidence of influence of seasons and therefore, every influencing parameter of seasonal recharge can be excluded.
- As a first result the average age of groundwater at Hagengebirge's main spring, Schwarze Torren, was calculated. The result is 0,4 years (average) and can be explained by its snow cover in winter season, residuum soil deposits in its vallies and in the epikarst.

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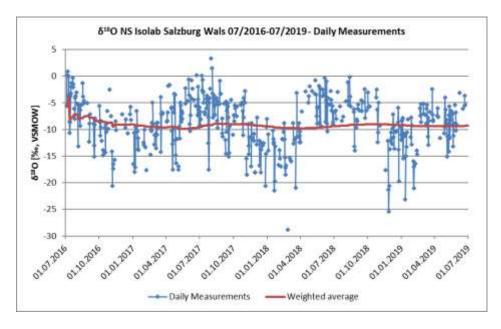
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Stable Isotopes of Daily Precipitation Sampling: 3 Years of Observation at Wals and Sonnblick Observatorium Salzburg, Austria

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In summer of 2016 a campaign of daily sampling of precipitation started in Wals/Salzburg and in autumn of the same year at Sonnblick Observatorium. Since then, every precipitation event > 0.5 mm was analysed for its content of 18 O, 17 O and 2 H; exceedingly now a 3 years period of observation with more than 1000 single analyses.

These daily analyses, later, are weighted and the weighted average over months, years and the entire observation period allows a comparison with measurements of the ANIP network. The values are computed absolutely and in relations among them.



The first relation is the 18 O against 2 H in order to depict the local meteoric water line and compare it to the GMWL (global meteoric water line). All calculations and statistics include the deuterium excess and the 17 O excess.

As consequence, the obtained dataset not only allows a comparison among itself but with other datasets as well: Precipitation origin, temperatures and moisture; as well as isotope data from other precipitation stations.

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Honey water: an easy solution to detect sugar syrup addition?

Horváth, B.1

Honey is one of the most often adulterated foods according to the EU Food Fraud Network. There is plethora of counterfeit honey, due to adulteration by added sugars, botanical or geographical origin. Especially challenging is to prove addition of sugar syrups. The easiest case is when C3 plant based honey is blended with the high amount (min 7%) of C4 sugars. This method is based on δ^{13} C measurement of sugars and proteins in honey (AOAC998.12). Blending honey with C3 sugars is much more difficult to detect. The published LC/EA-IRMS method is based on the comparison of the δ^{13} C of honey protein vs different sugar fractions (individual sugars). This method is relatively easy to fulfill for but not selective enough. One relatively efficient method to detect beet syrup (C3) known today is the site specific determination of the deuterium/hydrogen ratio of the honey sugar with SNIF-NMR. This method is elaborative as the honey must be fermented to alcohol and expensive because of the instrumentation. This latter problem is addressed by the EIM-IRMS method which measures the isotope ratio of the non-exchangeable hydrogen in sugars. This method however is still in development.

In this paper a completely different approach was used. Instead of concentrating on the sugar itself, the water part of the honey was analyzed. The idea is that authentic honey must have specific water fingerprint, which is different from meteoric water which is used by industrial processing of sugar containing plants. The preliminary results show that most honey waters are enriched in hydrogen and oxygen isotopes in contrast to sugar syrups. However there are exceptions in both honey and syrups, thus it was not possible to establish threshold values neither for oxygen nor for hydrogen isotope ratios. Nonetheless there was remarkably stable correlation between hydrogen and oxygen isotope ratios in honey water which was different from the meteoric water line. Using this correlation (honey water line) it was possible to distinguish known adulterated honeys from the authentic ones. Further investigations must prove the reliability of this approach.

