# SINA - Stable Isotope Network Austria

## 15th Stable Isotope Network Meeting

University of Vienna, Austria

24th & 25th November 2017

# Programme

**VENUE:**
University of Vienna
Lecture room 2, UZA 1
Althanstraße 14
1090 Vienna

# 15th Stable Isotope Network Meeting

## University of Vienna, 24th and 25th November 2017

## Scientific Organizing Committee

Wolfgang Wanek [1]
Andreas Richter [1]
Martin Kralik [2]
Jan Wiederhold [2]

[1] Department of Microbiology and Ecosystem Science, University of Vienna

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# PROGRAMME

## FRIDAY 24th NOVEMBER 2017

| | |
|---|---|
| 12:30 – 12:55 | Registration |
| 12:55 – 13:00 | Welcome & Introduction (**Wolfgang Wanek**) |
| **SESSION 1** | **Chair: Christoph Spötl** |
| 13:00 – 13:45 | *Key Note Address*<br>**Thomas Hofstetter** (Eawag, Swiss Federal Institute of Aquatic Science and Technology, Dübendorf, Switzerland)<br>New challenges for the stable isotope analysis of organic pollutant biodegradation |
| 13:45 – 14:05 | **Jan Wiederhold** (Dept. of Environmental Geosciences, University of Vienna, Austria)<br>Tracing sources and fate of mercury in soils and groundwater at industrial contamination sites with stable mercury isotopes |
| 14:05 – 14:25 | **Christin Müller** (Helmholtz Center for Environmental Research, Department Catchment Hydrology, Halle, Germany)<br>Tomography of anthropogenic nitrate contribution along mesoscale rivers in two European catchments |
| 14:25 – 14:45 | **György Czuppon** (Institute for Geological and Geochemical Institute, Hungarian Academy of Sciences, Budapest, Hungary)<br>Relationship between the air moisture source and the stable isotope composition of the precipitation in Hungary |
| 14:45 – 15:05 | **Stefan Wyhlidal** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)<br>Temporal and spatial distribution of isotopes in river water in Austria |
| 15:05 – 15:40 | **Coffee and Tea Break** |
| **SESSION 2** | **Chair: Jan Wiederhold** |
| 15:40 – 16:00 | **Christoph Spötl** (Institute of Geology, University of Innsbruck, Innsbruck, Austria)<br>Stable isotopes reveal the origin of caves |
| 16:00 – 16:20 | **Attila Demény** (Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Hungarian Academy of Sciences, Budapest, Hungary)<br>Middle Bronze Age humidity and temperature variations and societal changes in East-Central Europe |
| 16:20 – 16:40 | **Anna-Lena Grauel** (Godwin Laboratory for Palaeoclimate Research, Department of Earth Sciences, University of Cambridge, Cambridge, United Kingdom)<br>Magnitude of temperature change in the lowland Neotropics during the last Glacial |
| 16:40 – 17:00 | **Kathleen A. Wendt** (Institute of Geology, University of Innsbruck, Innsbruck, Austria)<br>775,000 years of climate history from the southwest USA: revamping the famous Devils Hole cave record |
| 17:00 – 17:20 | **Zoltán Kern** (Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Hungarian Academy of Sciences, Budapest, Hungary)<br>Stable isotope signatures in tree rings of Swiss stone pine (*Pinus cembra* L.) in Romania – annually resolved summer proxy archive for the past 600 years |
| 17:20 – 19:00 | **Poster session 1** and **Stable isotope laboratory tours** |
| from 19:00 | **Dinner** at Brandauers Bierbögen, Heiligenstädter Straße 31, 1190 Vienna |

## SATURDAY 25th NOVEMBER 2017

| | |
|---|---|
| **SESSION 3** | **Chair: Albrecht Leis** |
| 09:00 – 09:45 | *Key Note Address*<br>**Rolf Siegwolf** (Paul Scherrer Institute, Villigen, Switzerland)<br>Trees in a changing environment: Visualizing their responses with stable isotopes |
| 09:45 – 10:05 | **Patrick Meister** (Dept. for Geodynamics and Sedimentology, University of Vienna, Austria)<br>Carbon isotope fractionation during methanogenesis in marine sediments: An evaluation by reactive-transport modelling |
| 10:05 – 10:25 | **Micha Horacek** (HBLFA Francisco-Josephinum BLT, Wieselburg, Austria)<br>Identification of ivory with respect to animal species by stable isotopes |
| 10:25 – 11:00 | **Coffee and Tea Break** |
| 11:00 – 11:20 | **Danijela Smajgl** (GEOMAR Helmholtz Centre for Ocean Research Kiel, Kiel, Germany)<br>New analytical approach in monitoring of $CO_2$ cycle in aquatic ecosystems |
| 11:20 – 11:40 | **Filip Volders** (Elementar Analysensysteme GmbH, Langenselbold, Germany)<br>An LC-IRMS Interface for Flexible Compound-specific Stable Isotope Analysis |
| 11:40 – 12:00 | **Oliver Schlegel** (Air Liquide Austria GmbH, Schwechat, Austria)<br>Improving Measurement Reliability of Isotope Ratios |
| 12:00 – 14:00 | **Lunch** (on-site lunch is included in the registration fee!) and **Poster session 2** |
| **SESSION 4** | **Chair: Stefan Wyhlidal** |
| 14:00 – 14:45 | *Key Note Address*<br>**Michaela Dippold** (Dept. of Biogeochemistry of Agroecosystems, Georg-August University Göttingen, Germany)<br>From position-specific labeling to quantitative soil fluxomics - new dimensions in terrestrial biogeochemistry |
| 14:45 – 15:05 | **Yuntao Hu** (Dept. of Microbiology and Ecosystem Science, University of Vienna, Austria)<br>Multiple isotope tracing reveals the transformation processes of oligopeptides in soils |
| 15:05 – 15:25 | **Zsofia Kovacs** (Institute of Earth Sciences, University of Graz, Austria)<br>The influence of pelagic calcification on the Late Triassic seawater chemistry |
| 15:25 – 15:45 | **Lukasz Pytlak** (Applied Geosciences and Geophysics, University of Leoben, Austria)<br>Light Hydrocarbon Geochemistry of Oils in the Alpine Foreland Basin: Impact of Geothermal Fluids on the Petroleum System |
| 15:45 – 16:05 | **Magda Mandic** (Thermo Fisher Scientific, Bremen, Germany)<br>Studying various processes with $\delta^{13}C$ and $\delta^{18}O$ |
| 16:05 – 16:30 | **Coffee and Tea Break** |
| 16:30 – | **Generalversammlung SINA** - Verein für Stabile Isotopen Forschung<br>(General assembly of the Stable Isotope Network Austria – SINA.<br>Seminar room 'Ecology', UZA 1, Althanstr. 14, A-1090 Vienna) |

## POSTER SESSION 1 Friday 24th November

17:20 – 19:00

P1: **Cornelia Wilske** (Dept. of Catchment Hydrology, Helmholtz Centre for Environmental Research (UFZ), Halle/Saale, Germany)
Behavior of stable sulfate isotopes in a coupled Cretaceous and Quaternary aquifer system, Lower Jordan Valley

P2: **Katharina Schott** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
Isotope patterns of water at different time scales in the Hydrological Open Air Laboratory (HOAL) Petzenkirchen

P3: **Martin Kralik** (Dept. of Environmental Geosciences, University of Vienna, Austria)
Tracing stable isotopes ($\delta^2$H and $\delta^{18}$O) from meteoric water to spring-groundwater in small catchments of the Vienna Woods, Vienna, Austria

P4: **Stefan Wyhlidal** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
The Austrian Network of Isotopes in Precipitation (ANIP) and its application in hydrology

P5: **István Gábor Hatvani** (Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Hungarian Academy of Sciences, Budapest, Hungary)
Resampling of sedimentary proxy records to evenly spaced time-series using spectral control on the example of speleothem stable isotope records from Europe

P6: **György Czuppon** (Institute for Geological and Geochemical Research, Hungarian Academy of Sciences, Budapest, Hungary)
Multi-annual monitoring in the Béke and Baradla caves (NE Hungary): implications for the conditions for the formation cave carbonates

P7: **Izabela Bujak** (Dept. of Catchment Hydrology, Helmholtz Centre for Environmental Research (UFZ), Halle/Saale, Germany)
Multi-isotope approach to evaluate sources and fate of nitrogen in the Erlauf River catchment in Austria

P8: **Vera Winde** (LUBW, Institute for Lake Research, Langenargen, Germany)
Rhine river plume tracking with stable hydrogen and oxygen isotopes in Lake Constance

P9: **Giorgio Höfer-Öllinger** (Georesearch Forschungsgesellschaft mbH, Wals, Austria)
Stable Isotopes in Precipitation: A Case Study on Daily Analyses at Sonnblick Observatory (3.106m) and in Wals Isolab (446m), Salzburg, Austria

P10: **Máté Karlik** (Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Hungarian Academy of Sciences, Budapest, Hungary)
Natural and anthropogenic changes in a lake-forest system in South Bukovina based on stable C and N isotope ratios and lipid biomarkers since 1340 A.D.

P11: **Zoltán Kern** (Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, Hungarian Academy of Sciences, Budapest, Hungary)
Establishment of the lab protocol for simultaneous determination of stable isotope composition of carbon, oxygen, and non-exchangeable hydrogen in cellulose samples using a heated vacuum-equilibration device

## POSTER SESSION 2 Saturday 25th November

12:00 – 14:00

P12: **Kathrin Schmittner** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
Impacts of nitrogen deposition on forest biogeochemical processes across a trans-European gradient investigated using a tool kit of stable isotope methods

P13: **Elisabeth Ziss** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
Biochar: NET-2-U. Biochar: Negative Emission Technologies are nice to the environment - Using stable isotopes in a citizen science approach

P14: **Andrea Watzinger** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
Consequences of climate change for agroecosystem carbon and nitrogen cycling – an isotope labelling of green manure approach

P15: **Andrea Watzinger** (AIT – Austrian Institute of Technology GmbH, Tulln, Austria)
Determination of carbon isotope enrichment factors of metabolites after precursor amendment-illustrated by the example of chlorinated ethene degradation

P16: **Nora Fricko** (TU Wien, Institute for Water Quality, Resource and Waste Management, Vienna, Austria)
Project MINTOx – Microbial nitrogen transformation processes during waste degradation

P17: **Micha Horacek** (HBLFA Francisco-Josephinum BLT, Wieselburg, Austria)
Imported or already locally native? Stable isotope investigation of invasive tree pests can differentiate between pests grown in local trees (Austria) from pests grown elsewhere: a pilot study of *Monochamus*

P18: **Ingrid Rabitsch** (Dept. of Forestry and Soil Science, University of Natural Resources and Life Sciences, Vienna, Austria)
Impacts of habitat changes on food availability for the Warbler finch in the Scalesia Forest, Galápagos

P19: **Jan Wiederhold** (Dept. of Environmental Geosciences, University of Vienna, Austria)
Tracing Hg transformations in contaminated soil using Hg isotopes

P20: **Jan Wiederhold** (Dept. of Environmental Geosciences, University of Vienna, Austria)
Hg isotope analysis and sequential extractions of industrially contaminated soils in Valais, Switzerland

# New challenges for the stable isotope analysis of organic pollutant biodegradation

Thomas B. Hofstetter[1,2]

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## Abstract

Enzyme-catalyzed oxygenations are among the most important biodegradation and detoxification reactions of organic pollutants in contaminated soil and water. Because such processes typically take place over timescales of decades and lead to products that are difficult to detect, it is very challenging to quantify the extent of oxidative biodegradation. These issues can be circumvented with compound-specific isotope analysis (CSIA), where the extent of transformation is inferred from the isotope fractionation measured in the remaining pollutant and *a priori* knowledge of isotope effects pertinent to enzymatic oxygenations. However, the kinetics of such reactions may be governed by enzymatic activation of molecular $O_2$ which do not involve a transformation of the organic pollutant. Little is known to date about the relative contributions of $O_2$ activation to the rate of oxidative pollutant removal from a contaminated environment. Based on our recent work with Rieske non-heme ferrous iron dioxygenases[1,2] and flavin-dependent monooxygenases,[3] I will illustrate how their catalytic cycles determine the observable pollutant isotope fractionation.

**References**

(1) Pati, S. G.; Kohler, H.-P. E.; Hofstetter, T. B. Characterization of substrate, co-substrate, and product isotope effects associated with enzymatic oxygenations of organic compounds based on compound-specific isotope analysis. In *Methods in Enzymology*; Harris, M. E., Anderson, V. E., Eds.; Academic Press, **2017**; pp 292–329.

