

Benelux Association of
BASIS
Stable Isotope Scientists

Annual Meeting 2017
May 3 & 4

Utrecht, The Netherlands



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Preface

Dear members of BASIS and attendees,

This booklet shows once again that the stable isotope community in the Benelux remains active. We hope to provide you again an exciting scientific and social program.

Also this year we highly appreciated your scientific contributions allowing us to provide an attractive program for the BASIS 2017 annual meeting, which follows BASIS 2015 as in 2016 no real BASIS meeting was organized because of the JESIUM 2016 event in Gent, Belgium.

The board is thankful and proud to be supported by a large number of industrial partners: *Thermo Fisher, van Loenen instruments/Sercon, Elementar, Campro Scientific, IVA, Buchem, Euriso-Top, IsoLife* and *OEA Labs* and *Air liquide (Expertise centre)*

We hope you are happy with the BASIS organization and the local organizers.

On behalf of the board of BASIS, I wish you all a very pleasant meeting.

With kind regards,

Pascal Boeckx

President

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BASIS 2017 Annual Meeting

May 3 & 4, 2017

Utrecht, The Netherlands

Wednesday, May 3

10:00 – 12:00 Arrival and registration of industrial partners
Arrival and registration of attendees/members

12:00 – 13:00 **Lunch and sponsor trade show**

13:00 – 13:15 **OPENING BASIS 2017**

Session 1 **Chairman: Ivonne Nijenhuis**

13:15 – 13:45 **Forensic in-depth investigations using IRMS and ICPMS results.**

Gerard van der Peijl, (Netherlands Forensic Institute, The Hague)

13:45 – 14:05 **Plant phenological water cycle and implications for using $\delta^2\text{H}$ -alkanes as paleo proxy in a semi-arid tropical climate**

Lien De Wispelaere (ISOFYS, Gent)

Candidate for Young Scientist award

14:05 – 14:25 **Chromatography-based EA-IRMS : Redesigning the combustion elemental analyzer around modern chromatographic principles**

Andreas Hilkert, (Thermo Scientific Bremen, Germany)

14:25 – 15:05 **COFFEE BREAK - TRADE SHOW – POSTER SESSION**

Session 2 **Chairman: Kristin Verbeke**

15:05 – 15:25 **Brain Plasticity: Measuring Protein Synthesis Rates in the Human Brain**

Joey SJ Smeets, (Maastricht University Medical Centre)

Candidate for Young Scientist award

15:25 – 15:45 **Using the $\delta^{13}\text{C}$ of general biomarkers at CO₂ vents on Shikine Island, Japan to calculate past pCO₂**

Caitlyn R. Witkowski (NIOZ and Utrecht University)

Candidate for Young Scientist award

15:45 – 16:05 **An LC-IRMS Interface for Flexible Compound-specific Stable Isotope Analysis**

Filip Volders (Elementar)

16:30 – 18:00 **Activity**

18:30 – 23:00 **Reception/ Dinner Stadskasteel Oudaen
Oudegracht 99
3511 AE Utrecht
Tel: 030 231 18 64**

Thursday, May 4

Session 3

Chairman: Marcel van der Meer

9:00 – 9:20

Effects of climate and geochemistry on soil organic matter stabilization and greenhouse gas emissions along altitudinal transects in different mountain regions

Marco Griepentrog (ISOFYS, Gent)

9:20 – 9:40

Compound specific hydrogen isotope measurements of algal chemical fossils

Gabriella Weiss (NIOZ, Den Burg)

Candidate for Young Scientist award

9:40 – 10:00

Latest Developments in 10 kV Isotope Ratio MS

Andreas W. Hilkert, (Thermo, Bremen)

10:00 – 10:40

TRADE SHOW – POSTER SESSION

Session 4

Chairman: Klaus Wutzke

10:40 – 11:00

Unusually high sea ice cover influences resource use by benthic invertebrates in coastal Antarctica

Michel, L. N. (University of Liège)

11:00 – 11:20

Post-prandial protein handling following ingestion of different amounts of protein during post-exercise recovery in older males

Andrew M. Holwerda, (Maastricht University Medical Centre)

Candidate for Young Scientist award

11:20– 11:40 **A 15N tracing technique to disentangle the gross dynamics of nitrogen in tropical forest soils**

Marijn Bauters, (ISOFYS, Gent)

Candidate for Young Scientist award

11:40 – 12:30 **LEDENVERGADERING – Members meeting 2017**

12:30 – 13:45 **Lunch and sponsor trade show**

Session 5 **Chairman: Samuel Bodé**

13:45 – 14:05 **Characterisation of microbial dehalogenation of halogenated organic contaminants using compound-specific stable isotope analysis**

Ivonne Nijenhuis, (Helmholtz-Centre for Environmental Research, Leipzig)

14:05 – 14:25 **Deuterated water dosing and measurement of skeletal muscle protein synthesis rates *in vivo* in humans**

Joy P.B. Goessens (Stable Isotope Research Center (SIRC), Maastricht)

14:25 – 14:45 **The effect of resistant starches on fat oxidation in healthy adults as measured by a 13CO₂-breath test**

Wutzke, Klaus (University Medicine Rostock, Children's Hospital)

14:45 – 15:30 **Young Scientist award - Closing - Coffee 'to go'**

Posters:

1/ Verification of the geographical origin of European plaice (*Pleuronectes platessa*)

Annemieke M. Pustjens,
RIKILT Wageningen.

2/ ^{13}C -labelled polysaccharide fibers as tracers in health research

Ries de Visser, Ton Gorissen.
IsoLife b.v., Wageningen.

3/ Factors affecting carbon dioxide evasion and degassing from three rivers and streams on Tibetan Plateau: $\delta^{13}\text{C}_{\text{DIC}}$ constraints

Xiao-Long Liu
Tianjin Normal University.

4/ The use of stable isotopes to study the origin of methane trapped in Antarctic sea ice

Caroline Jacques
Université libre de Bruxelles

6/ High temporal resolution measurements of the isotopic composition of CH_4 at the Lutjewad station, The Netherlands

T. Röckmann
IMAU, Utrecht University.

7/ Calibration Mixtures for Improving Accuracy of Stable Isotope Measurements

Tracey Jacksie
Air Liquide, Newark.

Abstracts oral presentations:

Forensic in-depth investigations using IRMS and ICPMS results

Dr. Gerard van der Peijl, Dr. Andrew van Es and Ing. Wim Wiarda
Netherlands Forensic Institute, Laan van Ypenburg 6, The Hague, The Netherlands.

IRMS and (LA-)ICPMS are used in combination as part of in-depth forensic casework investigations within the Netherlands Forensic Institute (NFI). Various applications were developed that demonstrate the strong discriminating power of this technique combination and the additional value it can provide for a forensic investigation to establish links with *e.g.* materials found at a crime scene and visually similar materials that are found with a suspect. One of the advantages of this technique combination is the versatility that allows for using IRMS and (LA-)ICPMS even for materials that have never been investigated before with these techniques.