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Drought and recovery effects on belowground respiration dynamics of recent carbon in managed and abandoned grassland

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The supply of soil respiration with recent photoassimilates is an important and fast pathway for respiratory loss of carbon (C). To date it is unknown how drought and land-use change interactively influence the dynamics of recent C in soil respired CO₂ in grassland. In an in situ common-garden experiment, we exposed soil-vegetation monoliths from a managed and an abandoned grassland to an experimental drought. Using two ¹³CO₂ pulse labelings, we traced recently assimilated C in soil respiration during drought, rewetting and early recovery. Independent of land use, drought reduced the allocation of recent C to soil respiration. Rewetting triggered a rapid and significant loss of recent C through a pulse of high respiration. Afterwards, respiratory dynamics of recent C were quickly restored. While abandonment shifted the belowground partitioning of recent C from root storage towards belowground metabolic activity under ample water supply, this land-use effect was reversed under drought. We conclude that drought effects override effects of land-use change on the dynamics of respiratory utilization of recent C, and that abandonment of grassland management can alter the preferential allocation and thus the belowground partitioning of recent C to storage versus metabolic use. Our results demonstrate interactive effects of drought and land-use change on belowground C dynamics in grassland and highlight trade-offs of the processes underlying grassland resilience to drought.

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Laser-spectroscopic analysis of CO₂ clumped isotopes

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The abundance variation of the doubly substituted $^{13}C^{16}O^{18}O$ molecule in CO_2 and carbonates (mass spectrometrically quantified as Δ_{47}) has become an exciting new tool for research in Earth Sciences, namely related to paleothermometry and reconstruction of mineral formation conditions. Sample amount, achievable precision, isobaric interferences and measurement time constraints are currently limiting the full exploitation of this new tool. Laser spectroscopic analysis has the potential to provide complete isotope information ($\delta^{13}C$, $\delta^{18}O$, $\delta^{17}O$, clumped Δ_{368} , Δ_{288} , ...) simultaneously with one measurement setup. A pilot laser-based CO_2 isotopologue analyzer has been built with capability for rapid analysis and simplified sample preparation. An important advantage of the new instrument design is that it can unambiguously measure all isotopologues. The current measurement precision for clumped Δ_{368} is better than 50 ppm within about 60 to 90 minutes.

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Stable isotope studies (δ^2 H, δ^{18} O) of precipitation and soil water movement in forested ecosystems at Solling, Germany

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Two-year time series of stable isotopes (δ^{18} O and δ^2 H) in precipitation and soil water were collected at European beech and Norway spruce ecosystems at Solling, in Northwest Germany. The aim of the isotope study is a better understanding of soil water movement and groundwater recharge at the long-term ecosystem monitoring sites [1]. Precipitation samples from collectors at an open, beech and spruce forest site, were taken as bi-weekly totals. Soil water samples were collected in bi-weekly totals from suction lysimeters installed at four different depths at the beech and the spruce site, whenever soil water was available. All samples were analyzed for stable isotope composition at the Federal Institute of Geosciences and Natural Resources (BGR) in Hannover, using a Picarro L2120-i water vapor analyzer with long-term accuracies for quality check samples better than 0.2% and 0.8% for δ^{18} O and δ^{2} H, respectively. A discussion of the two-year precipitation and soil water series (about 500 samples) will be presented. Besides general patterns (seasonality and trends) we also focus on importance of amount weighing procedures, corrections for minor rain amounts and dry season, and aspects of sample storage.

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Calcium isotope record of the Late Triassic

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The Ca isotope composition ($\delta^{44}/^{40}$ Ca) of Late Triassic – earliest Hettangian bulk carbonates from Austria were analyzed using TIMS and double-spike technique. Throughout the Norian the $\delta^{44/40}$ Ca_{carb} values exhibit a slight increase. Afterwards the most characteristic change of the dataset follows: an abrupt drop of ~0.5 % at the beginning of the Rhaetian. This negative shift is followed by a decreasing trend until the end of the Rhaetian. A rebound starts in the basal Hettangian. This dataset fills a long $\delta^{44/40}$ Ca data gap in the Triassic and earliest Jurassic. Due to the low amount of data during the Lower Jurassic, the question remains open when the $\delta^{44/40}$ Ca_{carb} values return to Norian level, but the negative excursion lasted at least 4 Ma.

Magnitude changes of the input fluxes alone are insufficient to explain this duration. Furthermore there is no indication of globally switching calcite vs. aragonite sea, which could theorethically also serve as an explanation. Sr concentration and $\delta^{44/40}\text{Ca}_{\text{carb}}$ values indicate a hyperbolic trend. Based on this relation, local primary mineralogy differences can partly explain the Rhaetian negative shift. Sections with originally higher aragonite content tend to have lower $\delta^{44/40}\text{Ca}$ signature. To detect a secular change and distinguish the significance of global vs. local factors further measurements are required from different peleogeographic and paleoenvironmental settings.