(2) Pati, S. G.; Kohler, H.-P. E.; Pabis, A.; Paneth, P.; Parales, R. E.; Hofstetter, T. B. Substrate and enzyme specificity of the kinetic isotope effects associated with the dioxygenation of nitroaromatic contaminants. *Environ. Sci. Technol.* **2016**, *50* (13), 6708–6716.

(3) Wijker, R. S.; Pati, S. G.; Zeyer, J.; Hofstetter, T. B. Enzyme kinetics of different types of flavin-dependent monooxygenases determine the observable contaminant stable isotope fractionation. *Environ. Sci. Technol. Lett.* **2015**, *2* (11), 329–334.

# Tracing sources and fate of mercury in soils and groundwater at industrial contamination sites with stable mercury isotopes

Jan G. Wiederhold[1], F.M. Brocza[1], A.R.C. Grigg[1], S.M. Kraemer[1]

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## Abstract

Mercury (Hg) is a toxic pollutant of great environmental concern. The "Minamata Convention on Mercury", a global treaty initiated by the United Nations Environment Programme and designed to reduce negative impacts of Hg on the environment, entered into force in August 2017. Although Hg is now being phased out in most industrial applications, a large number of legacy sites have been affected by historical industrial Hg releases. Elevated Hg levels in soils and waters at these sites represent a serious threat for the environment at local and regional scales. The long-term fate, mobility, and bioavailability of Hg strongly depend on its speciation, which is determined by the initial Hg compound from the industrial contamination source as well as biogeochemical transformation processes after release into the environment. Understanding the governing processes and controls on Hg speciation at contaminated sites is thus essential for risk assessment and site management.

There are seven stable Hg isotopes ($^{196}$Hg, $^{198}$Hg, $^{199}$Hg, $^{200}$Hg, $^{201}$Hg, $^{202}$Hg, and $^{204}$Hg) and recent studies have revealed that environmental processes can cause significant stable Hg isotope fractionations. Importantly, both mass-dependent (MDF) and mass-independent (MIF) fractionation can occur, affecting even-mass and odd-mass Hg isotopes to a different extent. This opens up the possibility of using Hg isotope signatures as two-dimensional tracer. High-precision Hg isotope analyses can be performed using multiple collector inductively coupled plasma mass spectrometry (MC-ICP-MS) using a cold vapor introduction system and Tl addition for mass bias correction. With this method an analytical precision of about ±0.1‰ (2SD) for $\delta^{202}$Hg (MDF) and $\Delta^{199}$Hg (MIF) can be achieved. In the context of industrial contamination sites, variations in Hg isotope signatures may help identifying contamination sources and quantifying transformation processes of Hg species.

Here, we present data from two projects investigating Hg speciation and Hg isotope signatures in soil and groundwater collected at industrial legacy sites in Germany and Switzerland. Data from a former wood treatment facility, where Hg(II)-chloride had been used as preservative, demonstrate that significant Hg isotope variations exist between different depths of contaminated soil cores as well as between soil extracts targeting different Hg species. For instance, water-extractable Hg in the most contaminated zone is enriched in heavy Hg isotopes compared with the bulk soil suggesting that the initial Hg isotope fingerprint of the contamination source was altered during biogeochemical transformations in the subsurface. This finding is corroborated by groundwater samples collected downstream which also exhibit positive $\delta^{202}$Hg values. In contrast, data from a second contamination case, where Hg emissions from an industrial facility resulted in severe soil contamination adjacent to a drainage canal, exhibited no significant Hg isotope variations in soils collected downstream of the facility and between different soil extracts. This suggests that the source signature of the contamination source is preserved at this site. The implications of these results for using Hg isotope signatures as source and process tracer for industrial Hg emissions will be discussed.

## ORAL PRESENTATIONS

# Tomography of anthropogenic nitrate contribution along mesoscale rivers in two European catchments

Christin Müller[1], Matthias Zink[2], Luis Samaniego[2], Ronald Krieg[1], Michael Rode[3], Ralf Merz[1], Kay Knöller[1]

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## Abstract

The challenge of the presented project is to characterize and quantify large (regional) scale dynamics and trends in water and solute fluxes from two European catchments representing changing environmental conditions (e.g. elevated inputs, land use and climate change). One investigated area is part of the TERENO (Terrestrial Environmental Observatory) project, the test side of the Bode region in the Harz Mountains, Germany. The second catchment is the Erlauf hydrographic basin in the alpine foothills in Austria. Differences between the two regions in terms of precipitation patterns and altitudes have a major impact on the isotopic signatures of sampled surface waters and have to be considered in further comparisons.

The Bode River catchment (total size of 3200 m$^2$) in the Harz Mountains in Germany was intensively investigated by a monthly monitoring approach with 133 sampling points representing the same number of sub-catchments which starting in 2012. The area is characterized by a strong anthropogenic gradient, with forest conservation areas in the mountain region, grassland, and intensively mixed farming in the lowlands (Mueller at al. 2015). Consecutive discharge simulations by a mesoscale hydrological model (mhM) allow a quantitative analysis of nitrate fluxes for all observed tributaries (Samaniego, Kumar et al. (2010) and Kumar, Livneh et al. (2013)). The investigation of nitrate isotopic signatures for characteristic landscape types allows the delineation of dominant $NO_3^-$ sources: nitrified soil nitrogen, fertilizer, and manure. Besides source delineation, the relationship between runoff and nitrate dynamics was analyzed for the entire Bode river catchment and, more detailed, for one major tributary with minor artificial reservoirs. Thereby, it becomes apparent that nitrate isotopic variations increase with decreasing discharge (Mueller et al. 2016). This effect might be due to a local, more intense impact of bacterial denitrification under low discharge conditions (higher residence time) in the anoxic soil zone, in the groundwater that discharges into the river and in the hyporheic zone. Generally, $\delta^{15}N_{NO3}$ and $\delta^{18}O_{NO3}$ decrease with increasing runoff at each sampling point, which can be caused by a preferential wash-out of more easily mobilizable, isotopically lighter fractions of the soil nitrate pool.

### References

Kumar, R., B. Livneh and L. Samaniego (2013). "Toward computationally efficient large-scale hydrologic predictions with a multiscale regionalization scheme." Water Resources Research **49**(9): 5700-5714.

Mueller, C., R. Krieg, R. Merz and K. Knoller (2015). "Regional nitrogen dynamics in the TERENO Bode River catchment, Germany, as constrained by stable isotope patterns." Isotopes Environ Health Stud: 1-14.

Mueller, C., M. Zink, L. Samaniego, R. Krieg, R. Merz, M. Rode and K. Knöller (2016). "Discharge Driven Nitrogen Dynamics in a Mesoscale River Basin As Constrained by Stable Isotope Patterns." Environmental Science & Technology **50**(17): 9187-9196.

Samaniego, L., R. Kumar and S. Attinger (2010). "Multiscale parameter regionalization of a grid-based hydrologic model at the mesoscale." Water Resources Research **46**(5): n/a-n/a.

# Relationship between the air moisture source and the stable isotope composition of the precipitation in Hungary

György Czuppon[1], Emese Bottyán[2], László Haszpra[3], Tamás Weidinger[2], Krisztina Kármán[1]

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## Abstract

In the last few years, the analysis of backward trajectories has become a common use for identifying moisture uptake regions for the precipitation of various regions. Hungary is influenced by meteorological (climatological) conditions of Atlantic, Mediterranean and North/East regions therefore this area is sensitive to detect changes in the atmospheric circulation. In this study we present the result of the investigation about the determination of air moisture source regions for six localities in Hungary for more than four years. To reconstruct the path of the air moisture from the source region, we ran the NOAA HYSPLIT trajectory model using the GDAS database with 1° spatial and 6 hours temporal resolution for every precipitation event, for heights of 500, 1500 and 3000 m. We determined the location where water vapour entered into the atmosphere by calculating specific humidity along the trajectories. Five possible moisture source regions for precipitation were defined: Atlantic, North European, East European, Mediterranean and continental (local/convective). Additionally, this study evaluates the regional differences in stable isotope compositions of precipitation based on hydrogen and oxygen isotope analyses of daily rainwater samples. Stable isotope variations show systematic and significant differences between the regions. The variability of moisture source shows also systematic seasonal and spatial distribution. Interestingly, the most dominant among the identified source regions in all stations is the Mediterranean area; while the second is the Atlantic region. The ratio of the precipitations originated in Eastern and Northern Europe seem to correlate with the geographic position of the meteorological station. Furthermore, the ratios of the different moisture sources show intra annual variability.

In each location, the amount weighted d-excess values were calculated for the identified moisture sources. The precipitation originated in the Mediterranean regions has systematically higher d-excess values than that originated in the Atlantic sector, independently from the absolute value which apparently changes from station to station. The precipitation fraction attributed to the Northern European sector has also relatively elevated d-excess values that might be related to the cold-season domination of moisture transport from this region. Thanks for the financial support of the National Research, Development and Innovation Office (project No. OTKA NK 101664, SNN118205/ARRS:N1-0054, PD 121387). György Czuppon also thanks for the support of the János Bolyai Research Scholarship of the Hungarian Academy of Sciences.

## ORAL PRESENTATIONS

# Temporal and spatial distribution of isotopes in river water in Austria

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## Abstract

The isotopic composition of hydrogen and oxygen in river water (Figure 1) is mainly determined by the isotopic composition in precipitation water in the drainage area (altitude effect, continental effect, seasonal variations and influence of storms). Several hydrological parameters and processes modify this isotopic signature and its temporal variation: delayed runoff of winter precipitation, residence time of groundwater discharged to the river, confluence with tributaries, evaporation from lakes in the river system as well as anthropogenic influences on the hydrological regime (e.g. reservoirs, irrigation).

The comparison of long-term trends of stable isotope ratios in precipitation and river water reveals that, in the case of the Upper Danube Basin (catchment area 101700 km$^2$, mean discharge 1925 m$^3$/s), river water quite well reflects the isotope trends of precipitation. Not only short-term signals (e. g. seasonal $\delta^{18}O$ variations or $^3H$ releases from nuclear facilities) but also long-term changes of isotope ratios in precipitation are transmitted through the catchment, for instance the significant increase of $\delta^{18}O$ values during the eighties. The different sources of air moisture (Atlantic, Mediterranean) are also clearly represented isotopically in the river system.

The river water isotope time series will serve as a basic data set for hydrological investigations as well as for assessing future impacts within the catchment areas. This includes climatic/hydrological changes (e.g. temperature changes, change of precipitation distribution) as well as anthropogenic impacts on the hydrological regime (e.g. reservoirs, change in land use). All these changes will more or less be reflected in the isotopic composition of river water.

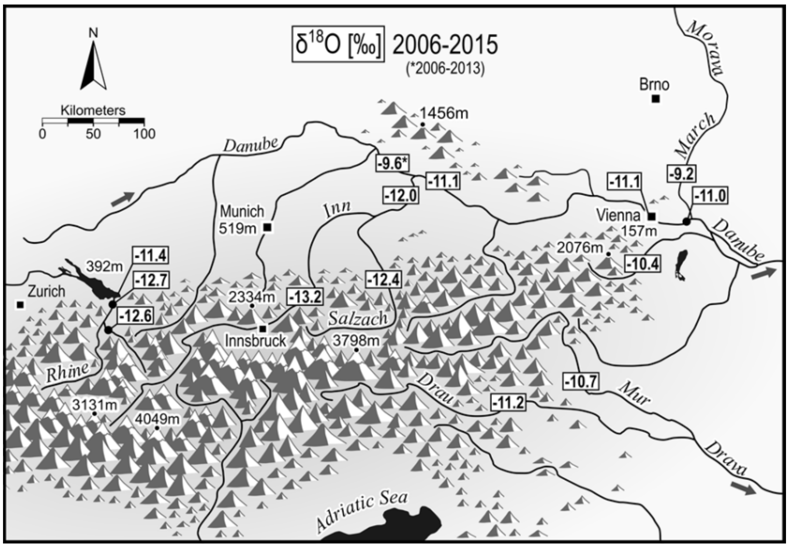

Figure 1: $\delta^{18}O$ (average values 2006-2015) of river water in Austria

**References:**

D. Rank, S. Wyhlidal, K. Schott, S. Weigand, A. Oblin, (2017): Temporal and spatial distribution of isotopes in river water in Central Europe: 50 years experience with the Austrian network of isotopes in rivers: Isotopes in Environmental and Health Studies (in press). http://dx.doi.org/10.1080/10256016.2017.1383906

## ORAL PRESENTATIONS

### Stable isotopes reveal the origin of caves

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**Abstract**

Soluble rocks such as limestones are characterised by a localised high permeability as a result of dissolution by water charged with carbon dioxide derived from the soil zone. This process, collectively referred to as karst, is held responsible for the worldwide formation of most cave systems. Recent research has shown that small- to large-scale caverns can also be created by a different process, i.e. upwelling of deep-seated groundwater. This second type, referred to as hypogene speleogenesis, gives rise to cave systems lacking natural entrances. Following uplift and erosion these networks of subsurface caverns may be modified by "normal" karst processes, thus masking their primary origin.