Materials for which IRMS (mostly together with (LA-)ICPMS) has been used are various tape types, adhesives, paper, safe wall filling materials (combination of potassium alum and saw dust), jeans, motor oils, polyester trousers, polypropylene rope, matches, candles, arson accelerants, jerrycans, cosmetics, explosives, drugs. Some NFI applications will be discussed in the presentation.

Specifically challenging investigations relate to materials that have been in a fire. Even then, materials can often be characterized and linked to other relevant materials, *e.g.* for remnants of burnt jerrycans.

For serious crimes such as murders, results are used for different purposes in different phases of a large scale police investigation and the judicial process. Four phases can be distinguished: pre-crime marking of materials/tracers; forensic intelligence for police investigation; court evidence phase and (potentially) follow-up study. These phases will be described as well as how the evidential value of the information is determined. To apply results in court procedures, often the background variation of the characteristics needs to be determined since this is the first time ever these characteristics are determined.

Plant phenological water cycle and implications for using $\delta^2\text{H}$ -alkanes as paleo proxy in a semi-arid tropical climate

Lien De Wispelaere¹, Samuel Bodé¹, Pedro Hervé-Fernández^{1,2}, Andreas Hemp³, Dirk Verschuren⁴, Pascal Boeckx¹.

¹Isotope Bioscience Laboratory – ISOFYS, Faculty of Bioscience Engineering, Ghent University, Coupure links 653, 9000 Gent, Belgium. ²Laboratory of Hydrology and Water Management, Faculty of Bioscience Engineering, Ghent University, Coupure links 653, 9000 Gent, Belgium. ³Department of Plant Systematics, University of Bayreuth, 95440 Bayreuth, Germany. ⁴Limnology Unit, Department of Biology, Ghent University, Ledeganckstraat 35, 9000 Gent, Belgium.

Lake Challa is a steep-sided crater lake situated in equatorial East Africa, a tropical semi-arid area with bimodal rainfall pattern. The $\delta^2\text{H}$ and $\delta^{18}\text{O}$ of precipitation, lake water, groundwater, plant xylem water and plant leaf water were measured across different plant species, seasons and plant habitats in the vicinity of Lake Challa, as well as the hydrogen-isotopic composition of leaf wax n-alkanes ($\delta^2\text{H}_{\text{wax}}$). Long chain n-alkanes of terrestrial plant leaf waxes provide information on plant-water relations and have been widely used as proxy in paleoclimate and paleovegetation reconstructions. In our study, we found that plants rely mostly on water from the 'short rains' falling from October till December (northeast monsoon), as these recharge the soil pores after the long dry season. This plant-available, static, water pool is only slightly replenished by the 'long rains' falling from February to May (southeast monsoon), in agreement with the 'two water world' hypothesis according to which plants rely on a static water pool separated from a more mobile water pool that recharges the groundwater. Spatial variability in water resource use exists in the study region with plants at the lakeshore relying on water of different isotopic composition, i.e isotopically evaporated lake water at the lakeshore vs. non- or slightly evaporated precipitation in the savannah and on the crater rim. This spatial resource partitioning is recorded by elevated $\delta^2\text{H}$ values in the leaf wax lipids of plants at the lakeshore. The distribution of n-alkanes in the fresh leaves shows a unimodal distribution pattern reaching a maximum at n-C29 and n-C31 for both shrubs and trees, while C4 grasses are dominated by n-C31. However, the relative abundance of n-C31 was higher at the lakeshore compared to the savannah and crater rim (when grasses were not included). According to our results, plant species and their associated leaf phenology are the primary factors influencing the enrichment in deuterium from xylem water to leaf water, with deciduous species giving the highest enrichment; while growth form and season have negligible effects. Growth form exerted a strong influence on $\delta^2\text{H}_{\text{wax}}$, with more depleted values for C4 grasses compared to shrubs and trees. However, the variability on $\delta^2\text{H}_{\text{wax}}$ within the group of woody species remains large (range of ~ 100 ‰). The variability in $\delta^2\text{H}_{\text{wax}}$ with season was plant-specific and ranged from no effect of seasonality to total dependency of seasonality. Our observations have important implications for the interpretation of $\delta^2\text{H}$ of plant leaf wax n-alkanes from paleohydrological records in tropical East Africa, given that i) the water used by plants reflects only a small portion of the annual temporal variability in isotopic composition of precipitation and that ii) large variability on apparent isotopic fractionation is observed, though yet not fully understood.

Chromatography-based EA-IRMS : Redesigning the combustion elemental analyzer around modern chromatographic principles

Chris Brodie, Oliver Kracht, Andreas Hilkert

The elemental analyzer (EA) was invented by Justus Liebig in 1830 and is deeply rooted in analytical chemistry, but the steps to make it an analytical tool for biology and geochemistry came in 1968, when Carlo Erba replaced trapping of the gases from combustion with isothermal gas chromatography using a packed GC column. In 1980, when Professor Tom Preston put a Carlo Erba EA onto an IRMS, inventing "continuous flow-IRMS". The technique was rapidly adopted and the work flow was extended from N to C and then S as well as to dual element (CN) and triple element (CNS) analyses. In 2016, Thermo Fisher Scientific introduced the EA IsoLink, an evolutionary change to the combustion elemental analyzer, based on the common "injector-chromatography-detector" philosophy. Every component of the elemental analyzer has been examined and either optimized or redesigned, from the autosampler to the TCD. The Dumas combustion products are now resolved on a GC column using variable He flow rates and GC temperature ramping, and for the first time, chromatographic terms like baseline and resolution are rigorously defined. The result is improved performance in every figure of merit, improved precision for every measurement (C, N, S, CN, CS, CNS) and sample size, at the same time as improving throughput and greatly reducing He consumption.

The presentation will focus on the principles of gas chromatography with respect to elemental analyzers showing examples of applications highlighting the essentials of the new EA-IRMS technology.

Brain Plasticity: Measuring Protein Synthesis Rates in the Human Brain

Joey SJ Smeets¹, Astrid MH Horstman¹, Olaf EMG Schijns², Jim TA Dings², Govert Hoogland², Annemie P Gijzen¹, Joy PB Goessens¹, Freek G Bouwman¹, Will KWH Wodzig³, Edwin C Mariman¹, and Luc JC van Loon¹

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Introduction: Brain plasticity is a term typically used to refer to the ability of the brain to reorganize itself throughout life. Though the term is often applied in neuroscience to express synaptic or non-synaptic plasticity, information on whether neuroplasticity is also accompanied by a high brain tissue protein turnover rate is scarce. Because of obvious limitations with regard to brain tissue sampling no study has ever directly measured brain protein synthesis rates *in vivo* in humans. In this study, we applied contemporary stable isotope methodology to assess protein synthesis rates *in vivo* in human brain tissue of patients operated for drug-resistant epilepsy.

