Technische Universität München

Annual Report 2013

Institute of Hydrochemistry and Chemical Balneology
Chair for Analytical Chemistry

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Chair for Analytical Chemistry
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Editor: Dr. Thomas Baumann

Editorial

Dear coworkers, friends & colleagues!

The new formulation of the drinking water guideline, now specifically limiting the occurence of *Legionella* in water distribution systems, Right2Water, the first european citizens initiative asking for the Human Right to water and favoring groundwater protection above drinking water treatment, the decisions on the natural purity of bottled mineral water and bio-mineral water, plastic particles not only in the Pacific ocean but also in rivers and lakes — water related issues received significant press coverage in 2013. After the first rush into renewable energy sources, biogas and geothermal energy have revealed technical problems, for instance the presence of siloxanes in biogas, causing abrasive deposits in engines or scalings in fuel cells or the delicate carbonate equilibrium, causing scalings in pipes, heat exchangers, and pumps in geothermal systems, both influencing the efficiency of renewable energy.

Meanwhile, the Institute of Hydrochemistry not only continued to provide scientific support to these core problems but also broadened its spectrum. With the new HEV microarray the acclaimed microarray read-out and multiplexed analysis system extended its scope to blood serum analysis, in direct competition with traditional analytical techniques. Flooding after longer droughts makes it more likely for viruses to enter groundwater resources. In order to monitor groundwater quality the enrichment of viruses from groundwater has been optimized and we are now able to meet the WHO suggestions by enrichment of a 90-m^3 water sample.

Large scale production of genetically modified tobacco plants provides a new source for antibodies against algae toxins which are the used for separation and enrichment. These plantibodies are a vision for new water treatment technologies. We rather like to apply selective separation for producing clean water than performing advanced oxidation processes with the risk of unknown secondary metabolite formation.

Following the water cycle you will find the Institute of Hydrochemistry also on the other end of the city's water system: together with my colleague Prof. Drewes, the newly appointed director of the Chair for Civil Engineering at TUM we started a project focussing on N_2O reduction at sewage water treatment plants as the greenhouse potential of N_2O is much higher than of CO_2. A prototype N_2O sensor, based on photoacoustics, will enable continuous N_2O monitoring of the gas and water phase, thus setting the base for any optimization.

The cooperation with Prof. Laforsch (Bayreuth University) on polymer microparticles in Lake Garda and its tributaries received significant attention. Again, Raman microspectroscopy provided the analytical key to assess a new threat to aquatic wildlife and has shown its superior performance as within several other projects in the field of hydrogeology, aerosol science, and microbiology.

With my sincerest thanks for your continuous support,

Reinhard Niessner

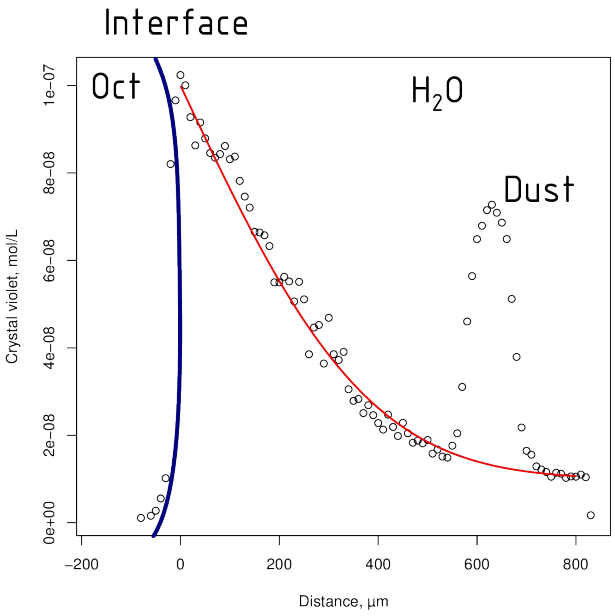
Hydrogeology (PD Dr. T. Baumann)

Investigating Biogeochemical Interfaces in Soil Using Micromodels and Raman Microscopy

Funding: DFG (Deutsche Forschungsgemeinschaft)
Cooperation: Partners in the DFG Priority Programme SPP 1315

Biogeochemical interfaces (BGI) in soil control the fate of organic chemicals and the functioning of soil as a filter to protect groundwater resources. Biogeochemical interfaces are transient in space and time, thus rendering batch tests under equilibrium conditions and without spatial restrictions inadequate to predict the overall behavior. Instead, the concentration gradients of organic chemicals have to be measured and the spatial and temporal dynamics of the BGI themselves have to be monitored.

Processes at BGI can be visualized and quantified using microfluidic structures mimicking the pore topology of the soil, so called micromodels. In combination with Raman microspectroscopy chemical information can be retrieved from a micromodel experiment with a spatial resolution on the order of 1 µm² and a temporal resolution in the seconds-range. To increase the sensitivity, silver nanoparticles have been added to the water phase flowing through the micromodel to make use of the amplifying surface-enhanced Raman effect. Currently chemical gradients of moderately lipophilic substances have been acquired with a limit of detection of 10^{-8} mol/L (see Fig. 1). Challenges to overcome include the interactions between silver nanoparticles and target analytes which might alter the mass transfer rates, and the settling of nanoparticles in the channel.



Concentration gradient of crystal violet at an 1-octanol-water interface

Imaging results at the interface and the development of the concentration gradient suggest, that the interface is highly dynamic in the beginning. Vortices with velocities in the upper µm/s range are reaching several dozens of µm from the interface into the solution. A diffusion controlled mass transfer is established at a later stage only. This observation might explain part of the first flush phenomenon.

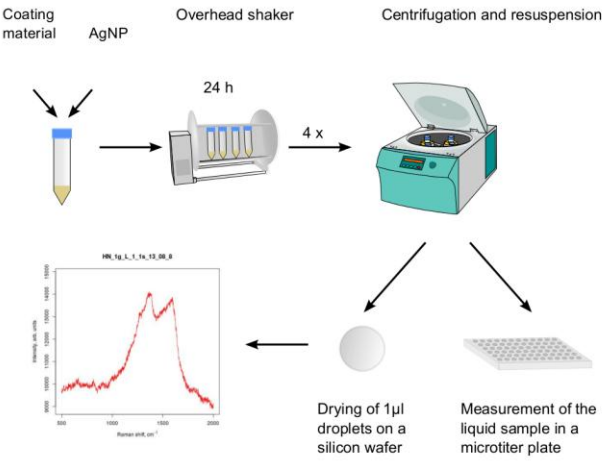
C. Metz

Effects of Physical and Chemical Surface Heterogeneity on the Transport of Engineered Inorganic Nanoparticles

Funding: DFG (Deutsche Forschungsgemeinschaft)
Cooperation: DFG Research Unit InterNANO (FOR1536)

Emissions of engineered inorganic nanoparticles into the environment are increasing sharply due to their growing applications in industry and consumer products. Simultaneously, there is an increasing number of reports regarding adverse impacts of EINP on aquatic ecosystems and possible health risks. EINP transport in aquatic and terrestrial environments is controlled by the physical and chemical heterogeneity of the matrix along the flow path and the properties of the EINP themselves.

While EINP are well characterized concerning their field of applications, there is still a lack of knowledge regarding their stability and transport behaviour when they are released into the environment. Additionally, EINP are likely to get coated with natural organic substances soon after release. Once the coating has formed, stability and transport behaviour of the nanoparticles will no longer be influenced by the core material, but mainly by the properties of the coating. Therefore, it is crucial to characterize the surface properties and stability of the EINP and their possible coating materials to predict their behaviour under environmental conditions.



Preparation of coated silver nanoparticles

However, analytical methods to characterize organic coatings on nanoparticles are scarce. One suitable tool for the investiagtion of coatings on silver nanoparticles is surface enhanced Raman spectroscopy (SERS). Silver nanoparticles are known to enhance the Raman signal of adsorbed or nearby substances by a factor of 10^3 - 10^6. This leads to a considerably higher sensitivity of SERS compared to normal Raman micro-spectroscopy.

Different silver nanoparticles were coated with humic acid and other organic substances, centrifuged and resuspended in deionised water. After this washing procedure, the samples were either dried on a silicon wafer or measured as liquid samples in a microtiter plate. Results indicate the formation of a stabilizing layer around the nanoparticles after contact with humic acid. Humic acid seems to form a quite stable coating around the nanoparticles that is even present after four steps of centrifugation and resuspension in deionized water.

M. Kühn

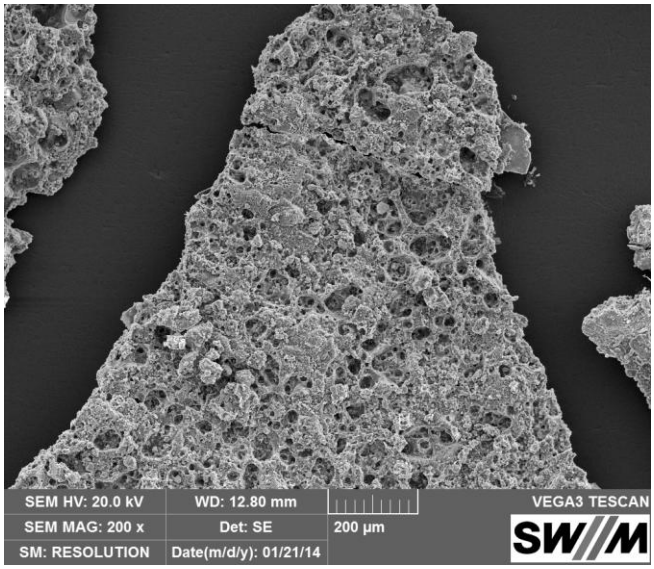
Minimizing Risks for Geothermal Power Plants

Funding: BMU (Federal Ministry for the Environment, Nature Conservation and Nuclear Safety)
Cooperation: SWM Services GmbH, Munich

The Malm aquifer in the Molasse basin, Bavaria, Germany, is ideally suited for the exploration of deep hydrogeothermal energy. Several ambitious projects for combined heat and power production were initiated over the last few years. Although the general conditions of the Malm aquifer are very favorable for geothermal power production with volume fluxes exceeding 100 L/s, temperatures above 140 °C, and low salinity, the technical details for long-term operation are far from trivial. Recent research projects have shown that higher concentrations of methane and H_2S can occur in the area around Munich and that carbonate equilibria are likely to be disturbed during production.

During the retrieval of the thermal water pump at the combined heat and power station at Sauerlach, operated by the Stadtwerke München GmbH, scalings were found on the production unit, inside the stand pipes, and in the ground level thermal water system. The thickness of the scalings was high immediately at the pump's outlet, decreased to minimum thickness roughly 200 m above the pump and increased again to the surface. The morphology and mineralogical composition of the scalings analyzed by SEM/EDX and XRD reveals mainly magnesium-calcite and some iron sulfides in the lower part of the stand pipes. The size of individual crystals in the scalings increases from the pump to the surface.

The results indicate a disturbance of the carbonate equilibria due to degassing. Indeed, gas bubbles can be observed in the thermal water at ground level inspection windows. These findings, however, are in contrast to model calculations under static conditions, which predict complete dissolution of all gases in the thermal water system under the given pressure conditions. A likely explanation to this discrepancy is a local pressure drop in the pump which leads to the formation of gas bubbles which, once formed, dissolve only very slowly in the thermal water flow due to little differences in the local partial pressures.



SEM image of scalings from the stand pipes

Scalings inside the pipes of the ground level facility where also found after filter systems and show little adhesion to the pipes due to the presence of an oil film. Although easily removable during maintenance cycles, these scales can also be mobilized by sudden pressure changes when starting and stopping the pumps. They might then clog the heat exchangers and reduce their efficiency significantly.

M. Herbrich

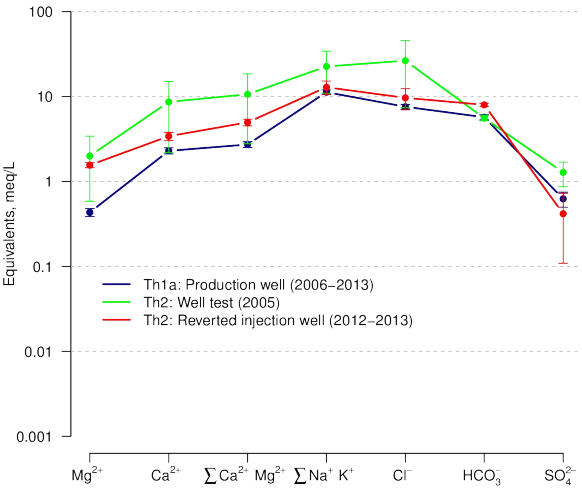
A Push-Pull Tracer Test at a Geothermal Well

Funding: BMU (Federal Ministry for the Environment, Nature Conservation and Nuclear Safety)
Cooperation: IEP GmbH, Pullach; Erdwerk GmbH, Munich; Aquasoil GmbH, Berlin

Geothermal exploration of the Malm aquifer in Bavaria is highly successful. Data about the long-term operation, however, is still scarce, although detailed knowledge about the processes occurring in the aquifer is a key requirement to run geothermal facilities efficiently and economically. While there usually is a constant flow of data from the production well (temperatures, hydraulic data, hydrochemical conditions, gas composition) not even the temperatures in the immediate surrounding of the reinjection well are accessible or known.

In 2011 the geothermal facility in Pullach was extended with a third geothermal well reaching into the Malm aquifer which is now used as a reinjection well. The former reinjection well was converted to a production well after 5 years of operation. This setting offers a unique opportunity to study the processes in the vicinity of a reinjection well and provides the data base to describe the hydraulic, thermal and hydrochemical performance of the reservoir.

As the viscosity of the injected cold water is at least 60% higher compared to the hot water in den reservoir, one would expect an increase of the reinjection pressure as the cold water plume spreads around the reinjection well. Measurements, however, show a significant decrease of the reinjection pressure at many geothermal sites in the area of Munich, suggesting processes in the aquifer which positively change the hydraulic properties and overcompensate the viscosity effects.



Schoeller diagram of the water injected into well Th2 (Th1a, blue) and produced water from well Th2 (red)

When the well Th2 was transformed into a production well in 2012, access was provided to injected thermal water. Not surprisingly many dissolved ions like Na^+, K^+, and Cl^- showed similar concentrations in the produced water compared to the injected water. However we also observe a significant increase of Ca^{2+}, Mg^{2+}, and the alkalinity as well as a decrease for sulfate. While the former indicate a dissolution process in the aquifer, the latter points to increased microbial activity,

The dissolution processes might explain the overcompensation of viscosity effects by either increasing the width of the flow paths in the matrix or reducing the skin effect, or both.

M. Lafogler

High-Temperature-Aquifer Storage

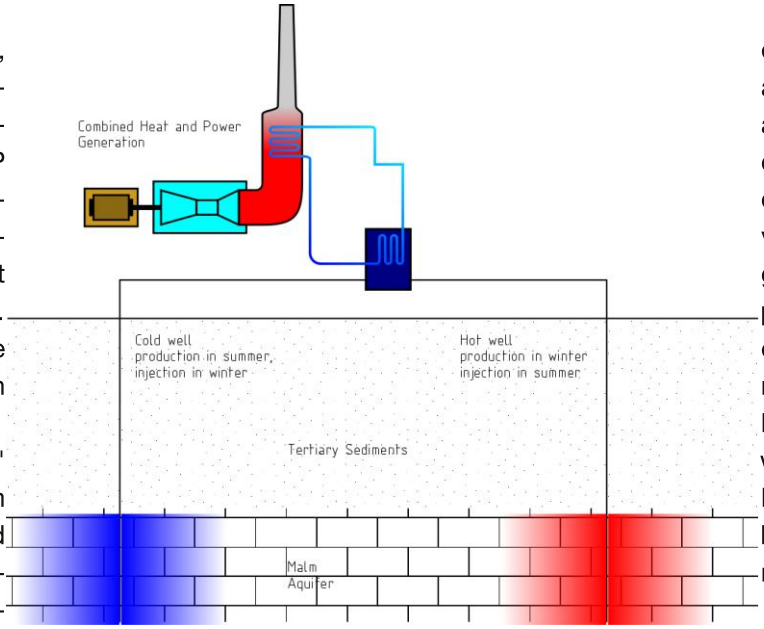
Funding: BMW (BayINVENT)
Cooperation: TUM Hydrogeology; Erdwerk GmbH, Munich; Aquasoil GmbH, Berlin

Combined heat and power generation (CHP) is highly efficient because excess heat is used for heating and/or process energy. However, the demand of heat energy is highly variable throughout the year while the demand of electrical energy is rather constant.