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Continental CH₄ concentration and ¹³C/¹²C isotope ratio measurements in the Carpathian Basin, Hegyhátsál: comparison with European clean air sites

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The Hungarian rural tall tower station, Hegyhátsál is widely used in long-term atmospheric greenhouse gas monitoring measurements. As the former CO₂ concentration and isotope measurements proved, it is an ideal place to collect background atmospheric air samples. Background ambient air samples were collected between 2013 and 2016, 4 bags per month at the Hungarian site for methane (CH₄) mole fraction and $\delta^{13}C_{CH_4}$ measurements. The results from the Carpathian Basin were compared with two NOAA atmospheric monitoring site, Mace Head and Zeppelin to determine the excess continental CH₄ level and isotopic shift. The measurement data was used coupled with back trajectory modelling and concentration weighted trajectories (CWT) to detect potential CH₄ source areas. The Hungarian station can be separated from the coastal and polar area based on the concentration results, that have higher seasonal amplitude and higher maxima (from 1876 to 2119 ppb), but the $\delta^{13}C_{CH4}$ results match with the NOAA stations, the mean $\delta^{13}C$ ratio is -47.55 ‰, except in a short period, when an intense negative isotopic shift was detected. The trajectory modelling reveal numerous potential source areas what influence the CH_4 level and its $\delta^{13}C$ signal in the Carpathian Basin. Our study presents the first, supplementary $\delta^{13}C_{\text{CH4}}$ dataset in the Carpathian Basin, what shows the usefulness of the Hungarian measurement site in the greenhouse gas monitoring. The measurement and modelling campaign also illustrates that the monitoring data coupled with trajectory modelling is a powerful tool for the detection of potential source areas.

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Estimation of the natural variability of the tritium concentration of precipitation

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Tritium of the precipitation plays a very important role in the study of the waters of our planet because its tracer property is suitable for a better understanding of atmospheric and hydrological processes. Since the atmospheric test-ban treaty, the bomb tritium concentrations in precipitation have significantly reduced. Therefore, in the last two decades, global tritium concentration of precipitation (including anthropogenic and natural sources) have nearly reached a steady state level. The aim of this study is to explore the variability of natural tritium in the past by using the neutron monitor time series, which represents the variability of solar activity.

Many previous studies focused on the reconstruction of the bomb-tritium time series, taking into account the long tritium time series of some cities (Ottawa, Vienna, Tokyo). In our study, we primarily estimate the tritium of natural origin in the northern and southern hemispheres, which may be an important input parameter for atmospheric and hydrological modeling. Therefore, we been have developed a statistical model to estimate short and long term changes in natural tritium. This estimate is based on the relationship between the global tritium time series and the neutron flux. The statistical model provides a relatively accurate estimate for stations that are poor in technogenic sources of tritium since time series values closely follow seasonal and long-term changes. Whereas for time series that are richer in technogenic tritium, the estimation is less accurate because the tritium values of the time series do not change regularly, that is, they do not follow the natural change.

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Effect of summer drought on the coupling of photosynthesis and soil respiration under current and future climate

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Photosynthesis and soil respiration are the two largest fluxes of CO_2 in terrestrial ecosystems, and are immediately linked through the fast belowground translocation of recently assimilated carbon. Climate extremes such as drought can exert a major impact on the ecosystem carbon balance by altering carbon uptake, release processes and carbon allocation, with consequences for the coupling of photosynthesis and soil respiration. It is unclear how drought effects are altered by a future warmer climate under elevated atmospheric CO_2 , where positive effects of elevated CO_2 on photosynthesis and soil moisture could be counterbalanced by warming effects on evaporative demand.

ClimGrass is a comprehensive experimental facility with 54 plots for studying multilevel and multifactor interactions of elevated CO_2 , warming and drought on managed C3 grassland. Here, we focused on a subset of plots subjected to ambient precipitation or drought, both under current environmental conditions or a future climate (+3°C, + 300 pm CO_2). To trace the fate of recent carbon from photosynthesis to soil respiration we performed two replicated $^{13}CO_2$ pulse-labeling experiments during peak drought and recovery.