Methods: Six otherwise healthy patients (47±6 y), scheduled to undergo resective surgery for treatment of drug-resistant temporomesial epilepsy, were included in this study. Primed continuous intravenous infusions with L-[ring-13C6]-Phenylalanine and L-[3,5-2H2]-Tyrosine were initiated 2.5 h prior to surgery and continued during surgery. Throughout the surgical procedure the following tissue samples were obtained: temporal neocortex, hippocampus, *temporalis* muscle, and *vastus lateralis* muscle. Tissue-specific fractional protein synthesis rates (%/h) were assessed by measuring the incorporation of labelled L-[ring-13C6]-Phenylalanine in tissue protein and were compared between the different tissues using a paired *t* test.

Results: Serum L-[ring-13C6]-Phenylalanine enrichments averaged 8.1±0.7 MPE throughout the surgical procedure. Protein synthesis rates of temporal neocortex and hippocampus tissue averaged 0.17±0.01 and 0.13±0.01 %/h, respectively, with a significant difference between both tissues (*P*<0.05). Brain tissue protein synthesis rates were 3-4 fold higher compared to skeletal muscle protein synthesis rates (0.05±0.01 %/h; *P*<0.001).

Conclusion: This is the first study to present protein synthesis rates *in vivo* in the human brain. With brain protein synthesis rates being several-fold higher compared to skeletal muscle protein synthesis rates, the human brain displays a degree of tissue plasticity much higher than generally assumed.

Using the $\delta^{13}\text{C}$ of general biomarkers at CO_2 vents on Shikine Island, Japan to calculate past pCO_2

Caitlyn R. Witkowski¹, Sylvain Agostini², Stefan Schouten^{1,3}, Jaap S. Sinninghe Damsté^{1,3}

¹Royal Netherlands Institute for Sea Research, and Utrecht University, Den Burg, The Netherlands.

²Shimoda Marine Research Center, University of Tsukuba, Shimoda, Japan.

³Utrecht University, Utrecht, The Netherlands.

The atmospheric concentration of carbon dioxide (pCO_2) is the cornerstone of the carbon cycle, the biosphere, and climate. Although the latter has been a major topic of discussion, particular regarding global climate change and anthropogenic influences on pCO_2 , the long-term history and influences of pCO_2 remains poorly understood. One of the most common and highly-quantified methods for extrapolating past pCO_2 is via the stable carbon isotopic composition of preserved organic matter ($\delta^{13}\text{COM}$) in sediment and oil. During photosynthesis, the CO_2 -fixing enzyme Rubisco discriminates against ^{13}C , making the photosynthate isotopically lighter than the surrounding environment. This stable carbon isotopic fractionation associated with photosynthesis (ϵ_p) largely determines the $\delta^{13}\text{COM}$ but provides more context than $\delta^{13}\text{COM}$ alone, e.g. by considering the $\delta^{13}\text{C}$ of the utilized dissolved inorganic carbon. Here, we explore the $\delta^{13}\text{C}$ of the general phytoplankton biomarker phytol, the chlorophyll-a sidechain and diagenetic precursor of phytane, which offers a ubiquitous record over the past 500 Ma. To validate this potential proxy, seawater filters, phytoplankton net filters, and sediments were sampled along 300 to 2000 ppm transects from naturally occurring CO_2 vents in Shikine Island, Japan. CO_2 vents have been neglected in the literature partly due to their high sulfide concentrations which prevents microbial growth, a feature that Shikine Island lacks, allowing us to explore an otherwise uncommon field site. Unlike the more standard approaches, such as laboratory cultures and free-ocean CO_2 enrichment experiments, CO_2 vents more accurately reflect the natural environment and lack difficult and time-consuming experimental set-up and maintenance. Furthermore, the CO_2 vents offer nearly limitless sample collection, which is a major benefit considering the relatively high amounts of analyte necessary for compound-specific isotope analysis. Samples were extracted, saponified using base hydrolysis, eluted into polar fractions, analyzed using gas chromatography with a flame ionization detector and gas chromatography-mass spectrometry, and measured with isotope-ratio mass spectrometry. The acquisition of data is currently on-going. The results are expected to offer insights into the potential of these biomarkers as proxies for past pCO_2 .

An LC-IRMS Interface for Flexible Compound-specific Stable Isotope Analysis

Filip Volders¹, Christian Schmidt¹, Sam Barker², Paul Wheeler², Lutz Lange¹, Hans-Peter Sieper¹.

¹Elementar Analysensysteme GmbH,

²Elementar UK Ltd.

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

Effects of climate and geochemistry on soil organic matter stabilization and greenhouse gas emissions along altitudinal transects in different mountain regions

Marco Griepentrog¹, Samuel Bodé¹, Mathieu Boudin², Gerd Dercon³, Sebastian Doetterl⁴, Machibya Matulanya⁵, Anna Msigwa⁶, Pieter Vermeir⁷, Pascal Boeckx¹,

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Terrestrial ecosystems are strongly influenced by climate change and soils are key compartments of the global carbon (C) cycle in terms of their potential to store or release significant amounts of C. This study is part of the interregional IAEA Technical Cooperation Project “Assessing the Impact of Climate Change and its Effects on Soil and Water Resources in Polar and Mountainous Regions (INT5153)” aiming to improve the understanding of climate change impacts on soil organic carbon (SOC) in fragile polar and high mountainous ecosystems at local and global scale for their better management and conservation. The project includes 13 benchmark sites situated around the world. Here we present novel data from altitudinal transects of three different mountain regions (Mount Kilimanjaro, Tanzania; Mount Gongga, China; Rauris, Austria). All altitudinal transects cover a wide range of natural ecosystems under different climates and soil geochemistry. Bulk soil samples (four field replicates per ecosystem) were subjected to a combination of aggregate and particle-size fractionation followed by organic C, total nitrogen, stable isotope (¹³C, ¹⁵N) and radiocarbon (¹⁴C) analyses of all fractions. Bulk soils were further characterized for their geochemistry (Na, K, Ca, Mg, Al, Fe, Mn, Si, P) and incubated for 63 days to assess greenhouse gas emissions (CO₂, CH₄, NO, N₂O). Further, stable C isotopic signature of CO₂ was measured to determine the isotopic signature of soil respiration (using Keeling plots) and to estimate potential respiration sources. The following four ecosystems were sampled at an altitudinal transect on the (wet) southern slopes of Mount Kilimanjaro: savannah (920m), lower montane rain forests with angiosperm trees (2020m), upper montane cloud forest with gymnosperm trees (2680m), subalpine heathlands (3660m). Both forests showed highest C contents followed by subalpine and savannah. The largest part of SOC was found in particulate organic matter followed by microaggregates, except for the subalpine ecosystem which had most SOC stored in microaggregates. Silt and clay fractions stored the smallest fraction of SOC for all ecosystems. Cumulative soil CO₂ emissions (normalized to SOC, gCO₂-C kgSOC⁻¹) after 63 days of incubation were highest for savannah (15.2 ± 1.4) followed by subalpine (7.9 ± 0.5), upper forest (6.9 ± 1.0) and lower forest (4.8 ± 0.4). CO₂ emissions were negatively correlated with soil C contents, showing that soils with lower C contents loose higher relative amounts of their SOC through soil respiration. Keeling plot intercept is a measure for the isotopic signature of respired CO₂ and high offsets between Keeling plot intercepts and the isotopic signature of bulk SOC point towards labile (¹³C-depleted) SOC fractions as respiration sources. Highest offsets (and thus most labile respiration sources) were observed for savannah followed by subalpine, lower forest and upper forest and these were positively correlated with cumulative CO₂ emissions, showing that in savannah soils, which have lowest C contents and respire highest amounts of CO₂, mainly labile SOC is used as respiration source. Results from the other two altitudinal transects are currently under investigation and will be presented in conjunction with climatic and geochemical data.