Stable isotopes provide one of the most robust tools to identify such hypogene processes in the history of cave systems even millions of years after their formation. Examples presented in this talk include caves from the Vienna Basin and the Dolomites.

## ORAL PRESENTATIONS

### Middle Bronze Age humidity and temperature variations and societal changes in East-Central Europe

Attila Demény[1], Z. Kern[1], Gy. Czuppon[1], G. Schöll-Barna[1], Z. Siklósy[1], Sz. Leél-Őssy[2], G. Cook[3], G. Serlegi[4], B. Bajnóczi[1], P. Sümegi[5], Á. Király[4], V. Kiss[4], G. Kulcsár[4], M. Bondár[4], A. Németh[1]

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**Abstract**

Archaeological evidence pointed to substantial changes in Bronze Age societies in the European-Mediterranean region. Isotope geochemical proxies have been compiled to provide independent ancillary data to improve the paleoenvironmental history for the period of interest and support the interpretation of the archaeological observations in Hungary. Beside published compositions, in this study we gathered new H isotope data from fluid inclusion hosted water from a stalagmite of the Trió Cave, Southern Hungary, and compared the H isotope data with existing stable isotope and trace element compositions reported for the stalagmite. Additionally, animal bones and freshwater bivalve shells (Unio sp.) were collected from Bronze Age archaeological excavations around Lake Balaton and their stable C and O isotope compositions were measured in order to investigate climate changes and lake evolution processes during this period. The data indicate warm and humid conditions with elevated summer precipitation around 3.7 ka cal BP, followed by a short-term deterioration in environmental conditions at about 3.5 ka cal BP. The environment became humid and cold with winter precipitation dominance at about 3.5 to 3.4 ka cal BP, then gradually changed to drier conditions at ~3.2 ka cal BP. Significant cultural changes have been inferred for this period on the basis of observations during archaeological excavations. The most straightforward consequences of environmental variations have been found in changes of settlement structure and trading routes. The palaeoclimatological picture is well in line with other European-Mediterranean climate records, indicating that the climate fluctuations took place at regional scale.

## ORAL PRESENTATIONS

# Magnitude of temperature change in the lowland Neotropics during the last Glacial

Anna-Lena Grauel[1,2], David A. Hodell[1], Stefano M. Bernasconi[3]

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## Abstract

We present a reconstruction of temperature and precipitation changes over the last 42ka from a lake sediment core from Lake Petén Itzá, Guatemala, using three independent temperature proxies: pollen-based temperature estimates using the Modern Analog Technique (MAT), tandem measurements of $\delta^{18}O$ in biogenic carbonate and gypsum hydration water, and clumped isotope thermometry. The gypsum hydration method cannot be applied to the clay-rich intervals that were deposited during humid periods. The combination of temperature estimates from pollen MAT and isotope geochemical methods has the potential to provide a continuous record of tropical temperature change in lowland Central America.

The temperatures derived using the three methods generally agree during interstadials and some stadials (e.g., Heinrich Event 2 and HE3), but diverge during other stadial events (e.g., HE1 and HE4). The most arid conditions coincide with stadials, especially those associated with HEs when pollen assemblages are dominated by xeric-tolerant taxa. In contrast, during interstadials and the last glacial maximum, pollen from temperate pine-oak forest indicate more humid conditions in the lowland Neotropics. During HE1 and HE4 the gypsum hydration and clumped isotope methods show a severe cooling of 6 to 10°C, whereas the pollen MAT suggests more moderate cooling of 3 to 6°C. The reason for this divergence is likely that no modern analogues exist for the pollen assemblage during these cold, arid stadials when the MAT is not applicable.

Overall, the results of our study show that temperature decline in the lowland Neotropics was much greater during HE1 and HE4 than previously assumed. The extreme dry and cold conditions during HSs in lowland Central America were associated with fresh water forcing into the North Atlantic, which led to reduced Atlantic Meridional Overturning Circulation, cooling of the North Atlantic, southern advance of sea-ice, and southward shift of the Intertropical Convergence Zone. Although some models correctly predict the sign of temperature and precipitation changes, they consistently underestimate the degree of observed cooling and decreased precipitation over land in lowland Central America.

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Grauel, A-L., Hodell, D.H., Bernasconi, S.M., 2015. Quantitative estimates of tropical temperature change in lowland Central America during the last 42ka. Earth and Planetary Science Letters 438, pp 37-46.

# 775,000 years of climate history from the southwest USA: revamping the famous Devils Hole cave record

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## Abstract

Variations in oxygen isotopes recorded in calcite deposits within Devils Hole cave provide insight into the paleoclimate of the southwest USA. Early Devils Hole climate records [1,2] have been a source of controversy for over 3 decades, as they conflicted with accepted global climate mechanisms tied to orbital forcing. A resolve to this controversy was presented in 2016, when samples from the neighbouring cave Devils Hole 2 (100m northeast from Devils Hole) corroborated with the accepted insolation-forced timing of glacial termination II and provided evidence for geochemical processes biasing the original Devils Hole chronologies [3]. Moving forward, we have extended the oxygen isotope record from Devils Hole 2 cave (DH2) to 775,000 years before present. 100 high resolution $^{230}$Th-$^{234}$U ages and 10 independently-calculated $^{234}$U-$^{238}$U ages form the basis of our preliminary DH2 chronology. In addition, evidence for a paleomagnetic reversal was discovered in DH2 samples at the onset of marine isotope stage (MIS) 19, as determined by stable isotope curve matching and $^{234}$U-$^{238}$U ages, and thus corresponds in time to the Brunhes-Matuyama reversal [4]. O isotope values derived from DH2 samples ($\delta^{18}O_{DH2}$) principally reflect variations in temperature and precipitation source in the southwest USA [1]. Depleted $\delta^{18}O_{DH2}$ represent cool and wet periods corresponding to glacial periods, during which regional paleoclimate evidence suggests an increase in northerly-sourced moisture likely due to a southward migration of the jet stream. Preliminary results show a strong temporal agreement with 65°N summer insolation over the past eight glacial-interglacial cycles. The preliminary $\delta^{18}O_{DH2}$ record corresponds with distant records worldwide reflecting orbital-scale climate change, including the timing of glacial terminations II to VII as determined by the precisely dated East Asian monsoon record [5]. Ongoing work on the DH2 record aims to shed detailed insight into atmospheric circulation changes in the Northern Hemisphere mid-latitudes over the past 775,000 years.

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# Stable isotope signatures in tree rings of Swiss stone pine (*Pinus cembra* L.) in Romania – annually resolved summer proxy archive for the past 600 years

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## Abstract

Swiss stone pine (*Pinus cembra* L.) presents the greatest dendroclimatological potential in the Carpathians owing to the significant longevity of the species, strictly constrained ecological preference (i.e. timberline habitat) and the related pronounced temperature regulated growth, and well-preserved snags and subfossil findings. Stone pine has a prominent importance in tree-ring studies also in the Alps. This is the only species with continuous availability over the multi-millennial dataset of the Eastern Alpine Conifer Chronology upgraded recently to cover practically the entire Holocene (Nicolussi et al., 2014). Despite these facts, the potential of stone pine dendroisotope signal was never tested against the instrumental climate records.

The aim of this study is 1) to calibrate the relationship between stable isotopes of oxygen and carbon in Stone pine cellulose and main climatic parameters 2) to determine the potential of *Pinus cembra* for palaeoclimatological reconstruction based on analysis of stable isotopes of oxygen and carbon in tree rings from samples collected in Călimani Mts. and 3) to test the hypothesis that the nearby sulphur mines have not biased the climate signal of the stable isotope composition, which is a proxy indicator better than tree-ring width (TRW).

For this study we analyzed wood samples of Swiss stone pine from living and dead trees from Călimani Mts., NE Romania that grew between 2012 and 1400 AD. The stable isotope composition of cellulose oxygen and carbon ($\delta^{18}O$ and $\delta^{13}C$) was measured at the Institute for Geological and Geochemical Research, Budapest, Hungary, using a high-temperature pyrolysis system (Thermo Quest TC-EA) coupled to an isotope ratio mass spectrometer (Thermo Scientific™ Delta V) following a ring by ring (i.e., non-pooled) approach.

The $\delta^{18}O$ and $\delta^{13}C$ values showed a strong positive correlation with maximum air temperature (r=0.60), mean temperature (r=0.60), and sunshine duration (r=0.69) and negative correlation with precipitation amount (r=-0.50) and nebulosity (r=0.60) during the summer months (June, July and August). Correlation with TRW is less than 0.3, for all parameters. Since temporal stability of the proxy-climate correlation is maintained also over the period of sulphur exploration when the growth-climate relation was found to break down, we conclude that sulphur did not influenced the climate signal archived in the stable isotope composition in comparison with TRW. Based on this data we suggest that $\delta^{18}O$ and $\delta^{13}C$ is a better indicator proxy for paleoclimatic reconstruction, and sulphur mining had less impact on this correlation than for TRW.

Acknowledgement: The research was supported by the European Union and the State of Hungary, co-financed by the European Regional Development Fund in the project of GINOP-2.3.2.-15-2016-00009 'ICER', as well as by projects LP2012-27/2012 (Hungary), CLIMFOR 18SEE, and Ro-Hu CTR 663 (Romania).

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## ORAL PRESENTATIONS

### Trees in a changing environment: Visualizing their responses with stable isotopes

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**Abstract**

Along with an increase in $CO_2$, changes in the hydrological regimes and mean annual temperatures, in particular an increase in the frequencies of high temperature extremes can be observed. This results in an increase of **V**apor **P**ressure **D**eficit (VPD) the driver for evapotranspiration, causing an increase in drought stress for the vegetation.

In lab experiments it is observed that an increase in $CO_2$ causes a distinct increase in net Photosynthesis ($A_N$), and often enhanced growth rates, while stomatal conductance ($g_s$) decreases significantly. Long-term lab experiments even showed a down regulation of photosynthesis (acclimatization), i.e. a gradual reduction of $A_N$ under elevated $CO_2$. These results led to the expectation of finding similar response patterns in FACE experiments in the field. However, the results were quite sobering, in particular for forest ecosystems. Although photosynthesis increases at high $CO_2$ concentration the whereabouts of the additionally gained carbon can currently not be determined. Also the responsiveness of stomatal conductance to changes in $CO_2$ is diminished or disappeared.

In most FACE experiments, the applied $CO_2$, for exposing plants to elevated $CO_2$, originated from biomass or fossil fuel burning. Thus, the added $CO_2$ is depleted in the heavier isotope ($^{13}C$) relative to that of the atmosphere. This provides an efficient and cheap label, allowing the tracing of the newly assimilated carbon, besides studying the plants responses to $CO_2$ exposition.

In a further example the effect of acute and severe drought stress on tree growth is shown, resulting in a halt of tree growth during the vegetation period and an isotopic decoupling between leaves and tree rings. When plants (i.e. trees) are resupplied with sufficient water we can observe an enhanced photosynthetic rate, which is higher than that of control plants, which were not exposed to drought. The recognition of such decoupling and recovery phenomena is crucial for the interpretation of tree ring isotope data.

Finally, time series of tree ring isotopes for the last 150 years show a clear increase of the of the carbon water relations (intrinsic Water Use Efficiency) iWUE as a response to these environmental changes. Yet trees show different strategies how they respond to these changes.

In this presentation some of the unexpected and interesting results about the response or the lack of it to elevated $CO_2$, drought and recovery from drought will be shown. In particular the use of stable isotopes will be demonstrated as a tool that facilitated the investigation of C- translocation from the leaves to the soil under elevated $CO_2$ or drought or drought-recovery processes. FurthermoreFurthermore, the changes in iWUE for the last 150 years and the various strategies of how trees cope with environmental changes will be shown. Most of these results would not have been possible without the tool of stable isotopes or only under great costs and efforts.

