

UNIVERSITÄT  
DUISBURG  
ESSEN



2017

Jahresbericht  
Annual Report



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# Instrumental Analytical Chemistry



Dear friends and colleagues,

after long hesitation we finally decided to compile an annual report as some of our colleagues already do regularly. We feared the work involved and indeed this first issue took us much longer than anticipated and thus becomes available only several months into 2018. Based on this first issue we hope to be faster in its production in the future though. The outcome was surely worth the effort in the end since also for ourselves it is good to look back once in a year and to realize what we have accomplished. And that is quite a lot:

We are in particular proud of our seven current or former group members who won various awards last year ranging from best graduate or thesis over conference poster or lecture distinctions to the FIM Environmental award 2017.

Several new projects were granted and commenced in 2017 that will be introduced together with still running projects on the next pages.

Six PhD students finished successfully their theses works and were awarded their doctoral degrees. 15 Master and 5 Bachelor students carried out their thesis at our department or at external partner institutes with a home supervisor at IAC.

We have also been very successful in publishing our scientific work with more than 20 peer-reviewed publications in the group and a classical German textbook on Analytical Chemistry coauthored from the 3<sup>rd</sup> edition onwards by me together with my colleagues Prof. Georg Schwedt and Prof. Oliver Schmitz.

In addition to the annual meeting of the Water Chemistry Society in Donaueschingen in May 2017, we have organized together with Prof. Schmitz and Dr. Michael Arlt of Merck a successful 7<sup>th</sup> Spring School "Industrial Analytical Chemistry" in Essen with almost 40 graduate students from all over Germany in March 2017.

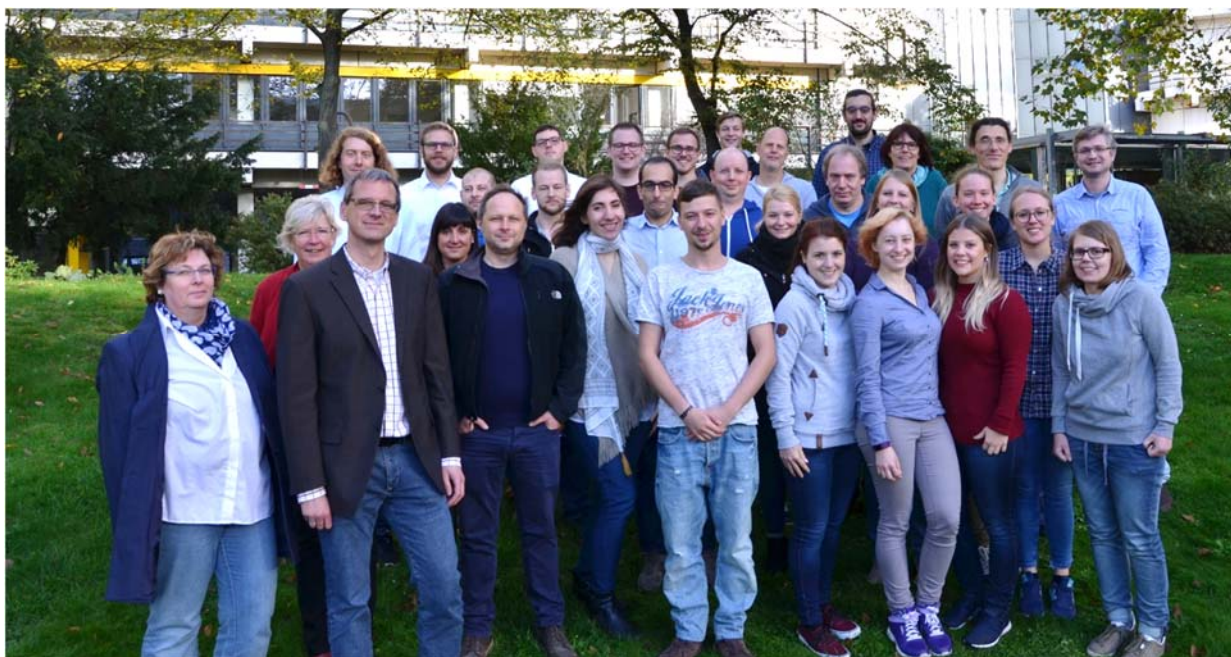
We participated in several outreach activities, in particular UNI Kids 2017 with an experimental lecture on "Water builds bridges, but does it also have a memory?" in front of more than 600 excited kids in the largest lecture hall at the Essen campus.

Finally, I would like to thank all group members and students for their excellent work at IAC and all partners from academia, industry and funding agencies for their great support and fruitful collaborations

I hope you enjoy reading the following report and wish all of you the best for the remaining year 2018,



Torsten C. Schmidt



From left to right (front row): Lydia Vaaßen, Torsten Schmidt, Maik Jochmann, Mischa Jütte, Vanessa Wirzberger, Michelle Lüling, Wiebke Kaziur, Katharina Hupperich,

(second row) Claudia Ullrich, Rosangela Elliani, Nerea Lorenzo Parodi, Vanessa Hinnenkamp, Alexandra Fischbacher, Laura Wiegand, Lotta Hohrenk

(third row) Daniel Köster, Lokman Coban, Robert Marks, Sajjad Abdi, Florian Metzelder, Klaus Kerpen

(last row) Oliver Knoop, Nenad Stojanovic, Jens Terhalle, Frank Jacobs, Joel Geldermann, Robert Knierim, Sasho Joksimoski, Ursula Telgheder, Holger Lutze, Andriy Kuklya

## Head of Chair

Prof. Dr. Torsten C. Schmidt

## Secretarial Office

Lydia Vaaßen

## Regular Staff

Dipl.- Ing. Gerd Fischer	IT Administrator/Project Administration
Dr. Maik Jochmann (AOR)	Stable Isotope Analysis/Sample Preparation and Gas Chromatography
Dr. Klaus Kerpen	2D-Fluorescence Spectroscopy/Advanced Oxidation Processes/Laser Commissioner
Dipl.- Ing. Robert Knierim	Laboratory equipment/Glassware/Support of guest scientists
Michelle Lüling	Laboratory assistant
Dr. Holger Lutze	Advanced Oxidation Processes
Robert Marks	Technical support
PD Dr. Ursula Telgheder	Ion Mobility Spectrometry/2D-Fluorescence Spectroscopy/Expert advice for course of studies/Radiation Protection Commissioner
Claudia Ullrich	Laboratory assistant/Safety officer

## Post Docs

Dr. Andriy Kuklya	Ion Mobility Spectrometry
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## Apprentices

Jaqueline Bruckmann	Laboratory assistant
Christin Herrmann	Laboratory assistant

## Ph.D. Students

### Internal

Mohammad Sajjad Abdighahroudi	An investigation of Pharmaceutical and Personal Care Products (PPCPs) as potential precursors for Nitrogenous Disinfection Byproduct (N-DBPs)
Christian Becker	Response analysis for a newly developed flame ionization detector for liquid chromatography
Lotta Hohrenk	Suspect and non-target screening of diffuse immissions into aquatic systems
Sasho Joksimoski	Novel coupling techniques for the determination of organic compounds in complex samples by ion mobility spectrometry
Alexandra Fischbacher	Formation and Quantification of ·OH in oxidative water treatment
Wiebke Kaziur	Development of a generic protocol for automated sample preparation in GC-MS
Daniel Köster	Development of a wet-chemical interface for online nitrogen isotope analysis of organic compounds using liquid chromatography-isotope ratio mass spectrometry (LC-IRMS)
Oliver Knoop	Formation and Effects of Transformation Products During the Ozonation of Tamoxifen
Xolelwa Lamani	Determination of aromatic amines as relevant toxicological components in aqueous and biological samples using comprehensive multi-dimensional GC-MS
Nerea Lorenzo Parodi	Aromatic amines as biomarkers in human urine: Analytical method development and epidemiological studies
Florian Metzelder	Investigation of sorption properties of carbon nanomaterials using packed columns and inverse liquid chromatography
Alyson Ribeiro	Ozonation and ecotoxicological assessment of two veterinary antibiotics: Cefapirin and Ceftiofur
Syed Mohammad Syed Khademi	Development of an on-line monitoring system for the analysis of organic contaminants in aquifers based on ion mobility spectrometry
Manuel Stephan	Microextraction techniques in GC/IRMS
Nenad Stojanovic	Development and application of advanced stable isotope methods to study compound degradation at real field sites
Jens Terhalle	Transformation processes and Isotope effects in natural and technical aqueous systems

Florian Uteschil	Drift tube ion mobility spectrometry laser induced fluorescence detection
Laura Wiegand	Characterisation of reactive species in the Fenton reaction
Sarah Willach	Stable isotope analysis to characterize oxidative and photochemical transformation processes
Vanessa Wirzberger	Oxidative water treatment: mechanistic aspects and matrix effects
Jens-Benjamin Wolbert	Characterization of glyphosate degradation by liquid chromatography-isotope ratio mass spectrometry (LC-IRMS)
Cornelia Zscheppank	Development of an analytical method for Identification and fast Screening Analysis of Microbial Metabolites

## External

Anastasia Barion (Günter)	Disk SPE-based sensitive determination of organochlorine pesticides and polybrominated diphenyl ethers in water
Nico Bätz	Effect-directed identification and reduction of diffuse immissions in rivers
Matthias Dumm	Cyanides in furnace gas scrubbing slurry
Lina Gessner	Evaluation of a simple quantification method of drugs of abuse in dried blood spots
Vanessa Hinnenkamp	Development and application of suspect and non-target screening of water samples using high resolution mass spectrometry (HRMS)
Frank Hitzgrath	Determination of diffusible hydrogen in high-strength steel related to lattice structure
Oliver Höcker	Characterisation of anaerobic processes in biogas generation using non-target screening
Fabian Itzel	Evaluation of innovative wastewater treatment processes using instrumental and effect-based analysis
Susann Pristat	Development of procedure tests for hot and cold coils considering the optimization of transport logistics of slabs
Kirsten Puschke	Suspected Target Screening of organic compounds in industrial wastewater
Gerrit Renner	Development of new spectroscopic and multivariate chemometric methods for the characterisation of (micro)plastics in the marine environment