Unfortunately, at higher ambient temperature the CHP has to be ramped down, since the heat cannot be used.

Within the project "High temperature aquifer storage" scientists from mechanical and electrical engineering, geology, hydrogeology, and mathematics investigate storage and recuperation of excess heat energy into the Malm aquifer in Bavaria. Pilot studies indicate that high temperature energy storage is beneficial because of an increase of the total working hours of the CHP and a significant reduction of the CO_2 footprint.

Heat storage in a calcareous aquifer at injection temperatures above 100 °C is a challenge, not only because of the changes of the carbonate equilibrium at all levels of the facility. Therefore, a pilot scale heat storage test will be performed at an industrial site. This test includes a push-pull tracer test with conservative and reactive tracers and will later be expanded to a full scale demonstration project.



Schematic of the high temperature aquifer storage

Laboratory experiments in an autoclave and hydrogeochemical modelling will provide the background for the pilot scale tests, especially with regard to the handling of the waters at ground level and the behaviour of the reservoir.

Besides findings about transport and heating behaviour in the core samples, the interaction of the injected tracers with the matrix under reservoir conditions will be quantified. An integrated camera system delivers information about precipitation processes.

M. Ueckert

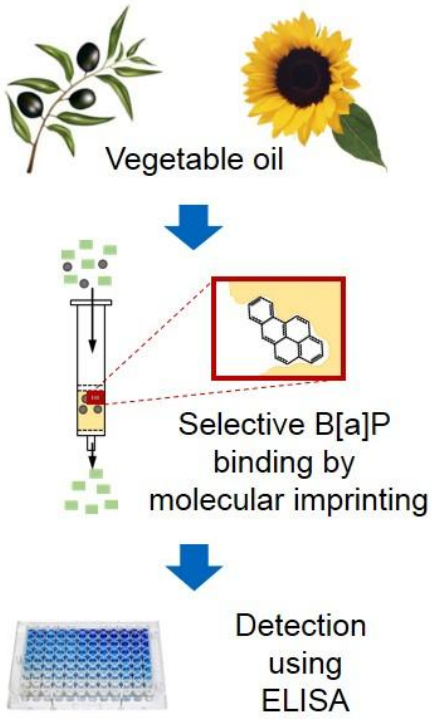
Bioanalytics (Apl. Prof. Dr. D. Knopp)

Paratope-Epitope Interactions of Benzo[a]pyrene Antibodies and Extraction of B[a]P in Edible Oils

Funding: Hanns-Seidel-Stiftung, IWC
Cooperation: Lehrstuhl für Biologische Chemie, TU München, Prof. Dr. A. Skerra; Oil Crops Research Institute, Chinese Academy of Sciences, Wuhan, China, Prof. Dr. Peiwu Li, Dr. Ran Li

Polycyclic aromatic hydrocarbons (PAHs) are formed during incomplete combustion of organic compounds. Because of their high toxicity, limit values were set by the European Commission (Council Directive 98/83/EC) for benzo[a]-pyrene (B[a]P) of 10 ng/L in tap water and 2 µg/kg in edible oils. Sensitive and reliable analytical methods are needed to detect B[a]P at these low concentrations.

In the past, we reported on the development of recombinant antibodies (scFv) based on the genetic information of three highly affine monoclonal antibodies, which displayed comparable assay sensitivity to the corresponding complete antibody molecules. For further characterization of the binding mechanism and an evaluation of possibilities to increase assay sensitivity by genetic manipulation the X-ray crystallography should be used. For production of crystals suitable for X-ray scattering Fab fragments have been produced by recombinant techniques. The Fab properties have been optimized for crystallization and the produced Fabs were characterized for their affinity towards B[a]P using ELISA and Surface Plasmon Resonance (SPR).



Schematic procedure for Benzo[a]pyrene determination in edible oils

With the existing highly affine murine monoclonal antibodies an immunological method for the determination of B[a]P in edible oils should be established. To reach this goal, matrix effects of the lipidic matter have to be minimized, e.g., by effective solid phase extraction of B[a]P to guarantee maximum affinity of the antibodies and thus high sensitivity of the immunoassay. Selective polymeric materials have been prepared using molecular imprinting technology and an extraction procedure for a sensitive immunological determination of B[a]P in different edible oils has been established allowing the detection around the limit value for B[a]P set in the EU directive. Still, further optimization and validation of the method is necessary to achieve a fast and reliable immunological test method.

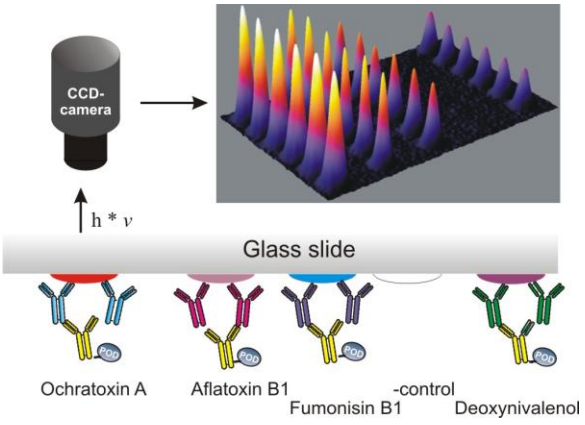
M. Pschenitza

Hapten Microarray-Based Screening of Mycotoxins in Cereals

Funding: AiF-FEI (German Federation of Industrial Research Associations), Verband Deutscher Mühlen

Cooperation: Lehrstuhl für Hygiene und Technologie der Milch, LMU München, Prof. Dr. E. Märtlbauer, Dr. R. Dietrich

The mycotoxins are secondary metabolites from fungal species such as Aspergillus, Fusarium, Alternaria and Penicillium growing on agricultural commodities in the field or during storage. Since, they represent a potential health hazard to humans and animals, maximum levels for several mycotoxins in food have been set by the European Community (EC). Because multi-mycotoxin methods are highly desirable in order to keep analysis time and costs low, the biosensor development increasingly focuses on parallel analysis of several mycotoxins. In this project, an indirect competitive immunoassay on regenerable, reusable glass microchips for the parallel determination of four mycotoxins, i.e., aflatoxins, ochratoxin A, deoxynivalenol, and fumonisin B1 in three different cereal extracts on a fully automated flow-through device (MCR 3) with chemiluminescence readout was developed.



Principle of the immunoassay using the microarray chip

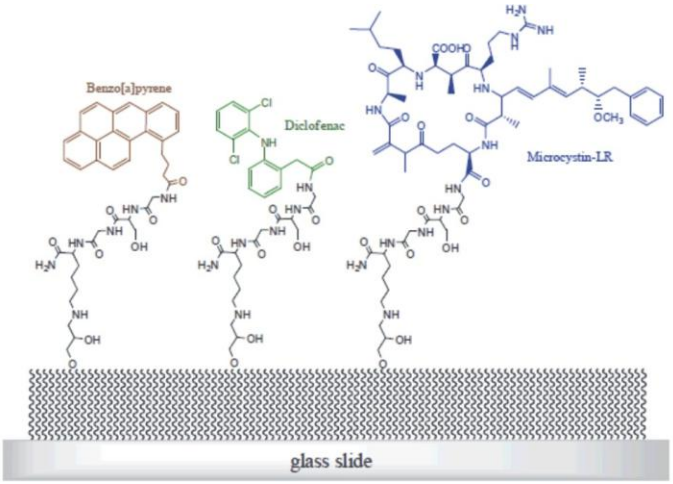
The total assay time, including extraction, extract dilution, measurement and surface regeneration, was only 19 min and the crude extracts could be used without further purification other than filtration and dilution. Thus, a rapid sample analysis was possible. The prepared microarray chip was reusable for at least 50 times. Due to a batch-to-batch variation of the maximum signal intensity of more than 20 %, each new batch requires a new calibration of the analytical system demonstrating the need for further optimization of the chip preparation step. Cleanrooms with controlled environment are indispensable in this context. In-lab validation of the chip revealed that oat extract could be used as representative sample matrix for preparation of mycotoxin standards and determination of different types of cereals such as oat, wheat, rye, and maize at relevant concentrations set by the European Commission. The use of methanol/water (80:20, v/v) as extraction solvent yielded a good compromise to obtain acceptable recoveries of the four mycotoxins with distinct polarity features. This could be demonstrated with both fortified and naturally contaminated samples (obtained from Rosenmühle, Landshut, Germany) and commercially available reference materials (obtained from IRRM, EU Joint Research Centre, Geel, Belgium and from Biopure, Tulln, Austria). Some further improvement of the extraction yield might be expected by fine-tuning of the extractant mixture. Future efforts will be devoted to the inclusion of other mycotoxins relevant to cereals.

S. Oswald

Nanoscaled Architectures for Highly Sensitive Biosensing of Small Molecules

Funding: DFG (Deutsche Forschungsgemeinschaft)/ANR (Agence Nationale de la Recherche)
Cooperation: Université Pierre et Marie Curie, Paris, France, Prof. Dr. S. Boujday, Prof. Dr. A. Proust, Prof. Dr. C.-M. Pradier

Pharmaceuticals, combustion products and pollution-borne toxins are harmful to living organisms, and wildlife. Consequently, the water-related environmental legislation in Europe defines an urgent need for regular monitoring of the so-called priority substances down to nanogram per liter levels (EC Directive 2000/60/EC).



Antigens of different chemical structures immobilized on a glass slide for regenerable immunoassay measurements

The challenge is to separate these pollutants at very low concentrations from complex matrices. The detection methods should be rapid, cheap, simple and reliable in order to handle increasing sample loads. Immunoassays provide inherent features for such economical screening methods. They can be selective, sensitive and fast screening methods for environmental samples.

The micropollutants diclofenac, microcystin-LR and benzo[a]pyrene are used as master analytes for the development of a sensitive and selective indirect competitive enzyme-linked immunosorbent assay for the multiplex analysis of water samples.

Commonly, immunoassays are carried out using microtiter plates or in a tube format. In contrast, we make use of the Microarray Chip Reader (MCR 3), a platform for the automated screening of the samples for simultaneous measurements of the three analytes.

For the development of the screening test an antigen microarray is transferred to a modified glass slide. In cooperation with the project partners, the microarray surface is studied in-detail on the molecular level and in the nanometer range. This analysis allows the establishment of a quality control of the chip preparation. The created surface with immobilized antigen is robust enough to remove the bound antibody for at least 30 times. For the elaboration of these sensitive immunoassays, the best antibodies for each target have to be available. For benzo[a]pyrene and microcystin-LR, highly specific and affine monoclonal antibodies already exist. For diclofenac, highly affine and specific monoclonal antibodies are prepared during this project. In the course of the project, these antibodies will be used to develop new surface preparations with nanoscaled architectures by the project partners. These nanostructured surfaces are created by the use of polyoxometalates, layered silicates or gold nanoparticles.

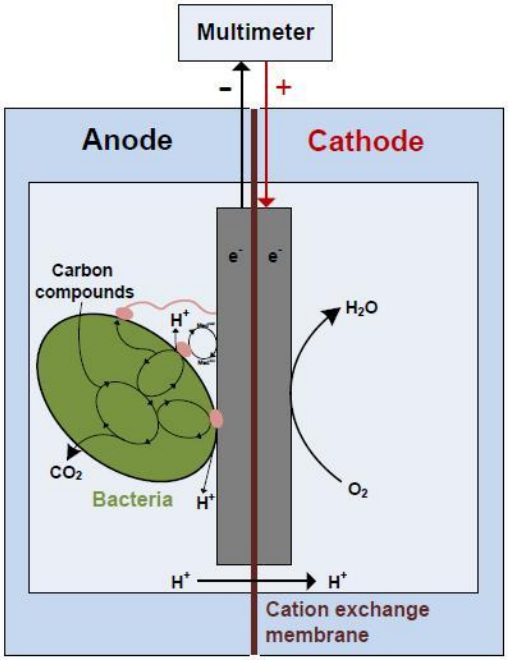
M. Hübner

Microbial Fuel Cell - Development and Application as Biochemical Oxygen Demand Sensor

Funding: IWC
Cooperation: Max von Pettenkofer-Institut, Prof. Dr. S. Schubert, SGL Carbon, Dr. R. Schweiss, Helmholtz-Zentrum Neuherberg, Dr. J. Bosch, TUM, Dr. W. Ludwig, Dr. F. Hasché

Due to demographic change small wastewater treatment plants become more important in rural areas of Germany. The development of an in-situ sensor to determine the biological oxygen demand (BOD) might be a conceivable approach to reduce their energy consumption and costs. A correlation between the pollution of the inlet wastewater and the attended amount of oxygen to decompose organic substances can be created in-situ and thus the aeration can be regulated immediately. In this project a microbial fuel cell (MFC) should be the principle of the BOD-sensor where microorganisms react sensitive on changes in the wastewater pollution. First, the aim of this study was to establish a basic state of knowledge to comprehend effects on the operating mode.

Measurements in batch-mode and continuous flow were possible and when Lysogen-Broth-Medium was flushed through the MFC maximum values of 0.9 mA and 640 mV could be generated. Measurements in batch-mode delivered a correlation between the inserted mass of bacteria and the maximum signal in the first two hours of the record. Continuous flow measurements showed a dependency between the obtained current signal and three copper concentrations, as an example, could be observed and at this juncture a response time for the MFC could be determined. It takes 2.4 hours until the plateau of the curve is reached and as a result of this the signal changed significantly after the medium arrives in the MFC. Continuous flow measurements with temperature change from 25 to 37°C yielded an association between the yielded current signal and van't Hoff's



Schematic setting of a MFC

rule according to which the enzyme activity of bacteria doubles with increasing the temperature by 10°C.

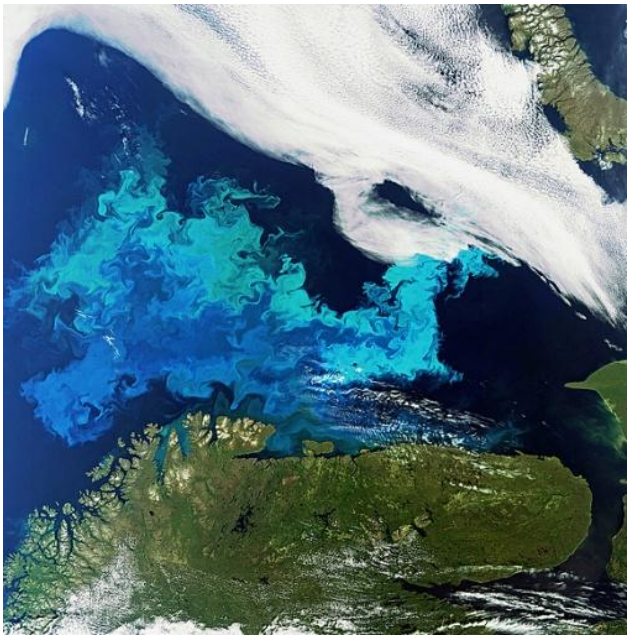
Based on these early results, further investigations will focus on parameters like the used bacterial strains (e.g., usage of mixed cultures directly from the wastewater treatment plant), temperature, and substrate supply.

A.-C. Neumann

Depletion of Algal Toxin-Contaminated Water Using Selective Biofilters Based on Plant-Produced Antibodies (Plantibodies)

Funding: DFG (Deutsche Forschungsgemeinschaft)/FWF (Fond zur Förderung der wissenschaftlichen Forschung)
Cooperation: University of Natural Resources and Life Sciences (BOKU), Vienna, Austria, Prof. Dr. E. Stoeger

Untreated sewage effluent and agricultural run-off carrying fertilizers are examples of human-caused eutrophication in lakes and rivers. The enrichment of nutrients generally promotes excessive plant growth and decay, favouring simple algae and plankton over other more complex plants, and causes a severe reduction in water quality. Organisms that are responsible for these changes are photosynthetic bacteria called cyanobacteria, which are originally classified as algae and commonly called blue-green algae. Cyanobacteria produce a wide variety of unique secondary metabolites like anatoxin, saxitoxin, nodularin and microcystin.