# Carbon isotope fractionation during methanogenesis in marine sediments: An evaluation by reactive-transport modelling

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## Abstract

Carbon isotope compositions of dissolved inorganic carbon (DIC) and methane ($CH_4$) in porewater of marine sediments at seafloor temperatures show very large variation covering a $\delta^{13}C$ range from -100‰ to +30‰. These extreme values are the result of fractionation during microbial carbon metabolism, but the combined effect of all factors controlling the isotope distributions is still incompletely understood. We used a model approach to evaluate the effects of reaction and transport on carbon isotope distributions in modern sediment porewater under steady state. Simulated $\delta^{13}C_{DIC}$ profiles generally show negative values in the sulphate reduction zone and more positive values in the methanogenic zone. The model shows that the $\delta^{13}C$-offset between DIC and $CH_4$ in the methanogenic zone is in the order of 70‰, which is similar to the offset commonly observed in marine sedimentary porewaters (e.g. Meister et al., 2011). However, to reach such a large offset would require much larger fractionation factors than the ones reported from culture experiments with both acetoclastic and autotrophic methanogens (Londry et al., 2008).

The discrepancy between field observation and experimental results raises the question whether alternative fractionation mechanisms than generally assumed enzymatic kinetic fractionation may play a role. In fact, the observed offset between DIC and $CH_4$ is near to isotopic equilibrium at in situ temperature. Even though DIC and $CH_4$ do not spontaneously exchange isotopes, an equilibration may occur via a microbial enzymatic pathway. Indeed, such a partial isotopic equilibration was recently shown for anaerobic methane oxidizing consortia (Yoshinaga et al., 2014) by tracing a reverse flux of $^{14}C$-labeled DIC at low thermodynamic driving force. We propose a similar mechanism may be active during methanogenesis.

Both further model studies and incubation experiments are necessary to clarify the fractionation mechanism during methanogenesis. This will fundamentally contribute to interpreting carbon isotope signatures in the geological record, as often, early diagenetic carbonates are formed or altered in methanogenic zones. A better understanding of these processes may furthermore help using carbon isotope signatures for the reconstruction of ancient microbial life on Earth.

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## ORAL PRESENTATIONS

### Identification of ivory with respect to animal species by stable isotopes

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**Abstract**

Ivory is an expensive and regulated good. Elephant ivory is a CITES (Convention on International Trade in Endangered Species of Wild Fauna and Flora, also known as the Washington Convention) relevant good and can only be traded under special circumstances together with the obligatory CITES documents. When trading with illegal ivory people sometimes claim that it is mammoth ivory, which does not fall under the CITES treaty, or that the ivory is coming from walrus, which is not so strictly protected.

About fifty ivory samples from different species (elephant, mammoth, walrus) were investigated for their respective isotope signatures to learn whether this method is suitable for a quick identification of species.

The obtained results show a clear separation of elephant ivory from mammoth and walrus ivory, with dominantly oxygen isotopes giving different values. A study of a larger sample set should be carried out to confirm the findings.

## New analytical approach in monitoring of $CO_2$ cycle in aquatic ecosystems

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### Abstract

The isotopic signatures of carbon ($\delta^{13}C$) and oxygen ($\delta^{18}O$) can be used as a tool to understand pathways, processes and the fate of $CO_2$ molecules in particular in the marine environment. This is because $\delta^{13}C$-values are controlled by species specific metabolic processes of respiration and photosynthesis, while the $\delta^{18}O$-values are affected by the oxygen exchange between the molecules of $CO_2$ and the ambient water. Applying a new analytical approach to determine $\delta^{13}C$ and $\delta^{18}O$ changes using a mid-infrared laser (IRIS) absorption spectrometer, Thermo Scientific™ Delta Ray™ IRIS with URI Connect, it is possible to record online changes of carbon and oxygen isotopes with time resolutions of seconds. This new approach was tested in a 600 L reef coral culturing tank with controlled day-night cycle, at GEOMAR. In our study we continuously measured $\delta^{13}C$ and $\delta^{18}O$ of air from a semi-closed column constantly mixed with water from the main tank. Measurement per sample was 3 minutes and with integration time of 60 seconds we achieve internal precision better than 0.05 ‰ for both $\delta^{13}C$ and $\delta^{18}O$ per sample run. Along with continuous measurement of air, we collected water samples every 1 to 3 hours during a period of 24 hours. Results of air measurement showed that air and water are in equilibrium and that isotopic change occurs rapidly when light intensity is changed. The average day-night amplitudes were about 2.7 ‰ and about 0.6 ‰ for $\delta^{13}C$ and $\delta^{18}O$, respectively. The $\delta^{13}C$-values of dissolved inorganic carbon (DIC) were analyzed following the usual gas evolution method. The results follow the same trend as it was observed on air measurements, with an average amplitudinal change in $\delta^{13}C$ of about 2.4 ‰. Future field experiments of this technique will show if a large-scale measurement of $CO_2$ fluxes across the air-seawater interface above coral reefs is possible and whether this system may be used to monitor the status of coral reefs and the influence of human activity in coastal areas.

## An LC-IRMS Interface for Flexible Compound-Specific Stable Isotope Analysis

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**Abstract**

Introduction:

In aqueous samples compound-specific stable isotope analysis (CSIA) plays an important role. Environmental and forensic sciences are prominent examples of such applications, utilizing naturally occurring fractionation processes during transport and transformation processes to, e.g., allocate contaminants or drugs sources. The broad range of involved application areas includes e.g. the food industry (food fraud) and sport (doping). However, the currently available LC-IRMS solutions are limited to stable carbon isotope analysis only and therefore the use of pure aqueous solvent. This considerably limits the application possibilities and analyzable compound classes. No direct method (without sample preparation) for stable isotope analysis of nitrogen and sulfur of non-volatile compounds is known yet.

Methods:

A novel high-temperature combustion interface was developed to hyphenate high-performance liquid chromatography with isotope ratio mass spectrometry in a more flexible way. The system is capable to analyze stable isotopes other than carbon, which also abolish the limitation of pure aqueous solvent usage. In continuous operation virtually for all peaks in a chromatogram the stable isotope ratio can be analyzed.

Results:

Experimental data of different examples proof the performance and flexibility of such a system. Compounds were determined typically with a precision and trueness of ≤0.5‰ for different stable isotopes.

Conclusion:

The development of a novel LC-IRMS interface resulted in the first system reported that is not limited to stable carbon isotopes anymore. Furthermore, the use of organic solvents is possible which open up new possibilities in CSIA-based research fields.

Keywords:
Instrumentation, LC-IRMS, IRMS interface, stable isotope analysis

## Improving Measurement Reliability of Stable Isotopes

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### Abstract

The dependence of stable isotopic analyses in geochemistry and environmental measurements are steadily increasing. However, the ability to compare measurements between laboratories can be quite challenging owing to differences in measurement and calibration (standards and methods).

The key to obtaining reliable data is by designing experiments which utilize sampling methodologies that represent the environment intended for the study and meet the data-quality objectives. These samples in turn must then be calibrated against suitable reference materials containing low levels of uncertainty.

The precision and accuracy of the analytical result is directly related to the precision and accuracy of the standard used to calibrate the analytical instrument.

The reliability of measurements can be approved by choosing suitable equipment. The composition of a calibration gas mixture can be altered by using inappropriate transfer lines and/or insufficient purging. Results of our investigations will be presented.

The production of stable isotopic calibration gases requires transfilling between cylinders. Results of our study concerning potential alteration of isotopic compositions will be discussed in detail.

# From position-specific labeling to quantitative soil fluxomics - new dimensions in terrestrial biogeochemistry

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## Abstract

Past decade of biogeochemical research had strong focus on the understanding of soil and sedimentary organic matter (SOM) dynamics. A detailed understanding of the transformation pathways and its controlling factors is required to unravel the relation of organisms, fluxes and C transformations in soils. Biogeochemists' view on C cycling by microorganisms has rarely exceed a strongly simplified concept assuming that C gets either oxidized to $CO_2$ via the microbial catabolism or incorporated into biomass via the microbial anabolism. Biochemists, however, identified in the past decades the individual reactions of glycolysis, pentose-phosphate pathway and citric acid cycle underlying the microbial catabolism. At various points within that metabolic network the anabolic fluxes feeding biomass formation branch off. Recent studies based on position-specific isotope labeling allowed metabolic tracing in soils in situ, an approach which is quantitatively complemented by metabolic flux modeling. This approach has reached new impact by the combination with compound-specific isotope analysis of microbial biomarkers which allows the identification of microbial group-specific pathways. Thus, the combination of position-specific labeling, compound-specific isotope incorporation in biomarkers and quantitative metabolic flux modelling provide the toolbox for quantitative soil fluxomics.

Application of position-specific labeled glucose showed that up to 55% of glucose, incorporated into the amino sugar glucosamine, first passed glycolysis before allocated back via gluconeogenesis. Similarly, glutamate-derived $^{13}C$ is allocated via anaplerotic pathways towards fatty acid synthesis and in parallel to its oxidation in citric acid cycle. Thus, oxidizing catabolic pathways and anabolic pathways occurred in soils simultaneously, a combination rarely occuring in pure cultures, where constant growth conditions under high C supply allow a straight unidirectional regulation of C metabolism. However, unstable environmental conditions, C scarcity and interactions between a still unknown diversity of microorganisms in soils are likely to induce the observed metabolic diversity. Thus, implementation of this metabolic backlux into metabolic C models is required to describe life conditions and metabolic adaptations in soils and could be shown to improve our predictions of parameters such as carbon use efficiency based on metabolic flux modelling.

To understand how microorganisms catalyze the biogeochemical fluxes in soil a profound understanding of their metabolic adaptation strategies such as inracellular backfluxes or recycling of extracellular compounds is crucial. Metabolic flux models adapted to soils will not only deepen our understanding on the microorganims' reactions to environmental changes but also create the prerequisits for a quantitative prediction of biogeochemical fluxes based on the underlying microbial processes.

## Multiple isotope tracing reveals the transformation processes of oligopeptides in soils

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### Abstract

Environmental changes such as global warming, rising atmospheric $CO_2$, and increasing N deposition have increased the need to advance our understanding of the global biogeochemical cycles, especially of the soil carbon and nitrogen (N) cycles. In the soil N cycle, the rate limiting step is believed to be the depolymerization of high molecular weight (HMW) organic N which is mainly comprised of proteinaceous substances. The proteins are decomposed to bio-available short chain oligopeptides and free amino acids by extracellular hydrolytic enzymes excreted by soil microbes (and plants). In the few published studies, organic nitrogen uptake by soil microorganisms only takes into account amino acids. However, the extractable soil oligopeptide pool comprises a much larger proportion of the dissolved organic nitrogen pool compared to amino acids and it has been demonstrated that oligopeptides can be directly utilized by plants and microbes without prior hydrolysis to free amino acids. Although several $^{14}C$ oligopeptide experiments have been conducted which increased our understanding of microbial C utilization and mineralization ($^{14}CO_2$ production) of some model dipeptide and tripeptide structures, we still lack a method to determine in situ rates of soil oligopeptide transformation processes and related N cycling.

Here we report a novel approach to estimate gross transformation rates of oligopeptides and amino acids utilizing liquid chromatography / high resolution mass spectrometry (LC/HRMS) and numerical modeling. The oligopeptide pool is labeled with uniformly $^{15}N$ labeled oligopeptides and the amino acids pool is concurrently labeled with uniformly $^{13}C$ labeled amino acids. After tracer addition, the $^{15}N$ enrichment in the oligomer pool will be diluted due to depolymerization of high-molecular-weight N compounds (proteins) to oligopeptides and the consumption of the oligomers (same as with isotope pool dilution theory). At the same time, the $^{13}C$ enrichment in the monomer pool will be diluted as well, but it also concomitantly undergoes $^{15}N$ enrichment due to further depolymerization of $^{15}N$-labeled oligomers to free amino acids. With our newly developed liquid chromatography /high resolution mass spectrometry (LC/HRMS) platform, different stable isotope labeled ($^{15}N$ and $^{13}C$) isotopic analogues of these compounds can be well separated. The transformation process rates are calculated by a novel $^{13}C$-$^{15}N$ version of the *Ntrace* tracing model.

Initial application of the approach to a forest soil showed that the direct uptake of oligopeptides by microbes accounted for 0.6-fold of that of amino acids, indicating that oligopeptides represent an important N source for soil microbes. In addition, the depolymerization rate of oligopeptides to free amino acids is ten times smaller compared to that of protein to free amino acids and of protein to oligopeptides.