During drought, plant carbon assimilation, soil respiration and ¹³C respired per amount of C assimilated (rel. ¹³C) were reduced. The impact of drought was stronger under future climate conditions. During the recovery period, the soil respiration dynamics did not differ between current and future climate conditions, however, assimilation and rel. ¹³C respired recovered more rapidly in a future climate. We conclude that under future climate conditions, drought resistance of soil respiration and its use of recent C decreases, while drought recovery of assimilation and the coupling of assimilation and soil respiration are accelerated.

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Ten Years of Elementar and IsoPrime: Pioneers in simultaneous CNS isotope ratio analysis

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In 2008 Elementar and IsoPrime (now Elementar UK) joined forces and consolidated their expertise in elemental analyser inlets and stable isotope analysis of organic elements. This unique partnership lead to unprecedented technological advances such as the first simultaneous analysis of carbon, nitrogen and sulphur isotope ratios. Here, we briefly review the working principle of Elementar's CNS isotope analysis setup and present key papers using this method.

Samples enter the elemental analyser (EA) inlet via an autosampler and a He-purged ball valve, which guarantees blank-free sample introduction. Subsequently, organic sample components are quantitatively converted to a homogenous gas mixture of CO₂, N₂, and SO₂ within a high temperature furnace. CO₂ and SO₂ gases are retained by adsorption columns, while N₂ passes through. Trapped gases can be released individually and in a pulse-like fashion by heating up the respective adsorption column. This technology guarantees complete baseline separation of C, N and S peaks even for extreme elemental ratios without isotopic fractionation and is capable of handling high elemental concentrations (up to 20mg C). Furthermore, it allows for pulse-like injection of each individual sample gas into the isotope ratio mass spectrometer (IRMS), which results in narrower and taller peaks compared to isothermal GC-based systems. Accordingly, we observe an excellent signal to noise ratio and exceptional instrument sensitivities.

For the analysis of substances with a very low S or N content, the sample weight can easily be increased. When the sample weight is increased, the major carbon isotope will reach very high concentrations. To avoid amplifier saturation, our instruments feature a 100V amplifier and a fully automated sample gas dilution system.

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Stable isotope-based derivation of a conceptual hydrogeological model of the Vögelsberg landslide (Tyrol, Austria)

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Continuously creeping deep-seated gravitational slope deformations (DSGSDs) pose a serious threat to settlements, infrastructure and economic well-being in mountainous areas. Their spatio-temporal pattern of movement is typically controlled by pore-pressure variations which generally correlate with groundwater level fluctuations. Conceptual hydrogeological models deliver essential information about source areas and flow paths of water causing these fluctuations. They provide constraints on the slope's hydrological parameters along a 2D profile and serve as fundamental input for numerical models aiming at identifying mitigation measures in order to reduce the landslide's velocity. We present results of a hydrogeological study of the Vögelsberg DSGSD located in the Innsbrucker Quartzphyllite complex of the central Eastern Alps (Tyrol, Austria). Monthly measurements of discharge, temperature, electrical conductivity and stable water isotopes of 80 springs and two groundwater wells between autumn 2018 and autumn 2019 were combined with field mapping and precipitation sampling at elevations between 880 and 1980 m a.s.l. The aim of these investigations was to (i) estimate the mean recharge area of each spring, (ii) determine the fraction of winter and summer precipitation of spring recharge, (iii) classify springs according to different aquifer types, and (iv) combine this information to derive a conceptual hydrogeological model. Preliminary results covering eight months of observation and using a literature-compiled δ^{18} O-elevation gradient of -0.15 % per 100 m for precipitation show that the spring water infiltrates between 1200 to 1680 m a.s.l. Generally increasing $\delta^{18}O$ values with increasing elevation of the springs indicate that water emerging at lower elevation must have recharge areas at higher elevation compared to water emerging at the midslope. Winter precipitation dominates the annual groundwater recharge budget. Ongoing and future work includes water balance calculations and numerical groundwater modelling based on the derived conceptual model to better understand the cause of landslide acceleration phases. Further measurements will be carried out to extend the observation period for obtaining e.g. robust transit time estimates.

