

The miniRUEDI Symposium 2019

Eawag, 8600 Dübendorf, Switzerland

Thursday, 4 April

9:30 – 10:00	Welcome coffee
10:00 – 10:10	Opening
10:10 – 11:10	Presentations
11:10 – 11:30	Break
11:30 – 12:30	Presentations
12:30 – 14:00	Lunch at Eawag
14:00 – 15:00	miniRUEDI demonstration (ponds)
15:00 – 15:20	Break
15:20 – 16:00	Presentations
16:00 – 16:20	Break
16:20 – 17:00	Presentations
19:00 (open end)	Group dinner in Zürich

Friday, 5 April

9:30 – 10:00	Welcome coffee
10:00 – 10:40	Presentations
10:40 – 11:00	Break
11:00 – 11:50	miniRUEDI demonstration (ponds, slides)
11:50 – 12:30	Presentations
12:30 – 14:00	Lunch at Eawag

End of miniRUEDI Symposium

Thursday 4 April 2019 / 10:10–10:30

What is the miniRUEDI, and how did it come to be?

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Environmental aquatic systems are dynamic and hard to predict. Collecting the “right” samples to study the aquatic environment using dissolved (noble) gases as tracers is therefore notoriously difficult, and subsequent sample analysis in the lab is time consuming and laborious. In addition, combining these noble gas data with biogeochemically active gases for the integrated interpretation of physical and biogeochemical processes typically requires additional samples and labs, which introduces further obstacles that prevent efficient scientific investigations of the natural gas dynamics in environmental systems.

To overcome these challenges, we developed a mass-spectrometric system for “real-time” analysis of (dissolved) He, Ar, Kr, N₂, O₂, CO₂, CH₄ and other gas species in the field. The first version of this instrument was based on a commercially available mass spectrometer (MS) system aimed for leak detection in gas apparatus, coupled to a gas-equilibrium membrane inlet (GE-MIMS) for dissolved gas analysis in aquatic environments. This instrument (the RUEDI) showed the great potential for environmental research – but it was still too large and bulky, did not allow easy automated operation, and consumed too much sample gas.

We therefore developed the miniRUEDI, which builds on our experience with the RUEDI, but was developed from scratch to suit the needs for (dissolved) gas analysis in environmental systems. The miniRUEDI does not require any purification or other preparation of the sampled gases and therefore allows maintenance-free and autonomous operation. The gases are continuously sampled and transferred through a capillary pressure reduction system into a vacuum chamber, where they are analysed using a quadrupole mass spectrometer with a time resolution of about 1 min. The low gas consumption rate (< 0.1 ml/min) minimises interference with the natural mass balance of gases in environmental systems, and allows the unbiased quantification of dissolved-gas concentrations in water by GE-MIMS.

In this presentation, we will discuss the technical history of the miniRUEDI and outline a number of field applications illustrating the utility of the miniRUEDI in environmental research.

Thursday 4 April 2019 / 10:30–10:50

In-situ Mass Spectrometry Improves the Estimation of Stream Reaeration from Gas-Tracer Tests

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The estimation of gas-exchange rates between streams and the atmosphere is of great importance for the fate of volatile compounds in rivers. For dissolved oxygen, this exchange process is called reaeration, and its accurate and precise estimation is detrimental for the determination of metabolic rates. A common method for the determination of gas-exchange rates is through artificial gas-tracer tests with a proxy gas. We present results from gas-tracer tests using krypton and propane as tracer compounds, which are detected in-situ by a portable gas-equilibrium membrane inlet mass spectrometer (GE-MIMS). This field-compatible device uses signals of atmospheric measurements for concentration standardization, and allows recording the dissolved-gas concentrations at a high temporal resolution, leading to overall low measurement uncertainty. Furthermore, the in-situ approach avoids loss of gas during the steps of sampling, transport, storage, and analysis required for ex-situ gas measurements. We compare obtained gas-exchange rate coefficients, reaeration and metabolic rates from the in-situ measurements to results obtained from head-space sampling of propane followed by laboratory analysis, and find much lower uncertainties with in-situ methods.