Compound specific hydrogen isotope measurements of algal chemical fossils

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The stable hydrogen isotope ratio of C37 alkenones ($\delta DC37$) derived from haptophyte algae has been investigated as a means to track hydrologic shifts and reconstruct paleosalinity of the surface ocean (e.g. M'Boule et al., 2014, Simon et al., 2015, Schouten et al., 2006). Alkenone specific hydrogen isotope ratios are measured using GC/TC/irMS with a CP-Sil 5 25m column in the GC. The CPsil 5 column has an apolar stationary phase, which does not allow for baseline separation of alkenones with different amounts of double bonds. Therefore, alkenones of the same chain length but different degrees of unsaturation (i.e. C37:2 and C37:3) are integrated as one peak and one hydrogen isotope value is assigned to both compounds (i.e. van der Meer et al., 2013).. Here we tested an Rtx-200 60m column with a midpolarity stationary phase to achieve better separation on alkenones of the same chain length, but different degrees of unsaturation, as well as separating methyl ketones from ethyl ketones. Clean alkenone fractions can be base line resolved but environmental samples with relatively low abundance of targeted compounds compared to other components in the sample prove difficult to separate due to rapid overloading. However, an additional clean-up step by silver nitrate impregnated silica gel column separation prior to isotope analysis allows for cleaner samples and better separation. Preliminary results are promising and show stability in isotope measurements between runs. Furthermore, separated peaks can still be integrated together, allowing for comparison with samples run using the previous method on the CP-Sil 5 column.

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Latest Developments in 10 kV Isotope Ratio MS

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10 kV accelerating voltage is used for the highest performance in isotope ratio mass spectrometers, currently the 253 Plus and 253 Ultra (IRMS), Triton plus TIMS, Neptune plus MC ICPMS, and Helix MC and SFT static source mass spectrometry. 10 kV is needed for applications, which require the highest sensitivity, best peak shape, peak stability, and abundance sensitivity and, most recently, achieving high mass resolution on the 253 Ultra. These attributes are important for very small isotope signatures related with small isotope abundances and extended dynamic ranges, i.e. the analysis of isotopomers and of isotopologues containing clumped isotopes signatures.

The new 253 Plus 10 kV IRMS has improved ion optics and multicollector capabilities, including the new $10^{13} \Omega$ technology, which allows the analysis of isotope clumping on even smaller samples and isotopologue signatures. The new long integration dual inlet mode (LIDI) redefines the way of the classical dual inlet analysis with much lower sample amounts required and faster acquisition times. We will present an update on our latest improvements and recent developments on the 253 Plus 10 kV IRMS. Recent applications performed on the 253 Plus will be presented.

Unusually high sea ice cover influences resource use by benthic invertebrates in coastal Antarctica

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Antarctica currently undergoes strong and contrasted impacts linked with climate change. While the West Antarctic Peninsula is one of the most rapidly warming regions in the world, resulting in sea ice cover decrease, the sea ice cover of East Antarctica unexpectedly tends to increase, possibly in relation with changes in atmospheric circulation. Changes in sea ice cover are likely to influence benthic food web structure through modifications of benthic-pelagic coupling, disruption of benthic production and/or modifications of benthic community structure (i.e. resource availability for benthic consumers). Here, we studied shallow (0-20 m) benthic food web structure on the coasts of Petrels Island (Adélie Land, East Antarctica) during an event of unusually high spatial and temporal (two successive austral summers without seasonal break-up) sea ice cover. Using stable isotope ratios of C and N and the SIAR mixing model, we examined importance of 4 organic matter sources (benthic macroalgae, benthic biofilm, sympagic algae, suspended particulate organic matter) for nutrition of dominant primary consumers and omnivores. 14 invertebrate taxa including sessile and mobile polychaetes, gastropods, bivalves, sea stars, sea urchins and sea cucumbers were studied. Our results indicate that most benthic invertebrates predominantly relied on sympagic algae. Despite its very high abundance, trophic role of benthic biofilm seemed limited. However, interpretation of data was complicated by the peculiar ecophysiological features of Antarctic invertebrates, whose very low metabolic rates could be associated to low isotopic turnover and long time to reach isotopic equilibrium with their food items. Resource use by consumers from Adélie Land markedly differed from literature data about invertebrate diet in coastal Antarctica, suggesting 1) important influence of increased sea ice cover on benthic food web structure and 2) high spatial and/or temporal variation in the feeding habits of studied organisms, likely linked with a high degree of trophic plasticity. Our results provide insights about how Antarctic benthic consumers, which have evolved in an extremely stable environment, might adapt their feeding habits in response to sudden man-driven changes in environmental conditions and trophic resource availability.

Post-prandial protein handling following ingestion of different amounts of protein during post-exercise recovery in older males

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Purpose: The age-related decline in skeletal muscle mass is, at least partly, attributed to anabolic resistance to food intake. Resistance-type exercise sensitizes skeletal muscle tissue to the anabolic properties of amino acids. Data are warranted to define the amount of ingested protein needed to maximize post-exercise myofibrillar protein synthesis rates in older individuals.

Methods: In a parallel group design, forty-eight healthy older men (66±1 y) were randomly assigned to ingest 0, 15, 30 or 45 g milk protein concentrate (MPC80) after performing a single bout of resistance type exercise. Post-prandial protein digestion and absorption kinetics, whole body protein metabolism and myofibrillar protein synthesis rates were assessed using primed, continuous infusions of L-[ring-²H₅]-phenylalanine and L-[ring-²H₂]-tyrosine combined with the ingestion of intrinsically L-[1-¹³C]-phenylalanine labeled milk protein.