## The influence of pelagic calcification on the Late Triassic seawater chemistry

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**Abstract**

Pelagic life plays an immense role in the ocean-atmosphere interaction due to forming the basis of biological pump, modifying the alkalinity and providing feedback mechanisms. Coccolithophores, belonging to red-plastid phytoplankton appeared in the Early Mesozoic. In the Austrian Alps the oldest record of them dates back around the Norian/Rhaetian boundary. Modelling study (Zeebe and Westbroek, 2003) pointed out the determining role of pelagic calcifiers on the regulation of seawater chemistry permitting a buffering effect. Sections from different paleogeographic environments and latitudes were chosen to confront the model with geochemical data. The isotope systems in focus are: carbon, strontium, calcium, magnesium. Calcium isotope system is highly relevant to the pelagic life as it is related the calcification rate, however additional proxies are needed to better constrain the input and output fluxes. As a first step towards this, Sr isotope measurements were carried out in sections from Austria, Turkey and Oman. Different solution techniques were used to get reliable data. Careful diagenesis estimation with the use of light microscope, SEM, ICP-OES and cathodoluminescence helps to sort out the altered samples. From all the sections the Austrian Steinbergkogel seems to provide the less overprinted material, probably due to the early diagenetic stabilization of the Hallstatt Limestone in hemipelagic high environment. Overall, we are able to reconstruct the $^{87}Sr/^{86}Sr$ trend in the Norian—Rhaetian as that is known from the literature, and besides complete the curve across the stage boundary and better constrain the characteristic drop in the Sr isotopes, which serves as an important background to evaluate other geochemical signatures.

ORAL PRESENTATIONS

## Light Hydrocarbon Geochemistry of Oils in the Alpine Foreland Basin: Impact of Geothermal Fluids on the Petroleum System

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### Abstract

Oil is produced in the Austrian sector of the Alpine Foreland Basin from Eocene and Cenomanian reservoirs. Apart from petroleum, the basin hosts a significant geothermal potential, which is based on the regional flow of meteoric water through Malmian (Upper Jurassic) carbonate rocks. Oils are predominantly composed of n-alkanes, while some samples are progressively depleted in light aromatic components. The depletion in aromatic components relative to abundant n-alkanes is an effect of water washing. Waters coproduced with oils that are affected by water washing show a progressive reduction in salinity and depletion in $^2$H and $^{18}$O isotopes, indicating that the degree of water washing is mainly controlled by the inflow of meteoric water from the Malmian aquifer. In some fields with Cenomanian reservoir rocks, a hydraulic connectivity with the Malmian aquifer is evident. However, water washing is also recognized in Eocene reservoirs and in areas where the Malmian aquifer is missing. This shows that existing flow models for the regional Malmian aquifer have to be modified. Therefore, the results emphasize the importance of combining data from the petroleum and geothermal industry, which are often handled separately.

## ORAL PRESENTATIONS

### Studying various processes with $\delta^{13}C$ and $\delta^{18}O$

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**Abstract**

Stable isotopes are a useful tool to provide insights into different biological, ecological or geological processes. They are also providing information to analyze the underlying mechanisms on different scales ranging from laboratory discreet measurements to global scale continuous measurements.

Using the field-deployable Thermo Scientific Delta Ray Isotope Ratio Infrared Spectrometer with automatic calibration system, we measure the isotopic composition of $^{13}C$ and $^{18}O$ of $CO_2$.

We will be presenting you how this simple and easy to use instrument can be used in various applications from industrial to academia. We will go in details how the instrument is applied in environmental and life science research, biotechnology, food authenticity checking, and other applied fields.

POSTER

# Behaviour of stable sulfate isotopes in a coupled Cretaceous and Quaternary aquifer system, Lower Jordan Valley

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## Abstract

The Lower Jordan Valley comprises the transition zone between the Cretaceous aquifers and the Dead Sea system. Discharging Cretaceous groundwater into the Quaternary section of the Lower Jordan Valley is marked by the chemistry of inhomogeneous sediments, residual pore waters and ascended brines. Based on the composition of the distinct sulfur sources and sulfur depositions, $\delta^{34}S$ and $\delta^{18}O$ in the dissolved sulfate are analyzed to evaluate the groundwater origin, flow paths and mixture processes in the Cretaceous aquifers, Quaternary aquifer and discharging spring water (northern coast of the Dead Sea). Groundwaters in the recharge area stored in the Cretaceous aquifers show different $\delta^{34}S$ and $\delta^{18}O$ patterns due to the specific aquifer composition and groundwater-rock interaction: the upper aquifer represents the highest $\delta^{34}S$ (12 to 7 ‰) and the lowest $\delta^{18}O$ (to -0.4 ‰) isotopic signatures. Contrastingly, the lower aquifer tends to lower $\delta^{34}S$ (7 to -3 ‰) and higher $\delta^{18}O$ values (6 to 10 ‰). Further input of different sulfate sources in the Quaternary section can be identified by the specific isotope patterns in combination with $SO_4$/Cl ratios.

## POSTER

# Isotope patterns of water at different time scales in the Hydrological Open Air Laboratory (HOAL) Petzenkirchen

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## Abstract

Hydrological flow path information at a catchment scale is critical to the preservation of public water supplies. Knowledge of the hydrological and environmental processes underlying discharge formation, discharge concentration and associated transport of nutrients and pollutants in catchment areas is essential in order to construct predictive models to effectively manage water resources sustainably. In the light of climate and land use change these models will be used predictively at a policy level to address issues of flood protection, flood prediction, groundwater pollution as well as soils erosion and losses to the environment. To construct hydrological models and effectively predict and control flooding, we need to understand water dynamics at a cascade of scales both temporally and spatially.

The Petzenkirchen Hydrological Open Air Laboratory (HOAL) is located 100 km west of Vienna. The 64 ha catchment is dominated by agricultural land use and well equipped with numerous climatological, hydrological and water-quality measuring devices. The catchment is special in that many runoff generation processes (surface runoff, spring runoff, tile drainage, runoff from wetlands) can be observed simultaneously.

We present preliminary results of isotope patterns of precipitation and discharge at different time scales as well as component analysis for temporally high-resolved hydrological events.

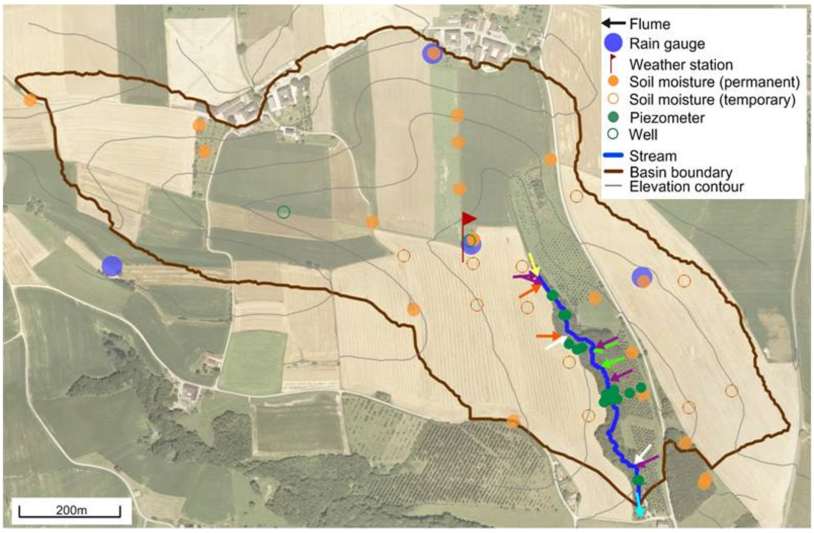

Figure 1: Catchment and instrumentation overview. Blöschl G et al., 2016: The Hydrological Open Air Laboratory (HOAL) in Petzenkirchen: A hypotheses driven observatory. Hydrol. Earth Syst. Sci., 20, 227-255. doi:10.5194/hess-20-227-2016.

POSTER

# Tracing stable isotopes ($\delta^2H$ and $\delta^{18}O$) from meteoric water to spring-groundwater in small catchments of the Vienna Woods, Vienna, Austria

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## Abstract

The isotopic ratios of oxygen and hydrogen in water ($^2H/^1H$ and $^{18}O/^{16}O$) are important tools to characterise waters and their cycles. To trace the recharge area of spring waters the mean annual $\delta^{18}O$ values in precipitation changes due temperature and fractionation factors with altitude and a representative fraction infiltrates in the ground without any changes. In this study, the characteristics of $\delta^{18}O$ and $\delta^2H$ in rain water and groundwater from two springs have been used to understand the transformation mechanism of rain water to groundwater. Two small recharge areas in the Vienna Woods (outskirts of Vienna) above Flysch sandstones and marls were studied. One spring drains a small catchment totally covered with an old beech-oak forest and the other one a catchment covered by lawns and partly by weekend cottages. The springs were sampled every month and the precipitation in monthly samples in a Palmex rain-collector close by over a period of three years (2013-2016). The altitude of the recharge areas is in the range of 310 – 464 m with a yearly precipitation sum of 593-754 mm. The mean temperature of this Pannonian climate range in this period from 2.2° C in January and 24.1° C in July.

Precipitation, stream water and groundwater from each site plot approximately along the $\delta^2H/\delta^{18}O$ slope ($\delta^2H=7.9x\delta^{18}O+7.4$) of local precipitation inputs. The spring water of the recharge area with mainly lawns and weekend cottages shows a clear seasonal variation between -11.84 ‰ in April and -9.99 ‰ in September. The recharge area with an old beech-oak forest shows a nearly constant $\delta^{18}O$-value of -11.0 ‰ in the spring water comparable to the mean of the winter half-year of the precipitation station. The isotope data and the considerable smaller discharge suggest that in the forested recharge area only precipitation water of the winter half year is added to the groundwater and rain water of the summer half-year is nearly totally transpired by the forest vegetation.

This indicates that shallow groundwater in forested recharge areas with moderate precipitation input cannot be traced by applying conventional altitude effects from precipitation data and estimates of the mean residence time (MRT) using the seasonal variation of the $\delta^2H/\delta^{18}O$ from precipitation stations are misleading.

POSTER

# The Austrian Network of Isotopes in Precipitation (ANIP) and its application in hydrology

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## Abstract

Austria runs one of the longest-standing and most dense isotope precipitation collection networks worldwide, resulting in unique isotope time series. Stable isotope variations in precipitation are a consequence of isotope effects accompanying each step of the water cycle. Therefore, stable isotope ratios of oxygen ($^{18}O/^{16}O$) and hydrogen ($^{2}H/^{1}H$) in precipitation provide important information about the origin and atmospheric transport of water vapour. Deuterium excess is a second-order isotopic parameter which is often interpreted as a tracer of the evaporation conditions of water vapor at the moisture source in terms of relative humidity, wind speed, and sea surface temperature, but can also be modified by local influences, such as below-cloud evaporation and equilibrium fractionation under very cold conditions. The separation of remote moisture source signals from local influences is thereby challenging.

The amount of precipitation in Austria is highly influenced by the Alpine mountain range (400-3.000 mm/a). The annual precipitation amount increases towards the mountain ranges. However, strong regional differences exist between the north and south of the Austrian Alps because the Alpine range acts as weather divide. The differences in $\delta^{18}O$-values of sampling stations at similar altitudes can be explained by the origin of the air moisture. An Atlantic influence causes lower $\delta^{18}O$-values than sources from the Mediterranean due to the different distances to the sea.

The isotope time series of the stations of the Austrian precipitation network show significant but not uniform long-term trends. While the 10-year running mean of some mountain stations exhibit a highly significant increase in $\delta^{18}O$ of about 1 ‰ since 1975, the change of $\delta^{18}O$ at the valley stations is less pronounced. The increasing $\delta^{18}O$ values can be correlated to an increase of mean air temperature in the Alpine area and can be used as an additional indicator of climate change in this region. The long-term variations of d-excess in precipitation at selected stations show a significant difference at mountain and valley stations.

Deuterium excess and $\delta^{18}O$ studies will be used to explore climate effects on precipitation signatures observed and to improve global climate models.