Algal Bloom in the Barents Sea as seen from space (Photo by Keith Cowing, 2011)

The joint project is focussed on microcystin-leucine-arginine (MC-LR) because it is the most toxic microcystin congener, destroying liver cells for example. The World Health Organization recommends that the amount of free and cell bound MC-LR in drinking water should be limited to 1 µg/L. Germany and the United States aspire this proposal.

A monoclonal anti-microcystin antibody (MC 10E7) was generated previously at the Institute of Hydrochemistry and based on the antibody-antigen-interaction the development of an efficient, selective and cost-effective biofilter for the depletion of the antigen (microcystin) from water samples is conceivable. As a biofilter, in this context, could serve a suitable support material which is doped with anti-microcystin antibodies.

Little attention has so far been paid to the production of antibodies in plants and their ex vivo application in selective depletion. Therefore, highly affine and specific antibodies against algal toxins using microcystin as an example will be produced in plants (so-called plantibodies) at low cost within this research project. The project will go beyond to confirm the theoretical possibility of the production of plantibodies against environmental contaminants, but rather to achieve the production of practically relevant antibodies for further applications in several formats.

A.-C. Neumann

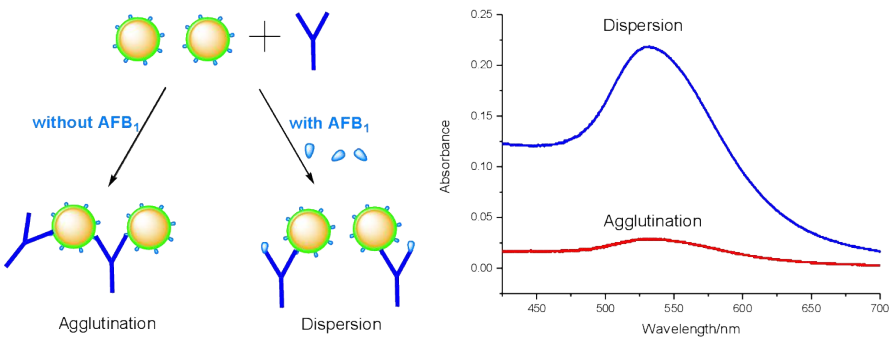
Simple and Rapid Detection of Aflatoxin B_1 Using Gold Nanoparticles

Funding: IWC, China Scholarship Council

Aflatoxins are highly toxic secondary metabolites produced by *Aspergillus flavus* and *Aspergillus parasiticus*. They may be present in a wide range of food and feed commodities. Among them, aflatoxin B_1 (AFB1) is most prevalent and toxic even at very low concentrations. Although various analytical methods and detection techniques have been applied for the qualitative and quantitative analysis of AFB1 in foods, still faster and simpler methods are increasingly being requested by the food industry.

Optical biosensors coupled with nanotechnology provide a new approach to address this issue. Among the numerous nanoparticles synthesized at the present, gold nanoparticles (GNPs) are among the most attractive ones in nanobiotechnology due to their special physical and chemical properties, such as strong absorption, non-photobleaching and blinking, ease of synthesis, simplicity of conjugation chemistry and excellent biocompatibility. The GNPs-based assay is perhaps one of the most powerful nanosensing methods available so far. The use of GNPs as a colorimetric platform is based on the unique distance-dependent optical property of GNPs. That is, monodisperse GNPs take on red, but as the interparticle distances decrease to less than approximately the average particle diameter, the absorbance decreases greatly. This special property of GNPs is widely used in bio-analysis.

A simple, rapid and cost-effective nanoprobe was fabricated for AFB1 determination based on the aggregation of GNPs. Free AFB1 molecules dispersed solution compete with nanoprobes to combine with the anti-AFB1 antibodies. Nanoprobes aggregate when the amount of free AFB1 is not enough, leading to a large decrease in absorbance. And fewer amount of GNPs aggregate with the increase of AFB1 concentration, as a result of competition. The amount of nanoprobe and antibody, incubation time, as well as the buffer solutions were investigated and successfully optimized. A linear correlation exists between the absorbance of nanoprobe and the AFB1 concentration over a distinct range. Higher sensitivity could be achieved by converting the absorption signal to fluorescence or light scattering.



Schematic representation of the AFB1 determination using GNPs

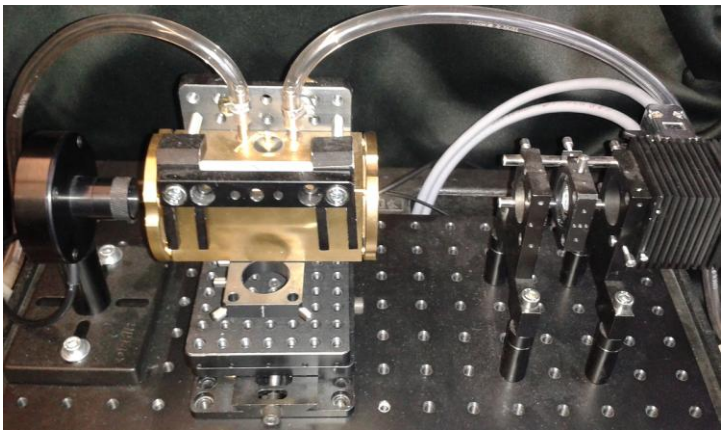
X.Wang

Applied Laser Spectroscopy (PD Dr. C. Haisch)

Photoacoustic Spectroscopy for Gas Analysis

Funding: IWC, International Graduate School of Science and Engineering (IGSSE)
Cooperation: Nanoplus GmbH, Meiningen-Dreißigacker

Trace gases like nitrous oxide is a strong greenhouse gas and contribute to the depletion of the ozone layer. One anthropogenic source for climate-relevant trace gases is the exhaust gas of combustion engines, another source are wastewater treatment plants. Both these sources require for a continuous monitoring system in order to reduce emissions. Although these sources are so different, the analytical challenges in monitoring the corresponding emissions are similar. Both require for time-resolved analysis with a resolution between seconds and minutes, the sensitivity should be as low as 300 ppb, which is the range of the natural background concentration.



Experimental setup for photoacoustic gas monitoring

Photoacoustic (PA) spectroscopy has proven to be a very sensitive method for gas analysis. The principle of PA spectroscopy is the conversion of absorbed light energy into acoustical waves which can be detected by means of a microphone. The robustness and simplicity of some PA techniques allow for in situ monitoring. A typical PA setup consists of a laser, a PA cell, a microphone and a lock-in amplifier. The cell design can be modified to optimize the signal-to-noise ratio. Resonant cell designs, like Helmholtz resonant cells, can be used in PA systems and achieve high amplification factors. Combined with differential lock-in amplification a highly sensitive detector system is possible.

The aim of the presented research is to build a detector system dedicated to in situ monitoring of N_2O. According to the spectroscopic data of N_2O, a suitable infrared laser has been chosen and an LOD of 1.5 ppm N_2O in dry air has been achieved. With an enhanced setup and an optimized PA cell design LODs in the sub-ppm region should be possible, which allows for numerous other applications. Beyond the instrument itself, the necessary infrastructure for the gas sampling from exhaust gas as well as from the liquid phase in the wastewater treatment plant has to be established.

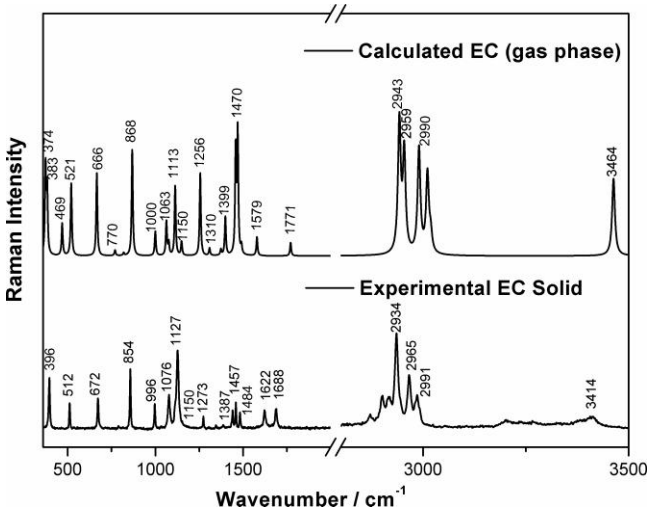
C. Berger

Surface Enhanced Raman Scattering Detection of Ethylcarbamate Detection in Alcoholic Beverages

Funding: IWC

Ethylcarbamate, a by-product of fermentation and storage with widespread occurrence in fermented food and alcoholic beverages, is a compound potentially toxic to humans. In this work, a new approach for quantitative detection of ethyl carbamate in alcoholic beverages, based on surface-enhanced Raman scattering (SERS), is reported. Individual silver-coated gold nanoparticle colloids are used as SERS amplifiers, yielding high Raman enhancement of ethyl carbamate in three kinds of alcoholic beverages (vodka, Obstler, and white rum). The Raman bands observed for EC in solid phase are characteristic for the carbonyl group, C–C, C–H, and N–H stretching and deformation vibrations. These spectral features coupled with a pKa study allowed establishing the neutral species of EC present in the aqueous solutions experimentally tested at different concentrations. In addition, by performing a density functional theory study in the gas phase, the calculated geometry, the harmonic vibrational modes, and the Raman scattering activities of EC were found to be in good agreement with our experimental data and helped establish the surface-enhanced Raman scattering (SERS) behavior and EC adsorption geometry on the silver surfaces. The strongest and best reproducible peak in the SERS spectra at 1006 cm^{-1} was used for a quantitative evaluation of EC. The limit of detection, which corresponds to a signal-to-noise ratio of 3, was $9.0 \cdot 10^{-9}$ M (0.8 µg/L), $1.3 \cdot 10^{-7}$ M (11.6 µg/L), and $7.8 \cdot 10^{-8}$ M (6.9 µg/L), respectively. Surface-enhanced Raman spectroscopy offers great practical potential for the in situ assessment and identification of ethyl carbamate in the alcoholic beverage industry.

D. Yang, N. E. Mircescu

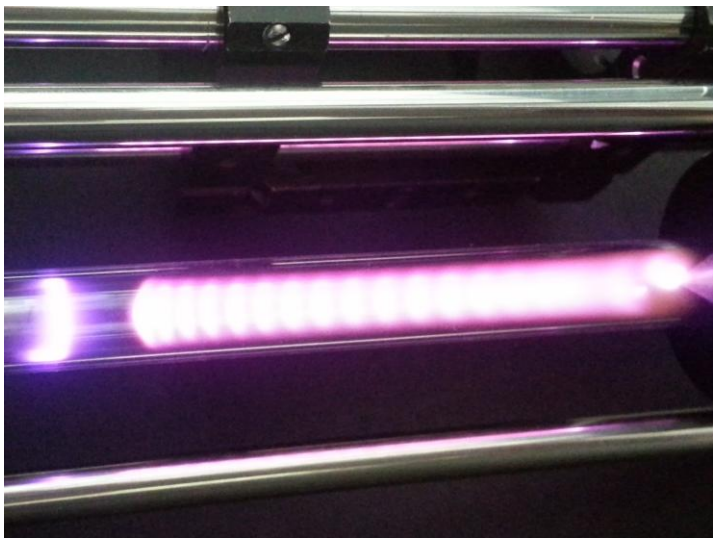


Comparison of calculated and measured Raman spectra of ethyl carbamate

A Novel Plasmaspectroscopic System for Online Monitoring of Siloxanes in Biogas

Funding: IWC

Due to rising energy consumption and the drive to renewable energies, biogas is increasingly used as an energy source. The number of biogas plants has more than quadrupled in the last ten years in Germany.



Gas discharge plasma as used for siloxane monitoring

Depending on the way of energy exploitation, determination of the composition and in some cases the removal of certain components is required. Siloxanes are undesirable trace component, which can be found in varying concentrations in biogas from wastewater treatment. If these organosilicon compounds reach the combustion engines, they get oxidized to silicon oxide, which gets deposited in form of a white precipitate on inner parts of the engine. The crystalline SiO_2 can clog valves and cause overheating of motor parts, because the deposits act as a thermal insulator. Operators of landfills and sewage treatment plants must ensure that the siloxane concentration in combustion gas remains below the limits recommended by the engine manufacturer.

The goal of this project is the quantification of these compounds in biogas. A novel method, based on plasma emission spectrometry, is used. A gas discharge, generated by high voltage, served as a plasma source. Its optical emission is detected by a spectrometer. By comparing several measuring cells, optical devices, and high-voltage sources low detection limits are achieved.

The construction of a mobile measurement system was prepared, which will allow measurements under practical conditions. This instrument will fit into a 19˝ rack and it will be robust enough to be installed in a biogas production environment.

In a next step, this mobile system will be installed in a wastewater treatment plant or a similar installation for performance testing under routing operation conditions. In the future, this system could be used as a tool for routine analysis in biogas plants.

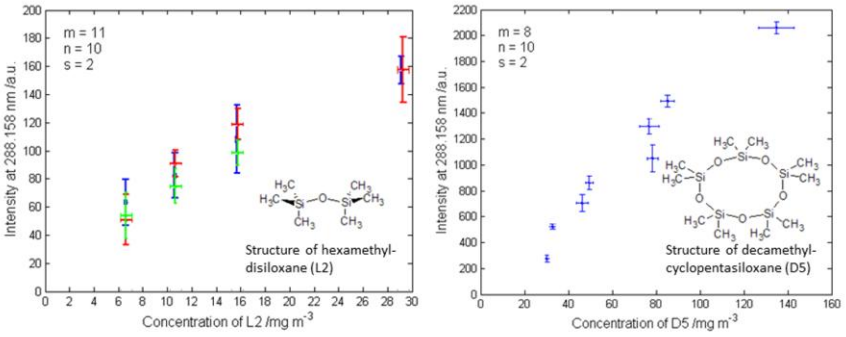
C. Thaler

Continuous Monitoring of Biogas Contamination Using Laser Induced Breakdown Spectroscopy and Raman Spectroscopy

Funding: EU
Cooperation: Italian Agency for New Technologies, Energy and Sustainable Economic Development, Italy; TUBITAK Marmara Research Centre, Turkey; University of Genoa, DICAT department, Italy; Royal Institute of Technology KTH, Sweden; OVM-ICCPET Institute, Romania; Joint Research Centre JRC, Belgium

As part of the research and development of Molten Carbon Fuel Cells (MCFC), the use of biogas as a renewable energy source is tested. Fuel cells gain energy by transformation of hydrogen which is produced by reformation of the methane. The composition of biogas depends on the substrates used for the fermentation. It consists of 40-75% methane, 25-55% carbon dioxide and different trace components in the ppm range. These are halogenated hydrocarbons, sulfur-containing compounds and siloxanes. These contaminants cause undesired reactions at the anode and cathode, which reduce efficiency and lifetime of the cell. In order to protect the MCFC from damage, a continuous monitoring of the cleaning process is necessary. An analytical method should be able to quantify low concentrations and detect a wide range of different components.

Two spectroscopic methods are combined for this analytical problem: The laser-induced breakdown spectroscopy (LIBS) and Raman spectroscopy. For LIBS, a pulsed laser beam is tightly focused into the sample, and generates a plasma plume with a temperature of more than 10000 K. The molecules are atomized and ionized by the energy input. The plasma first emits a nonspecific radiation (bremsstrahlung and recombination radiation) and then element-specific radiation of the atoms. The emission intensity correlates with the concentration (see Figure). Raman spectroscopy is used to determine the molecular composition of the biogas. The light beam is scattered by the sample. This generates a frequency shift; the difference to the incident light corresponds to the characteristic molecular vibration of the material, which can be detected. For the combination of both measurement methods, we used a special spectroscope that has two separate light paths available and just one CCD camera is needed.