Results: A total of 76±2% (11.4±0.3 g), 63±3% (18.9±0.9 g) and 60±3% (26.8±1.2 g) of the protein derived amino acids were released in the circulation during 6 h after ingesting 15, 30 or 45 g protein (P<0.01). Ingestion of 15, 30 and 45 g protein resulted in higher whole-body protein synthesis when compared to the control treatment (0.61±0.01, 0.64±0.01 and 0.67±0.02 vs 0.53±0.02 μmolPhe·kg⁻¹·min⁻¹, respectively; P<0.01). Whole body protein breakdown rates were lower after ingestion of 45 g when compared with 0 g (0.45±0.01 vs 0.52±0.02 μmol Phe·kg⁻¹·min⁻¹; P<0.01). Whole-body protein oxidation rates were increased after ingestion of 45 g when compared with 15 g and 0 g protein (0.061±0.003 vs 0.050±0.003 and 0.046±0.002 μmol Phe·kg⁻¹·min⁻¹, respectively; P<0.05). Whole-body protein balance increased in a dose-dependent manner after the ingestion of 0, 15, 30 and 45 g protein (0.02±0.0, 0.11±0.0, 0.16±0.01, and 0.22±0.01 μmol Phe·kg⁻¹·min⁻¹, respectively; P<0.001). Muscle tissue analyses are currently being performed.

Conclusions: Dietary protein ingested after resistance-type exercise is rapidly digested and absorbed, with 60-75% of the protein derived amino acids being released in the circulation within 6 h after ingestion. Whole body protein synthesis rates and net protein balance are increased in a dose dependent manner following the ingestion of 15, 30 and 45 g milk protein in older males.

A ^{15}N tracing technique to disentangle the gross dynamics of nitrogen in tropical forest soils

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The tropical forest of the Congo basin remain very poorly investigated and understood; mainly because of logistic, political and research capacity constraints. Nevertheless, characterization and monitoring of fundamental processes in this biome is vital to understand future responses and to correctly parameterize Earth system models. Nitrogen (N) fluxes are key in these processes for the functioning of tropical forests, since CO_2 uptake by terrestrial ecosystems strongly depends on site fertility, i.e. nutrient (and hence) availability. In addition to an ecosystem N budget, process based understanding of N soil transformations is vital to our understanding of the cycling of this element in these complex ecosystems. We conducted an in situ ^{15}N labeling experiment in the remote forest of the Democratic Republic of the Congo to assess these gross soil N dynamics. We show how important the soil transformations are compared to the ecosystem inputs and outputs, and compare the results of two tropical lowland forest types to tropical montane forest. This study provides new process-based understanding of the N cycle in tropical forests, and we identify new challenges and knowledge gaps for the current state of the art of the in-situ ^{15}N labeling technique.

Characterisation of microbial dehalogenation of halogenated organic contaminants using compound-specific stable isotope analysis

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Over the last decades, concepts involving compound-specific stable isotope analysis (CSIA) have been developed allowing the qualification and quantification of in situ (bio)transformation of common groundwater contaminants such as the chlorinated ethenes and ethanes. The advance in methods now enables, besides carbon, also the analysis of chlorine, bromine and hydrogen isotopes of halogenated organic substances, allowing detailed insights into biotransformation reactions. Triple-element stable isotope composition was applied to investigate the dihaloelimination of 1,2-dichloroethane (1,2-DCA) by *Dehalococcoides mccartyi* strain 195, isolated from an anoxic digester sludge, and BTF08, enriched from contaminated groundwater in Bitterfeld (Germany). Dihaloelimination of 1,2-DCA to mainly ethene was observed with relatively higher conversion rates for strain 195 compared to strain BTF08, similar to previous observations. In addition to carbon and chlorine, for the first time, the compound-specific hydrogen stable isotope composition was analyzed for both the substrate, 1,2-DCA, and the product ethene. Similar fractionation for carbon and chlorine isotopic composition changes, for both *Dehalococcoides mccartyi* strains, was observed, leading to the assumption that dehalogenation was taking place via the same reaction mechanism. Concurrently, hydrogen isotope fractionation of the substrate was observed. For verification, additional experiments with deuterated water were conducted to assess hydrogen exchange and substitution behavior during the reaction.

Deuterated water dosing and measurement of skeletal muscle protein synthesis rates *in vivo* in humans

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Background: Skeletal muscle protein synthesis rates can be assessed using stable isotope-labeled amino acids. The traditional approach involves an acute intravenous infusion of ¹³C-phenylalanine combined with blood and skeletal muscle biopsy sampling. However, tracer infusion methodologies are limited by the short incorporation period (<12 h) relative to the time course of muscle reconditioning (days-to-weeks). Oral deuterated water (²H₂O) administration has recently re-emerged as an isotope-based method for assessing muscle protein synthesis rates over days-to-weeks *in vivo* in humans. Deuterated water administration introduces deuterium into the body water pool, which serves as a precursor for endogenously synthesized alanine. Deuterium-labeled alanine is then incorporated into newly-synthesized muscle proteins which can be assessed in muscle biopsy samples.

Objective: To assess the ability to detect ²H in saliva, free ²H-alanine in plasma and mixed muscle bound ²H-alanine enrichment to assess muscle protein synthesis rates over multiple days *in vivo* in humans.

Methods: Twelve healthy young male participants followed a dosing protocol which involved ingestion of 400 mL of 70% ²H₂O on the first day and ingestion of 50 mL of 70% ²H₂O once per day on each of the following 6 days. Saliva and blood samples were collected each day to assess precursor enrichments. Skeletal muscle biopsies were collected on days 4 and 7. Saliva deuterium enrichments were prepared using a hydrogen gas exchange protocol before being measured on a GC-IRMS. Blood plasma was deproteinized and derivatized using MTBSTFA for measurement of free ²H-alanine enrichments on a GC-MS using a 30M DB5MS column. Mixed muscle protein was isolated from the biopsy samples and hydrolyzed overnight. Amino acids were then purified and made volatile using an ethoxycarbonyl-ethylester derivatization method before being measured on a MAT253 GC-IRMS using a 60M DB-17MS column. In comparison to the measurement of ¹³C-phenylalanine in muscle, deuterium measurements required a pyrolysis oven with different calibrations and settings for the IRMS.

Results: Over the duration of the study protocol, body water deuterium enrichment in saliva averaged 0.68±0.03 APE. At corresponding time points, free ²H-alanine enrichments in blood plasma averaged 2.2±0.1 MPE. Deuterium-labeled alanine enrichments in mixed muscle protein reached 0.058±0.004 MPE on day 4 and increased over time to 0.179±0.012 MPE on day 7. Stability of the measurement was assessed across 4 replicates per sample, and was 3.3% on average (range: 0.1 - 10.2). Using saliva enrichments as the precursor and mixed muscle protein enrichments as the product, muscle fractional synthetic rates (FSR) were calculated to be 0.063±0.005 %·h⁻¹.