Sara Schäfer (Saß)	Oxidative transformation of organic compounds in ultrapure water by ozonation and UV photolysis
Tobias Uber	Adsorption and desorption processes on polymers in aquatic systems

### Guest Scientists

Prof. Sina Dobaradaran, Bushehr University of Medical Sciences, Iran

Prof. Hiroto Kawashima, Akita Prefectural University, Japan

M. Sc. Rosangela Elliani, University of Calabria, Arcavacata di Rende, Italy

## Awards



**Jens Terhalle**

Water Science Awards for best master theses:  
„Chlorine dioxide in wastewater  
treatment -potential and by-products” (Kurita Award)  
Essen, 11.12.2017



**Carina Schönsee**

Young Scientist Award, Centre for Water and Environmental  
Research (ZWU) 2017



**Daniel Köster**

Best Poster Award for “Determination of Compound specific carbon  
isotope ratios for xylitol in chewing gum by LC-IRMS”  
ANAKON, Tuebingen, 03.-06.04.2017



**Wiebke Kaziur**

Award for best Master Degree in Water Science  
Essen, 29.11.2017



**Nerea Lorenzo Parodi**

Poster Award for „Aromatic amines in human urine: sample preparation using liquid phase microextraction (LPME)“  
Euroanalysis 2017, Stockholm, 28.08.-01.09.2017



**Terence Hetzel**

Award for best lecture on „Development of a fast method for the analysis of cytotoxic drugs using micro-LC-MS/MS“  
Ph.D. student seminar of AK Separation Science, Hohenroda, 08.-10.01.2017



**Claudia Ullrich**

Environmental award 2017 awarded by Fédération Internationale de Motocyclisme in (FIM) in Andorra la vella. The FIM was honoring an inspiring example of how nature conservation, youth education and motorsport can cooperate and work together in a trusting and open way. Marc Márquez (6-time world Motor GP champion), the ambassador for environmental, was

impressed about the amphibian protection and the particular work to save the natter jack and European green toad, the Biodiversity Project to save the nature and thanked the people who involved.

Andorra la vella, 24.11.2017

## Running Projects 2017

### **NRW Graduate School Future Water: Global water research in the metropolitan region Ruhr (Future water)**

**Involved staff:** Oliver Knoop, (IAC PhD student), Dr. Holger V. Lutze, Simon Kresmann, Claudia Freimuth (Coordinators), Prof. Dr. Torsten C. Schmidt (Speaker)

**Involved students:** Lotta Hohrenk, Fabian Berg

**Partners:** Prof. Dr. Bernd Sures, Prof. Dr.-Ing. André Niemann, Prof. Dr. Martin Denecke, Prof. Dr. Rainer Meckenstock, Prof. Dr. Jens Boenigk, Prof. Dr. Nicolai Dose (University of Duisburg-Essen), Prof. Dr. Marc Wichern, Prof. Dr. Michael Wilhelm (Ruhr-University Bochum), Dr. Jochen Türk, Fabian Itzel, Nico Bätz (IUTA), Prof. Dr. Sigrid Schäfer, Dr. Daniel Dangel (EBZ Business School Bochum), Prof. Dr. Mark Oelmann (HRW Mülheim), Prof. Dr. Claus Leggewie (KWI Essen) and many mentors and collaborators from the water and wastewater sector

**Funding:** Ministry for Innovation, Science and Research of the State of North-Rhine Westphalia (NRW) through the joint project “Future Water: Global water research in the metropolitan region Ruhr”

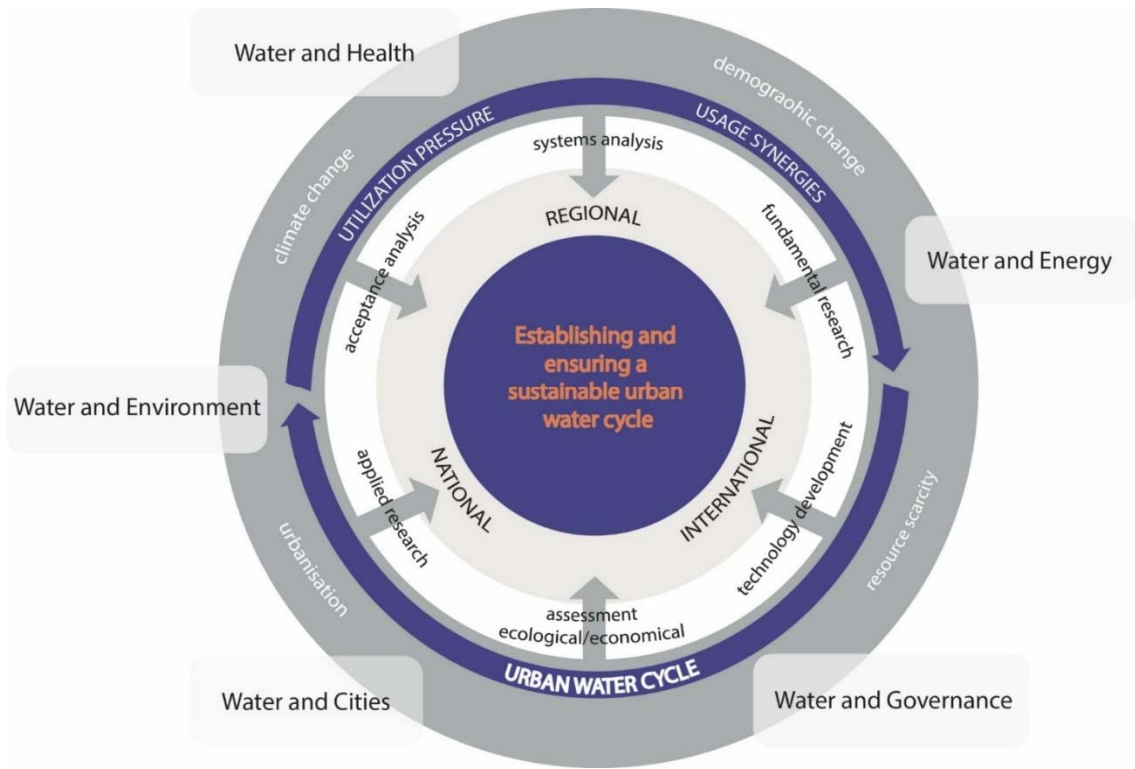


The multiperspectivity of inter- and transdisciplinary approaches allows to conduct innovative and path-breaking research. Combining knowledge and methods across disciplines makes possible to identify hitherto unnoted research questions. Tackling questions from different and novel angles allows to find answers that have not been conceived before. The integration of practitioners into these processes can enhance the relevance of the research questions, the fit of the methods applied, the effectiveness of research processes and the applicability and outreach of the results.

This approach is at the very heart of the graduate school “Future Water”, which is located in the Ruhr metropolitan area in Western Germany.

In 2014, a variety of academic and applied institutes joined forces to develop strategies for a sustainable water management with a special focus on the urban water cycle. The following figure describes the many facets of that work. The wide array of disciplinary backgrounds represented in the graduate school made building bridges between the natural sciences, applied engineering and social sciences both possible and necessary. Altogether, in Future Water 12 PhD students and a coordinator position are funded. Coordination is done by the Centre for Water and Environmental Research (ZWU) at UDE.

At IAC, in a strongly interdisciplinary project, transformation of the anti-estrogenic pharmaceutical tamoxifen by ozonation was studied, including the experimental investigation of changes of endocrine effects by cell-based tests systems and of effects on aquatic organisms.

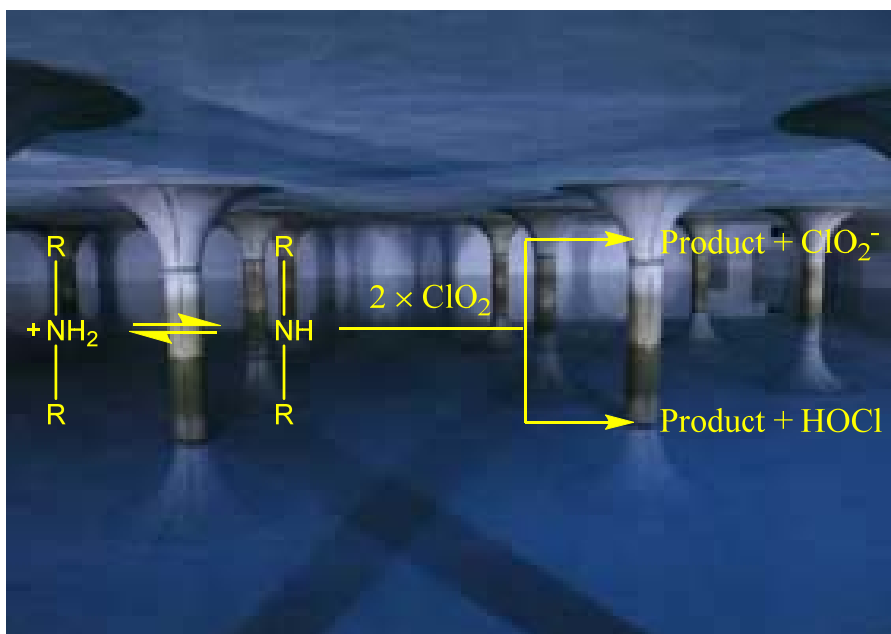


## Reactions of chlorine dioxide with nitrogen containing compounds

**Involved staff:** Mohammad Sajjad Abdighahroudi, Dr. Holger V. Lutze, Prof. Dr. Torsten C. Schmidt

**Involved students:** Xenia Mutke

**Funding:** German Academic Exchange Service (DAAD) via the Federal Ministry for Education and Research (BMBF) Program "Sustainable Water Management"



*Reaction of the disinfectant chlorine dioxide with nitrogen containing compounds*

The majority of anthropogenic pollutants are nitrogen containing compounds. Especially the reactions of these compounds in natural and technical processes are very complex and yet not fully understood. Many nitrogen containing compounds react fast with chlorine dioxide which is applied in drinking water disinfection. Traces of anthropogenic pollutants are also present in drinking water, which itself may cause no harm. However transformation products formed by chlorine dioxide products may reveal a higher toxicity than their precursors. Hence, the understanding of these reactions is very important.