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Simulating bypass flow in a two water worlds context

Radolinski, Jesse¹ et al.

Ecohydrological separation has been observed across climates and biomes, suggesting that mobile infiltrating water resists mixing with more tightly-bound and plant available water for extended periods of time; however little mechanistic evidence exists to explain this separation at a process scale. Non-equilibrium flow in the vadose zone may partially account for widespread perception of distinct hydrological domains yet no studies have weighed its contribution. Using a simple stable isotope mixing technique, we sought to determine the amount of preferential flow necessary to maintain a two water worlds scenario (i.e., physical separation between mobile and immobile water pools). We constructed 60 cm soil columns (20 cm-ID PVC) containing low soil structure (sieved soil material), subsoil structure (intact B horizon), and soil structure without matrix exchange (tubing reinforced macropores) to simulate multiple preferential flow scenarios. Columns were subjected to 3 rain storms of varying rainfall intensity (~2.5 cm h ⁻¹, ~5 cm h ⁻¹, and ~11 cm h ⁻¹) whose stable isotope signatures oscillated around known baseline values. Isotopic analysis was performed on collected leachate and matrix water sampled via direct vapor equilibration. Soil matrix water signatures indicate up to 100% mixing with infiltrating rain water under low rainfall intensity (2.5 cm h ⁻¹) in subsoil structure columns, whereas high intensity rain (11 cm h ⁻¹) produced separation between columns with intact or artificial soil structure and those controlled for structure (low structure treatment). These preliminary data suggest that distinct flow-domain separation may only be possible under extreme precipitation intensity.

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Speleothem-based isotopic variation during the Holocene period in southern Iran

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Iran's average annual precipitation is less than a third of the world's average. Some studies on lakes, marine sediments and caves have suggested that Iran has experienced wetter periods in the past. Among all proxies, stalagmites are a prominent archive in paleoclimate science, as they reveal paleoenvironmental and paleo-climatological information with a high-precision age model which covers a long range of events and terrestrial sedimentation. We have recently visited several caves in southern and central Iran and collected 51 cave sediment samples in total. Our collection includes stalagmites, flowstone, cave corals and drill cores. We have used the ²³⁰Th dating method to determine when the samples initially started precipitating, and when growth terminated. The samples show that southern Iran stalagmite growth occurred within the past 600 thousand years, during both glacial and interglacial periods. Here we present our initial oxygen isotopic data of the cave samples that grew in southern Iran during the Holocene. All samples have an oxygen isotopic ratio that ranges between -2‰ to -6‰. We compare these values with the oxygen isotopic values of regional modern precipitation as well as the average values of other regional stalagmites, including samples from Oman, the Eastern Mediterranean, and northern Iran.

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Stable isotopes in cave-ice, a link to past precipitation?

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Alpine ice caves preserve overlooked and largely untapped archives, which formed during several centuries to millennia Before Present. Firn-derived ice in caves forms through the metamorphism of in-falling snow, contributing positively to the mass of underground glaciers. Vertical (sag-type) pits are ideally suited to this type of ice build-up and may provide a complementary record of historical climate in the Alps, with respect to well-established proxies, in particular decadal to centennial trends of water stable isotopes in precipitation. Liquid precipitation at the cave entrance or through the rock overburden may alter the isotopic composition of the firn-derived ice by infiltrating and refreezing inside the ice-body. Poor constraints also exist concerning the stable isotopic evolution of the near-cave snow pack prior to its mechanical transport into the cave or its thawing and contribution as karst drip-water. We therefore present stable isotopes results from transects of modern ice in two Austrian caves and compare them to regional records of the Austrian Network of Isotopes in Precipitation in order to evaluate the faithfulness and strength of this underground archive.

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Oxygen isotope equilibration of phosphate in the soil /plant system – an ¹⁸O-PO⁴ organic fertilizer test

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In natural ecosystems the phosphorus (P) cycle is "tight", which means transfer between soil and biota is efficient and little P is lost from the system. However, the increasing human P input via fertilizers from rock phosphate has increased losses of phosphate from the terrestrial system into freshwater systems, leading to eutrophication issues. Targeting PO_4 by stable isotopic methods better enables us to define the origin of P sources and hence improve mitigation measures. However, during PO_4 uptake and cycling in cells the original isotope oxygen signature equilibrates, which complicates identification of the source.