Thursday 4 April 2019 / 10:50–11:10

Groundwater dating on timescales from weeks to months including Argon-37 other noble gases

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Groundwater-river water exchange dynamics in gravel aquifers typically occur at timescales of less than one year. Often water residence times and mixing processes both must be considered to characterize such dynamic systems. In this study we investigate the suitability of ^{37}Ar as it constrains water residence times of weeks to months. ^{37}Ar is a radioactive noble gas mainly produced and accumulated in the subsurface. With its half-life of 35 days, ^{37}Ar fills the gap between ^{222}Rn (used for days to weeks) and $^3\text{H}/^3\text{He}$ (for timescales of month to years). In the subsurface, ^{37}Ar is produced by multiple reactions, including neutron and muon capture (1). These phenomena result in an exponentially decreasing secular equilibrium concentration with depth. Once this concentration profile is parameterized and assuming an ^{37}Ar -free atmosphere as well as mainly horizontal underground flow, ^{37}Ar can be used as a conservative tracer for groundwater dating, which is sensitive to the depth of water percolation (2). This property suggests a high potential for the calibration of numerical flow models. Additional tracers such as ^{222}Rn , $^3\text{H}/^3\text{He}$ and ^4He can help elucidate the mixing between stream water and older groundwater. We used a Rad7 and a MiniRuedi to continuously analyze short term changes of mixing ratios. This is not feasible with ^{37}Ar due to the large sample volume required (~ 2 tons of water). The present study takes place in the context of a large-scale pumping test, which was carried out from January to March 2019 in the Aeschau aquifer in the Emmental. Our preliminary results indicate that ^{37}Ar and ^{222}Rn are decreasing with increasing pumping rates, which could suggest enhanced infiltration of river water correlated to increased pumping.

(1) J.T. Fabryka-Martin, Production of radionuclides in the earth and their hydrogeologic significance, with emphasis on Chlorine-36 and Iodine-129, University of Arizona, 1988.

(2) O.S. Schilling, C. Gerber, D.J. Partington, R. Purtschert, M.S. Brennwald, R. Kipfer, D. Hunkeler, P. Brunner, Advancing Physically-Based Flow Simulations of Alluvial Systems Through Atmospheric Noble Gases and the Novel Argon-37 Tracer Method: Integrating tracers with models, Water Resources Research. 53 (2017).

Thursday 4 April 2019 / 11:30–11:50

CH₄, N₂, Ar and Kr concentrations in lake sediment bubble gas

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Recently, Langenegger et al. (2019) have shown how the composition of gas bubbles from lake sediments can be used to better quantify the sediment methane dynamics in terms of CH₄ production and diffusive and ebullition fluxes. Langenegger et al. (2019) have focused on CH₄ and N₂ measurements, the theory behind their approach, however, applies to other gasses as well, e.g. Ar and Kr. According to theory, both Ar and Kr should be depleted with respect to atmospheric concentration and Ar/N₂ and Kr/N₂ ratios should be enhanced.

With the miniRUEDEI system, we have measured the content of CH₄, CO₂, N₂, Ar, Kr and O₂ in bubble gas released at various depths of 4 different lakes at two different times of the year. In all 4 lakes, the total moles of bubble gas consist mainly of CH₄ + N₂. Indeed as hypothesized, we observe that all measured sediment bubbles are depleted of atmospheric gasses (N₂, Ar, Kr) whereas at the same time Ar/N₂ and Kr/N₂ ratios are enhanced. The composition of bubbles shows little variation in time and depends mainly on water depth. This information can be used to infer methane pathways and emissions.

Besides preliminary results, we would like to share information on the measurement methodology with the miniRUEDEI system and stimulate a discussion for novel noble gas measurement techniques in the sediments with the miniRUEDEI system.