In frame of a DAAD student ship the reactions of nitrogen containing compounds with chlorine dioxide are investigated. Organic pollutants, though, are very complex and often reveal several reactive sites which can react with chlorine dioxide, what aggravates their investigation. Hence, the present project focuses on simple model compounds which reveal only one site of reaction towards chlorine dioxide such as saturated *N*-heterocycles (e.g., piperidine).

So far the project has shown that chlorine dioxides reacts fast with saturated *N*-heterocycles (amines) and rather slow with aromatic *N*-heterocycles. Since, only deprotonated amines react with chlorine dioxide the kinetics increases with pH. 2 moles of chlorine dioxide are consumed per mole model compound transformed. In this reaction chlorine dioxide gives rise to chlorite (electron transfer) as well as another unknown product. This behavior of chlorine dioxide was already observed by other researchers for aromatic compounds (e.g., phenol) and it was postulated that the unknown product could be hypochlorous acid. This may also apply to nitrogen containing functional groups. For investigating this, a new method was developed to determine intrinsically formed hypochlorous acid. This method is based on the reaction of hypochlorous acid with glycine yielding chloroglycine. Chloroglycine is fairly stable and can be ion-chromatographed.

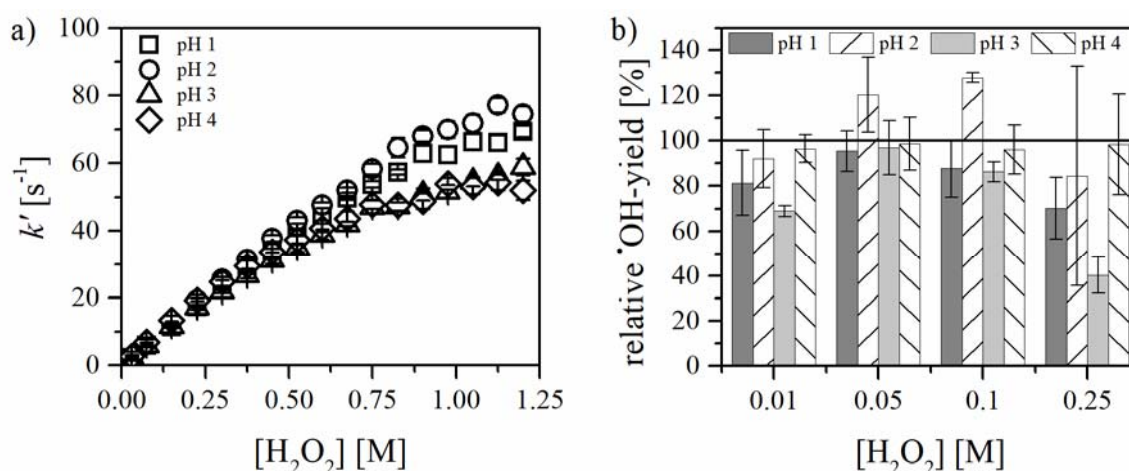
## Characterisation of reactive species in the Fenton reaction ( $\text{Fe}^{\text{II}} + \text{H}_2\text{O}_2$ ) - $\bullet\text{OH}$ vs. $\text{Fe}^{\text{IV}}$ (Fenton II)

**Involved staff:** Hanna Laura Wiegand, Dr. Holger V. Lutze, Dr. Klaus Kerpen, Prof. Dr. Torsten C. Schmidt

**Involved students:** Timon Orths, Katharina Hupperich, Mischa Jütte, Anke Dinter

**Partners:** Prof. Dr. Clemens von Sonntag (deceased)

**Funding:** German Research Foundation (DFG)



Observed pseudo first order reaction rate constants ( $k'$ ) (a) and relative  $\bullet\text{OH}$ -yields (b) at increasing  $[\text{H}_2\text{O}_2]$  determined at pH 1 – 4 ( $[\text{Fe}^{\text{II}}] = 300 \mu\text{M}$ )

For a better understanding of mechanistic aspects of the Fenton reaction ( $\text{Fe}^{\text{II}} + \text{H}_2\text{O}_2$ ) the influence of pH and presence of various organic chelates at pH 2 – 6 has been investigated with respect to existence and lifetime of an intermediate  $\text{Fe}(\text{H}_2\text{O}_2)^{2+}$ -complex and corresponding  $\bullet\text{OH}$ -yields in the Fenton reaction. It was found that the postulated intermediate  $\text{Fe}(\text{H}_2\text{O}_2)^{2+}$  is generated and quantitatively transformed into  $\bullet\text{OH}$ ,  $\text{OH}^-$ , and  $\text{Fe}^{\text{III}}$  ( $\sim 100\%$   $\bullet\text{OH}$ -yields per  $[\text{Fe}^{\text{II}}]_0$ ) regardless of pH and type of chelate. This may allow to use the Fenton reaction for remediation or as part of industrial wastewater treatment in absence of organic chelates at pH 1 – 4 as well as in presence of those ligands at pH 2 – 6. Formation of  $\text{Fe}^{\text{IV}}$  as a rather mild oxidant would largely limit the range of pollutants, which could be degraded. Furthermore, real water matrix constituents may serve as complexing agents similar to the investigated chelates.

In summary, results obtained in WPs 1, 3, 4 and 5 did not indicate appreciable formation of  $\text{Fe}^{\text{IV}}$ . Indeed, only an indirect approach was applied to rule out  $\text{Fe}^{\text{IV}}$ -formation in Fenton experiments but nevertheless, in case of considerable formation of  $\text{Fe}^{\text{IV}}$  in these reactions,  $\bullet\text{OH}$ -yields must have been markedly lower than 100 % per  $[\text{Fe}^{\text{II}}]_0$ . However, it is still possible that at reaction conditions not assessed in the course of this project  $\text{Fe}^{\text{IV}}$  might become relevant.

After those mechanistic investigations applicability of the Fenton reaction in terms of contaminant degradation was examined using bisphenol S (BPS) as model compound. It could be shown that degradation efficiency strongly depends on different parameters such as the ratio of  $[\text{H}_2\text{O}_2]$  to  $[\text{Fe}^{\text{II}}]_0$  or pH. With respect to the application of Fenton's reagent for degradation of contaminants in real samples these parameters must be adjusted considering actual conditions. Furthermore, it must be balanced between degradation time and chemical demand.

## Wastewater treatment on basis of chlorine dioxide (ABC)

**Involved staff:** Dr. Holger V. Lutze, Jens Terhalle, Prof. Dr. Torsten C. Schmidt

**Involved students:** Mischa Jütte, Johanna Buss

**Partners:** a.p.f. Aqua System AG

**Funding:** Federal Ministry for Economic Affairs and Energy (BMWi) by the Central Innovation Programme for SMEs (ZIM)



*Chlorine dioxide pilot plant*

This project deals with the application of chlorine dioxide for degradation of micropollutants in wastewater.

Wastewater treatment plants are an important source for micropollutants in the aquatic environment. Hence, for mitigating micropollutants wastewater treatment plants are currently retrofitted with advanced treatment steps based on activated carbon or ozone. Both steps have a similar efficiency in removing micropollutants from wastewater. In contrast to activated carbon, ozonation can also partially disinfect wastewater. However, ozonation also results in formation of undesired products such as cancerogenic bromate (US-EPA drinking water standard  $10 \mu\text{g L}^{-1}$ ). Chlorine dioxide is also a strong oxidant, which does not form bromate and may thus be an alternative process to wastewater ozonation.

The project has shown that pollutants with activated aromatic moieties (e.g., ethinylestradiol, sulfamethoxazole, diclofenac) can be readily degraded by chlorine dioxide. Interestingly, a degradation of some compounds, which barely react with chlorine dioxide, was also observed (e.g., atenolol). This was explained by the formation of the secondary oxidant hypochlorous acid, which reacts fast with atenolol. However, ozonation still appeared to degrade a larger range of micropollutants than chlorine dioxide. As by-product chlorine dioxide forms chlorite (US-EPA drinking water standard  $1 \text{ mg L}^{-1}$ ). Yet, the relevance of chlorite formation in wastewater treatment is investigated.

First results of the present project also indicated that chlorine dioxide may be a very good disinfectant. Currently, importance of wastewater disinfection increases since antibiotic resistant microorganisms in the environment were observed.

Results from bench scale were successfully up-scaled to pilot scale at a real wastewater treatment plant, indicating that a full-scale application of chlorine dioxide in wastewater treatment may be possible.



## Treatment of Tai Hu water

**Involved staff:** Dr. Holger V. Lutze, Prof. Dr. Torsten C. Schmidt

**Involved students:** Mischa Jütte, Pascal Kaiser, Johanna Buss, Robert Marks

**Funding:** Federal Ministry of Education and Research (BMBF)

Under contract of Research Centre Jülich (Dr. Stephan Küppers)

Close cooperation with Inge GmbH (Christian Staaks), bbe Moldaenke GmbH (Christian Moldaenke), TZW Dresden (Dr. Wido Schmidt) and institute of mechanical engineering (University Duisburg-Essen, Prof. Panglich, Sucipta Laksonon, M. Sc.), IWW Water Centre (Dr. Tim aus der Beek, Dr. Andreas Nahrstedt)



*Planned treatment chain for purification of Tai Hu water and ozone depletion after flocculation and membrane filtration (bench scale experiments)*

The Tai Hu (Tai Lake) is used as a raw water reservoir for approximately 10 million inhabitants predominantly in the Jiangsu province, China. Algae/cyanobacterial blooms occur frequently in the eutrophic shallow lake and represent a challenge for drinking water treatment. Furthermore, occasionally taste & odor (T&O) problems have been reported in drinking water. Due to the impact of wastewater and surface water runoff, also presence of pesticides and emerging pollutants such as pharmaceutical compounds have to be considered.