The effect of resistant starches on fat oxidation in healthy adults as measured by a $^{13}\text{CO}_2$ -breath test

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A resistant starch mixture (MIX) consisting of fibre of potatoes (FP) and wrinkled pea starch (WPS), and high amylose maize starch (HAMS) were supplemented in adults to evaluate their effects on fat oxidation by means of a $^{13}\text{CO}_2$ -breath test [1,2]. Sixteen subjects (10 female, 6 male, aged 18-58 years, body weight 48.0-102.0 kg) received a regular diet either without or with the supplementation of MIX and HAMS in randomized order. After administration of a U- ^{13}C algal lipid mixture, (dosage: 0.668 mg/kg corresponding to 0.0385 mmol/kg ^{13}C) at 8 00 a.m. exhaled air was collected over 14 h in 0.5- and 1 h-intervals. The ^{13}C -abundances were measured by nondispersive infrared spectroscopy (Fischer Analysen Instrumente GmbH, Leipzig). In comparison to the dry run (DR), supplementation with MIX and with HAMS increased the cumulative percentage dose recovery (DR: 16.7%, MIX: 16.9%, HAMS: 18.0%) but without statistical significance. The colonic degradation of MIX and HAMS to short chain fatty acids tends to lower the formation of carbohydrate-derived acetyl-CoA and contributes to a postprandial lipid oxidation increase by using fat-derived acetyl-CoA as a compensatory fuel source.

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Posters abstracts

1/ Verification of the geographical origin of European plaice (*Pleuronectes platessa*)

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Fish and fish products are common targets for food adulteration. During production, fish types can be completely interchanged or mixed with cheaper or less sustainable fish types. European regulation states that catch area and production method should be mentioned on the label. Therefore, analytical methods need to be found to verify the geographical origin and production method of fish. European plaice (*Pleuronectes platessa*) is the principle commercial flatfish in Europe. Plaice live predominantly in the North Sea, but extend to the Baltic Sea, the Barents Sea, the sea around Ireland, and the sea around Iceland. The aim of this study is to evaluate several techniques that are frequently used in determining the geographical origin of food and feed products for the applicability in the verification of the geographical origin of European plaice. Volatile organic compounds were analysed using Proton Transfer Reaction Mass Spectrometry (PTR-MS), the fatty acid composition was analysed using gas chromatography with a flame ionisation detector (GC-FID) and isotope ratios were analysed using Isotope Ratio Mass Spectrometry (IRMS). For this purpose, in total 24 plaice samples were collected from the North Sea (6), the Baltic sea (3), the Barents sea (5), the sea around Ireland (5), and the sea around Iceland (5). Partial least squares discriminant analysis (PLS-DA) was used to develop a hierarchical classification model. Based on the fatty acid composition, 86% of the fish could be correctly classified according to their geographical origin. If one is only interested in distinguishing plaice from the North Sea from the others, using IRMS 100% of the plaice could be classified correctly. The model is used to check the geographical origin of 23 samples obtained in the supermarket.

2/ 13C-labelled polysaccharide fibers as tracers in health research

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Accumulating evidence suggests that the gut microbiota play an important role in the harvest, storage, and expenditure of energy obtained from the diet. The composition of the gut microbiota has been shown to differ between lean and obese mice and humans; however, until recently, the specific roles that individual gut microbes play in energy harvest remain uncertain (Krajmalnik-Brown et al. 2012). Here, we give an overview of the progress made by the use of 13C-labelled polysaccharide fibers as in vivo tracers in gut microbial research in relation to nutrition and health. Some plant polysaccharides, including resistant starches and inulins show fiber properties as they may pass undigested to the large bowel (colon) where they become carbon substrates for fermenting gut bacteria. Identification of the bowel bacterial species involved in fermentation processes has led to a better insight of the functioning of the bowel in relation to effects of prebiotics or functional foods. U-13C starch has been used in RNA-SIP in an in vitro system (TIM) representing the human colon (Kovatcheva-Datchary et al. 2009; Venema et al 2010). Investigations using natural bacterial communities inhabiting living animals have not been reported until recently (Tannock et al., 2014), showing decomposition of (13C-) inulin and fructo-oligosaccharides (FOS) in the bowel of rats. This stable isotope probing (SIP) study concluded that *Bacteroides uniformis*, *Blautia glucerasea*, *Clostridium indolis*, and *Bifidobacterium animalis* were the main users of the 13C-inulin. *B. uniformis* utilized Fibruline-inulin for growth, whereas the other species used FOS and monosaccharides. Thus RNA-SIP provided new information about the use of carbon from inulin in microbiota metabolism. More recently, Butts et al. (2016) confirmed that the presence of inulin in the diet positively influences large bowel microbial fermentation. In a study on the regulation of satiety, Frost et al. (2014) demonstrate through 13C high-resolution magic-angle-spinning NMR that 13C acetate from fermentation of 13C-labelled chicory inulin in the rat colon increases hypothalamic 13C acetate (a satiety regulator) above baseline levels. A recent human trial (Deroover et al., 2017) shows that wheat bran does not affect plasma SCFAs from U-13C inulin fermentation. In a review in 2015, Vogt et al. (7) conclude: 'Inulin-type fructans are potent immunomodulating food components that hold many promises for prevention of disease. However, more studies into the mechanisms, dose-effect relations, and structure-function studies are required.' As shown by the recent progress reported in the literature, U-13C inulin plays an important role in this challenge. References Butts CA, G Paturi, MH Tavendale, D Hedderley, H Stoklosinski, T Herath, D Rosendale, N Roy, JA Monro, J Ansell 2016. The fate of 13C-labelled and non-labelled inulin predisposed to large bowel fermentation in rats. Food and Function doi: 10.1039/C5FO01056J. Deroover L., J. Verspreet, A Luypaerts, G VanderMeulen, C.M. Courtin, K. Verbeke 2017. Wheat Bran Does Not Affect Postprandial Plasma Short-Chain Fatty Acids from 13C-inulin Fermentation in Healthy Subjects. Nutrients 9: 83. DOI: 10.3390/nu9010083. Frost G, ML Sleeth, M Sahuri-Arisoylu, B Lizarbe, et al. 2014. The short-chain fatty acid acetate reduces appetite via a central homeostatic mechanism. Nature Communications 5. DOI: 10.1038/ncomms4611. Kovatcheva-Datchary P, Ergert M, Maathuis A, Rajilic-Stojanovic M, de Graaf AA, Smidt H, de Vos W, Venema K. 2009. Linking phylogenetic identities of bacteria to starch fermentation in an in vitro model of the large intestine by RNA based stable isotope probing. Environ. Microbiol. 11:914-926. Krajmalnik-Brown R, ZE Ilhan, DW Kang, JK DiBaise 2012. Effects of gut microbes on nutrient absorption and energy regulation. Nutr Clin Pract. 27: 201-214. doi: 10.1177/0884533611436116. Tannock GW, B Lawley, K Munro, IM Sims, J Lee, CA Butts, N Roy. 2014. RNA-stable isotope probing (RNA-SIP) shows carbon utilization from inulin by specific bacterial populations in the large bowel of rats. Applied and Environmental Microbiology 80: 2240-2247. Venema K, AA de Graaf, AJH Maathuis, P Kovatcheva-Datchary, H Smidt. 2010. Fermentation in the large intestine unravelled using 13C-labelled substrates: implications for obesity and gut health. In: Dietary fibre: new frontiers for food and health: 539-554. Vogt L., D. Meyer, G. Pullens, M. Faas, M. Smelt, K. Venema, U. Ramasamy, H. A. Schols & De Vos P. 2015. Immunological Properties of Inulin-Type Fructans, Critical Reviews in Food Science and Nutrition, 55:3,414-436, DOI: 10.1080/10408398.2012.6567728.