Calibration of the new system for different siloxanes

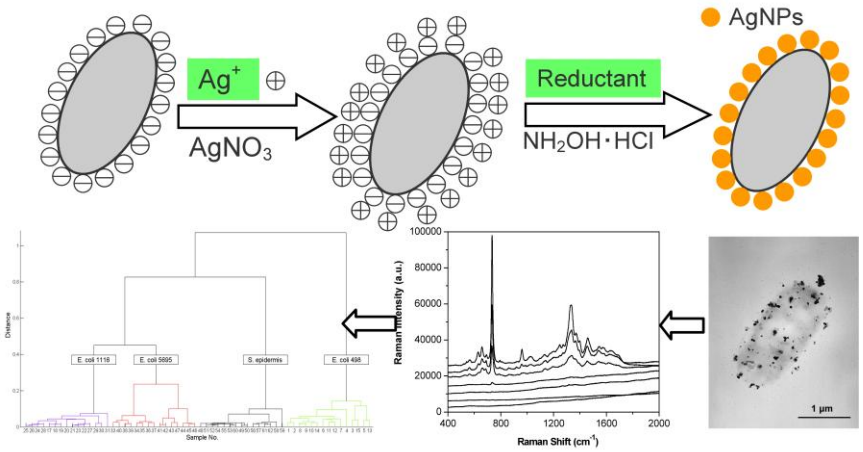
The determination of the various pollutants of the biogas is possible by the combination of the mentioned methods. It can be detected the atomic and molecular composition. An important aspect of the device is that it can cover a wide range of concentrations of various components.

K. Schwarzmeier

SERS Detection of Bacteria in Water by In Situ Coating With Ag Nanoparticles

Funding: IWC, China Scholarship Council

Biosensing for the convenient detection of bacteria has been widely explored with the use of various sensing materials and techniques. It is still a challenge to achieve ultrasensitive and selective but simple, rapid, and inexpensive detection of bacteria. We report on surface-enhanced Raman scattering (SERS) for living bacteria detection in drinking water by employing a synthesis of silver nanoparticles coating the cell wall of bacteria (Bacteria@AgNP). We found that the Raman signals intensity of bacteria after AgNP synthesis mainly depends on the zeta potential of the cell wall. The enhancement of the Raman signal of bacteria using this strategy is about 30fold higher than that in the case of simply mixed colloid-bacterial suspension (Bacteria-AgNP). The total assay time is only 10 min and the total reagent volume needed in order to analyze bacteria in a real environment is as low as 1 mL. Particularly, only one droplet of 3 µL sample is necessary for each SERS measurement.



Schema of the novel SERS colloid production and corresponding results (cluster characterization of different bacteria)

Furthermore, we were able to use this novel strategy to discriminate three strains of E. coli and one strain of S. epidermidis by hierarchy cluster analysis (HCA). Finally, we can detect bacteria down to 2.5 · 10^2 cell/mL on a hydrophobic glass slide by SERS mapping. Thus, our detection method offers prominent advantages, such as reduced assay times, simple handling, low reactant volumes, small amount of sample, and higher sensitivity and selectivity compared to previously reported label free methods. This novel strategy may be extended to open an avenue for developing various SERS-based biosensors.

H. Zhou

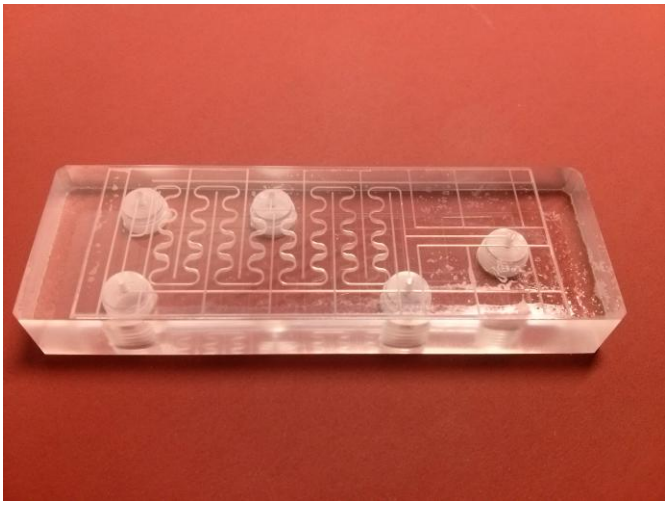
Microfluidic Devices for the SERS Detection of Bacteria

Funding: IWC, China Scholarship Council, DAAD

Reliable and fast detection of microorganisms is required in many fields, such as food production and medical diagnostics. Although there are many conventional methods for microorganisms detection, such as plating and culturing, flow cytometry, enzyme-linked immunosorbent assay, and polymerase chain reaction have been developed, these methodologies are limited either regarding sensitivity, speed, or throughput for routine low level bacteria detection. Since the SERS effect was discovered at rough silver electrode surface, SERS technique has become a subject of interstate for the detection of bacteria, motivated by the potential application on single whole microorganism. Even though SERS is highly specific and sensitive and well suited for bacteria samples, all SERS measurement still suffer from low reproducibility of spectra. To solve this disadvantage, one of the choices is the implementation of a microfluidic device. The integration of lab-on-a-chip fluidic device also provides many other advantages, such as limiting the assay time, reduced reagent volume, and possible automatic operated.

Within this study, we reported on SERS for living bacteria detection in a microfluidic cell by employing a synthesis of silver nanoparticles coating the cell wall of bacteria. Different low-priced microfluidic devices were developed and compared regarding device production as well as regarding applicability for routine SERS application.

We found that the Raman signals intensity of bacteria obtained by this way is higher than that in the case of pre-synthesis method. Furthermore, in order to increase the concentration of bacteria at the detection point in the microfluidic cell, we used the electrostatic attraction force strategy for capturing the bacteria at the corresponding modified microchannel area.



Possible microfluidic device for lab-on-the-chip SERS characterization of bacteria

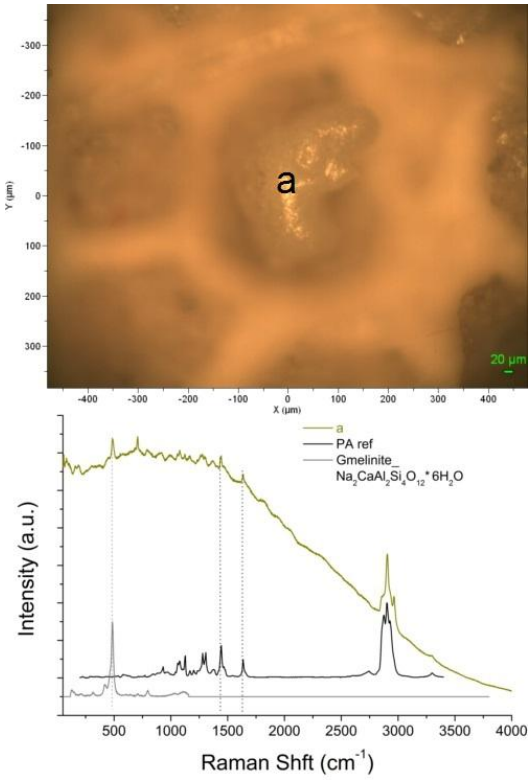
H. Zhou, C. L. do Lago

Raman Competence Center (Dr. N. P. Ivleva)

Raman Microspectroscopic Analysis of Microplastic Particles in Aquatic Sediments

Funding: DFG (Deutsche Forschungsgemeinschaft)
Cooperation: Prof. Dr. C. Laforsch, H.K. Imhof, University of Bayreuth

Plastic and especially microplastic (< 5 mm) particles are of increasing concern in marine ecosystems, posing risk to the biota. However, a large portion of the plastic waste is produced onshore and enters even headwaters. This may come along with all the associated harmful consequences which have been reported previously for marine ecosystems. Hence, microplastic particles may also harbor the risk of entering limnetic food webs. However, there is until now a considerable gap of knowledge on the contamination of freshwater ecosystems with (micro)plastic particles.



Optical microscope image of small microplastic (a) from Lake Garda, Italy; Raman spectra of the particle a and reference compounds

We analyzed the abundance of plastic particles in beach sediments from the subalpine Lake Garda, Italy. The sample preparation was based on density separation. Identification and quantification was performed using Raman microspectroscopy (RM). RM provides vibrational fingerprint spectra of plastic and other (in)organic compounds with spatial resolution of an optical microscope and allows us to analyze different types and size classes of plastic particles. We found the majority of plastic particles at the north shore of Lake Garda with 483 ± 236 macroplastic particles/m^2 and 1,108 ± 983 microplastic particles/m^2. At the south shore 8.3 macroplastic particles/m^2 and 108 ± 55 microplastic particles/m^2, respectively were detected. Similar to marine, we found primarily low density polymers, namely polystyrene (45.6%), polyethylene (43.1%) and polypropylene (9.8%). However, in the size class of very small microplastic particles (9–500 µm), also polyamide (Figure) and polyvinylchloride were identified. Further research and standardized surveillance guidelines to control for microplastic contamination in freshwater ecosystems should help to foster future risk assessment and conservation strategies.

N. P. Ivleva

Raman Microscopic Studies on Accumulation of Pollutants by Biofilms in Aquatic Systems

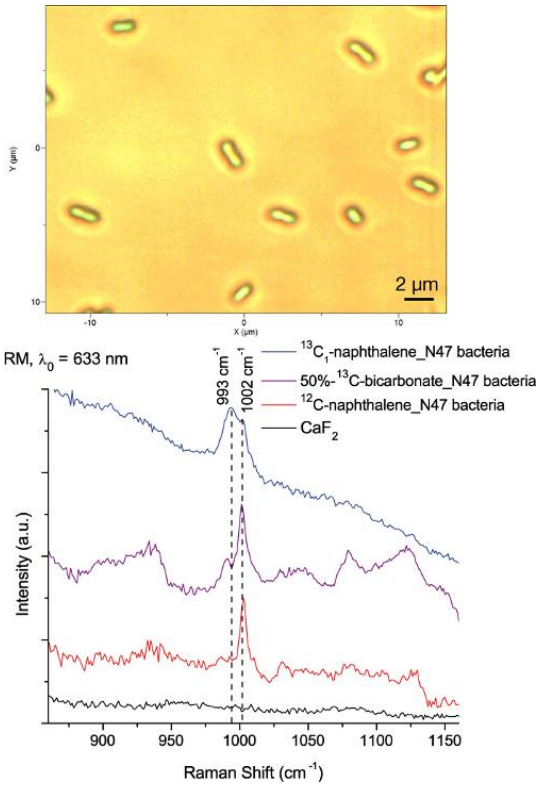
Funding: Helmholtz Zentrum München (Water Alliance)
Cooperation: Prof. R.U. Meckenstock, Dr. T. Lüders, Institute of Groundwater Ecology, Helmholtz Zentrum München

Most microbial cells live in the form of biofilms on our planet. These multicellular communities of microorganisms (bacteria, protozoa, algae and fungi) surrounded by a hydrogel matrix of extracellular polymeric substances (EPS, biopolymers such as polysaccharides, proteins, nucleic acids, lipids) are called biofilms. They play an important role in the degradation of pollutants, but they are very sensitive to varying boundary conditions. Therefore a rapid and noninvasive analytical tool for chemical characterization with high spatial resolution and sensitivity is required.

Raman microspectroscopy (RM) is a powerful tool for an in situ nondestructive chemical characterization of biofilm matrix in the µm-range. This vibrational spectroscopy allows a noninvasive acquisition of Raman spectra without the interference of water. However, the quantum efficiency of 10^{-6} - 10^{-8} for the Raman effect and therefore the sensitivity is rather limited. Hence surface-enhanced Raman scattering (SERS) can be used to enhance the intensity of the Raman bands significantly. SERS occurs if a molecule is attached to, or in immediate proximity of a nanometer-roughened metal (e.g. Ag, Au) surface. With this improvement of the sensitivity of RM with SERS a reproducible and rapid analysis of the biofilm matrix even at low biomass concentration is possible.

In this project we analyze the accumulation/degradation of pollutants by biofilms related to ground water. Stable-isotopes (i.e. ^{13}C-tracer) are used to achieve a better understanding of degradation pathways of pollutants. It is already known that the Raman bands of proteins or nucleic acids in ^{13}C-labeled microorganisms show a characteristic red-shift in the Raman spectrum. The analysis of sulfate reducing N47 bacteria cultivated with ^{13}C-naphthalene show a clear red-shift of the bands in the Raman spectra of the microbiologic cells. In addition Raman and SERS measurements of $^{12}C/^{13}C$-glucose and phenylalanine mixtures revealed good correlation between changes in spectra and ^{13}C-content. The results should help in understanding the influence of biofilms on the flux, turnover and fate of natural and anthropogenic pollutants in regional water cycles.

P. Kubryk



Optical microscope image of single N47 cells; Raman spectra of N47 cells cultivated with ^{12}C- or $^{13}C1$-naphthalene with red shift of the Phe band from 1002 cm^{-1} to 993 cm^{-1}

Raman Analysis of Soil Organic Matter for Improving the Water Holding Capacity

Funding: International Graduate School of Science and Engineering (IGSSE), TUM
Cooperation: Prof. Dr. I. Kögel-Knabner, Chair of Soil Science, TUM

To minimize the economic and environmental consequences of drought an adequate amount of rainwater has to be captured and stored in soil. The plant roots further need to penetrate and proliferate the soil. In literature a correlation between the available water capacity (AWC) and soil organic matter (SOM) is stated. The water holding capacity is discussed to be increased through the influence of the SOM on the structure and aggregation formation. Despite some publications on this topic there is still the need to investigate the causative mechanism.

Soil with biochar amendments at the field trial (Lusignan, France)

The complex composition of soils poses a challenge for the analytical method. Therefore Raman microspectroscopy (RM) will be applied in combination with state-of-the-art soil science methods. RM provides a non-destructive spectroscopic analysis which is based on the inelastic scattering of light and enables the study of soil amendments with a high spatial resolution and a comparison of optical and spectroscopic images. Different types of organic matter amendments from several field trials will be examined. The samples undergo a physical fractionation to identify the soil fractions in which the degradation and storage of SOM occurs. With the comparison of RM and established soil science techniques like NMR new informations on the storage and degradation of SOM will be gained.

The combination of RM with stable isotope techniques will also be applied (Stable isotope RM). After the output of ^{13}C labelled organic matter to different soils, the fate and transformation of the organic matter in bulk can be analyzed by RM due to a specific redshift of the bands of the labeled compounds in spectra.

By application of surface enhanced Raman scattering (SERS) the low intensities of RM can be increased. A strong enhancement of the Raman signal occurs close to metal surfaces like gold or silver. This can be explained by an electromagnetic enhancement due to surface plasmon resonance and a chemical enhancement due to charge transfer complexes.

The obtained data will be compared to the soil properties to gain information on the effects of SOM on the formation and structure of aggregates and therefore on the water holding capacity.