It was shown, that part of the T&O compounds present in finished drinking water are formed from reactions of amino acids during water treatment (e.g., by chlorination). These amino acids may be part of the algae organic matter that may be released after cell lysis in conventional water treatment using pre-ozonation, flocculation and sedimentation. Our results have shown that ozonation can cause lysis of algae with subsequent release of intracellular material. Hence ozonation should be performed after a filtration step. The process consisting of 1. flocculation, 2. ultrafiltration and 3. ozonation was investigated in a combination of pilot- and bench scale experiments. The results revealed that the pretreatment by flocculation and ultrafiltration does not only remove intact algae cells but also improves the overall performance of the ozonation step. The combination of flocculation and ultrafiltration removes a part of the dissolved organic matter which reduces the required ozone dose. First results have also shown, that direct ozonation of the Tai Hu water may be prone to form halogenated organic compounds. This was deduced from the observation that after addition of ozone bromide was oxidized, while the final product bromate (undesired disinfection by-product with a US-EPA and EU drinking water standard of  $10 \mu\text{g L}^{-1}$ ) was formed to a minor extent (incomplete bromine mass balance). The remaining fraction of oxidized bromide (e.g., hypobromous acid) might have reacted with organic matter to form brominated organic compounds. After flocculation and ultrafiltration though, bromide recoveries (sum of bromide and bromate) very much improved. It is worth mentioning that halogenated organic compounds may also originate from chlorinated wastewater discharge into the Tai Hu.

The formation of halogenated products and strategies for bromate minimization are currently investigated. Furthermore, the treatment chain shown above will be investigated in pilot scale with additional bench scale experiments.

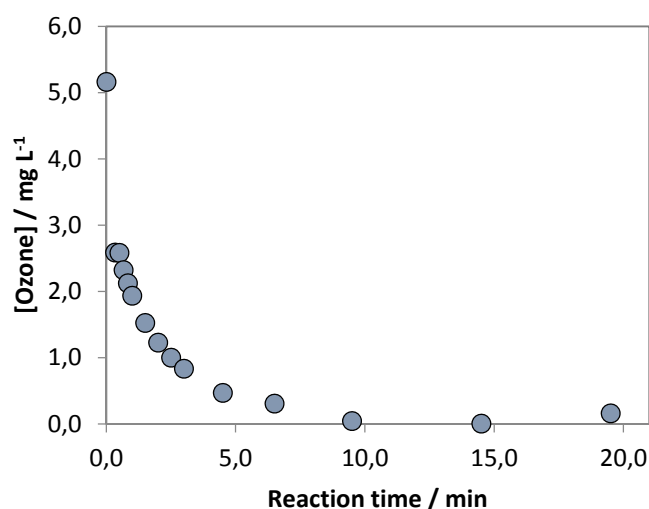
## Water and wastewater ozonation feasibility studies

**Involved staff:** Dr. Holger V. Lutze

**Involved students:** Mischa Jütte, Pascal Kaiser, Johanna Buss, Robert Marks

**Funding:** Partners from water industry

Cooperation with IWW Water Centre (Marcel Koti, Anil Gaba, Dieter Stetter, Andreas Nahrstedt) and IUTA (Andrea Börgers, Jochen Türk)



*Ozone depletion in a wastewater*

In cooperation with IWW and IUTA several feasibility studies on wastewater ozonation were performed. One important factor in water treatment is energy demand, which can represent a large part of the operational costs. One strategy to reduce costs is a dynamic treatment performance taking fluctuations of electricity prices into account (.e.g., reduced treatment performance at high costs for electricity and vice versa). Such kind of concepts were investigated in the project Enerwa. A part of the work dealt with energy saving strategies for ozonation, which were investigated in pilot scale and bench scale experiments.

Ozonation is also applied in wastewater treatment for removal of residual anthropogenic trace compounds. Since, trace compound removal in wastewater treatment was supported by government in NRW many operators of wastewater treatment plant engaged bench scale studies in wastewater ozonation to assess the operational conditions for a full scale ozonation plants. These bench scale experiments investigated the amount of ozone needed for degradation of pollutants as well as the required hydraulic retention time of the ozonation reaction tanks. In wastewater ozonation higher ozone dosages have to be applied. This is mainly due to the concentration of organic matter (OM) in wastewater (average app. 5 – 10 mg L<sup>-1</sup> OM) which is higher compared to source water used for drinking water purification (average app. 0.5 - 2 mg L<sup>-1</sup> OM).

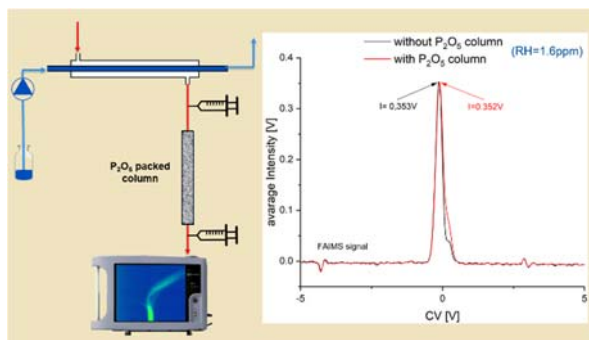
## Development of a combined in-situ-remediation- and monitoring system for the treatment of groundwater damages (LUKE)

**Involved staff:** Sasho Joskimoski, Dr. Andriy Kuklya, Michelle Lüling, PD Dr. Ursula Telgheder

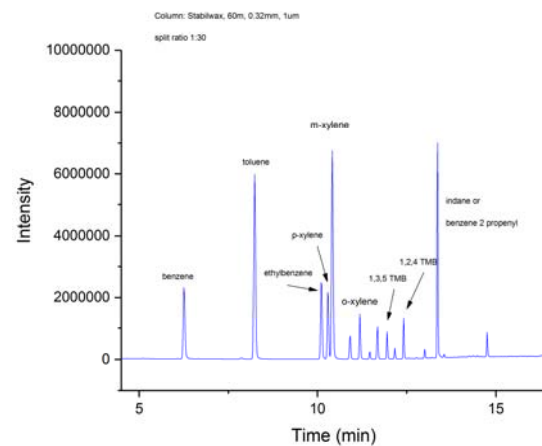
**Partners:** Fabricius Pro Terra GmbH

**Funding:** Federal Ministry for Economic Affairs and Energy (BMWi) by the Central Innovation Programme for SMEs (ZIM)

a)



b)



*Gas-water separation unit for direct monitoring of BTEX contaminated groundwater with FAIMS (a) Experimental set-up (b) GC-MS analysis of BTEX in contaminated groundwater*

The aim of this project is the development of a cost-effective in-situ-procedure for the remediation of ground water contaminated with BTEX. The design of a novel multiparameter – spear system allows the introduction of a gas injection system, a continuous sampling of the headspace and the direct determination of pH-value,  $O_2$ ,  $CO_2$  and  $CH_4$ -amount, conductivity and humidity in the aquifer.

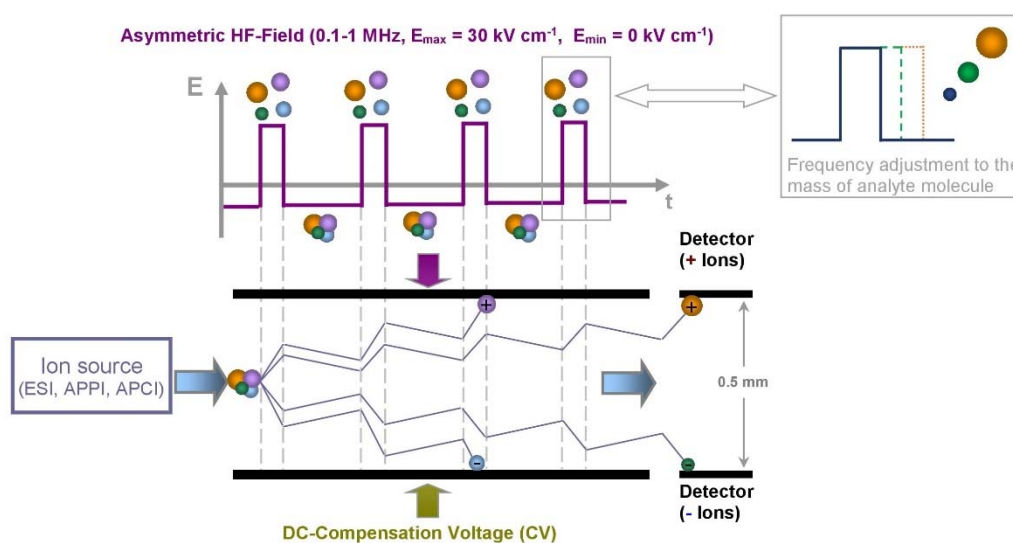
Applying the designed system, monitoring of vitality parameters and generation of  $CO_2$  and  $CH_4$  by metabolic processes are possible. The remediation takes place by reversible introduction of a glass fibre reinforced polymer membrane into the aquifer releasing oxygen by diffusion. The introduction of further nutrients by using the spear system is possible. The multiparameter – spear system is coupled with a modified remote controlled High-Field Asymmetric Waveform Ion Mobility spectrometer (FAIMS), which allows the monitoring of the BTEX concentration in the groundwater monitoring wells. A fast evaluation of the remediation efficiency is possible using the developed system.

## Fast on-site analysis for the detection of pesticides in water by 2D-DMS (Hydro-2D-DMS)

**Involved staff:** Dr. Klaus Kerpen, PD Dr. Ursula Telgheder

**Partners:** Ampegon PPT GmbH

**Funding:** Federal Ministry for Economic Affairs and Energy (BMWi) by the Central Innovation Programme for SMEs (ZIM)



*Schematic illustration of the functional principle of 2D-differential mobility spectrometry system*

The aim of this project is the development of a sensitive, cost-effective analytical system for monitoring of selected pesticides (quinoxifen, cybutryne, dichlorvos, terbutryn) in different water matrices with regard to the legally binding limit values. The analytical system is based on differential mobility spectrometry (DMS) technique. Commercially available DMS-systems are limited to fixed frequencies (1MHz) of the electrical field. In the frame of this project, a detector will be developed with variable frequencies in order to enlarge the detectable range of masses with high sensitivity. The sample introduction system as well as the ionization source have to be adapted to the novel detector. The experimental parameters have to be optimized. Calibration and the analysis of real water samples with different compositions shall give an evaluation of the performance of the prototype.