3/ Factors affecting carbon dioxide evasion and degassing from three rivers and streams on Tibetan Plateau: $\delta^{13}\text{C}_{\text{DIC}}$ constraints

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Riverine systems act as important role for carbon exchange between terrestrial, atmospheric and oceanic reservoirs. Estimated carbon dioxide (CO₂) evasion rates of 1.80 ± 0.25 Pg C per year in rivers and streams was reported (Raymond et al., 2013), accompanied with a large uncertainty, implying that the affecting factors and sources of riverine CO₂ are still not well known. CO₂ evasion from rivers and streams controlled by various factors such as climatic conditions, water chemistry, lithological distribution, hydrological cycles, source characteristics, and so on. Basically, CO₂ emission is closely related to dissolved inorganic carbon (DIC), of which sources include carbonate dissolution, silicate weathering, aquatic respiration, atmospheric CO₂ invasion and soil respiration derived CO₂ (Atkins et al., 2017). As called “the roof of the world”, Tibetan Plateau has typical and unique ecosystems which are vulnerable to climate change. However, little is known on quantities and influence factors on CO₂ evasion in rivers and streams on Tibetan Plateau, which led to noticeable uncertainties for global CO₂ estimation. For that, three rivers and streams of Duilongqu (DLQ), Golmud River (GR), and Bayin River (BR) were sampled to investigate CO₂ concentration, CO₂ emission fluxes and influence factors. The chosen rivers and streams differed a lot on climate characteristics, elevation drop, lithological feature, and vegetation cover. The pCO₂ in BR, GR, and DLQ averaged 3125 μatm , 9871 μatm , and 1632 μatm in the surface water, suggesting rivers and streams represented as an obvious sources of CO₂ with respect to atmosphere. The calculated CO₂ emission fluxes averaged 53 mmol m⁻²d⁻¹, 190 mmol m⁻²d⁻¹, and 24 mmol m⁻²d⁻¹ in DLQ, GR and BR, respectively, showing moderate evasion rates when compared with other rivers worldwide. The $\delta^{13}\text{C}_{\text{DIC}}$ value of BR, GR and DLQ ranged from -4.2 to +3.7‰, -4.1 to +1.3‰, and -7.1 to -2.3‰, with an average value of -1.5‰, -2.8‰, and -5.6‰. The $\delta^{13}\text{C}_{\text{DIC}}$ value of BR and GR is close to carbonate-rock weathering isotopic value, much higher than that in rivers reported before, while soil organic matters seems to play an important role in DIC production in DLQ. Correlation analysis and mixed model methods suggested that DIC in GR, BR and DLQ were significantly affected by snowmelt water, groundwater, and lithological distribution, no obviously relationship found with elevation drop. Reference: Atkins, M.L., Santos, I.R., Maher, D.T., 2017. Seasonal exports and drivers of dissolved inorganic and organic carbon, carbon dioxide, methane and delta C-13 signatures in a subtropical river network. *Science of the Total Environment*, 575: 545-563. Raymond, P.A. et al., 2013. Global carbon dioxide emissions from inland waters. *Nature*, 503(7476): 355-359.

4/ The use of stable isotopes to study the origin of methane trapped in Antarctic sea ice

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Methane (CH₄) is a strong greenhouse gas, with a global warming potential 28 times higher than that of CO₂ over a 100-year period. Since the beginning of the Industrial Era, its atmospheric concentration has increased by more than 150 %, mainly because of anthropogenic activities. Large uncertainties exist on the contribution of different CH₄ sources. One of the largest unknown is the contribution of the ocean. In polar ocean, sea ice has long been considered as an inert and impermeable barrier, however recent studies have highlighted the existence of gas fluxes at the atmosphere-sea ice and sea ice-seawater interfaces. Because of the lack of measurements and the heterogeneity of the system, these fluxes are to date poorly understood and quantified. To improve future climate projections, we aim to investigate the control exerted by sea ice on the CH₄ atmospheric budget. We measure the stable isotopic composition of CH₄ ($\delta^{13}\text{C}$ and δD) in cores of landfast sea ice from the Ross Ice Shelf, Antarctica, collected between 2011 and 2012 in the frame of the YROSLAE (Year Round survey of Ocean-Sea Ice-Air Exchanges in Antarctica) project. These isotopic signatures help us to distinguish the processes responsible for CH₄ formation and removal in sea ice. In the ocean, CH₄ can be of biogenic or thermogenic origin. Characteristic isotopic fractionation processes are associated to each type of formation/removal process hence the analyses of the $\delta^{13}\text{C}$ and δD of CH₄ help to unravel the origin of the gas. Thermogenic CH₄ is typically enriched in ¹³C and D whereas biogenic CH₄ is typically depleted in both heavy isotopes. Our first results show the presence of CH₄ highly enriched in both heavy isotopes that might be of thermogenic origin. However, further work is performed to investigate if potential aerobic production process, using as substrate e.g. DMSP or methylphosphonate could as well take place in seawater, at the sea ice-seawater interface or in the sea ice and explain the observed isotopic enrichment.

5/ Planktic foraminiferal stable-isotopes across the EECO: investigating the coupling between temperature and the exogenic carbon pool (ODP Site 1263, Walvis Ridge)

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The Late Paleocene to Early Eocene warming trend is characterized by a gradual temperature rise of 5-6°C resulting in the Early Eocene Climatic Optimum “EECO”. This warming trend was punctuated by several so-called “hyperthermals”, which were geologically brief (<200kyr) episodes of extreme warmth. Recently, a new, ~4.7 million year (Myr) long, high-resolution benthic foraminiferal stable isotope record of ODP Site 1263 has been presented, which encompasses the peak of the early Eocene “hothouse” (~49.5 - 54.2 Ma). This record confirms the presence of hyperthermals during and at the termination of the EECO as was previously found for ODP Site 1258. In addition, the record reveals a highly significant linear relationship between $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ for these events, similar as for their early Eocene counterparts. This indicates a strong coupling between global warming and the release of isotopically light carbon into the ocean-atmosphere system throughout the EECO. Whilst the coupling between temperature changes and perturbations in the exogenic carbon pool remain stable on short-term time scales, they do not for the long-term trends at ~52 Ma when a rapid ^{13}C enrichment in carbon data is not accompanied by changes in the oxygen record. It was hypothesized that enhanced carbonate and organic carbon burial rates might be responsible for this shift in average isotopic values during a temporary reduced efficiency of the biological pump. To test this hypothesis, we will present our first stable isotopic results of two planktic foraminiferal species derived from the same samples of ODP Site 1263, which portray changes in surface water (*Acarinia* ssp.) and thermocline waters (*Subbotina* ssp.).