Thursday 4 April 2019 / 11:50–12:10

A new method for tracing denitrification in-situ in riparian groundwater

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Nitrate is among the most prevalent groundwater pollutants globally. Despite centuries of research on denitrification—the main process converting nitrate to nitrogen gas (N₂)—we still lack a comprehensive understanding of its spatio-temporal dynamics. The high atmospheric background concentration of N₂ has been recognized as a major impediment for advancement in this field. Previous research has shown that noble gases can be used as conservative proxies for N₂ gas exchange in groundwater. Thus, by a combined analysis of dissolved noble gases and N₂, one can account for the atmospheric background concentration of N₂ and quantify the amount of N₂ stemming from denitrification. Conventional dissolved gas analysis, however, is both expensive and labour intensive. We used a portable Gas Equilibrium Membrane Inlet Mass Spectrometer (GE-MIMS) system, which allows for a fast and efficient in-situ analysis of dissolved gases (N₂, O₂, CO₂, He, Ar, Kr). We employed the GE-MIMS system at three piezometers located in the riparian zone over a six months period. By accounting for atmospheric N₂, we obtained spatio-temporal time series of N₂ production due to denitrification. Moreover, we observed processes associated with nitrate respiration such as CO₂ production and O₂ depletion in the riparian zone. We can link spatial differences in denitrification dynamics to differences in stream-bank hydraulic conductivity and microbial activity. Our results demonstrate that the GE-MIMS system can accelerate a better understanding of denitrification dynamics as it allows for high resolution sampling of groundwater.

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Ruedi vs oil spill: on-site mass spectrometry to investigate gas dynamics in the vadose and saturated zones of a contaminated groundwater

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We investigated biogenic gas dynamics in a shallow unconfined aquifer contaminated by a crude oil spill, near Bemidji, MN, USA by on site-gas analysis of He, Ar, Kr, O₂, N₂, CH₄ and CO₂ concentrations in groundwater and mixing ratios in soil gas. Environmental contaminants are decomposed by microbial respiration and methanogenesis. While the end products of these processes, CO₂ and CH₄ respectively are indicators for contaminant decomposition, the distributions of gases are also influenced by advective and diffusive transport in groundwater and vadose zone and by naturally occurring soil respiration. Atmospheric noble gas signatures were successfully used to trace biogeochemical and transport processes both in the vadose zone and in the saturated zone in biologically active unconfined aquifers. Depletion of noble gases in the saturated zone are an indication of bubble formation and possibly ebullition due to high gas built up e.g. from methanogenesis. Elemental fractionations in noble gases due to advective gas transport can be used to characterize pressure gradients that can be established between different biogeochemical process zones (1).

In this study, we mapped with on-site mass spectrometry the gas composition of a cross-section along the groundwater flow-path across the contaminated site during a two week field campaign. The mass spectrometer was equipped with a gas-equilibrium membrane-inlet for water (GE-MIMS aka Ruedi system (2)) and a capillary inlet was used for soil gases. The method needed to be adapted to the high methane content that changes air viscosity and to the local presence of hydrocarbons causing interference in the MS on the Kr mass to charge ratio. The acquired data set reproduced the before mentioned depletion and elemental fractionations of noble gases demonstrating the potential of Ruedi-like systems for the investigation of biogenic gas processes in contaminated aquifers.

(1) Amos, R.T. and Mayer, U. *Investigating the role of gas bubble formation and entrapment in contaminated aquifers: Reactive transport modelling*, Journal of Contaminant Hydrology 2006

(2) L. Mächler et al, *Membrane inlet mass spectrometer for the quasi-continuous on-site analysis of dissolved gases in groundwater*, ES&T, 2012

Thursday 4 April 2019 / 15:20–15:40

Gas Quantification in an Arsenic Contaminated Aquifer

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The noble gases; He, Ne, Ar, Kr, Xe, are commonly used as tracers in groundwater. They are most typically used in the reconstruction of past climatic conditions and to understand the current/future hydraulics of a particular system. Combining noble gas measurements, with quantification of other dominant gas species in groundwaters, provides a powerful and useful tool in the field of environmental science. One such gas species, which is of particular interest for this investigation, is methane. Studies have found (1,2), that groundwaters with high CH₄ concentrations are inextricably linked to low noble gas concentrations. Furthermore, CH₄ can be related to high arsenic concentrations in groundwaters where arsenic mobilization is believed to be microbially driven.