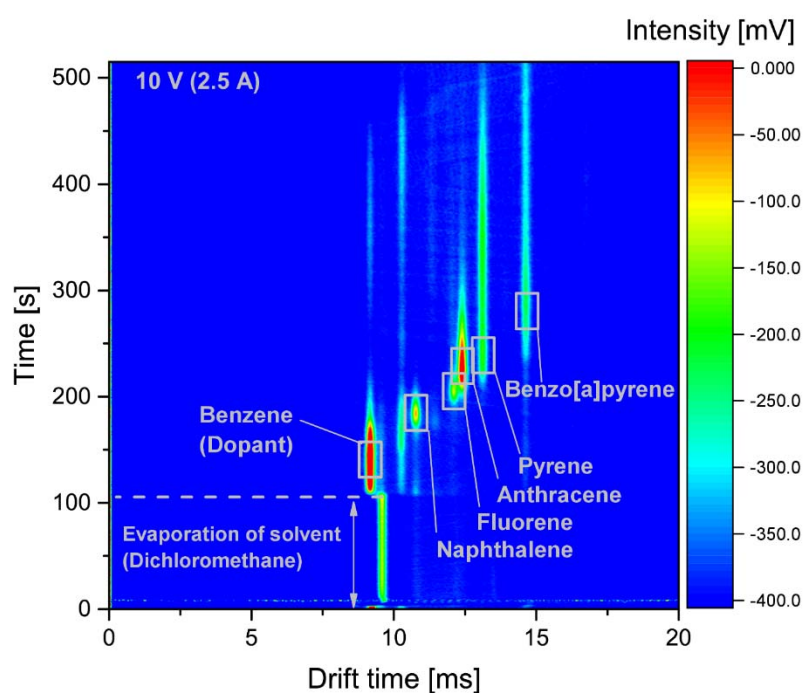
## Development of a Direct Inlet Probe - Atmospheric Pressure Photoionization – Ion Mobility Spectrometers (DIP-APPI-IMS) -system for quality control of chemical and pharmaceutical products (DIPPI-IMS); Development of a miniaturized IMS coupled to direct inlet probe (DIP)

**Involved staff:** Dr. Andriy Kuklya, PD Dr. Ursula Telgheder

**Involved students:** Lokman Coban

**Partners:** SIM GmbH

**Funding:** Federal Ministry for Economic Affairs and Energy (BMWi) by the Central Innovation Programme for SMEs (ZIM)



*Three-dimensional plot of the analysis of a polycyclic aromatic hydrocarbon mixture by DIP-APPI-IMS*

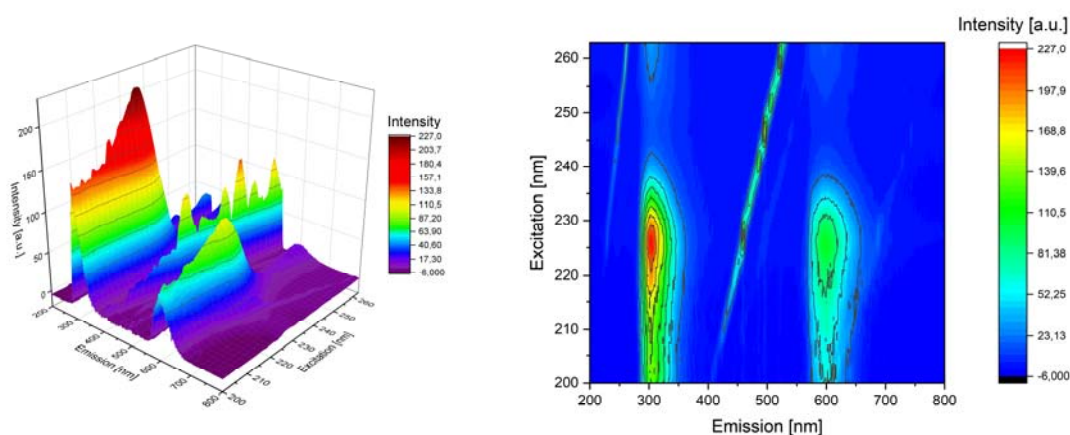
A sensitive and cost-effective system for the quality control of chemical and pharmaceutical products will be developed by coupling of Direct Inlet Probe with Atmospheric Pressure Photoionization – Ion Mobility Spectrometry (DIP-APPI-IMS). The direct inlet probe enables the introduction and evaporation of solid and liquid samples. Matrix compounds will be removed by application of a suitable temperature program. Atmospheric photo ionization is used for the selective ionization of the analytes. The generated ions will be separated and detected by a miniaturized ion mobility spectrometer in milliseconds. After optimization of the prototype selected real samples will be analyzed in order to evaluate the performance of the system. The validation of the methods is made by GC-MS analysis.

## Fast on-site-detection of sulfonamides in ground- and surface water by 2D-fluorescence spectroscopy (MultiDimSpec)

**Involved staff:** Dr. Klaus Kerpen, PD Dr. Ursula Telgheder

**Partners:** GO Systemelektronik GmbH

**Funding:** Federal Ministry for Economic Affairs and Energy (BMWi) by the Central Innovation Programme for SMEs (ZIM)



*Fluorescence spectra (left side: 3D-spectra; right side: contour plot) of an aqueous solution of sulfadiazine ( $c = 1 \text{ mg}\cdot\text{L}^{-1}$ ). Excitation in the range of 200 nm to 263 nm in 1 nm steps. Emission in the range of 200 nm to 800 nm in 0.5 nm steps*

A miniaturized system for the fast on-site analysis of selected sulfonamides in different types of water samples based on 2D-fluorescence spectroscopy has to be developed in compliance to legally binding limit values. The sensitivity of the fluorescence is high enough to determine the required concentrations. The selectivity will be achieved by multivariate data analysis of the of the emission spectra at different excitation wavelengths. The selected indicator substance are sulfamethoxazol, p-toluenesulfonamide, o-toluenesulfonamide, benzenesulfonamide, sulfadiazine and sulfamethazin. The prototype will be integrated in a multi sensor system after optimization of the experimental parameters. The complete system will be tested in field studies. The development of control software and complex software moduls for the evaluation of data is part of the project.

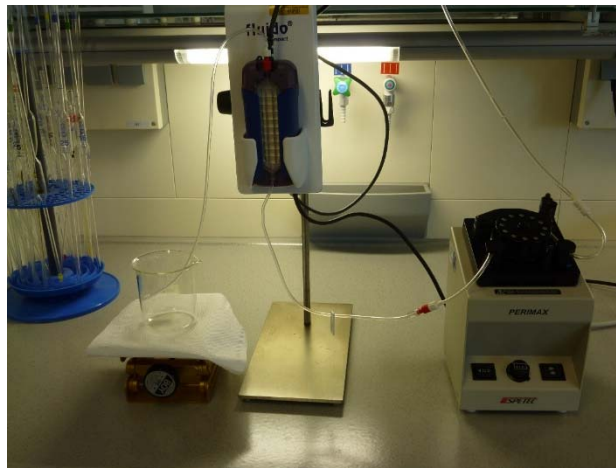
## Investigation of the aluminum discharge of warming systems for inline fluid warming of infusions by GFAAS (Al-dis-infu)

**Involved staff:** PD Dr. Ursula Telgheder

**Involved students:** Anna Lena Röhrig

**Partners:** The 37°Company

**Funding:** Industry cooperation project



*Experimental set-up of the batch system "fluido compact"*

Novel fluid warming systems for inline fluid warming of infusions and blood products provide small, effective devices in the range up to 500 mL/h volume. The systems consist of a controller, a warming module and a disposable including the warming unit. Generally, the warming unit is made of aluminum. Different designs of the warming units are available. Beside heating-elements made of pure aluminum, also Parylene-coated devices exist. In order to answer the question, whether a significant amount of aluminum compounds is released from the disposables during use with various infusions, systematic investigations has to be carried out.

The aim of this project includes the quantitative determination of aluminum by graphite furnace atomic absorption spectrometry (GFAAS) in selected infusion solutions after warming with an uncoated disposable as well as a Parylene-coated device.



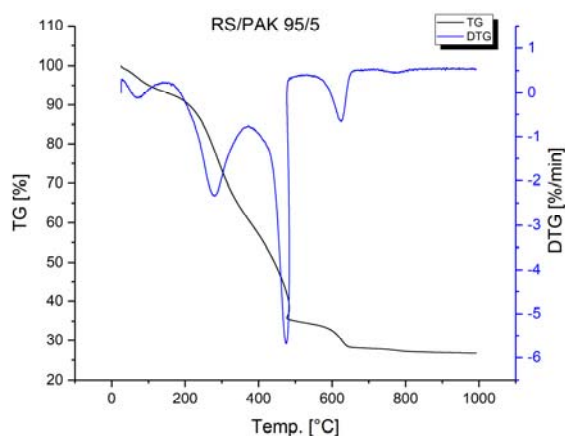
## “Den Spurenstoffen auf der Spur – Teil 3” (DSADS)

**Involved staff:** Michelle Lüling, PD Dr. Ursula Telgheder

**Involved students:** Anna Lena Röhrig

**Partners:** Lippe Verband, Ruhr-Universität Bochum, Fachgebiet Siedlungswasser- und Abfallwirtschaft Universität Duisburg – Essen, Fakultät für Biologie, Abteilung Aquatische Ökologie Universität Duisburg - Essen

**Funding:** District Government Münster, North-Rhine Westphalia



*Thermogravimetric Analysis by using the instrument STA 7200 Firma Hitachi* 95 % Dry substance (Return sludge RS) / 5 % Activated carbon powder (PAK)  
weight: 9.5/0.5 mg

In the course of 4th purification step in waste water treatment, the application of activated carbon is tested in several treatment plants as a further purification step. Up to now, it is not known if a small proportion of the loaded activated carbon is passing the sand filter or is released into the water bodies.

The aim of this project is the development of a reliable method for evaluation of the suitability of the application of activated carbon as a 4<sup>th</sup> purification step in waste water treatment.

A further possibility for the determination of activated carbon in presence of natural organic matters is the analysis by thermogravimetry in combination with differential thermoanalysis. For the determination of unknown amounts of activated carbon in purified sewage water or return sludge, the mass losses of pure dried substance released from the treatment plant and from the activated carbon will be determined. Furthermore, the mass losses of defined mixtures of both compounds will be determined. The unknown amount of activated carbon in a mixture will be calculated by applying of stoichiometric ratio.