This unique isotope date base is also used in hydrology issues to provide a basis for decision making for sustainable use of water resources such as:

Calculation of the Mean Transit Time (MTT, “water ages”)

- Identification of water catchment areas to protect sources of drinking water
- Interaction between ground- and river water
- Environmental monitoring e.g. Tritium pulses in river waters from nuclear power plants or tritium contaminations from waste deposits
- Isotope based hydrograph separation to identify the different components (e.g. base flow, precipitation, water from the vadose zone) in a stream flow

POSTER

# Resampling of sedimentary proxy records to evenly spaced time-series using spectral control on the example of speleothem stable isotope records from Europe

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## Abstract

Uneven spacing of sedimentary paleoclimate records causes difficulties in the application of classical statistical and time series methods in many cases. Although dedicated statistical tools do exist to assess unevenly spaced data directly, the difficulty of transforming such data to a temporarily equidistant time series applicable to commonly used statistical tools remains. The aim of the study therefore was to introduce an approach to obtain evenly spaced time series (using cubic spline fitting) from unevenly spaced speleothem records with the application of a spectral control to avoid the spectral bias caused by interpolation and retain the original spectral characteristics of the data. The methodology was applied to stable carbon and oxygen isotope records derived from two stalagmites of the Baradla Cave (NE Hungary) dating back to the late 18th century and to a well-dated and high-resolution stable isotope records from the Han-sur-Lesse Cave (Belgium). To show the benefit of these equally spaced records to climate studies, their coherence with primary and complex climate indices is explored using wavelet transform coherence. The results shed light on clear relationships with climate and NAO indices, lending support to the approach utilized in this study. Moreover, these suggest that the Baradla composite stable isotope data can serve as regional reference records for the past ~200 years. The obtained equally spaced time series are available at doi: 10.1594/PANGAEA.875917.

POSTER

## Multi-annual monitoring in the Béke and Baradla caves (NE Hungary): implications for the conditions for the formation cave carbonates

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### Abstract

In order to use speleothems in the reconstruction of past climate and environmental changes it is necessary to understand the environmental and hydrological processes that determine the physico-chemical conditions of carbonate precipitation and hence speleothem formation. Therefore, in this study an extended monitoring program was conducted in the Béke and Baradla caves located in the Aggtelek region (Northeastern Hungary). The studied caves are rich in speleothems and flowstone occurrences with great potential for paleoclimatology studies. The monitoring activity included measurements of atmospheric and cave temperatures, $CO_2$ concentration in cave air, as well as chemical and isotopic compositions of water samples (drip water, precipitation) and in situ carbonate precipitates. The hydrogen and oxygen isotope compositions of drip waters showed no seasonal variation at any of the collection sites, indicating a well-mixed karstic aquifer. This implies that the isotopic compositions of local speleothems were able to record multiannual isotopic changes inherited from stable isotopes in the drip water. $CO_2$ concentration showed seasonality (high values in summer and low values in winter) in both caves, likely affecting carbonate precipitation or corrosion and consequently stalagmite growth. Systematic variations among Mg/Ca and Sr/Ca, Na/Ca, and Si/Ca element ratios were detected in the drip water suggesting Prior Calcite Precipitation (PCP). As PCP is characteristic of periods of reduced infiltration during drier weather conditions, the variations in drip water chemistry and drip rates indicate that the hydrological conditions also varied significantly during the studied period. This hydrological variability appears to affect not only trace element composition but also the isotopic composition of modern carbonate precipitates. In summary, these findings imply that the speleothems from the studied caves were able to record the hydrological changes resulting from alternating wet and dry periods, and therefore the geochemical data can be used to reconstruct past climate and environmental changes. Thanks for the financial support of the National Research, Development and Innovation Office (project No. OTKA NK 101664, PD 121387). György Czuppon also thanks for the support of the János Bolyai Research Scholarship of the Hungarian Academy of Sciences.

**POSTER**

## Multi-isotope approach to evaluate sources and fate of nitrogen in the Erlauf River catchment in Austria

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### Abstract

In the last decades the N-balance has been altered due to the extensive agricultural and urban development, which resulted in raising the nitrogen fluxes into surface and groundwater. Anthropogenic changes, like the extensive use of industrial fertilizers, have elevated inorganic nitrogen concentrations, deteriorating water quality, and causing eutrophication. Appropriate nitrogen assessment and management is still challenging for water resource managers and policymakers because of the unclear origin of nitrate in the watersheds characterized by the mixed land use. A deep understanding of the interactions between sources, microbiological transformations processes, and hydrological transport is crucial to predicting the effects of anthropogenic changes in the N-cycle on aquatic ecosystems.

Former studies conducted in river catchments deduced information about N sources and N transformation processes by the analysis of nitrate concentrations and corresponding isotopic signatures ($\delta^{15}N$, $\delta^{18}O$), but predominantly underestimated the advantages of multi-isotope approach or neglected the importance of hydrological conditions influence on N-cycling. The use of hydrological models and probabilistic methods such as Bayesian networks opens the opportunity to better estimate the nutrients fate on a larger scale. Although, without a reliable input data based on field studies and understanding of the custom hydrological conditions and ongoing processes, such models cannot produce trustworthy results.

Tracing nitrate in a regional scale is a challenging task, but the combination of multi-isotopic approach with hydrological modelling and probabilistic methods can answer the question what is the impact of different hydrological conditions (base flow and high flow) on the N sources contribution and N-turnover processes. The present study examines the Erlauf River catchment with an area of 525km2 in Alpine foothills in Austria. The seasonal surface water monitoring programme started in 2012. The approach based on the combination of multi-isotope techniques ($\delta^{15}N_{NO3-}$, $\delta^{18}O_{NO3-}$, $\delta^{2}H_{H2O}$, $\delta^{18}O_{H2O}$, $\delta^{34}S_{SO42-}$, $\delta^{18}O_{SO42-}$ and $\delta^{13}C_{DIC}$) with data on water chemistry, hydrological modelling, and Bayesian isotope mixing model, is used to quantify the contribution of different N sources in stream water during contrasting hydrological conditions, and to address subsequent biogeochemical processes.

## POSTER

# Rhine river plume tracking with stable hydrogen and oxygen isotopes in Lake Constance

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## Abstract

Lake Constance is an intensively used lake (e.g. fishing) and is also serving as drinking water reservoir for more than 4 million people. The main tributaries of the lake (Rhine, Bregenzerach) are the dominant influence on the physical and chemical properties of the lake and thus a potential source for pollutants. The water body circulation of the lake have been intensively examined and modelled, but the river plumes, e. g. intrusion depth and mixing behaviour of the main rivers input, are not yet well known. The BMBF–ReWaM Project “SEEZEICHEN” was established with one focus on the investigation of propagation paths of these river plumes. River water and lake water each show characteristic stable isotope signatures, so it is expected that where mixing of both occurs there will be a significant change in the isotope signature of the lake water as has been shown by Halder at al. (2013) for the Lake Geneva with well comparable conditions. The aim of the project is to examine the expansion, thickness and mixing of the river plumes through Lake Constance by a proxy package combining isotope and element measurements for the identification of river water. These measurements involve the stable isotope composition of $^{18}$O and $^{2}$H of water as a natural conservative tracer, the principal chemical composition (Cl, $SO_4$, Mg, Ca, Na) and the in situ investigation of physical parameters (electrical conductivity, temperature, oxygen) using multi parameter probes.

To identify river plumes in Lake Constance at 4-7 transects through the Lake each with 4-6 stations depth profiles has been sampled in October 2016, February 2017 and July 2017. At these sites vertical profiles have been measured with a multi parameter probe and water samples for ion concentration and isotope signatures were analysed in selected depths. The isotope signatures have been measured on board with Wavelength-Scanned Cavity Ringdown Spectroscopy (WS-CDRS). To gain a better insight on the input signal of the incoming river monitoring stations (Rhine, Bregenzerach) and the outflow (Seerhein) from the Lake Constance region has also been sampled including long-term isotope data from the river Rhine from the official Austrian and Swiss stations (UBA, BAFU).

Results from the three measuring campaigns are presented, showing interflow layer in different water depths depending on the season and river water discharge. The Rhine River is loading with isotopic light melt water in the summer months, which is mixing with the homogenous heavier isotope signal of the lake water body. The specific isotopic compositions of the water body and the rivers indicate an isotopic stratification in the metalimnion between 15-30 m during summer and fall. The river water layer is in about 20m and can be tracked over the whole lake until the Überlinger Lake part. The intrusion depths of the river plume are depending on the temperature and ion concentration (salinity) of river and lake water. In winter no river plume was detectable because the lake showed no stratification both lake and river had no significant differences in temperature and ion concentration and could be directly mixed. A previous study (Roßknecht el al., 2003) used chloride concentration as a tracer to identify river plumes in the Lake Constance. Our study found that the isotope composition of the Rhine and Lake Constance allowed the tracking of their plumes over the whole lake even other parameters like temperature, conductivity or chloride do not indicate an intrusion. A quantitative estimation of the river mixing part can be expected. The results show that stable isotopes are representing an “ideal” conservative tracer to detect and quantify river water plumes in Alpine lakes with input originating from high mountainous areas.

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UBA (2017): Isotope data from ANIP

BAFU (2017): Isotope data from ISOT

POSTER

## Stable Isotopes in Precipitation.

## A Case Study on Daily Analyses at Sonnblick Observatory (3.106m) and in Wals Isolab (446m), Salzburg, Austria

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### Abstract

Since autumn of 2016, at weather stations Sonnblick Observatory and Wals (Isolab) daily precipitation probes are analyzed for oxygen 17, oxygen 18 and Deuterium against SMOW. The daily measurement concept allows determination of extreme values, the complete fluctuation range and avoids alteration by evaporation in collection buckets.

The range of $\delta^{18}$O lies between -4,55 and -23,91 ‰ at Sonnblick Observatory and 3,33 and -20,58 ‰ at Wals, the weighted average of time span July 2016 to July 2017 is -13,47 (N = 145) and -9,50 ‰ (N = 159), respectively. The values for Deuterium ($\delta^2$H) are -97,76 (D-Excess: 10,0) and -68,03 ‰ (D-Excess: 8,0); and for $^{17}$O -7,07 and -5,03 ‰.

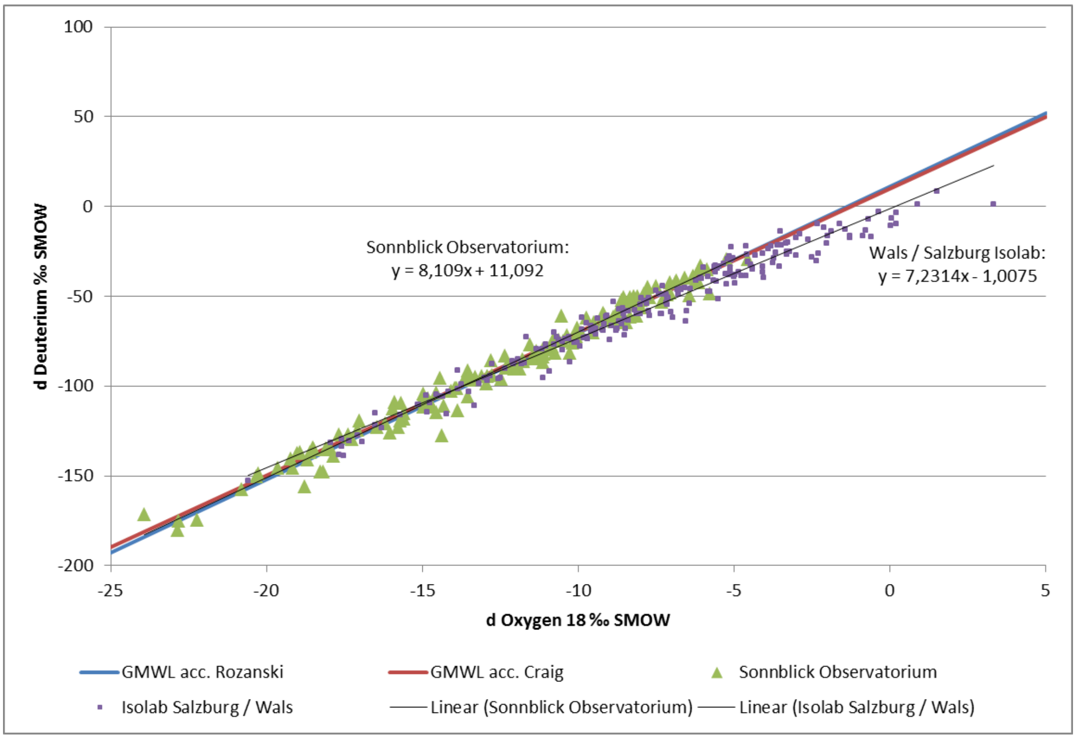

Fig. 1: MWL according [2] and [3].