Arsenic contamination of groundwater remains a problem for many of the river deltaic areas in South-East Asia; where concentrations regularly exceed the 10 µg/L currently recommended by the World Health Organization. The focus of this study, is to determine gas concentrations in groundwaters, in an area where arsenic mobilisation is active, to reveal information about the sites hydrology. The small village of Van Phuc, Vietnam presents an ideal opportunity for such a study as is it well documented and accessible, however still not well understood.

Gas concentrations in 21 wells at varying depths and locations were taken in Van Phuc with the miniRUEDI; a portable mass spectrometer capable of measuring noble gases; He, Ar, Kr, and additional gases; H₂, CO₂, CH₄, N₂ and O₂. Results show a clear depletion of the gases Ar, Kr and N₂, with increased CH₄ concentrations. He, shows the opposite behaviour such that it increases in concentration as CH₄ saturates. The conceptual picture these results indicate, is that the production of methane bubbles reduce the hydraulic conductivity in the aquifer; allowing enough time for He to accumulate, whilst simultaneously depleting Ar, Kr and N₂ in the groundwater as a result of their partitioning into the CH₄ gas bubbles.

(1) Amos, R.T. and Mayer, U. *Investigating the role of gas bubble formation and entrapment in contaminated aquifers: Reactive transport modelling*, Journal of Contaminant Hydrology 2006

(2) Brennwald, M.S., Kipfer, R. and Imboden D. *Release of gas bubbles from lake sediment traced by noble gas isotopes in the sediment pore water*, Earth and Planetary Science Letters, 2005

Thursday 4 April 2019 / 15:40–16:00

Temporal Variation in Noble Gas Signatures of Captured CO₂

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In the ICO2P project (1), a monitoring scheme for carbon capture and storage (CCS) employing noble gases as tracers is developed. Noble gases have successfully been used to refute alleged leakage in the past, for example, at an onshore enhanced oil recovery project (2). During a feasibility study (3) and the first year of the project, sampling campaigns were conducted at two CO₂ capture facilities. Single samples were analysed from two CO₂ capture facilities: at the capture research centre Technology Center Mongstad (TCM) and at Melkøya, a natural gas processing plant with CO₂ storage. At both sites, CO₂ capture is performed by amine gas treating.

The concentrations in captured CO₂ are typically several orders of magnitude lower than in air. In order to cover temporal development with on-site mass spectrometry, a calibration gas was configured. Further, not only the captured gas stream but also the incoming gas is monitored, giving information about the captured gas, assuming absorption is constant. With this approach, significant temporal variation in argon and helium were measured. Argon variation correlates with changes in the capture process; while helium varies due to changes in the composition of the incoming flue gas.

Noble gas concentrations, and subsequently traceability of injected CO₂ in future storage schemes, are significantly different for the two sites. Capture is performed of CO₂ from combusted (TCM) and non-combusted (Melkøya) gas. Further, observed variation in gas signatures will affect estimations of fluid mixing by several percent. Documenting variation in noble gas signatures will improve leakage detection schemes.

(1) www.mn.uio.no/geo/english/research/projects/ico2p

(2) Gilfillan et al. [2017] Int. Jour. of Greenhouse Gas Control, 63, 215-225

(3) Sundal et al. [2019], GHGT-14

Thursday 4 April 2019 / 16:20–16:40

Quantification of Air-Water Gas Exchange with the miniRUEDI

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The gas flux between air and water is caused by a disequilibrium of aquatic and atmospheric gas concentrations. The gas transfer velocity (k) determines the speed of equilibration. Experimental quantification of k often relies on spiking the water with a tracer gas and follow-up measurements (e.g. 1,2).