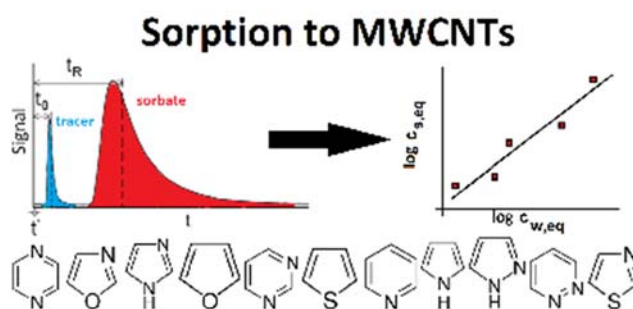
## Characterization of sorption of non-ionic organic compounds on carbon-based nanomaterials (Sorption II)

**Involved staff:** Florian Metzelder, Prof. Dr. Torsten C. Schmidt

**Involved students:** Matin Funck

**Partners:** Dr. Thorsten Hüffer, University of Vienna

**Funding:** German Research Foundation (DFG), Bruno-Werdelmann-Stiftung



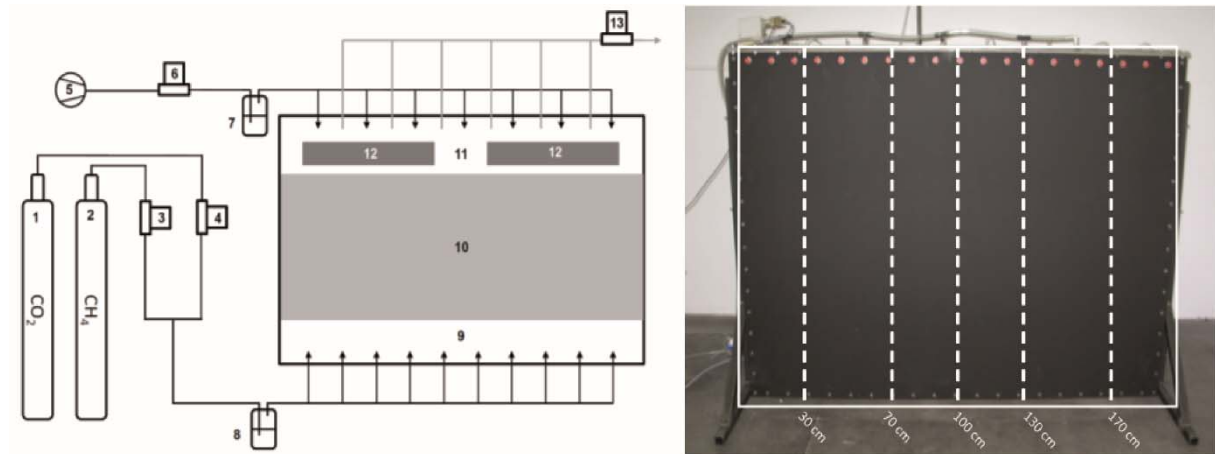
The interaction between carbon nanomaterials (CNM) and organic or inorganic compounds is studied by many scientists, because of the strong sorption of CNM. Although already many studies have been conducted with different compounds and materials in batch experiments, a general understanding of the underlying sorption mechanisms was missing. The results of the first project phase helped to improve the knowledge in this field concerning carbon nanotubes. Different models were developed, which give information about the relevant sorption mechanisms, but they are only valid for certain environmental conditions (pH, ionic strength, temperature) and are not transferable to other conditions. The influence of environmental conditions on sorption was studied in literature only for a limited number of compounds and materials with the consequence that even here a general understanding of the underlying processes is still missing. The batch experiments used in literature and the first project phase had disadvantages, namely the long equilibration time and limited amount of sorbent and hence the variation in environmental conditions and material characteristics was limited. In the proposed follow-up project to the running project SCHM 1372/10-1 the mechanistic investigations concerning sorption should be continued and the focus is shifted on the variability of sorption to different types of CNM and on the effect of different environmental conditions on sorption. For this purpose, a column-based method shall be developed, that is able to substitute the classical batch approach and thus to enable the faster variation of environmental conditions with less time-consuming work. First, it is investigated with validated columns how comparable results to batch experiments can be obtained. After establishment of this method, various sorbates are used as chemical probes to characterize the different sorbents. Sorption experiments will be performed at different environmental conditions to investigate the influence of these parameters on sorption. The results of these measurements are implemented in existing models or used to develop new models, which enable the prediction of sorption of organic compounds to CNM for different environmental conditions. Based on an improved process understanding, columns packed with CNM will be exemplary applied for separations in the field of analytical chemistry in comparison with a classical C18-phase.

## Characterization of boundary conditions and failure of biological methane oxidation in landfills: experimental investigation, chemical analysis, mechanical modelling and numerical simulation (Methane Oxidation II)

**Involved staff:** Dr. Marcel Schulte, Dr. Maik A. Jochmann, Prof. Dr. Torsten C. Schmidt

**Partners:** Prof. Dr.-Ing. Tim Ricken, Andrea Thom (TU Dortmund), Prof. Dr. Martin Denecke, Tobias Gehrke (University of Duisburg-Essen)

**Funding:** German Research Foundation (DFG)



*Experimental set-up for the investigation of methane oxidation in a model reactor*

Methane formed in reductive degradation of organic matter in landfills contributes substantially to greenhouse gas emissions. Therefore, an active management of methane emissions is implemented on modern landfills. Critical in that regard is the time period when methane contents in gas emissions are too low for energetic use. Further mitigation of emissions in this passive phase can be achieved by soil covers that facilitate methane oxidation by methanotrophic bacteria. However, up to date it is not fully understood how the oxidation potential changes and in a worst case ceases under varying boundary conditions such as temperature, substrate concentration or humidity. Based on results of the first study period focus of the second phase of the research project therefore is to foster our understanding and prediction of biological-chemical coupled diffusion-convection-reaction processes under such conditions. Furthermore, an important question within the project addresses the potential for recovery of oxidation capacity after cessation phases. Results of the IAC project part are summarized in the abstract of the PhD thesis of Marcel Schulte below.

## Marie Skłodowska-Curie Actions Innovative Training Network (MSCA-ITN) “Improved decision-making in contaminated land site investigation and risk assessment” (REMEDiate)

**Involved staff:** Nenad Stojanovic (ESR), Dr. Maik A. Jochmann, Prof. Dr. Torsten C. Schmidt

**Involved students:** Felix Niemann

**Partners:** Dr. Rory Doherty (Queen’s University Belfast), Dr. Brian Kelleher (Dublin City University), Dr. Frederic Coulon (Cranfield University), Prof. Dr.-Ing. Tim Ricken (TU Dortmund), Dr. Kristian Brandt (University of Copenhagen), Dr. Domenico De Luca (University of Turin),

**Funding:** EU Commission



The REMEDiate ITN will develop innovative research and training for the contaminated land site investigation and risk assessment industry in Europe. The REMEDiate ITN comprises 11 Partners from 6 Member States. The Partnership includes leading academic/ research organisations as well as large and small motivated representatives from the private sector. Together they will provide training across the disciplines of microbiology, chemistry, toxicology, engineering, materials science and environmental modelling. All Partners are committed to the common goal of providing a superior level of training to 15 young researchers for the contaminated land sector in Europe. Furthermore, the strong representation and commitment of the contaminated land sector through Associated Partners (4 commercial enterprises, 5 public sector bodies and 2 international higher education institutes) will ensure relevance of the training to future industry needs, and will ensure entrepreneurial thinking permeates every aspect of the ITN. REMEDiate will deliver overarching and advanced approaches to management of contaminated sites. It will address site investigations, risk assessment, life cycle assessment and the effectiveness of site remediation. The ultimate goal is to provide a superior level of training for the contaminated land sector, to generate improved decision-making skills for more effective and sustainable remediation. The problem of land contamination across an expanding EU is still highly significant. As a result over €2,000 million is currently spent annually on site risk management. As the EU further develops a common framework for the management of contamination sites, the burden on sustainability, characterisation, risk management and remediation is expanding. REMEDiate will not address specific remediation technologies but will instead address the overarching multidisciplinary demands needed for enhanced leadership and decision-making in the future.

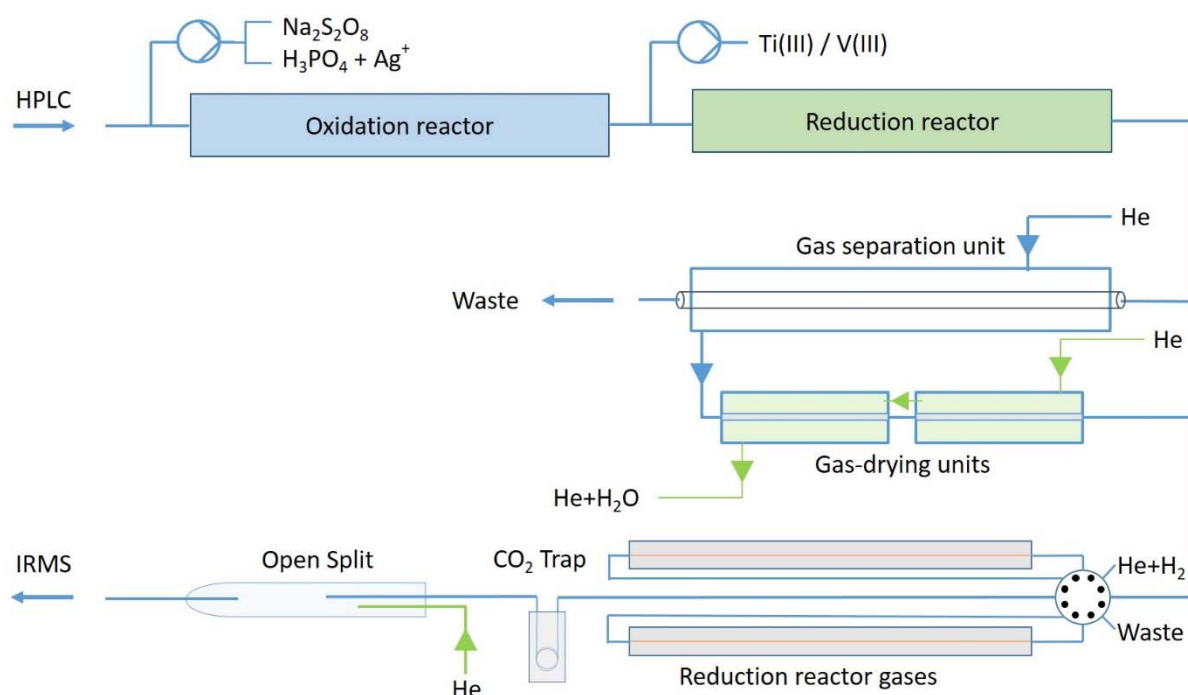
At IAC, the project looks specifically into the use of advanced stable isotope methods for site assessments in order to better characterize biodegradation processes of organic contaminants.