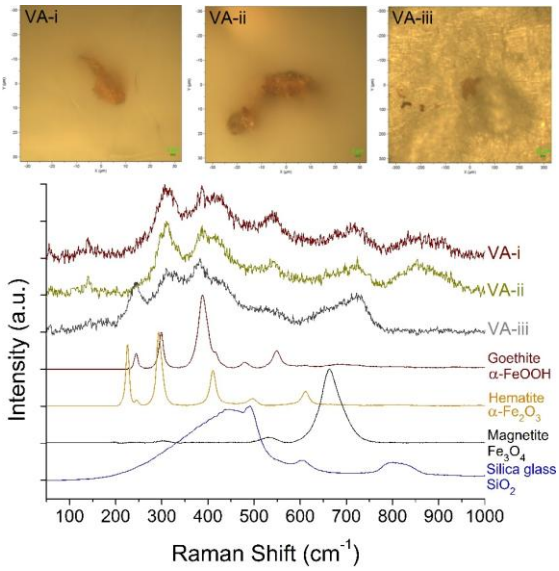
A. Wiesheu

Raman Microspectroscopic Identification and Characterization of Individual Airborne Volcanic Ash Particles

Funding: International Graduate School of Science and Engineering (IGSSE), TUM
Cooperation: Prof. Dr. B. Weinzierl, Deutsches Zentrum für Luft- und Raumfahrt, DLR

Volcanic ash particles can have significant impact on climate, public health and air traffic safety. During the eruption of the volcano Eyjafjalla in Iceland in April/May 2010, volcanic ash (VA) plumes were ejected into the atmosphere up to 9 km a.s.l. and distributed over Europe, which ceased air traffic in 23 European countries for several weeks. Different methods for the analysis of VA were applied during the last three years, however, no work concerning identification and/or characterization of individual airborne VA particles from Eyjafjalla (or from other volcanoes) using Raman Microspectroscopy (RM) was done before. Samples of fresh and aged airborne VA particles suitable for single-particle RM analysis were collected during aircraft research flights, operated by DLR on May 2th (from Keflavik to Stornoway; top part of the fresh plume over the North Atlantic) and May 18th (aged ash plume survey over Germany and North Sea). In addition, aerosol samples were collected at ground level at the IWC in Munich (TUM), using the Electrical Low Pressure Impactor (ELPI). Furthermore, volcanic ash particles taken at the ground near the eruption (June, 2010) and basaltic rock were studied by RM. We analyzed similarities between Eyjafjalla VA particles, collected at the different sampling sites, and compared them with the spectra of glassy and crystalline minerals by applying cluster analysis (CA).

The Figure shows representative VA particles from fresh plume, the corresponding Raman spectra, and spectra of some reference compounds. The fresh and aged plumes VA particles are brownish and exhibit similar spectral pattern with relatively broad bands near 310, 390, 415, 540, and 720 cm^{-1}, suggesting the presence of different minerals with rather disordered crystalline structure. A very broad band (200 - 700 cm^{-1}) is typical for quartz glasses. We assume a complex structure of VA particles, where different mineral phases are embedded in glassy matrix. Slight differences in the spectra of fresh and aged VA particles suggest variable proportions of the different minerals, partial precipitation, or chemical aging of VA during the transport. These results show that RM is indeed an effective method for identification and chemical characterization of individual airborne volcanic particles.



Optical microscope images of VA particles; Raman spectra of VA and reference compounds

N.P. Ivleva, C. Haisch, T. Baumann

Bioseparation and Microarray Technology (Dr. M. Seidel)

Fast Concentration and Multiplex Microarray Analysis for the Simultaneous Detection of Pathogens and Toxins in Food

Funding: *BMBF (Federal Ministry for Education and Research; 13N12613, LEVERA)*
Cooperation:Chair of Hygiene and Technology of Milk, LMU Munich; Institute of Veterinary Food Science, JLU Gießen; R-Biopharm AG (Darmstadt)

One of the main goals of governmental regulations in the field of public health aspects is to supply the population with hygienically and toxicologically harmless food. Especially in case of a widespread contamination outbreak, the source of infection and all contaminated products have to be identified as quickly as possible. Therefore, fast and multiplexed analytical methods are required to provide a rapid diagnosis and identification of pathogens and their toxins in/from food.

Experimental setup for combined concentration (right) and detection at the MCR 3 (left)

In this project, fast concentration methods and multiplex microarray assays will be developed for the simultaneous detection of pathogens and toxins in milk and food samples. We are mainly focused on the pathogens *Campylobacter spp*., *Cronobacter spp.*, *Clostridium spp.* and EHEC, the hygiene indicators *Staphylococcus aureus* and *Bacillus cereus*, and the toxins Staphylococcal enterotoxins, Shiga toxins and *B. cereus* enterotoxins. Research on new antibodies for concentration and detection will be carried out by our partners.

The concentration of the microorganisms and toxins from liquid (milk) and solid (meat, fish) matrices will be directly combined with the multiplex microarray analysis at the MCR 3. The aim of the project is to concentrate bacteria and toxins from 100 mL milk or 25 g homogenized meat and quantitatively detect them within a total analysis time of 1 h.

Two concentration methods are under development: monolithic immunofiltration (liquid samples) and immunomagnetic filtration (solid samples). The monolithic immunofiltration takes place in monolithic columns with immobilized capture antibodies. The respective microorganisms and toxins are separated from liquid samples and eluated, producing a concentrated pathogen/toxin solution. In contrast, the immunomagnetic filtration tolerates more complex matrices, therefore suitable for solid food samples. The sample is homogenized and mixed with our laboratory-made magnetic nanoparticles, previously coated with capture antibodies. After interaction, the particles are trapped by a strong magnet or a magnetic column, providing an analyte concentrated suspension.

The technology that will be developed in this project is of crucial importance to respond to shortcomings for food safety and product control.

E. Linares, V. Meyer

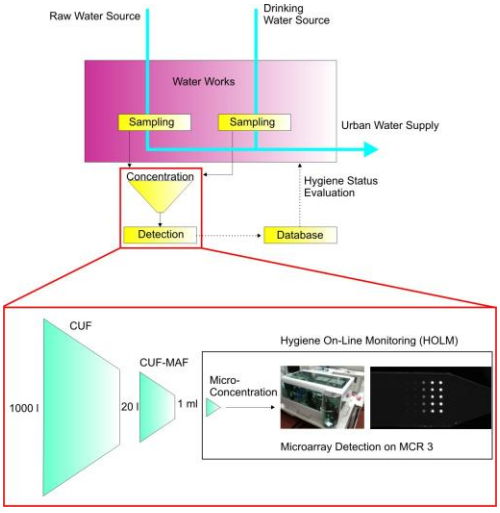
Concentration and Detection of Waterborne Pathogens for the Inline-Monitoring of Drinking- and Raw Water

Funding: BMBF (Federal Ministry for Education and Research; 033W010, EDIT)
Cooperation: Helmholtz-Zentrums für Umweltforschung GmbH; Institut für Mikrosystemtechnik, Universität Freiburg (IMTEK); DVGW-Technologiezentrum Wasser Karlsruhe (TZW); R-Biopharm AG (Darmstadt); GWK Präzisionstechnik GmbH (München); Fraunhofer Anwendungszentrum Systemtechnik (Ilmenau); Berliner Wasserbetriebe (BWB)

Waterborne pathogens are a threat to human health. Unfortunately, currently available methods for water quality monitoring are not well suited to detect and quantify viruses and bacteria. These pathogens occur in very low concentrations which makes it necessary to concentrate large amounts of water prior to detection: Intelligent processes need to be established that take representative samples from water pipes and concentrate, analyze and evaluate them. In the EDIT project this is the Hygiene-On-Line-Monitoring (HOLM) system. Fast multiplexed concentration- and analysis methods will be assembled, to deliver the hygiene status within an hour, thus enabling automated appropriate regulation mechanisms.

Large volume samples (> 1 m³/h) will be taken from raw- and drinking water pipes of water works and concentrated by continuous ultrafiltration. The concentrated sample will be transferred to a fully automated multiplex rapid test system consisting of a two-step concentration device combining cross-flow ultrafiltration (CUF) with an adsorption-elution method (monolithic affinity filtration MAF), a microsystem based micro-concentration- and nucleic acid extraction module, developed by IMTEK, and the multiplex microarray analysis platform (MCR 3, device provided by GWK). On the MCR 3, extracted nucleic acids will directly be amplified and subsequently detected by a chemiluminescence reaction. The systems for concentration and detection are being developed at our institute. Technical design for industrial application will be done by GWK.

To quantify indicator organisms as well as pathogens and for live/dead discrimination (development by TZW), on-chip based amplification methods for the multiplex microarray analysis on the MCR 3 will be investigated (IWC-TUM, R-biopharm). Data will be interpreted on the MCR 3 and provided to a database to achieve an evaluation of the hygiene status as fast as possible. The finished system will be tested on a test track (at Berliner Wasserbetriebe) under real conditions.



Schematic of the HOLM system in the water supply

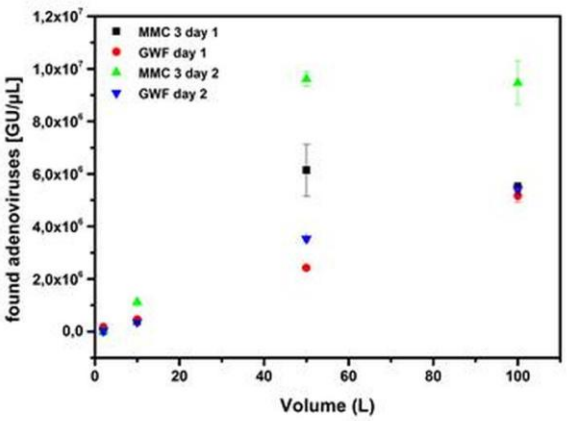
The fast concentration of water by a factor of 10^8 in a closed system and a multiplex analysis based on isothermal nucleic acid amplification will enable the automated on-site surveillance of water hygiene which can be implemented into existing monitoring systems.

D. Elsäßer, A. Kunze

Multiplexed Analysis of Pathogens and Indicator Organisms in Water – Combining Rapid Concentration Methods with Microarray Technology

Funding: BMBF (Federal Ministry for Education and Research; 02WU1142, PATH$_2$OGENSCAN)
Cooperation: Technologiezentrum Wasser, Landesgesundheitsamt Baden-Württemberg, GWK Präzisionstechnik GmbH

Water is the most important resource to sustain life. To ensure the high quality of drinking water, it is important to have a rapid, efficient and very sensitive method to quantify waterborne viruses at low concentrations in large volumes. With the MMC 3 (Munich Microorganism Concentrator), it is possible to concentrate microorganisms and viruses, using a combination of crossflow-ultrafiltration (CUF) and an adsorption–elution method based on monolithic affinity filtration (MAF) in water samples.



Recovery Comparison of MMC 3 and glass wool filtration with spiked adenoviruses in 10 L drinking water (1.9 · 10^7 GU/µL). Best results with MMC 3 for 50 L, time for concentration 1.5 h.

The MS2, PhiX174 and hAdV2 could be considered as indicator viruses for a viral contamination of drinking water. They were used by our cooperation partner, Landesgesundheitsamt Baden-Württemberg, to compare the MMC 3 system with the conventional glass wool filtration. On the example of the hAdV2 (1.9 · 10^7 GU/µL), it was shown that the MMC 3 system could concentrate more adenoviruses in 50 L than the glass wool filtration in 100 L drinking-water. Furthermore, the MMC 3 needs only 1.5 h with a flowrate of ~0.7 L/min in contrast with the glass wool filtration (flowrate ~1 L/min), which required 6 h due to the subsequent flocculation. As a conclusion, the MMC 3 is more efficient and faster for the concentration of viruses than the conventional glass wool filtration.

The detection of viruses in real samples was tested with surface water samples provided by Technologiezentrum Wasser. Samples were concentrated by glass wool filtration or flocculation. Analysis was performed via quantitative real-time PCR and DNA-microarray measurement on the Munich chip reader MCR3. Generally, found concentrations of hAdV2, bacteriophage MS2 and PhiX174 were at the detection limit. A concentration factor of at least 10^4 – 10^5 is needed to enable detection of viruses with qPCR or microarray. In samples with high concentrations, quantitative analysis was possible. In conclusion large volume concentration methods have to be combined with virus detection methods for water analysis to enable quantification of waterborne viruses.

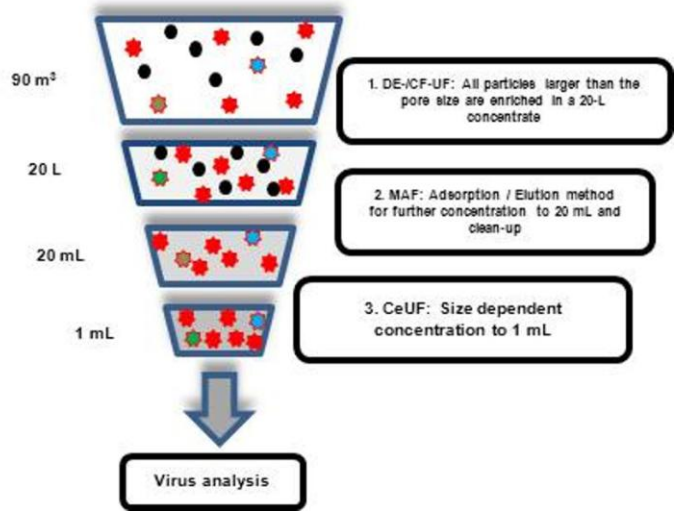
A. Stanojlovic-Collin, D. Elsäßer

Detecting Viruses in Drinking Water Samples up to 90 m³

Funding: DFG (Deutsche Forschungsgemeinschaft)
Cooperation: Centre of Infectiology and Infection Prevention, University of Bonn; Federal Environment Agency, Berlin; Institute of Groundwater Ecology, Helmholtz Zentrum München; Institute for Sanitary Engineering and Waste Management of the Leibniz University, Hannover

The risk of a viral infection when consuming contaminated drinking water is 10 to 10,000 folds greater than for pathogenic bacteria at similar level of exposure. Therefore, verification of microbial water quality needs quantitative detection methods, which are able to analyze enteric viruses in large-volume drinking water samples. By risk assessment, the WHO has increased the volume of drinking water, which should not contain more than one rotavirus from 32 m³ (WHO, 2004) to 90 m³ (WHO, 2011). The monitoring of viruses in such large volumes is very elaborate, as the detection limits of microbiological assays are not sufficient.

We designed a three-step concentration process which is able to concentrate viruses in water volumes of up to 10^5 liters to a final volume of 1 mL, ready for microbiological analysis. The process, combining cross-flow ultrafiltration (CUF), monolithic affinity filtration (MAF) and centrifugal ultrafiltration (CeUF) is mobile, robust and easy to perform. The ultrafiltration system can be driven independently from an external power supply, thus being applicable for experiments in the field. At flow rates of 1.5 m³/h, 21.4 ± 0.02 % of spiked bacteriophage MS2 could be recovered in a 90 m³ sample volume. The affinity filtration, based on a macroporous monolith could recover 102 ± 23 % of bacteriophage MS2 over 6 orders of magnitude. Other viruses like phiX174, human adenovirus (hAdV) and murine norovirus (MNV) were recovered with 40 ± 17 %, 67 ± 57 % and 12 ± 6 %, respectively.



Three-step enrichment procedure for concentrating viruses in 90 m³ of drinking water to a final volume of 1 mL.

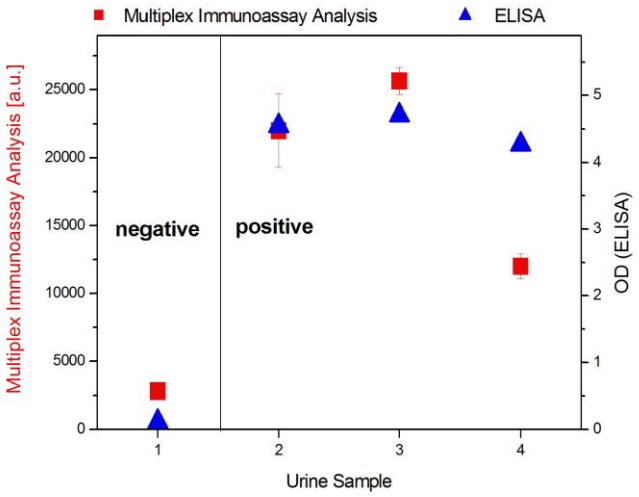
Concentrating bacteriophage MS2 in tap water samples with a volume of 1 to 90 m³, independently from the sample volume our three-step concentration process reached recoveries of 1 to 5 %, displaying an enrichment factor of 10^5.

A. Kunze

Multiplex-Immunoassay-Microarray for *Legionella pneumophila* Serogroups 1 – 15 in Water, Bioaerosols, and Human Urine

Funding: DFG (Deutsche Forschungsgemeinschaft)
Cooperation: Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit; Prof. Lück, TU Dresden

In August 2013 there was an outbreak of legionnaires' disease in Warstein, Germany. The contamination of river water with *Legionella* was caused by a clarification plant. That contaminated water has been found in the pretreatment tank of the Warsteiner brewery and also has been used by a factory for being nebulized in their recooling system. 165 People got infected with *Legionella* by inhaling contaminated aerosols, 3 of them died. The bioaerosols were contaminated with human pathogenic bacteria like *Legionella* (e.g. *L. pneumophila*) next to the clarification plant and brewery. Regarding the high risk of infection, the detection of *Legionella spp*. has a great impact on the environmental hygiene and medicine.