This first year of continuous monitoring of precipitation isotopes allows some preliminary conclusions:

- Continental and altitude effects are slightly overprinted by the origin of the precipitation. The effects would be too low if interpreted without considering a higher contribution of Adriatic meteorology at central alpine Sonnblick observatory.

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- The meteoric water line fits well to the GMWL at Sonnblick Observatory (D = 8,1 $^{18}$O + 11), whilst at Wals Isolab some kind of shift can be observed (D = 7,2 $^{18}$O - 1). This shift disappears by filtering precipitation to events > 3 mm/day (D = 7,9 $^{18}$O + 6,5).
- In comparison with long-term monitoring from Salzburg precipitation station (e.g. 188 measurements in monitoring period 1987-2002: -10,08 ‰ $^{18}$O / -70,34 ‰ D, [1]), the water in the current observation period seems to be enriched slightly in $^{18}$O and D.

The program will be continued with the purpose of obtaining a longtime stable isotope monitoring database and shall be extended with additional strategic weather stations in alpine environment.

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POSTER

# Natural and anthropogenic changes in a lake-forest system in South Bukovina based on stable C and N isotope ratios and lipid biomarkers since 1340 A.D.

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## Abstract

Lake Bolătău-Feredeu is a small (lake surface: 0.3 ha; catchment area: 31 ha) landslide dammed lake in Bukovina (Eastern Carpathians, Romania). Elemental concentration and stable isotope composition of carbon and nitrogen and n-alkane composition of saturated hydrocarbon fraction of the organic material were analyzed along the sediment profile extracted from Lake Bolătău -Feredeu covering the past ~700 years.

This research is one of the first in this region focusing on organic geochemistry of young lake sediment sequence. The biological productivity in the lake-catchment system respond to the environmental changes now and did it also in the past.

The sediment chronology was established including $^{210}$Pb ages for the top 20 cm (Bihari et al., 2017) and 8 AMS radiocarbon dates from terrestrial macrofossils (Mindrescu et al., 2016) into a Bayesian age-depth model using the P_Sequence function of the OxCal v.4.2 (Bronk Ramsey, 2009).

In this study the used proxies for the interpretation were: $\delta^{15}N$; $\delta^{13}C$; C/N (atomic ratio); C%; N%; n-alkane indices: $TAR_{HC}$; $P_{aq}$; $P_{wax}$; $P_{hw}$. Stable nitrogen isotope record was corrected for diagenetic trend (Brahney et al., 2014).

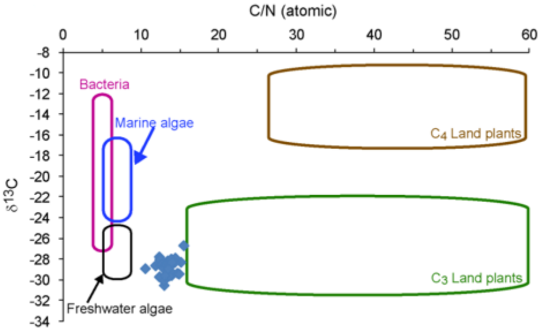

Figure 1: Cross-plot of $\delta^{13}C$ and C/N atomic ratios of organic matter of the sediment of Lake Bolătău -Feredeu (blue dots) and the fields of potential source materials in the aquatic environment.

The ranges of $\delta^{13}C$ values (−30.5 to −26.6 ‰) and C/N atomic ratios (10.5 and 15.4) placed the organic material of the recent Bolătău -Feredeu sediment between the $C_3$ Land Plants and the lacustrine algae (Fig. 1.). The n-alkane proxies help to make a better separation of the organic matter inputs. Detected major shifts in the n-alkane composition and the C and N concentrations together with the stable isotope compositions pointed out three distinct stages in the environmental history of the lake-catchment system.

- Modern landscape change (~1820-2013): The proxy information point to a substantial landscape change started by a deforestation and increase of open pastures with herbaceous plants.
- Variable lacustrine environment decreased productivity in the catchment (c. 1640 – 1760) The C/N, $\delta^{15}N$, and $\delta^{13}C$ values showed the highest variability. It can be assumed that extended periods of lake ice cover during the cold decades experienced at the turn of the 17-18th centuries were translated to diminished biological productivity in the lake and its catchment.

## POSTER

- Afforestation and decline in lake productivity (~1470 – 1560): The $\delta^{13}C$, $\delta^{15}N$, and C% reached a peak, but the C/N ratio decreased suggesting increased lacustrine contribution. In the n-alkane proxies the $TAR_{HC}$ had a small peak in this period indicating a higher terrestrial contribution. The $P_{wax}$ index rose to a higher level while the $P_{hw}$ reached a lower level. This information indicated an afforestation effect in this region.

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POSTER

# Establishment of the lab protocol for simultaneous determination of stable isotope composition of carbon, oxygen, and non-exchangeable hydrogen in cellulose samples using a heated vacuum-equilibration device

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## Abstract

Simultaneous analysis of carbon-, oxygen-, and hydrogen-isotope composition of cellulose samples can offer better insights into plant physiological processes. An obstacle plant-based physiological, palaeoclimatological, archaeological, and forensic studies are usually facing is the strongly limited sample material prohibiting replicate analyses for each isotopes.

A commercial vacuum equilibration system (Uni-Prep™, Eurovector, Milan, Italy) (Wassenaar et al., 2015) was coupled to a high-temperature elemental analyser interfaced with an isotope-ratio mass spectrometers at the Stable Isotope Laboratory of the Institute for Geological and Geochemical Research, Research Centre for Astronomy and Earth Sciences, MTA . When cellulose samples are pyrolyzed at high temperature (>1400 °C) the glucose-polymer will be converted C, CO, and $H_2$ making possible to determination of isotopic composition using continuous-flow isotope mass spectrometry (Leuenberger and Filot 2007, Loader et al., 2014). Agitating the cellulose samples with steam produced from water of known isotopic composition will facilitate isotopic exchange of the ~30% exchangeable hydrogen atoms (i.e. OH-bounded) in the cellulose polymer. After that the hydrogen-isotope ratio of the non-exchangeable fraction, can be also calculated (Filot et al., 2006).

Here we summarize the experimental results when equilibration time was increased with 30 min steps up to 2.5 hours, and repeating the experiment using a very depleted ($\delta^2H_{VSMOW}$:-841‰, $\delta^{18}O_{VSMOW}$:- 188.3 ‰) and a super heavy ($\delta^2H_{VSMOW}$:+1132‰, $\delta^{18}O_{VSMOW}$: -13.4 ‰) water for equilibration.

Based on the results the equilibration conditions have been set to 400 µL of $H_2O$ at 110°C for 1.5 hours in the lab protocol for triple-isotope analysis of cellulose samples.

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**POSTER**

# Impacts of nitrogen deposition on forest biogeochemical processes across a trans-European gradient investigated using a tool kit of stable isotope methods

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## Abstract

Meta-data analyses and the model based hypotheses state that global soil C storage is controlled by microbial scale processes of fungal competition for available nitrogen (N). Global trends of increasing atmospheric N deposition and the continuing use of inorganic N fertilizer in both agriculture and forestry mean that the soils vital function as a carbon sink is potentially under threat. We set out to experimentally investigate these hypotheses across a Trans-European gradient of forest soils and provide reliable information on soil microbial responses to nitrogen inputs for predictive climate change models.

Changes in soil nutrient status could result in a chain reaction of interacting microbial mechanisms which in turn could lead to the shifts in underlying ecosystem biogeochemical process rates. Recent meta-analysis has shown that plant fungal symbiont community structure, exerts a greater fundamental control over soil C storage than temperature, precipitation or net primary production. Based on the hypothesis that plant associated fungi effectively scavenge all available organic and inorganic N leaving little N for the growth of the free-living decomposer microbial community and preventing further breakdown of SOM.

The "Trans-European Nitrogen Deposition Experiment" (ALTER-net-MSII network). is embedded in the project „Functional response of forest ecosystems to Nitrogen deposition and climate change"

The objectives of this experiment are to measure the impact of nitrogen fertilization on carbon decomposition, particular organic matter residence time and carbon use efficiency across a biogeographic gradient. Experimental plots were set up in a random block design in Austria, Germany, Sweden and the United Kingdom.

We have set up an experiment in which a series of dual isotope labelled C and N in-growth beech litter bags have been incubating in-situ in the forest. Moreover the treatment plots have received additional inputs of inorganic nitrogen fertilizer over an eight year period. We have studied both nitrogen and carbon dynamics in these systems using a tool box of stable isotope techniques.

POSTER

# Biochar: NET-2-U. Biochar: Negative Emission Technologies are nice to the environment Using stable isotopes in a citizen science approach

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**Abstract**

The aim of this project was to communicate FWF funded and further biochar research findings to a wider public through a participation model of communication. Making the public aware of nature-based negative emission technologies (NETs), specifically biochar addition to soil as a viable climate change combating strategy. In creating this interest so we wanted to engage the public in a horizontal dialogue on climate change and inspire attitude and behavioral changes. We wanted to highlight and publicize the role science plays in ensuring that these potential technologies are risk free and are grounded in evidence based findings.

Moreover we wanted to offer the opportunity of participating in their own experiment, so at each event we encouraged the public to participate in our pot-scale Citizen Science endeavor.

In temperate Austria we have identified that biochar could be particularly useful in extreme drought years. We saw ten percent greater yields in biochar amended soils than in control soils in the very dry year of 2011. We set out to investigate if this increase in soil water holding capacity is a generic property of adding wood-based biochar to different soils across Austria. There is preliminary evidence that biochar can reduce water usage and increase resilience to extreme heat events whilst capturing significant quantities of carbon and improving nutrient and soil retention. We wanted to test this hypothesis in using a citizen science approach and provide scientific evidence of biochar's water use improving properties in a wide range of Austrian soils. We used the simple $\Delta^{13}C$- stable isotope method to assess plant water use efficiency (WUE). The beauty of this method is that it allowed us to assess WUE over the growing season and that it is incredibly simple to apply, in a citizen science context. Moreover it gave the citizen scientists the chance to run the experiment from seed to harvest and observe the effects of the biochar directly in their garden or on their balcony. But it also gave us the chance to collect useful data on the impact of biochar WUE across a range of soils. In doing so it provided an entry point for the citizen scientist's into the more technical world of stable isotopes and allowed them to expand their knowledge in this area. The action therefore functioned at two levels, the more basic level of growing and seeing the effects of biochar on the plants for the children, with easy to conduct experiments about soil, needs of plants etc., but also at a higher level for the parents. In these experiments we wanted to create an awareness of stable isotopes and their vital role in advancing soil and climate science.

Finally we collected, collated, discussed and interpreted the data bringing together all social actors and Citizen Scientists in a Biochar Day where we fostered a global outlook by making tangible links to on-going projects in developing countries.

The results and conclusions from this project will be presented.

POSTER

# Consequences of climate change for agroecosystem carbon and nitrogen cycling – an isotope labelling of green manure approach

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## Abstract

Climate change will have an impact on soils, with consequences for organic matter turnover and soil fertility. A useful method to trace the fate and behaviour of nutrients in agricultural soils is through soil amendments of isotopic enriched green manures. In this study, **white mustard** (*Sinapis alba)* will be labelled with **$^{13}$C** and **$^{15}$N isotopes**, and then transferred as a green manure to the crops planted in long term lysimeter trials – which consists of 18 lysimeters including three different types of soils (Sandy calcaric chernozem, Gleyic phaeozem and Calcic chernozem, representing the Marchfeld region) and two different precipitation regimes (current situation and a future climate based on a the slightly pessimistic scenario RCP6.0 according to the 5th IPCC Assessment Report) are used.

The overall aim of the study is to analyze how different precipitation regimes and soil types affect the nutrient turnover in soil and plant uptake. The study presented here will contribute to a better understanding of the soil-water-plant interaction processes under changing conditions and will allow for a better planning of adaptation measures to mitigate climate change effects.

As a first step, wheat from the lysimeter station was harvested to measure differences in plant biomass under different climate conditions and soil types and to analyse the C/N ratio and the $\delta^{13}$C and $\delta^{15}$N values of leaves stalks and grain using isotope ratio mass spectrometry. In further experiments, white mustard will be dual labelled with $^{13}$C and $^{15}$N isotopes under controlled conditions and used as green manure for the next crop at the lysimeter station.

First results showed that lower precipitation of the future climate regime decreased plant biomass and increased $\delta^{13}$C indicating drought stress. Lower precipitation also slightly decreased $\delta^{15}$N in the leaf but not the C/N ratio. Different soil types showed differences in the C/N ratio and the $\delta^{15}$N, with lower $\delta^{15}$N values and higher C/N for the calcaric chernozem. The soil type did not affect wheat biomass production. Next steps will clarify how climate and soil type affect the nutrient turnover in agricultural soils and therefore allow for a better planning of adaptation measures.