6/ High temporal resolution measurements of the isotopic composition of CH₄ at the Lutjewad station, The Netherlands

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Isotope measurements can help constraining the atmospheric budget of the greenhouse gas methane (CH₄) because different sources emit CH₄ with slightly different isotopic composition. In the past, high precision isotope measurements have primarily been carried out by isotope ratio mass spectrometry on flask samples that are usually collected at relatively low temporal resolution. We have recently developed a fully automated gas chromatography - isotope ratio mass spectrometry system (GC-IRMS) for autonomous and unattended CH₄ isotope measurements (dD and d13C) in the field. The first deployment at the Cabauw Experimental Site for Atmospheric Research (CESAR) indicated that CH₄ emissions from fossil fuel sources are overestimated in this region. To further exploit the potential of this approach, the in situ system has been installed in November 2016 at the Lutjewad atmospheric monitoring and sampling site in the North of the Netherlands. This site is expected to sample also emissions from the large Groningen gas fields. The isotope measurements are expected to allow distinguishing these emissions from the agricultural emissions, which are also strong in this region. We will present the results from these ongoing measurements of dD and d13C in CH₄.

7/ Calibration Mixtures for Improving Accuracy of Stable Isotope Measurements

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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. 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 preparation of high quality calibration mixtures which can be used as analytical standards typically requires the use of cylinders manufactured from aluminum alloys. The exact choice of alloy may be governed by local authorities. The cylinder valve selected must also consider material compatibility of the various components in the mixture to ensure stability of the mixture. Traces of moisture and oxygen contained within the cylinder are known to cause stability issues in mixtures and pure gases. Metal surfaces can adsorb water that is tightly bound to the surface. Therefore the use of suitable vacuum baking procedures must be followed. Calibration gases can either be prepared gravimetrically, volumetrically or by dynamic blending. Each of these methods has its own advantages and limitations. Gravimetric techniques have the advantage of high mass discrimination and the use of weights which are directly traceable to national metrology institutes. One of the most significant limitations of this technique is that the balance used must have a large range and suitable sensitivity for the addition of the minor components. Gravimetric mixtures are prepared by accurately measuring gas additions as measured by the increase in mass of the cylinder. The composition of the mixture is calculated based on the mass of each gas as well as some additional parameters. Chief among them is the purity of the source materials used. The individual source materials must be carefully characterized to take impurity constituents (both molecular and isotopic) into account in the final calculation.

Errors and uncertainties can arise from three primary sources (1) analyzer calibration (2) analyzer repeatability (precision) and (3) uncertainties in concentration of reference materials used for calibration.¹ The contribution to the uncertainty can be obtained by propagating the standard uncertainties of each source. To decrease the uncertainty, each parameter must be minimized. This entails calibrating the instruments with the highest accuracy standards.

Solid isotopic primary reference materials, which can be combusted, are available in extremely limited quantities from the International Atomic Energy Agency (IAEA) and the US National Institute of Standards and Technology (NIST), formerly known as NBS. However, quantities of these materials are typically limited to one reference material per laboratory every 3 years. Laboratories are encouraged to develop their own working standards that can be used to calibrate samples. Preparation of these standards often increases the total uncertainty of the measurements. It is good analytical practice to matrix match the standard and sample as well as bracket the standards around the concentration of the samples. Typically, 3 standards are the minimum required to assess linearity and accuracy. How can this be accomplished with the limited range of standards available?

This presentation will examine the isotopic mixture preparation process, for both molecular and isotopic concentrations, for a range of components and delta values. The role of precisely characterized source material will be presented. Analysis of individual cylinders within multiple batches will be presented to demonstrate the ability to dynamically fill multiple cylinders containing identical compositions without isotopic fractionation. Additional emphasis will focus on the ability to adjust isotope ratios to more closely bracket sample types without the reliance on combusting naturally occurring materials, thereby improving analytical accuracy.

References

1Bell, S., 1999. A Beginners Guide to Uncertainty of Measurement, National Physical Laboratory

Agenda 11th general BASIS member meeting

4 May 2017

Utrecht, The Netherlands

1. Welcome, opening and attendances

2. Minutes 10th general meeting

3. Financial report (01-01-2016 to 31-12-2016)

4. Board members and renewals
President: Pascal Boeckx
Secretary: Katja Van Nieuland
Treasurer: Marcel Van der Meer
Scientific committee: Henk Schierbeek

5. Meeting 2017

6. Meeting 2018 and 2019
2018: University of Liège, Liège, Belgium
2019: NIOZ, Texel, The Netherlands

7. Various

Minutes 10th general BASIS member meeting

6 September 2016, Gent, Belgium

1. Welcome and opening

The president (Pascal Boeckx) welcomes the participants and opens the meeting at 17:30.

2. Announcements

None.

3. Attendances

The secretary registers the attendances and announces that there are 15 members present.

4. Minutes 9th general meeting

The present members accepted the minutes of working year 2015.

5. Financial report (01-01-2015 to 31-12-2015)

Pascal Boeckx explained the financial status of BASIS. The result for 2015 was negative because no BASIS meeting was organized in 2016 and hence no income was generated. The financial report was approved by the financial control committee Samuel Bodé and Ries de Visser. This approval results in the discharge of the treasurer.

Marcel Van der Meer is the new treasurer of BASIS.

The president explains the aims of the BASIS fund again. Three persons used the BASIS fund in 2015.

6. Board members

President: Pascal Boeckx

Secretary: Katja Van Nieuland

Treasurer: Marcel Van der Meer

Scientific committee: Henk Schierbeek.

7. Meeting 2017

The 2017 BASIS meeting will be held in Utrecht on May 3 and 4. The local organiser is Tom Bosma. P. Boeckx will check if the 2018 meeting can be held in Liège, Belgium. M. Van der Meer offered NIOZ, Texel as venue for the 2019 BASIS meeting.

8. Various

BIG + BASIS exists 15 years in 2016. This will be celebrated during the 11th BASIS meeting in Utrecht.

Frans Stellaard will check if the laboratory information on the BASIS website is still up to date.

The president closes the meeting at 18:00.

Katja Van Nieuland
Secretary BASIS

Aims of BASIS fund

The BASIS fund will support pre- and postdoctoral researchers for active oral contributions to large conferences with an international audience. In addition the BASIS fund will support technical staff to visit other stable isotope laboratories to receive training on a specific analytical stable isotope tool.

Each person can request for funding only once during his/her pre- or post-doctoral career. Technical staff can apply twice during their professional career.

Application procedure

Who can apply?

All pre- and post-doctoral researchers and technical staff that are member of BASIS can apply. BASIS membership starts after participation in an annual BASIS meeting and is valid for one year.

What can be requested?

Financial support is limited to 750 Euro.

Reimbursement is always based on effective costs.

When to apply?

Submissions are open any time of the year. Hence, there is no deadline.

Note that the BASIS fund budget is limited and the funding system can be closed anytime. The BASIS board will decide the latter.

How to apply?

The applicant fills in the above application form and sends it with annexes to bestuur@basis-online.be and the BASIS board will assess each application.

Reimbursement will be carried out upon receipt of original proofs of expenses by BASIS treasurer (Marcel van der Meer).