Comparison for urine of infected patients tested with MIA/CR3 and ELISA

To investigate the correlation between the bioaerosol distribution, the origin of the contamination and infected patients, the exposure of the staff from breweries and related industries, environmental samples (water, air) and patient urine should be analysed in parallel.

The analysis of samples of patients suspected to have legionnaires' disease is routinely done by urine antibody analysis. However, actual standard analysis methods only allow for detection and identification of *L. pneumophila* serogroup 1, causing more than 80% of all counted legionnaires' diseases, and serogroups 2 - 15 only as sum parameter. An automated microarray immunoassay (MIA) is a promising serotyping method to rapidly detect all possible serogroups in parallel. In a first proof of principle study, a sandwich MIA for *Legionella pneumophila* serogroup 1 was established on the MCR 3 by using commercially available polyclonal antibodies. Urine of patients infected with legionellosis was tested using ELISA and sandwich chemiluminescence MIA. Similar results were obtained. The next step is the establishment of the MIA for *Legionella pneumophila* serogroups 1 - 15. Afterwards, environmental samples (bioaerosol and water) and human urine will be measured on the MCR 3.

A. Wunderlich

Fast and High-Parallel Detection of Zoonoses Antibodies by Means of Chemiluminescence Microarray Immunoassays

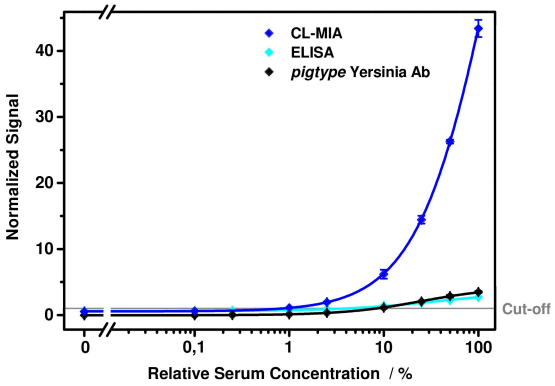
Funding: Bavarian Research Foundation. SK 37-10
Cooperation: Mikrogen GmbH (Munich); Chair for Food Hygiene, LMU Munich

Zoonoses are infectious diseases which are transmittable from animals to humans. Zoonotic agents are e.g. bacteria, viruses, or parasites. In case of porcine meat, the pathogens Hepatitis E virus, *Yersinia*, *Campylobacter*, *Salmonella*, *Trichinella*, and *Toxoplasma* are of great interest for food safety and human health. Currently, the screening for only *Salmonella* and *Trichinella* is regulated by law. Fast and multiplexed screening methods are required to monitor meat products and improve the hygiene status in animal husbandry.

In this project, a method for fast and parallel detection of IgG antibodies against these zoonotic pathogens in serum samples of slaughtered pigs was developed. For this purpose, a chemiluminescence microarray immunoassay (CL-MIA) was performed at the microarray chip reader platform MCR 3 which combines the flow-through principle with the microarray technology.

Recombinant or extracted antigens (rAg) of the various pathogens are immobilized on functionalized glass slides. The microarray chip is then incubated with serum samples. In case of present zoonoses antibodies, these antibodies bind to the chip surface and are detected by horseradish peroxidase (HRP) labelled secondary antibodies. The chemiluminescence readout is accomplished via CCD camera and incubation times are considerably reduced compared with ELISA test formats performed in titer plates or line blots. Moreover, multiple analytes can be detected simultaneously.

Signal reproducibility and specificity were determined based on ORF2C of HEV genotype 1 and 3 and Yersinia outer protein D (YopD) to characterize this new method. The analytical performance was compared with in-house ELISA as well as the nitrocellulose-based line assay recomLine HEV (Mikrogen, Neuried, adapted for swine)



Dose-response measurements with CL-MIA compared to in-house ELISA and ELISA test kit pigtype *Yersinia* Ab (Qiagen Leipzig)

and the ELISA test kit pigtype *Yersinia* Ab (Qiagen Leipzig), respectively. The immunochip achieved the highest analytical sensitivity and detection capability in dilution experiments.

Within an assay time of 7 minutes, serum samples can be analyzed directly without interfering matrix effects or cross reactivity. Integrating antigens of Trichinella, Toxoplasma, and Campylobacter on the immunochip led to a multiplexed detection method that is able to support the meat-producing industries concerning food safety and public health aspects.

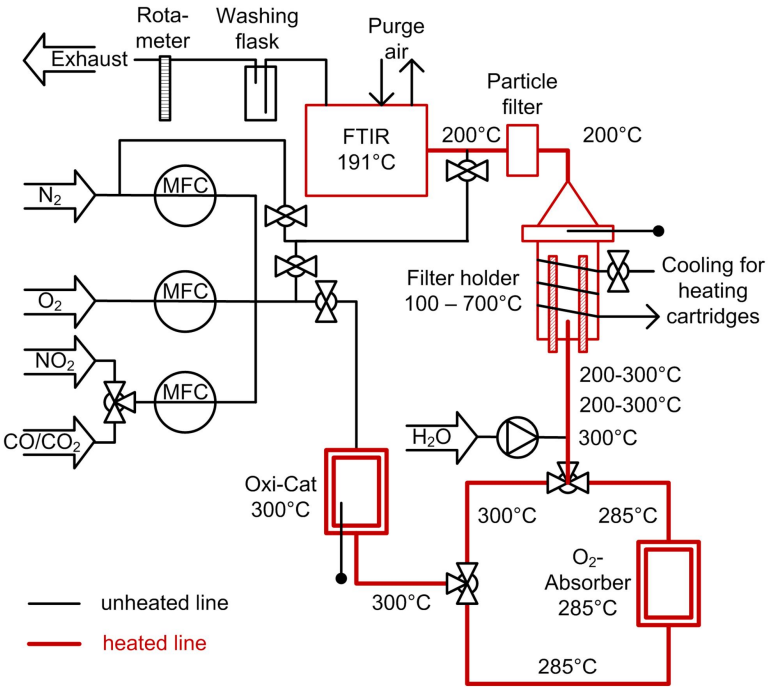
V. Meyer

Aerosol Research (Prof. Dr. R. Niessner)

Oxidation Reactivity of Biofuel Generated Soot

Funding: FVV (Association for Combustion Engine Research), FNR (Fachagentur Nachwachsende Rohstoffe e. V.)
Cooperation: Universität Bayreuth (LTTT/BERC), Universität Stuttgart (ICVT), Bergische Universität Wuppertal (Abt. Maschinenbau)

Biofuel offers the potential to completely or partially substitute fossil fuels such as diesel, gasoline or natural gas, if used as pure fuel or additive to fossil fuel, respectively.



Remodelled TPO setup.

Nowadays, there has been a worldwide need for an increased usage of fuel generated from renewable sources. In Europe, the use of biofuels is regulated by the EU guideline 2009/28/EG (“Erneuerbare-Energien-Richtlinie”; engl.: renewable energies guideline). It obligates the increment of the blending quota of biofuel to fossil fuel to up to 10 % until 2020.

There is a general lack of knowledge about the physico-chemical properties of diesel engine emissions generated from biofuels in contrary to fossil fuels. So far, there has been little fundamental research on structure and reactivity of biodiesel soot, which is subject of this cooperation project.

Task of our institute is to establish the method of Multiwavelength Raman Microspectroscopy (MWRM) for the analysis of biodiesel generated soot. For this purpose, a correlation of soot structure characterized by Raman Microspectroscopy at different laser excitation wavelengths and soot oxidation reactivity analyzed by Temperature-Programmed Oxidation (TPO) needs to be generated. MWRM promises to be a rapid method for the prediction of soot structure and oxidation reactivity.

In the framework of this project, the previous TPO setup was remodeled. It is now possible to not only to conduct TPO experiments under oxygenic atmosphere, but also in the presence of NOx, and water. Thus, the simulation of soot oxidation by TPO is even closer to real gas conditions in the engine exhaust. Furthermore, it enables us to characterize the effect of distinct exhaust gases on soot oxidation.

H. Bladt, N. P. Ivleva

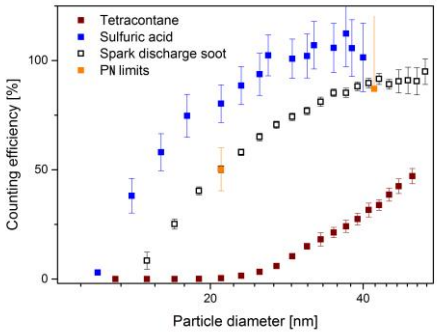
Validation of Particle Number Concentration Measurements

Funding: FVV (Association for Combustion Engine Research)
Cooperation: Institute for Internal Combustion Engines, TUM

Fine, and especially ultrafine, particles are considered to be harmful for human health. As diesel-powered vehicles are a significant source for particles in this size range, it is important to limit and consequently reduce the emissions caused by their exhaust. In 2011 a new maximum value, which is based on the number concentration of non-volatile aerosol particles in the exhaust, was established within the European Union. Additionally a new measurement procedure, that includes a pretreatment of the raw exhaust before determination of the particle number, was introduced. The pretreatment involves a separation of volatile and solid exhaust components with a so-called volatile particle remover (VPR), and a quantification of the residual non-volatile particle number concentration with a condensation nucleus counter (CNC).

One crucial point within this measurement procedure is the use of a CNC as detection instrument as its counting efficiency strongly depends on the particle surface composition. This may lead to errors in the quantification of the particles. To evaluate this source of defect a variety of model aerosols, mimicking real exhaust, was generated and measured with a CNC. It could be shown that the counting efficiencies for solid particles are almost similar and within the limits given by the EU. In contrast to that, volatile species were detected by the CNC with an up to 50 % differing efficiency compared to the solid ones.

To minimize the influence of the particle surface composition on the measurement, it is indispensable to completely or at least reproducibly separate volatile particulates from solid exhaust gas components. Up to now, the EU regulation only contains a type approval test that involves a determination of the VPR volatile particle removal efficiency with tetracontane aerosol. To evaluate, whether this test assures a proper elimination of the solid particles, different VPR types were constructed and tested at the IWC. It turned out that the different devices could easily remove this substance under most testing conditions. Further experiments will be performed in the future with a broader range of volatile exhaust components for better understanding of the removal efficiencies of different VPR types.



CNC counting efficiency for volatile and solid monodisperse particles.

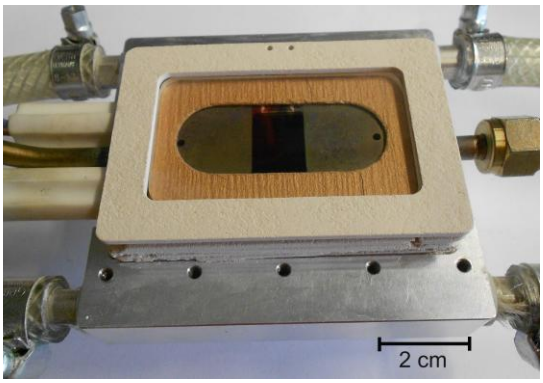
Furthermore, CNC devices from three manufacturers were compared among each other with different monodisperse test aerosols. These experiments were performed to reveal whether varying aerosols as well as calibration procedures used by the manufacturers have an influence on the counting efficiency. It could be shown that devices from different manufacturers have a differing counting efficiency for the same test aerosol of up to 10 %. A detailed specification of calibration aerosol and procedure in EU regulation may help to minimize these variations.

B. Kiwull, J.-C. Wolf

Temperature-Programmed Oxidation, Raman Microspectroscopy, and Particle-Conductivity for Diesel Soot Characterization

Funding: Audi AG, Ingolstadt
Cooperation: Audi AG, I/EA-821

Soot particles emitted by diesel engines influence the human health by penetrating into the lung and may cause diseases like lung cancer. In addition, direct and indirect effects on the environment and the climate, e.g. as cloud condensation nuclei or interference with the earth´s radiation balance, exist. To protect the environment and the people, the emission of diesel particles is regulated by law. Therefore, cars and trucks are equipped with diesel particle filters to trap soot particles and minimize their emission.



Measurement cell for the combination of TPO, Raman microspectroscopy and conductance measurements

When the pressure drop of the filter becomes too high, the filter is regenerated by oxidation of the deposited particles inside its structure. It is known that the reactivity of the soot, i.e. the temperature at which the soot can be oxidized, depends on its microstructure. Hence, effective tools for the determination of the soot reactivity and its microstructure are necessary to allow a better soot characterization and consequent engine optimization.

Currently, the most effective tools for soot characterization are temperature-programmed oxidation (TPO), where soot is burned in a defined environment and the oxidation products are measured to determine its reactivity, Raman microspectroscopy, where the relative intensity and integral of the carbon Raman integral is connected to the microstructure of the soot, and electric conductivity/ conductance which is also highly dependent on the microstructure. The aim is to combine these three individual tools in one setup in order to provide comprehensive results and to achieve a better understanding of the results of the different methods. Ideally, the time and cost intensive TPO or Raman microspectroscopy could be replaced by a more convenient conductance sensor provided this way the same information is obtained.

Therefore, a measurement cell was developed combining TPO, Raman microspectroscopy and electrical conductance in one setup. A heated steal block allows tempering of the cell´s inside up to 1000°C while the frame is water-cooled, so that the cell can be placed under the objective of a Raman microscope and the soot can be observed through a quartz glass window. A conductometric sensor with interdigitated electrodes is placed inside the cell. With this cell different kinds of test aerosols like spark discharge soot and graphite powder have already been measured by TPO. Conductance experiments and Raman measurements have been carried out separately inside the cell using the spark discharge soot. The next step is to perform the conductance experiments under the Raman microscope and later to also add the TPO measurements to the combined setup.

M. Eß, B. Grob

Charging of Ultra-Fine Aerosol Particles by an Ozone-Free Indirect UV Photo-Charger

Funding: IWC
Cooperation: University of Applied Sciences Northwestern Switzerland, Windisch, CH, Prof. Dr. H. Burtscher

The most common way for charging particles is diffusion charging by ions, generated by a radioactive source (bipolar) or corona discharge (unipolar). Radioactive sources, however, are unsuitable for field measurements. Corona charges, on the other hand, tend to produce ozone and to form additional particles by the plasma of the corona discharge. This can lead to measurement errors for the particle concentration and or sampling artefacts.

Therefore we are trying to establish a different method for charging particles unipolar as first described by Bucholski and Niessner in 1991. A surface is irradiated with UV light (254 nm) with a photon energy higher than the photoemission threshold of the surface, but below the threshold of the particles and that for ozone formation. Depending on the carrier gas, the emitted photoelectrons remain free (in noble gases) or attach to gas molecules (if an electronegative gas like oxygen is available). These ions then attach to the particles by diffusion. Indirect photoelectric charging) allows a very soft ion production, mainly without inducing chemical reactions or ozone formation.

Our devices are optimized to work with air as carrier gas, requiring a photoemitter with a low work function under ambient conditions and a stable surface under the influence of oxygen. We are using a non-porous, non-graphitized carbon (glassy carbon), which has a very inert surface with respect to degradation.

A possible application for the indirect photoelectric charger can be a calibration setup for PMP condensation particle counters.

So far no calibration standard is defined for the particle number concentration measurement systems for light or heavy duty vehicles (UN-ECE Regulation 83 & 49). The commercially available systems count the non-volatile particles larger then 23 nm by a condensation particle counter (CPC).

Picture of the indirect photoelectric charger

The CPCs have to be calibrated either by comparing the counter with an electrometer or another CPC (calibrated with an electrometer). Therefore a monodisperse aerosol is required, which can be produced in a repeatable manner. We could show, that a spark discharge generator equipped with graphite electrodes (GfG 1000, Palas) is the ideal aerosol source in combination with our newly developed indirect photoelectric unipolar charger and an electrostatic classifier.