## Development of an LC-IRMS interface for the component specific determination of the nitrogen isotopic ratios of organic compounds via wet chemical oxidation/reduction (LC-IRMS)

**Involved staff:** Daniel Köster, Dr. Maik A. Jochmann, Dr. Holger V. Lutze, Prof. Dr. Torsten C. Schmidt

**Involved students:** Tobias Hesse

**Funding:** German Research Foundation (DFG)



### *Experimental set-up of the proposed LC-IRMS interface for the component specific determination of nitrogen isotope ratios*

During the last years, compound specific stable isotope analysis (CSIA) has successfully been applied in the field of food authenticity control, doping analysis in sports and in the elucidation of degradation pathways of environmental pollutants. In addition to the widespread use of gas chromatography coupled to isotope ratio mass spectrometry (GC-IRMS), liquid chromatography can be applied to separate polar, not directly GC compatible compounds. With the availability of LC-IRMS interfaces, it became possible to separate polar compounds for example directly from an aqueous matrix. Currently commercially available LC-IRMS interfaces utilize wet chemical oxidation of the analytes eluting from the LC system. Peroxodisulfate is commonly used in the interface system to oxidize the carbon in the analyte molecules to carbon dioxide via sulfate radicals. In a second step the carbon dioxide is separated from the liquid mobile phase with the help of gas permeable membranes and directly transferred into the IRMS system by a helium carrier gas stream. In contrast to GC-IRMS, LC-IRMS systems are restricted to the determination of carbon isotopic ratios until now, as no uniform nitrogen reaction product for the IRMS measurement is formed during the oxidation. The oxidation products of nitrogen containing organic compounds after sulfate radical treatment have not been studied in detail yet. Estimated reaction products are on the one hand water soluble compounds like nitrate and nitrite,

on the other hand gaseous nitrogen oxides will be formed. The oxidation products of different classes of nitrogen containing compounds will be studied in this work. To be able to determine isotope ratios for nitrogen from the organic compounds, the contained nitrogen has to be converted into a single species for the IRMS measurement e.g. elemental nitrogen. Therefore the water soluble nitrogen species will be reduced to gaseous nitrogen oxides, which are separated from the liquid phase by a membrane system. In a second reduction step, the gaseous nitrogen oxides will be further reduced to elemental nitrogen with the help of copper. The resulting nitrogen gas will be treated similar to CO<sub>2</sub> and is directly introduced into the IRMS by the carrier gas. In the project, different pathways for the wet chemical reduction of nitrite and nitrate after the oxidation of the analytes will be compared. In addition, the conversion rates during the wet chemical oxidation of the analytes and the reduction of the different nitrogen species in the system will be studied. Finally, the nitrogen isotopic ratios which can be obtained for different model compounds with the LC-IRMS system will be validated with the help of an elemental analysis isotope ratio mass spectrometry (EA-IRMS) system and international isotope reference material.

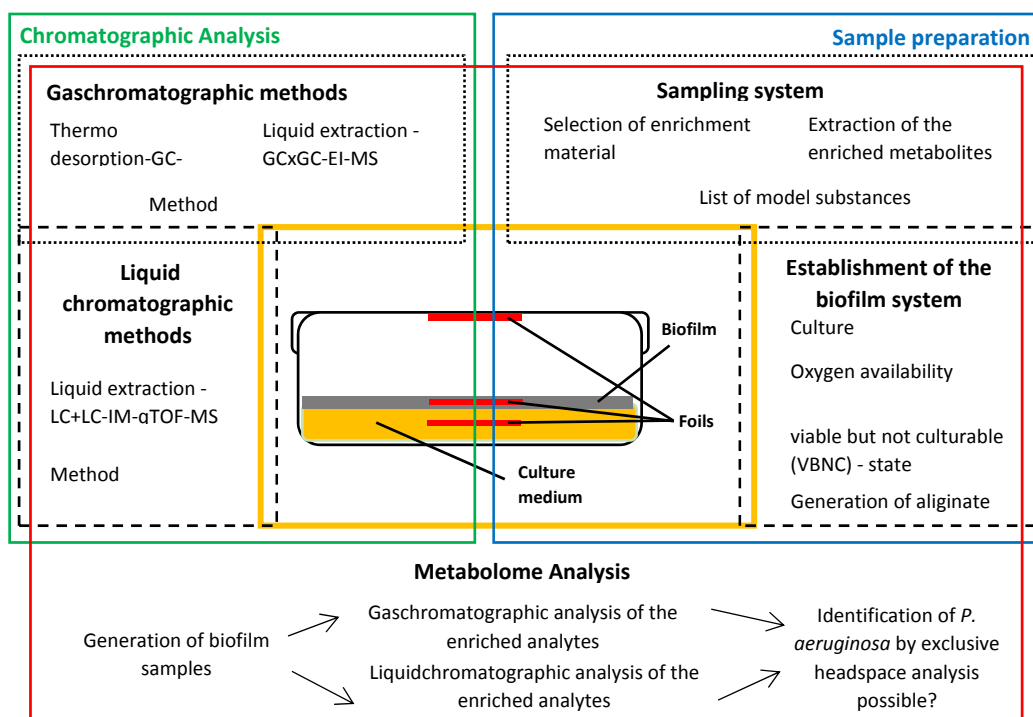
## Characterization of the metabolome of *P. aeruginosa* in biofilms as a lung infection model

**Involved staff:** Timo Köhler (AAC), Cornelia Zscheppank, PD Dr. Ursula Telgheder

**Involved students:** Maryam Taghavi, Frank Jacobs

**Partners:** Prof. Dr. Oliver Schmitz, AAC, University of Duisburg - Essen

**Funding:** German Research Foundation (DFG)



In this project, the metabolome of *P. aeruginosa* will be characterized considering lung adapted conditions by using a biofilm as a lung infection model. Therefore, a sampling system for the cultivation of *P. aeruginosa* will be developed, which enables the sampling of the culture medium, the biofilm and the headspace over time. This three-phase approach allows the classification of detected and characterized substances as substrates or metabolites. A comprehensive preconcentration of the metabolites shall be achieved by application of various extraction techniques.

The qualitative and quantitative determination of selected metabolites will be carried out by GCxGC-EI-MS and LC+LC-IM-qTOF-MS. The sample preconcentration and experimental parameters e.g. stationary phase, analysis time in the 2<sup>nd</sup> dimension (modulation time) and flow rates will be optimized.

As an internal standard, Benzothiazole will be added to the culture medium in order to investigate and control the preconcentration, diffusion and distribution of the potential metabolites.

Based on the results, it shall be verified if *P. aeruginosa* is represented by detecting only the metabolites in the headspace of a biofilm sample. This should answer the question whether the identification of *P. aeruginosa* is possible only by head space analysis or not.

## Aromatic amines as biomarkers in human urine using GCxGC: analytical method development and epidemiological studies

**Involved staff:** Nerea Lorenzo Parodi, Prof. Dr. Torsten C. Schmidt

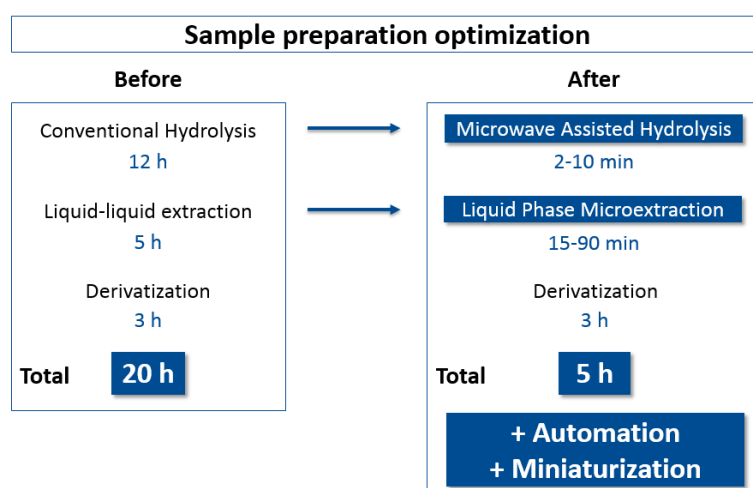
**Involved students:** Wiebke Kaziur

**Partners:** Dr. Astrid Gjelstad (University of Oslo), Prof. Dr. Susanne Moebus (Uniklinikum Essen)

**Funding:** Evonik Industries AG

Several aromatic amines pose a health threat due to their carcinogenic effects. One of the most important exposure routes to these compounds is through tobacco smoke. After being inhaled, the aromatic amines enter the bloodstream and can be transported to the liver, where they are metabolized, and eventually reach the bladder. Once in the bladder, the metabolites can either be excreted with the urine or form DNA and protein adducts that can induce bladder cancer.

Sample preparation procedures for the analysis of aromatic amines with GC-MS usually involve time consuming and labor intensive steps. The first part of the project aims to minimize this burden by substituting the conventional approach with a more efficient one. The individual steps are compared with alternative ones (conventional vs. microwave assisted hydrolysis and liquid-liquid extraction vs. liquid phase microextraction) and the best performing option is chosen for future experiments.