Similar to former experiments, a gas spike was added to the water in an experimental pond at Eawag to measure gas exchange. The decay of the gas concentrations was then measured with the miniRUEDI. The gas spike necessitated the use of a gas bag for the calibration gas. Further, the large range of concentrations measured, made the scaling of the Faraday detector to the multiplier detector necessary. With the high temporal resolution of the miniRUEDI, it was possible to observe the increase of k due to precipitation.

In a novel approach to quantify gas exchange, we only employ the natural, temperature-induced diurnal gas dynamics (DGD) (3). Variations in observed dissolved gas concentrations are damped and lagged with respect to equilibrium concentrations; more or less, dependent on k . Experiments were performed in a controlled experiment at Eawag and in a field study in a shallow lagoon in Southern France. With the current experimental setup, the method is sensitive to gas transfer velocities of 0.05–9 m/day (for N₂), at a water depth of 1 m, and a given daily water temperature variation of 10 °C.

(1) Watson et al. (1991), *Nature* 349, 145–147

(2) Crusius and Wanninkhof (2003), *Limnol. Oceanogr.* 48(3), 1010–1017

(3) Weber et al. (2019), *Environ. Sci. Technol.*, 53 (3), 1463–1470

Thursday 4 April 2019 / 16:40–17:00

MiniRuedi in the Danube Delta – Insights from mapping CO₂ and CH₄

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The release of greenhouse gases like CO₂ and CH₄ from freshwater systems is gaining increasing attention in the last decade. In-situ production and transport from terrestrial environments can create large spatial and temporal gradients, especially in heterogeneous settings like deltaic systems. Here, continuous measurements of dissolved concentrations of CO₂ and CO₄ can be very useful to gain a more detailed understanding of the greenhouse gas variability. To investigate the dissolved concentrations of CO₂, CO₄ and O₂ in the Danube Delta along our 200 long journey, we equipped a houseboat with a Mini-Ruedi and commercial sensors from KM Contros. Observing very large spatial gradients of all three gases allows us to compare the performance of the Mini-Ruedi with the commercial sensors under field conditions over a partial pressure range of several orders of magnitude (200 to 21000 μ atm CO₂, 100 to 9300 μ atm CO₄, 10 to 250 mbar O₂). While O₂ data showed a rather constant offset of -5% for large values and -10% below 50 mbar, both CO₂ and CO₄ showed a non-linear behavior below 800 μ atm, respectively.

Friday 5 April 2019 / 10:00–10:20

miniRUEDI analysis of small samples at low pressure

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We have developed methods to extend the utility of the Oxford miniRUEDI analysis platform for situations where sample size precludes analysis at atmospheric pressure. In order to assess the performance of the miniRUEDI system under the relevant conditions of low pressure and limited sample size, standards of varying composition have been systematically measured versus inlet pressure. Protocols developed to enable analysis of small samples will be presented, along with the first set of sample measurements from copper tubes (collected for high resolution noble gas mass spectrometry), where the bulk gas concentrations are unknown.

Friday 5 April 2019 / 10:20–10:40

Evolution of the gas composition in the FE experiment, an unsaturated spent fuel emplacement drift at Mt. Terri (Switzerland)

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The build-up of pressure due to gas generation is a critical factor in the safety assessment of deep geological repositories. Different processes are expected to produce and/or to consume gas which will have a direct influence on the composition of the gas phase and the pressure build-up. The accuracy of the evaluation and prediction of safety-relevant processes can be increased through the quantification of gas production and/or consumption rates and the understanding of the underlying mechanisms. In this context, the Full-scale Emplacement (FE) experiment is a unique opportunity to study the evolution of the gas composition in the near-field of an emplacement tunnel conceived for spent fuel (SF) during the early period after closure at full scale. Identification and quantification of processes controlling the composition of the gas phase in the near field of the FE experiment are targeted by the integration and interpretation of the following observational and experimental activities: 1) collection of data in the field by on-site monitoring of the gas composition in the FE drift by means of O₂ and H₂ sensors and a state-of-the-art on-line mass spectrometric system, 2) regular sampling of the gas phase for precise analyses (e.g., major and trace gases, noble gas isotopes, alkanes, alkenes, ¹³δCCH₄, ²δHCH₄), and 3) laboratory experiments which provide quantitative information on the interactions between different materials in the FE tunnel and the gas phase therein. The present contribution aims to stimulate a scientific discussion on the conceptual/technical challenges in understanding gas exchange between different geochemical reservoirs in an engineered system such as the FE experiment.