As the needed supersaturation within the CPC is influenced by the hydrophilicity of the particles, an ozone-free ion generation is important. Ozone would immediately change the particle surface wettability and therefore affect the response of the CPC.

B. Grob

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H. Bladt, Oxidation Reactivity of biodiesel generated Soot, EAC, 1.-6.09.2013, Prague.

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S. Oswald, R. Niessner, D. Knopp, Hapten Microarray-Based Screening of Mycotoxins in Cereals, 35th Mycotoxin Workshop, 22.-24.5.2013, Ghent, Belgium.

M. Seidel, A. Szkola, K. Campbell, C.P. Elliott, R. Niessner, R., Phycotoxin-Mikroarray zum schnellen Screening von marinen Biotoxinen, ANAKON 2013, 4.-7.3.2013, Essen.

M. Seidel, S. Lengger, J. Otto, S. Schneider, L. Pei, M. Rieger, A. Tiehm, J. Schneider, R. Niessner, Herausforderung, Vision und aktueller Stand in der Realisierung einer modernen Analytik von Viren im Wasser, WASSER 2013, 6.-8.5.2013, Goslar.

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T. Baumann, C. Metz, N. P. Ivleva, R. Niessner, Pore Scale Visualization of Chemical Gradients at Biogeochemical Interfaces Using Micromodels and Raman Microspectroscopy, Goldschmidt 2013, 26.-30.8.2013, Florence, Italy.

D. Knopp, Immunochemical Methods for Food Analysis With Special Emphasis on Mycotoxin Determination, Analytica Anacon India, 12.-14.11.2013, Mumbay, India.

S. Oswald, Entwicklung eines Biosensorarrays zur schnellen Bestimmung von Mykotoxinen in Getreide, 6. Wissenschaftliches Symposium des Verbandes Deutscher Mühlen zum Thema ‚Getreidequalität und –sicherheit', 8.11.2013, Würzburg.

C. Haisch, Recent Developments in Laser-Based Exhaust Gas Monitoring, 8.12. 2013, Tec.-Day of the AVL List GmbH, Graz, Austria.

C. Haisch, Raman-Scattering for Gas Phase Analysis, Seminar Series of the FVV (Research Association for Combustion Engines), 17.10.2013, Duisburg.

C. Haisch, Photoacoustic and Photophoretic Spectroscopy for Aerosol Characterization, GDCh Wissenschaftsforum Chemie (Science Forum Chemisty), 1.-4.9.2013, Darmstadt.

C. Haisch, Photoacoustic Spectroscopy as Tool in Exhaust Gas Monitoring, Advanced Emission Control Concepts for Gasoline Engines 2013, 13–15 May 2013, Bonn, Germany

N. P. Ivleva, P. Kubryk, R. U. Meckenstock, J. Kölschbach, R. Niessner, Raman Microspectroscopy and SERS for Biofilm Analysis: Focus on Stable-Isotope Technique, FT-IR Spectroscopy in Microbiological and Medical Diagnostics 2013, 24.-25.10.2013, RKI, Berlin.

R. Niessner, Analytische Mikroarrays: Hochleistungsanalytik für die Flüssigphase, ANAKON 2013, 4.-7.3.2013, Essen.

R. Niessner, a) Chemical on line Measurement Modern Spectroscopy as a Tool for Aerosol Characterization, Summerschool 2013, University of Vienna, July 5, 2013, Vienna.

M. Seidel, Herausforderung, Vision und aktueller Stand in der Realisierung einer modernen Analytik von Viren im Wasser. GDCh Fachgruppe Viren und Protozoen, 18.3.2013, Frankfurt, Deutschland.

M. Seidel, Kulturunabhängige Nachweisverfahren für Bakterien und Viren, VAAM Fachgruppensitzung „Qualitätssicherung, Diagnostik", 20.9.2013, Villingen-Schwenningen.

Poster Presentations

C. Berger, R. Nießner, C. Haisch, Photoacoustic Spectroscopy for Exhaust Gas Analysis, ANAKON 2013, 4.-7.3.2013, Essen.

M. N. Eß, R. Nießner, C. Haisch, Laser-Induced Plasma Optical Particle Counter, ANAKON 2013, 4.-7.3.2013, Essen.

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N. P. Ivleva, S. Huckele, B. Weinzierl, C. Haisch, T. Baumann, R. Niessner, Raman Microspectroscopic Identification and Characterization of Individual Airborne Volcanic Ash Particles, European Aerosol Conference, EAC 2013, 1.-6.9.2013, Prague, Czech Republic.

P. Kubryk, C. Karwautz, T. Lüders, R. Niessner, N. P. Ivleva, Raman- und SERS-Analyse im wässrigen Milieu: Fokus auf die Untersuchung von stabilisotopenmarkierten Bakterien und Biofilmen, WASSER 2013, 6.-8.5.2013, Goslar.

P. Kubryk, J. Kölschbach, R. Niessner, R. U. Meckenstock, N. P. Ivleva, Stable-isotope Raman Microspectroscopic Studies on Accumulation of Pollutants by Biofilms in Aquatic Systems, FT-IR Spectroscopy in Microbiological and Medical Diagnostics 2013, 24.-25.10.2013, RKI, Berlin

P. Kubryk, C. Karwautz, T. Lüders, R. Niessner, N. P. Ivleva, Stable Isotope Raman Microspectroscopy and SERS Analysis of Microorganisms, ANAKON 2013, 4.-7.03.2013, Essen.

M. Kühn, N. P. Ivleva, R. Niessner, T. Baumann, Untersuchung von Huminstoffbeschichtungen Künstlich hergestellter Silbernanopartikel mittels oberflächenverstärkter Raman-Spektroskopie, ANAKON 2013, 4.-7.3.2013, Essen.

A. Kunze, L. Pei, M. Rieger, R. Nießner, M. Seidel, A new Route to Concentrate Viruses in Large-volume Drinking Water Samples, Medical Biodefense Conference, 22.-25.10.2013, München.

M. Lafogler, G. Somogyi, R. Niessner, T. Baumann, Fluoreszenzspektroskopie zur Prognose der Reservoireigenschaften des Malmaquifers Für die Geothermie, ANAKON 2013, 4.-7.3.2013, Essen.

V. K. Meyer, K. Wutz, P. Krol, S. Wachek, M. Gareis, C. Nölting, F. Struck, E. Soutschek, O. Böcher, R. Niessner, M. Seidel, Fast Detection of Zoonoses Antibodies in Sera of Slaughtered Pigs by Means of Chemiluminescence Microarray Immunoassays, ANAKON, 4.-7.3.2013, Essen.

V. K. Meyer, K. Wutz, P. Krol, S. Wachek, M. Gareis, C. Nölting, F. Struck, E. Soutschek, O. Böcher, R. Niessner, M. Seidel, Fast Detection of Zoonoses Antibodies in Sera of Slaughtered Pigs by Means of Chemiluminescence Microarray Immunoassays, Medical Biodefense Conference, 22.-25.10.2013, München.

V. K. Meyer, K. Wutz, P. Krol, S. Wachek, M. Gareis, C. Nölting, F. Struck, E. Soutschek, O. Böcher, R. Niessner, M. Seidel, Fast Detection of Zoonoses Antibodies in Sera of Slaughtered Pigs by Means of Chemiluminescence Microarray Immunoassays, 11. Dresdner Sensor-Symposium, 9.-11.12.2013, Dresden.

C. Metz, N. P. Ivleva, R. Niessner, T. Baumann, Quantification of Chemical Gradients at Biogeochemical Interfaces, ANAKON 2013, 4.-7.3.2013, Essen.

S. Oswald, R. Dietrich, E. Märtlbauer, R. Niessner,, D. Knopp, Multiplex Microarray – Immunological Screening of Mycotoxins in Cereals, Medical Biodefense Conference, 22.10.-25.10.2013, München.

S. Oswald, R. Niessner, D. Knopp: Immunological Screening of Mycotoxins in Cereals, ANAKON 2013, 4.-7.3.2013, Essen.

M. Pschenitza, C. Stergiopoulos, M. Ochsenkühn-Petropoulou, R. Niessner, D. Knopp, Immunological Determination of Benzo[a]pyrene in Edible Oils, ANAKON 2013, 4.-7.3.2013, Essen, Germany.

K. Schwarzmeier, R. Nießner, C. Haisch, Time-Resolved Detection of Biogas Contaminants Using Laser-Induced Breakdown Spectroscopy and Raman Scattering, ANAKON 2013, 4.-7.3.2013, Essen.

K. Schwarzmeier, R. Nießner, C. Haisch, Continuous Monitoring of Biogas Contamination Using Laser Induced Breakdown Spectroscopy and Raman Spectroscopy, Dresdner Sensorsymposium 2013, 9.-11.12.2013, Dresden.

M. Seidel, S. Lengger, J. Otto, S. Schneider, A. Tiehm, J. Schneider, R. Niessner, Multiplex Quantification of Pathogens and Indicator Organisms in Water Using the MCR 3 Analysis for DNA Microarray Read-out. Annual Conference of the Association of General and Applied Microbiology (VAAM), 10.-13.3.2013, Bremen.

C. Stergiopoulos, M. Ochsenkühn-Petropoulou, M. Pschenitza, D. Knopp, R. Niessner, Immunological Determination of Polycyclic Aromatic Hydrocarbons in Edible Oils, IMA 2013, 15.-19.9.2013, Thessaloniki, Greece.

A. Szkola, E. Linares, B. Dorner, R. Dietrich, E. Märtlbauer, R. Niessner, M. Seidel, Simultaneous Detection of Different Molecular Weight Biotoxins Using a Flow-through Chemiluminescence Portable Microarray Analysis Platform, Medical Biodefense Conference, 22.-25.10.2013, München.

S. Wacheck, K. Wutz, V. K. Meyer, P. Krol, M. Gareis, C. Nölting, F. Struck, E. Soutschek, O. Böcher, R. Niessner, M. Seidel, New Route for Fast Detection of Antibodies Against Zoonotic Pathogens in Sera of Slaughtered Pigs by Means of Flow-through Chemiluminescence immunochips, 54. AT Lebensmittelhygiene, 24.-27.9.2013, Garmisch-Partenkirchen.

A. Wunderlich, C. Lück, R. Nießner, M. Seidel, Detection of *Legionella pneumophila* Serogroups 1 - 15 in Patient Urine by Means of the Automated Multiplex Analysis Platform MCR 3, 11. Dresdner Sensor-Symposium, 9.-11.12.2013, Dresden.

Scientific Committees

T. Baumann, Fate and Transport of Biocolloids and Nanoparticles in Soil and Groundwater Systems. EGU General Assembly, 7.-12.4.2013, Vienna (Co-convener)

Patents

C. Haisch and R. Niessner, Instrument and its application for the counting of particles suspended in gases. DE 202013008240 U1 (2013).

Hydrochemical consulting

Mineralisation control analyses: Bad Abbach, Bad Aibling, Bad Birnbach, Bad Füssing, Bad Griesbach, Bad Gögging, Bad Reichenhall, Bad Rodach, Bad Wimpfen, Bad Wörishofen, Bayreuth, Hölle, Kondrau, Treuchtlingen, Lipik (Croatia), Memmingen, Neumarkt i. d. Opf., Sibyllenbad, Straubing, Utting, Weißenstadt

Hydrogeological and hydrochemical expertises (mineral water, spa water): Sibyllenbad, Siegsdorf, Waldkraiburg

Deep Hydrogeothermal Energy Exploration: Bad Wörishofen, Pullach, Sauerlach, Waldkraiburg

Hydrogeochemical Modelling: Bad Wörishofen, Gas Storage, Pullach, Sauerlach

Magnetic water treatment: Landgericht Mosbach.

Theses

PhD Theses

Dipl.-Geol. Christina Mayrhofer: Hydrochemische Untersuchungen im Malmaquifer im bayerischen Molassebecken

MSc Chem. Susanna Oswald: Entwicklung einer immunologischen Multianalytmethode zur Detektion von Mykotoxinen in Getreide

MSc Chem. Jan-Christoph Wolf: Nitro-PAH-Bildung in Dieselpartikelfiltern & Partikelanzahl-Messverfahren für Dieselabgas

M.Sc. Theses

BSc Chem. Tobias Bauch: Versuche zur analytischen Nutzung der laserinduzierten Photofragmentierung organischer Stoffe

BSc Chem. Michaela Nicole Eß: Optical Aerosol Particle Counter Based on Laser-Induced Breakdown

BSc Chem. Fabian Knoller: Combination of Temperature-Programmed Oxidation, Raman-Spectroscopy and Conductivity Measurements for the Characterisation of Soot

BSc Chem. Patrick Kubryk: Optical Aerosol Particle Counter Based on Laser-Induced Breakdown

BSc ind. Chem. Antonio Meraldo: Synthesis of Formaldehyde-Free Microcapsules
BSc Chem. Verena Meyer: Zoonose-Monitoring bei Schlachtschweinen mittels analytischer Mikroarrays
BSc Chem. Anna-Cathrine Neumann: Microbial Fuel Cell: Development and Application as Biochemical Oxygen Demand Sensor
BSc Chem. Clement Ong: Surface Modification of Polyethersulfone Membranes
BSc Ramon, Edgar, Azpiri: Qualitiy Control of Monolithic Affinity Filtration Columns Using Monodisperse Suspensions of Polystyrene Microparticles
BSc Thaler, Klemens: Plasma Emission Spectrometry for the Detection of Siloxanes in Biogas
BSc Chem. Alexandra Wiesheu: Studies on Photophoretic Separation of Bacteria
Exam. Lebensm. Chem. Annika Wunderlich: Multiplex-Immunoassay für Legionella pneumophila aus Bioaerosolproben

B.Sc. Theses

Matthias Edelmann: Analysis of Soot Oxidation under the Impact of NO2 und H2O with Temperatur-programmed Oxidation
Catharina Kober: Regenerationsuntersuchungen von rekombinanten Antigen – Mikroarrays für das Zoonose – Monitoring von Schlachtschweinen
Katharina Hess: Sorption binärer Farbstoffmischungen an Gesteinen des Oberen Jura

Institute Colloquia

PD Dr. Rolf Altenburger, Helmholtz Zentrum für Umweltforschung Leipzig, Department Bioanalytische Ökotoxikologie: Status and Challenges in Aquatic Toxicology (15.1.2013)
Prof. Dr. Kristin Schirmer, Swiss Federal Institute of Aquatic Science and Techology, Department – Environmental Toxocology: Aquatic Toxocology – Perspective on Current status and Future Needs (30.1.2013)
Prof. Dr. Heinz Burtscher, University of Applied Sciences, Windisch, Switzerland: Charge-based Measurement of Aerosols (19.3.2013)
Prof. Dr. Stefan Zimmermann, Leibniz University Hannover, Institute of Electrical Engineering and Measurement Technology: Ion Mobility Spectrometry for Rapid Trace Gas Detection (26.3.2013)
Prof. Dr. Andreas Schütze, University Saarbrücken, Department of Mechatronics, Lab for Measurement Technology: Dynamic Operation of Chemical sensors for Improved selectivity and Novel Applications (9. 4.2013)
PD Dr. Carsten Engelhard, University of Münster, Institut für Anorganische und Analytische Chemie: Nanoparticle Characterization by ICP – Mass Spectrometry (14.5.2013)

Prof. Dr. Hans Bettermann, Heinrich Heine University Düsseldorf, AG Flüssigphasen-Laserspektroskopie: Laser Spectroscopie Methods to Characterize Molecules and Processes (15.5.2013)

Prof. Dr. Majid Hassanizadeh, Utrecht University, Faculty of Geosciences: Transport of Viruses in Partially Saturated Soil and Groundwater (17.5.2013)

Dr. Maik Jochmann, University Duisburg-Essen, Lehrstuhl Instrumentell Analytische Chemie: New Ways in Isotope Ratio Mass Spectrometry (28.5.2013)

Prof. Dr. Michael Tiemann, Universität Paderborn, Naturwissenschaftliche Fakultät, Department Chemie: Nanoporous Metal Oxides: Sythesis and Application as Gas Sensors (27.6.2013)

Prof. Dr. Wolfgang Frieß, Ludwig-Maximilians-Universität München, Zentrum für Pharmaforschung: The Particle Problem and Particle Analysis in Protein Drugs (1.7.2013)

Dr. Bügler und Dr. Uhl, Bayerisches Landeskriminalamt München, Kriminaltechnisches Institut: Chemical - Analytical Challenges in Forensic Sciences - Overview and Trends (9.7.2013)

Prof. Dr. Reinhard Renneberg, Hong Kong University of Science and Technology: Hunting for the Fastest Heart Attack Biotest of the World (17.7.2013)

Dr. Alexander Zybin, ISAS, Leibniz Institut für Analytische Wissenschaften (16.9.2013)

Dr. Karin Schütze, CellTool GmbH, Bernried: New Instrumental Solutions for Individual Cell-based Raman Spectroscopy (23.9.2013)

Prof. Dr. Claudimir do Lago, University of Sao Paulo, Department of Chemistry: Micro – and Macro – Fabrication Techniques for capillary Electrophoresis and Related Techniques (28.10.2013)

Prof. Dr. Helmut Cölfen, University Konstanz, Physikalische Chemie: Analytical Ultracentrifugation of Complex Colloids (5.11.2013)

Prof. Dr. Antje Bäumner, University of Regensburg, Bio- and Chemosensors: Nanofiber-integrated Microfluidic Biosensors for Pathogen Detection (6.12.2013)

Dr. Lars Ullerich, GNA Biosolutions GmbH, Martinsried: Ultrafast DNA Detection by Laser -heated Nanoparticles (12.12.2013)

Teaching

Chemistry (B.Sc./M.Sc.)