We monitored the extent of alterations of the oxygen isotope signature and hence defined the involvement of soil microbial processes in the desorption and transport of phosphate derived from an organic P fertilizer. The organic fertilizer was a biochar produced by pyrolysing apricot kernels, surface-modified with Mg. In a pot experiment, a modified Neubauer test, around 11 mg 18 O enriched PO4 with a δ^{18} O value of 302 % was bound onto one gram of biochar. We mixed 5 % of biochar (w/w) with a P deficient soil and planted rye. The alteration of the oxygen signature in the available (anion exchange membrane AEM bound), the HCl extractable PO4 soil pools and the available (TCA extractable) PO4 in plants were determined after 17 days.

The amount of AEM and plant extractable PO_4 was too little to achieve measurements by TC/EA-IRMS, while HCl extractable PO_4 was sufficient for measurements. The $\delta^{18}O$ of PO_4 extracted from biochar amended soil was 60.9 ‰, which was elevated in contrast to non ^{18}O -PO $_4$ biochar amended soil (18.1 ‰), but much less than assumed after mass balance calculation (260 ‰). Additionally, the increase in the amount of HCl extractable and purified PO_4 was only 4 % of the expected increase. To conclude, the ^{18}O -PO $_4$ added to the biochar was not sorbed or was not released by HCl extraction. After the 17 days lasting pot experiment, we expected a decrease of the amount and $\delta^{18}O$ values of HCl extractable PO_4 due to biological ^{18}O equilibration processes and plant uptake of biochar derived PO_4 . However, the $\delta^{18}O$ values of HCl extractable PO_4 had increased by 10 ‰ up to 71.2 ‰. HCl extractable PO_4 has not equilibrated due to microbial turnover. Furthermore, the HCl extractable and purified amount of PO_4 between the soil and the soil + biochar treatment was constant. This might indicate that plants have preferably taken up soil born PO_4 . Apparently the PO_4 attached to the biochar was not readily available for microorganisms and plants.

Additionally, we conducted an interlab comparison test with a Ag_3PO_4 BOKU standard including 4 well know international laboratories to account for the absence of certified standards for ¹⁸O measurements of Ag_3PO_4 . In total, 131 measurements determined the Ag_3PO_4 BOKU standard to be 13.9 ‰ \pm 0.4 ‰.

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Climate and land use as important drivers of N dynamics at a continental scale in Europe

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Nitrogen is a limiting element on primary productivity across global terrestrial ecosystems. Measuring the natural abundance of N isotope (15N: 14N) in plant and soil N pools is a useful tool to assess N cycling dynamics in natural and managed ecosystems, however, it is still not clear about the major factors controlling the variability of $\delta^{15}N$ values at a large scale. In this study, we sampled mineral soils (0-15 cm) along a Europe transect spanning from the Mediterranean to the Subarctic and measured the $\delta^{15}N$ values of a range of plant and soil N pools, including plant roots, foliar litter, bulk soil N, high-molecular-weight organic N, microbial biomass N and inorganic N. We found the $\delta^{15}N$ values of plant (roots and litter) and bulk soil N were constant and high in the managed cropland and grassland ecosystems while $\delta^{15}N$ values of bulk soil increased with increasing latitude from 2.0% to 4.3% in forest ecosystems, suggesting potential N losses causing ¹⁵N enrichment in soils at high latitudes. Furthermore, bulk soil $\delta^{15}N$ values were correlated with nitrate $\delta^{15}N$ values in cropland and forest soils, indicating denitrification as an important N loss pathway in both N-rich and N-poor ecosystems. Compared to δ^{15} N values of plant N, δ^{15} N values of inorganic N were more affected by climate and latitude in managed and natural ecosystems. Ammonium were more ¹⁵N enriched than nitrate in warm regions due to ammonia volatilization and active nitrification while ammonium became ¹⁵N depleted along latitude, being close to or smaller than nitrate δ^{15} N values in cold regions due to less N was released by microbes via N mineralization and provided for nitrifiers. Therefore, microbial decomposers and plant became limited by N along latitude due to climate factors and $\delta^{15}N$ values of soil N were more efficient in revealing ecosystem N status than that of plant.

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