POSTER

# Determination of carbon isotope enrichment factors of metabolites after precursor amendment- illustrated by the example of chlorinated ethene degradation

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## Abstract

Bacterial reductive dechlorination of the groundwater contaminant tetrachloroethene (PCE) involves the formation of lower chlorinated metabolites. Metabolites can be instantaneously formed and consumed in this sequential process; quantification and validation of their isotopic effects conventionally rely on separate laboratory microcosm studies. Here, we present an evaluation method enabling the determination of the carbon isotope enrichment factor ($\varepsilon$) for the intermediate cis ‐ dichloroethene (cis ‐ DCE) by a single laboratory microcosm study initially amending the precursor PCE only.

Environmental samples harboring organohalide ‐ respiring bacteria were incubated under anaerobic conditions and then successively and repeatedly amended with PCE and cis ‐ DCE in two separate laboratory microcosm studies. Reductive dechlorination was monitored by analyzing liquid samples using purge and trap - gas chromatography - isotope ratio mass spectrometry. The prerequisites of the presented evaluation method are mass and $\delta$ ‐ value balancing. The evaluation method was validated by agglomerative hierarchical classification of Rayleigh plot data points.

The sample ‐ sensitive range of $\varepsilon_{\text{cis - DCE}}$ extended from –10.6 ± 0.2‰to –26.8 ± 0.6‰ (R2 ≥98%). The maximum standard deviations of $\varepsilon_{\text{cis - DCE}}$ were ±1.8‰ for single microcosms, ±1.8‰ for replicates and ±1.0‰ for the compiled replicate data of PCE and cis ‐ DCE amendments. A linear regression of the $\varepsilon_{\text{cis - DCE}}$ for replicates obtained by each amendment study showed a slope of 95% (5 of the 7 data points are within a 95% confidence interval), demonstrating factor congruency and the practicability of the evaluation method.

We found metabolite degradation and formation to be sequential but also stepwise during bacterial reductive dechlorination. The stepwise phases of the degradation of the intermediate eliminate the impact of instantaneous precursor degradation. These stepwise sections were used to determine $\varepsilon_{\text{cis - DCE}}$ ‐ values. Our results showed the validity of $\varepsilon_{\text{cis - DCE}}$ ‐ values over a wide range at initial precursor degradation (PCE). The presented evaluation method could substantially decrease lab costs for microcosm studies designed for $\varepsilon_{\text{cis - DCE}}$ determinations. Moreover, the results indicated that the evaluation method can be applied to other PCE ‐ metabolites.

POSTER

## Project MINTOx –
## Microbial nitrogen transformation during waste degradation

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### Abstract

Landfills and old waste deposits pose a significant risk potential for humans and the environment. Leachate emissions are the main reason for this since their pollution burden will remain at an environmentally incompatible level for hundreds of years. In particular, the persistence of Ammonium-Nitrogen in the leachate constitutes one of the most significant long- term pollution problems. In-situ aeration of landfills is considered as practical means not only to reduce environmental impacts emanating from landfills containing organic waste but also to shorten the necessary post-closure period of MSW landfills. Besides the reduction of pollutants present in the leachate (e.g., COD, $NH_4$) also the avoidance/minimization of landfill gas emissions (in particular methane) and therewith associated risks are major arguments for the aeration of old landfills. Several studies[1][2] show a significant reduction in emissions emerging from such sites during in-situ aeration. However, it remains under question if this observation results from a sustainable decrease in accessible nitrogen or nitrogen is simply incorporated in biomass and thus only temporarily inaccessible. That later bears the risk that after ending aeration an increase in N loads has to be expected.

There is thus a need to determine the long-term impact of in-situ aeration on the emissions of old landfills in general and the metabolism of Nitrogen in particular. For this purpose, landfill simulation reactors (LSR) are operated under different aeration regimes. Measurements quantifying nitrogen remaining in the waste material as well as discharged over leachate and off- gas are conducted. The possibility of characterising future emissions is mainly based on knowledge regarding the importance of individual microbial conversion processes. Consequently, an incubation experiment with isotope-labelled material ($^{15}N$) is carried out and affected microbial communities are quantitative and qualitative detected.

Besides a better understanding of the long term metabolism of Nitrogen in landfills, project MINTOX is aiming to provide detailed Nitrogen balances for landfills also in order to optimize existing remediation measures. In-situ aeration reduces methane emissions of landfills dramatically. An increase in formation of nitrous oxide[3] might be the consequence, whereas optimized operation results in an overall minimization of greenhouse gases.

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POSTER

# Imported or already locally native? Stable isotope investigation of invasive tree pests can differentiate between pests grown in local trees (Austria) from pests grown elsewhere: a pilot study of *Monochamus*

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## Abstract

Global trade, imports and exports leading to shipments of vast amounts of goods between continents also resulted in spreading of unwanted stowaways: Invasive pests. Upon detection of such an alien pest in a trap there is an important question: Has this passenger just left its transport vehicle, or have already previous generations been introduced and the detected specimen is already a native local? This question is paramount, as it decides about the consequences of the detection: 1) just alerted surveillance, or 2) eradication measures.

In our study Monochamus specimens from Austria, Portugal, Poland and Russia have been investigated for their stable isotope patterns. *Monochamus* is important mainly due to the fact that it is the dominant vector for the pine wilt nematode, which is very common in North America, eastern Asia (China, Korea, Japan) and Portugal. We demonstrate that pests from different origin have significant differences in their isotope signature and can thus be differentiated, although a separation of samples from Austria and Poland is only incomplete. However, as the isotope signature of *Monochamus* from the regions of main concern (e.g. east and south-east Asia, and Northern America) can be assumed to stronger differ from the Austrian isotope pattern than the Portuguese isotope signature, which could be completely separated, this method is a potent tool to differentiate between “newcomer” and “locally-native” invasive pests and thus help to decide about the adequate counter-measure.

POSTER

# Impacts of habitat changes on food availability for the Warbler finch in the Scalesia Forest, Galápagos

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## Abstract

Darwin's finches in the Galapagos Islands have been affected by the presence of exotic invasive fly species, *Philornis downsi*, which parasitises the hatchlings and causes high mortality. Moreover, the finches' native habitat, *Scalesia* forest, has been heavily invaded by a raspberry species *Rubus niveus,* altering the forest understory conditions. There has been collaborative research on developing measures to control these invasive species. Three areas have been set-up to test the impact of specific *Rubus* management strategies resulting in three treatment areas of different control intensity (C3, C1, SC, long-term management, short-term management and no management, respectively). In this research, we set out to track the consequent changes in the dietary resources used and available to the Warbler Finch (*Certhidea olivacea*), across the breeding seasons (start and finish) in two years (2015 and 2016) and these treatments using traditional and stable isotope approaches. We analysed nitrogen ($\delta^{15}N$) and carbon ($\delta^{13}C$) in the birds' blood as indicators of resource use change and arthropod samples as indicators of the potential diet available. We aim to determine whether the birds exhibit dietary dependence on what is available or whether there are some selectivity and choice according to the availability and the quality of food. We also set out to investigate how the food-resource (arthropods) quality is, in turn, influenced by the quality of the vegetation and the precipitation. Our results suggest that the *Rubus* management "treatment" affects the finches' nutrient intake significantly. Changes in $\delta^{13}C$ are significant in the treated areas throughout seasons. In the area without treatment, there are no significant differences along time. Conversely, $\delta^{15}N$ in the area without treatment, $\delta^{15}N$ seems to increase with time. Areas with treatment show significant differences but fluctuating along time. We compared this results with the food resources patterns. Blood signatures overlap whether partially or entirely with arthropod samples suggesting that both have similar patterns under the same conditions. Moreover, arthropods showed greater variability, suggesting diversity on arthropod guilds and a broader pool of food resources, hence the partial overlaps. Finally, we analysed arthropod orders individually to seek dominant diet components. From our results, it seems that Warbler Finch present diet selectivity depending on the quality of food supply. Araneae, Hemiptera, Hymenoptera, Lepidoptera and Orthoptera are dominant components of the Warbler finch diet.

**POSTER**

## Tracing Hg transformations in contaminated soil using Hg isotopes

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### Abstract

Mercury (Hg) undergoes biogeochemical transformations whose products differ drastically in their bioavailability, mobility and toxicity. Knowing Hg speciation in natural systems is thus key to understanding its risk. The seven stable isotopes of Hg vary in their abundances in environmental samples. Mass-dependent and mass-independent isotope fractionation have also been shown to take place during biogeochemical reactions in nature and the laboratory. This allows for a new application: to identify and potentially quantify Hg transformation pathways and processes. Hg stable isotope ratios have been measured by CV-MC-ICP-MS [1] on vadose zone soil samples at a former industrial site in SW-Germany [2] to better understand the transformations of the pollutant $HgCl_2$ in the system. Despite oxidizing conditions in the bulk soil, $HgCl_2$ is partly reduced to metallic Hg(0). On two soil cores, total soil digests and sequentially extracted Hg pools were analyzed for Hg concentrations, Hg isotope ratios and speciation based on pyrolytic thermo-desorption. Digests of the most contaminated soil samples (up to 800 mg $kg^{-1}$ Hg) showed $\delta^{202}Hg$ values within the expected source signature of industrial Hg of around -0.43±0.09‰ (2SD, relative to NIST-3133). Among the sequentially extracted Hg pools within the samples, Hg isotope ratios varied significantly. Contaminated groundwater from a close-by well was strongly enriched in heavy isotopes (+0.75‰), contrasting with the light bulk soil signatures. Among the most Hg-rich soil samples, which contained a mixture of metallic Hg(0) and Hg(II) phases, it was the $H_2O$-extractable and most mobile fraction which was heaviest, exhibiting $\delta^{202}Hg$ values of up to +0.18‰. A series of sequential water extracts revealed slow dissolution kinetics of mobile Hg pools from the soil, continuously releasing isotopically heavy Hg into solution. The identified Hg pools showed significant Hg isotope fractionation among themselves, and also between different sampling depths of the soil cores. Our results demonstrate the potential of Hg isotopes to be useful tracers of Hg transformations in contaminated soil systems.

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POSTER

# Hg isotope analysis and sequential extractions of industrially contaminated soils in Valais, Switzerland

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## Abstract

The partitioning of mercury (Hg) into different pools within soils and sediments controls its fate and behaviour, but characterising these pools is a challenge. We examined industrially contaminated soils and sediments from Visp, Switzerland, by combining stable Hg isotope analysis and sequential extractions. Previous work suggested that the samples (up to 150 mg $kg^{-1}$ Hg) contained no elemental and only minor sulfide-bound Hg pools, and were dominated by Hg(II) species presumably bound to organic matter (OM) and/or mineral surfaces. We employed an extraction sequence of (1) water (for labile mercury), (2) NaOH or $Na_4P_2O_7$ (for organically bound Hg), (3) $NH_2OH$-HCl (for Mn and Fe (oxyhydr)oxides), and (4) aqua regia (residual pools). Hg isotope ratios were measured using CV-MC-ICP-MS. The proportions of the total Hg pool extracted by water (≤1.5%), NaOH (≤36%), $Na_4P_2O_7$ (≤18%), and $NH_2OH$-HCl (≤1.0%) were relatively small. Although both NaOH and $Na_4P_2O_7$ primarily dissolve OM and the Hg bound to it, differences between the two extractions indicate that they also attack other Hg pools. Hg associated with sulfide and/or Fe and Mn (oxyhydr)oxide colloids, were likely important in some samples and were extracted more efficiently by $Na_4P_2O_7$ than NaOH. The Hg that was not removed by the first three steps in the extraction procedure may be bound to recalcitrant OM or crystalline minerals. A series of consecutive H2O extractions showed that labile Hg species are released slowly. Surprisingly and in contrast to previous findings [1-3], Hg isotope signatures in sequential extracts and bulk digests were mostly indistinguishable (2SD ≤ 0.14‰). However, $NH_2OH$-HCl extractions produced heavy MDF and negative MIF values (max. difference from bulk: $\delta^{202}$Hg = +0.87‰, $\Delta^{199}$Hg = -0.15‰), presumably an extraction artefact caused by reduction of Hg(II) by Fe(II) followed by volatile Hg(0) loss. Our results provide insight into the action of sequential extractions and develop the application of Hg isotopes to understanding Hg partitioning in environmental media.

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