Friday 5 April 2019 / 11:50–12:10

N2ArCheck – A QC-tool for the evaluation of excess N₂-data

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In Germany there is currently a substantial demand for methods to quantify the initial nitrate input into aquifers, especially in regions with high nitrate input by agriculture. Thus, in recent years the analysis of N₂/Ar for Excess-N₂ determination (1) in groundwater has gained increasing importance. Several commercial laboratories started to offer excess N₂ measurements. Due to the lack of adequate round robin tests, the State Authority for Mining, Energy and Geology (LBEG) issued a series of interlaboratory tests in cooperation with the Lower Saxony Water Management, Coastal Defence and Nature Conservation Agency (NLWKN). Along with the test series, the LBEG developed the MS Excel based QC-tool “N2ArCheck”.

The tool offers a semi-automatic quality control and data evaluation of excess N₂-data for laboratories and users. It is based on gas equilibrium data (2) and several models for degassing effects. In this way the data can be checked for plausibility and certain processes in aquifers can be derived from the data. The current version of the tool covers a data check, definition of threshold/output values and the visualization of data. Furthermore, the tool evaluates the possible sources for observed degassing effects and performs cross checks for plausibility control. The results are then classified into five groups (I to V) with varying reliability and accuracy of the calculated excess N₂ due to degassing effects. Hence, N2ArCheck offers laboratories and users from other fields a comprehensive evaluation of excess N₂-data.

(1) Vogel, J. C., Talma, A. S. and Heaton, T. H. E., *Gaseous nitrogen as evidence for denitrification in groundwater*, J. Hydr., 50, 191-200, 1981

(2) Weiss, *The solubility of nitrogen, oxygen and argon in water and seawater*, Deep-Sea Research, 1970, Vol. 17, pp. 721 to 735, 1970

Friday 5 April 2019 / 12:10–12:30

Application of the miniRUEDI portable mass spectrometer for fugitive gas investigations in northeast British Columbia, Canada

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An undesired consequence of petroleum resource development is the release of fugitive natural gas into the surrounding environment. These leaks occur at a subset of active, orphan, and abandoned oil and gas wells. Once natural gas enters the sediments and aquifer surrounding a well, the fate of this fugitive gas and the consequences for water quality and greenhouse gas emissions are not well understood.

We conducted a controlled gas release experiment at the Hudsons Hope Field Research Station (HHFRS) in northeast British Columbia, a region of active oil and gas development. Over a 3 month period, 98 m³ of a synthetic natural gas (containing 85% CH₄, 0.5% He, and other gases) was released into a confined aquifer. Prior to the start of the injection, multilevel groundwater and soil gas wells were installed at the HHFRS. We used a miniRUEDI portable mass spectrometer to measure in real time the gas composition of groundwater and soil gas, and also analyzed samples for gas composition via traditional, laboratory-based methods. Measurements of CH₄, Ar, and He concentrations and ratios enabled us to monitor the transport of the gas and determine the fraction of the injected gas that was oxidized in situ.

During the field experiment, measurements of surface gas efflux obtained with soil gas survey chambers indicated that significant CH₄ uptake by surface soils and in situ oxidation to CO₂ occurred at some regions of the HHFRS. To investigate this process further, we conducted laboratory experiments where elevated levels of He and CH₄ were added to the headspace of a sealed chamber containing soil from the field site and the headspace concentrations of He, CH₄, and CO₂ were monitored over time. Helium acted as an inert tracer of diffusion. The lab data will be used with a reactive transport model (MIN3P) to parameterize the relative rates of CH₄ diffusion and oxidation.