Hydrogeological, Hydrochemical and Environmental Analysis Seminar (Hydrogeologisches, Hydrochemisches und Umweltanalytisches Seminar); Niessner, Baumann, Haisch, Knopp
Graduate Course in Analytical Chemistry: Lecture in Organic Trace Analysis-Physical and Chemical Separation Methods (Nebenfach Analytische Chemie:Vorlesung Organische Spurenanalytik-Physikalisch-chemische Trennmethoden); Niessner
Graduate Course in Analytical Chemistry: Lecture in Organic Trace Analysis-Applications of Selective Receptors (Nebenfach Analytische Chemie: Vorlesung Organische Spurenanalytik-Nutzung selektiver Rezeptoren); Niessner, Seidel
Graduate Course in Analytical Chemistry: Lab in Organic Trace Analysis (Nebenfach Analytische Chemie: Kurspraktikum Organische Spurenanalytik); Niessner, Seidel
Graduate Course in Analytical Chemistry: Research Lab in Organic Trace Analysis (Nebenfach Analytische Chemie: Forschungspraktikum Organische Spurenanalytik); Niessner, Seidel
Trace Analysis Techniques (Spurenanalytische Techniken); Niessner, Knopp, Haisch

Industrial Chemistry (M.Sc.) GIST TUM-Asia

Lecture in Bioengineering & Bioprocessing; Seidel
Water Chemistry; Niessner

Geosciences (B.Sc./M.Sc.)

Analytical Chemistry I: Instrumental Analysis for Geoscientists (Analytische Chemie I: Instrumentelle Analytik für Geowissenschaftler); Niessner
Analytical Chemistry II - Organic Trace Analysis for Geoscientists (Chemische Analytik II - Organische Spurenanalytik für Geowissenschaftler); Niessner
Applied Hydrogeology (Angewandte Hydrogeologie); Baumann
Contaminant Hydrogeology (Transport von Schadstoffen im Grundwasser); Baumann
Remediation Design (Erkundung und Sanierung von Grundwasser-schadensfällen); Baumann
Technical Hydrogeology (Technische Hydrogeologie); Baumann
Fluidflow in Porous Media Lab (Hydrogeologisches Laborpraktikum); Baumann, Haisch, Niessner
Hydrogeochemical Modelling (Hydrogeologische Modellierung II); Baumann
Hydrogeological Field Lab (Hydrogeologische Feldmethoden); Baumann
Hydrogeological Mapping (Hydrogeologische Kartierung); Baumann
Hydrogeological and Hydrochemical Field Trips (Hydrogeologische und Hydrochemische Exkursion); Baumann, Niessner
Water Chemistry I (Wasserchemie I); Niessner
Water Chemistry II - Hydrocolloids, Micellar Systems and Photochemical Transformations (Wasserchemie II - Hydrokolloide, micellare Systeme und photochemische Umsetzung); Niessner
Hydrochemical Lab (Hydrochemisches Praktikum); Knopp, Baumann

Biosciences (B.Sc./M.Sc.)

Biochemical Analysis (Biochemische Analytik); Görg, Gierl, Knopp, Nitz, Parlar, Schwab, Seidel

Analytical Chemistry - Separation Techniques, Chemical and Biochemical Sensors (Analytische Chemie - Trenntechniken, chemische und biochemische Sensoren); Knopp

Biochemical and Molecular Biological Methods in Environmental Analysis I - Immunological methods; Sensor techniques (Biochemische und molekularbiologische Verfahren ind er Umweltanalytik I - Immunologische Methoden, Sensor Techniken); Knopp

Biochemical and Molecular Biological Methods in Environmental Analysis II - Enzymatic methods; DNA Probes (Biochemische und molekularbiologische Verfahren ind er Umweltanalytik II - Enzymatische Methoden, DNA-Sonden); Knopp

External Tasks and Memberships

Prof. Dr. Reinhard Niessner

Bayer. Fachausschuss für Kurorte, Erholungsorte & Heilbrunnen	Member
Heinrich-Emanuel-Merck Award	Jury Head
Hong Kong University Grants Committee, Theme-based Research Scheme, Selection Committee	Member
Analytical Chemistry	Associated Editor
Analytical & Bioanalytical Chemistry	Advisory Board Member
Analytical Sciences	Advisory Board Member
Annual Review of Analytical Chemistry	Editorial Committee Member
Fresenius' Environmental Bulletin	Advisory Board Member
International Journal of Environmental Analytical Chemistry	Advisory Board Member
Microchimica Acta	Advisory Board Member
Talanta	Advisory Board Member
Toxicological & Environmental Chemistry	Advisory Board Member

Equipment

Hydrogeology
Two pilot scale tanks with flow lengths of up to 10 m allow transport experiments in a controlled environment while preserving almost natural conditions. Apart from studies on the transport behaviour of contaminants and colloids, these facilities are used for testing sensor prototypes and serve as a test bed for numerical models

Großhadern Unsaturated Zone field laboratory (10 m deep)

Dioxin Laboratory
3 High security labs with locks, separate activated carbon filter and high-performance particle filter systems

Aerosol Research
1 Aerosol chamber (1 m^3)
1 Aerosol flow tube (10 L)
1 Ozone analyzer (UV absorption)
1 NO/NO_2 analyser (Chemiluminescence)
2 Aerodynamic particle sizers (0.5-25 µm)
1 Berner impactor (9 stages, 50 nm - 16 µm)
1 Electrical low-pressure impactor (12 stages, 30 nm - 10 µm)
2 Low-Volume filter samplers (PM 10, PM2.5)
1 High-Volume filter sampler (PM 2.5)
2 Differential mobility particle sizer systems (10-1000 nm)
2 Diffusion batteries (5-300 nm)
5 Condensation nucleus counters
3 Electrostatic classifiers (10-1000 nm)
2 Spark-discharge soot aerosol generators (polydisperse ultrafine carbon aerosol)
1 Berglund-Liu aerosol generator (monodisperse aerosols, 0.8-50 µm)
1 Floating bed aerosol generator (powder dispersion)
1 Rotating brush aerosol generator (powder dispersion)
1 Tube furnace
1 Cyclone Impinger (Coriolis µ, Berlin)
1 Micro soot sensor with dilution unit

Bioseparation
1 Crossflow-Ultrafiltrationunit (6 m^2-hollow fibre module, Inge-AG)
1 Munich Microorganism Concentrator (MMC 3)
1 Monolithic Affinty Filtration Unit

Molecular Biology
1 Biacore X100, General Electric
1 Real-time PCR (Light Cycler 480, Roche)

Microarray Technology
2 Chemiluminescence Microarray Reader (Immunomat, IWC)
4 Chemiluminescence Microarray Reader (MCR 3, GWK GmbH)
1 Ink-Jet Microdispenser (SciFlexarrayer 31, scienion)
2 Contact Microarrayer (BioOdyssee Caligrapher, BioRad)

Microbiology
1 Flow Cytometer (Cell Lab Quanta SC, Beckman Coulter)
1 Water Microbiology (Colilert-18 and Quanti-Tray 2000, IDEXX)
3 Clean bench
1 Microbiological Incubator (BD 53, Binder)
1 Autoclave (Century 2100, Prestige Medical)
1 Autoclave (SHP Steriltechnik)

Standard Lab Equipment
1 Lyophilizer (Alpha 1-4 LSC, Christ)
1 Washer Disinfector (DS 500 Lab, International Steel CO.SPA)
1 Ultrapure Water System (Direct-Q 3 UV, Millipore)
1 Refrigerated Centrifuge (Universal 320R, Hettich)
1 Climatic chamber (Memmert HCP 108)
2 Fluorescence reader systems, time-resolving
3 Photometric reader systems
1 384-channel washer, Biotek
1 Turbidometer (WTW GmbH)
1 Nanophotometer (Implen GmbH)

Chromatography and Particle Separation

3 GCs with FID, NPD, ECD, TEA, and AED
1 Orbitrap-based benchtop MS, Exactive/HCD-System, Thermo Fischer
1 GC/MS, VG Autospec
1 GC/MS, Shimadzu
1 Portable Micro-GC, MITEC
1 Asymmetrical Field-flow-fractionation system, Postnova
2 Concentrators for dynamic headspace analysis
4 HPLC, UV/VIS array detector, programmable fluorescence detector
1 Capillary electrophoresis system
1 Ion chromatograph, Dionex 4500 i
1 Ion chromatograph, Dionex BioLC (Photodiode Array Detector, Electrochemical Detector)
1 LC system, ECONO
1 Preparative HPLC
1 Zetaphoremeter, SEPHY

Elemental Analysis

1 TXRF, Atomika EXTRA II a
1 Flame-Photometer, Eppendorf ELEX 6361
2 AAS systems with flame atomization, electrothermal atomization, hydrid system, Perkin-Elmer PE 3300, ELAN 4100
1 ICP-MS, Perkin-Elmer ELAN 6100

Laser

2 He/Ne-laser
5 Nd-YAG-laser, pulsed
3 Nd-YAG-laser, cw
1 CO_2-laser
3 Dye-laser (tunable with frequency doubler)
5 N_2-laser
8 Diode-lasers (600-1670 nm; up to 2 W CW)
1 Laserdiodearray with 10 diodes (0.8 µm - 1.8 µm)
1 Laserdiode with external resonator
2 Optical parameter oscillator (410 nm - 2.1 µm)

Optoelectronics/Spectrometer

1 Rowland spectrometer
2 Echelle spectrometer
1 FTIR-Spectrometer, Thermo Scientific Nicolet 6700
1 Fluorescence spectrometer, Perkin Elmer LS-50
1 Fluorescence spectrometer, Shimadzu RF 540
1 Fluorescence spectrometer, Shimadzu RF 5301 PC
1 UV/VIS spectrometer, Beckman DU 650
1 UV/VIS spectrometer, analytic jena Specord 250 plus
1 UV/VIS spectrometer, analytik jena Spekol 1500
2 Boxcar integrator
4 Digital storage oscilloscopes (400 MHz, 500 MHz)
3 Optical multichannel analysators with monochromators, time-resolving
3 Intensified CCD cameras
2 Wavemeter

SEM/Microscopy

1 SEM/EDX system
1 Polarisation microscope for phase analysis
1 Fluorescence microscope
1 Image analysis software for automated image processing
1 Inert gas glovebox
1 Laminar flow box

Raman-Microscopy

1 Laser Raman microscope, Renishaw 2000 (514/633/785 nm)
1 Laser Raman microscope, Horiba LabRam HR (532/633/785 nm)
1 Temperature controlled stage (-196°C - 600°C, Linkam THMS 600)

Sum Parameters

2 Coulostat for C quantification, Coulomat 702
1 DOC analysator, UNOR 6 N
1 TOC analysator, TOCOR 2
1 AOX/TOX, Sigma

Staff 2013

Permanent Staff

Univ.-Prof. Dr. Reinhard Niessner
PD Dr. Thomas Baumann
PD Dr. Christoph Haisch
Dr. Natalia P. Ivleva
apl. Prof. Dr. Dietmar Knopp
Dr. Elisangela Linares (9/13-)
Dr. Michael Seidel

Birgit Apel
Christine Beese
Roland Hoppe
Joachim Langer
Susanne Mahler
Cornelia Popp
Christine Sternkopf
Christa Stopp
Sebastian Wiesemann

Mira Kolar
Hatice Poyraz

PhD Students

Dipl.-Phys. Christoph Berger
Dipl.-Chem. Henrike Bladt
Dipl.-Bio.-Ing. Dennis Elsässer (8/13-)
MSc Chem. Michaela Eß (5/13-)
Dipl.-Phys. Benedikt Grob
Dipl.-Ing. Moritz Herbrich
MSc Chem. Maria Hübner
MSc Chem. Bettina Kiwull
MSc Chem. Patrick Kubryk (3/13-)
MSc Geol. Wiss. Melanie Kühn
MSc Chem. Andreas Kunze (2/13-)
MSc Ing. Hydrogeol. Mark Lafogler
MSc Chem. Verena Meyer (9/13-)
MSc Chem. Christian Metz (-9/13)
MSc Chem. Anna-Catherine Neumann (6/13-)
MSc Chem. Susanna Oswald (-8/13)
MSc Pharm. Anal. Lu Pei (-8/13)
MSc Chem. Michael Pschenitza
Dipl.-Chem. Kathrin Schwarzmeier
MSc Geol. Martina Ueckert (6/13-)
MSc Chem. Xu Wang
MSc Chem. Alexandra Wiesheu (9/13-)
MSc Chem. Jan-Christoph Wolf (-8/13)
Leb. Chem. Anika Wunderlich (5/13-)
MSc Chem. Klaus Wutz (-1/13)
MSc Chem. Haibo Zhou

External PhD Students

Dipl.-Biol. Carmen Kocot (Institut Klinische Pathobiochemie München)
Dipl.-Phys. Peter Menzenbach (INNOLAS, Krailling)
Dipl.-Ing. Daniel Müller (ABF München)
MSc Biol. Johannes Otto (DVGW-TZW, Karlsruhe)
Daniela Rascher (HelmholtzZentrum)
Michael Schmalenberg (Klinikum r. d. Isar)

Diploma Students / Master Students

BSc Chem. Tobias Bauch (-3/13)
BSc Chem. Michaela Eß (-3/13)
BSc Chem. Fabian Knoller (-4/13)
Bsc Chem. Patrick Kubryck (-1/13)
BSc Chem. Verena Meyer (-4/13)
BSc Geo. Elena Mraz (-4/13)
BSc Chem. Anna-Cathrine Neumann (-4/13)
BSc Chem. Edgar Ramon (-6/13)
BSc Chem. Aleksandra Stanojlovic-Collin (11/13-)
BSc Chem. Klemens Thaler (6/13-11/13)
BSc Chem. Alexandra Wiesheu (3/13-8/13)
BSc Lebensm. Chem. Anika Wunderlich (-4/13)

External Master Students

Antonio Meraldo (BASF, 8/12-3/13)
Clement Ong (BASF, 7/12-3/13)

Guests and Research Fellows

Prof. Dr. Heinz Burtscher (FH Nordwestschweiz, 3/13-5/13)
Prof. Dr. Claudimir do Lago (University of Sao Paulo, 10/13-11/13)
MSc Nicoleta Elena Mircescu (Babes-Bolyai University Romanian, 9/12-2/13)
MSc Danting Yang (University China, 9/12-9/13)

Student Assistants

Matthias Edelmann, (5/13-7/13)
MSc Geol. Martina Ueckert (2/13-5/13)