*First aim: automation, miniaturization and increased efficiency of the sample preparation steps.*

Thanks to the collaboration with Prof. Dr. Susanne Moebus, involved in the projects Heinz Nixdorf Recall study (over 4.800 participants) and Heinz Nixdorf MehrGenerationenStudie (3 generations), we have access to urine samples and medical information from the participants. In order to achieve a high throughput and have access to the limited archived samples, automation and miniaturization of the sample preparation procedure are also key components of the sample preparation optimization.

Finally, once the optimized method is established, a statistically relevant number of samples will be measured. Afterwards, the correlations between the aromatic amines present in the samples (amounts and types), the smoking status of the donor (smoker, non-smoker and passive smoker) and the development of smoking related diseases will be studied using complex statistical methods.

So far, microwave assisted hydrolysis was tested and compared to conventional hydrolysis, obtaining worse results than expected. Furthermore, three phase HF-LPME and PALME were studied and compared to the conventional liquid-liquid extraction step. PALME showed promising results and the further validation of the method is going to be studied. However, due to automation limitations of the PALME setup, the conventional hydrolysis step will simultaneously be automated.



## Theses completed in 2017

### PhD Theses



23.02.2017

**Sven Marcel Schulte**

“Characterization of the microbial methane oxidation for landfill studies by stable isotope analysis”

#### Summary

Next to water, carbon dioxide (CO<sub>2</sub>) and methane (CH<sub>4</sub>) are the most important greenhouse gases with regard to their radiative forcing effect. While the amount of CH<sub>4</sub> in the atmosphere is by two orders of magnitude lower than that of CO<sub>2</sub> its global warming potential is up to 28 times greater than that of CO<sub>2</sub>. The waste and wastewater sector contribute an important share to the total anthropogenic emissions of methane. In this particular case, landfills play an important role because worldwide the major amount of waste is deposited at landfill sites. Emissions of CH<sub>4</sub> at landfill sites arise due to the production of landfill gas mainly from fermentation by obligate anaerobic microorganisms. It can be collected for energy production which allows an economic use and the mitigation of CH<sub>4</sub> emissions to the atmosphere at the same time. Yet, both amount and concentration of CH<sub>4</sub> released from the waste body decrease throughout time. As a consequence for older landfills, the utilization of landfill gas for economic purposes is no longer possible. However, the German legislation demands the treatment of the landfill gas to reduce CH<sub>4</sub> emissions to the atmosphere. An extension or alternative to active gas extraction and treatment are landfill cover layers that serve as methane oxidation layers. On the one hand their advantage is that they represent passive systems and in contrast to active systems need no additional machinery and little maintenance. On the other hand a general problem with these systems is the assessment of the performance of methane oxidation by an appropriate method. One suitable technique for that purpose is stable isotope analysis (SIA). It relies on the preferential consumption of the lighter <sup>12</sup>CH<sub>4</sub> over the heavier <sup>13</sup>CH<sub>4</sub> by methanotrophic bacteria within the cover layer which can be described by the isotopic enrichment factor  $\epsilon$ . This preference results in an isotopic fractionation between the CH<sub>4</sub> produced in the anaerobic zone of the waste body and the partially consumed CH<sub>4</sub> emitted at the landfill's surface. Apart from the isotopic composition of the anaerobic and emitted CH<sub>4</sub>  $\epsilon$  is the crucial parameter for the estimation of the performance of the cover layer in terms of biodegradation. In this work the focus was on the determination of this parameter at different temperatures and for different methanotrophs (type I and II) by using gas chromatography isotope ratio mass spectrometry for SIA. The overall average  $\pm$  standard deviation was  $\epsilon = -0.021 \pm 0.004$  (mixed methanotrophs in topsoil:  $\epsilon = -0.0202 \pm 0.0047$  at 22°C and  $\epsilon = -0.0231 \pm 0.0059$  at 30°C; mixed methanotrophs enrichment culture at 22°C:  $\epsilon = -0.0136 \pm 0.0036$ ; type I enrichment culture:  $\epsilon = -0.0242 \pm 0.0007$  at 22°C and  $\epsilon = -0.0202 \pm 0.0030$  at 30°C; type II enrichment culture:  $\epsilon = -0.0204 \pm 0.0028$  at 22°C;  $\epsilon = -0.0232 \pm 0.0020$  at 30°C). Comparison with the literature revealed similar values for  $\epsilon$ . It was also shown that the high variability and uncertainty of  $\epsilon$  resulted in no statistical difference for either different temperatures or for type I or II methanotrophs. Further statistical analysis revealed that corrections suggested in literature based on a temperature dependency of  $\epsilon$  are of minor relevance when regarding the uncertainty in  $\epsilon$  (in the typical operational range of a cover soil of 10-40°C). Another factor influencing isotopic fractionation is diffusion. In landfills where methane transport to the surface is dominated by diffusion the estimation of

biodegradation by SIA has to be corrected for the former. The isotopic fractionation by diffusion was determined for a potential landfill cover material at 21°C and 30°C and compared with literature values. With an average of  $\epsilon_{\text{diff}} = -0.0212 \pm 0.005$  at 21°C and  $\epsilon_{\text{diff}} = -0.0218 \pm 0.003$  at 30°C it was of the same order of magnitude as the determined isotopic fractionation by methane oxidation and thus within the range of analytical uncertainty of the theoretical value for isotopic fractionation by diffusion of  $\epsilon_{\text{diff}} = -0.0191$ . Having determined these important parameters the biodegradation in a new reactor setup could be investigated by SIA for the study of topsoil as a potential cover layer. Apart from SIA, different methods based on mass balancing and stoichiometry were used and compared by statistical means in terms of correlation, measurement uncertainty, and biodegradation. The results based on SIA for a closed system and for stoichiometric balancing of product ( $\text{CH}_4$ ) and reactant ( $\text{CO}_2$ ) correlated well with the mass balance method. However, highest biodegradation was determined by mass balancing, followed by stoichiometry, and finally SIA that resulted in the lowest estimates, in general. The investigated topsoil proved to be very suitable as a potential cover layer by removing up to 99% of methane for  $\text{CH}_4$  loads of 35 - 65  $\text{gm}^{-2}\text{d}^{-1}$  that are typical in the aftercare phase of landfills. Finally, SIA and the stoichiometric approach were used to trace microbial activity within the reactor system and were able to validate a newly employed technique by thermographic imaging. It was shown that methane consumption and temperature increase -as a cause of high microbial activity- correlated very well. In future studies this will allow investigating the response of cover materials to additional simulated environmental changes such as fissure formation by plant root penetration and the influence of weather conditions such as desiccation or high precipitation. These exemplify events whose influences on the cover layer are not well understood, yet. All in all this work improves the understanding and comparability of techniques to evaluate methane oxidation. Based on this work, further opportunities in terms of forecasting and modelling the behavior of the methane oxidation layer are facilitated.



30.03.2017

**Ratnaningtyas Budhi Lestari**

“Defluoridation of drinking water by hybrid coagulation and filtration process”

## Summary

Fluoride contamination in drinking water can cause severe health problems, namely fluorosis. Defluoridation of drinking water is a practical option to overcome the problem of excessive fluoride in drinking water. Considering that most affected regions are located in less developed countries, it is necessary to find a safe and inexpensive defluoridation technique in order to remove the excess fluoride from drinking water. This study proposes two hybrid methods that have not been investigated with emphasis not only on the fluoride removal but also on the removal of the aluminum residue in the product water. The first step of the hybrid process is based on coagulation and co-precipitation based on Nalgonda technique. Alum ( $\text{Al}_2(\text{SO}_4)_3$ ) is used as coagulant, while lime ( $\text{Ca}(\text{OH})_2$ ) accounts for maintaining the system pH and as precipitation agent. However, the drawback of this process is a significant aluminum level in the product water. A systematic process study has shown that the removal of fluoride occurred very fast. It was best carried out at neutral pH and with an excessive amount of aluminum coagulant. Fluoride ions were adsorbed by precipitated aluminum hydroxide. After certain time the precipitated aluminum hydroxide collided and enmeshed fine particles and later

settled down. It is suggested that the removal reaction follows a sweep mechanism. Furthermore, to decrease fluoride concentration to the desired concentration, the optimum alum dosing was successfully determined. Reduction of fluoride concentration from an initial concentration of 10 mg/L to below 1.5 mg/L was best at an aluminum dosing of 100 mg/L that is corresponding to an  $\text{Al}^{3+}$  to F- molar ratio  $\geq 7$ . Meanwhile, for fluoride with initial concentration of 4 mg/L, an  $\text{Al}^{3+}$  to F- molar ratio  $\geq 4$ , equal to 17 mg/L aluminum, achieved the same purpose. This amount of aluminum is clearly lower than needed in the Nalgonda technique which is 16 to 181 mg/L or treating raw water with fluoride levels of 2 to 8 mg/L. Lower amounts of aluminum are preferred to avoid excess of aluminum residue in the product water and to minimize the sludge formation.

A species diagram shows that pH plays the most important role on the process especially in controlling the quality of product water. The removal of fluoride in raw water from the initial concentration of 10 mg/L to below 1.5 mg/L was achieved in the pH range 6 - 8. At this pH range  $\text{Al}(\text{OH})_3$  has low solubility and easily precipitates. Besides, at this pH the  $\text{pH}_{\text{pzc}}$  of  $\text{Al}(\text{OH})_3$  indicates that the precipitate is neutral to positively charged. In addition, by maintaining the pH on this level, the amount of  $\text{OH}^-$  ions as competing ion to fluoride to occupy  $\text{Al}(\text{OH})_3$  precipitate is also smaller.

As second step in the hybrid process a sand filter has been investigated to deal with excess residue of aluminium in the produce water. The insertion of a sand filter after the coagulation and co-precipitation step is proposed considering that the high level of aluminium in the product water is caused by suspended aluminium that still remained in product water after long term of settling down. In the laboratory scale, sand filtration as part of the hybrid process showed successful removal of aluminium to a concentration that will not lead to a risk for consumer health based on WHO standard (0.2 mg/L). Finally, a hybrid process of coagulation and co-precipitation with membrane ultrafiltration has been investigated. The hybrid process also successfully achieved 90 % reduction of aluminum concentration in the water after the coagulation and co-precipitation step by the UF membrane operation. The aluminum concentration after the hybrid process fulfilled the WHO standard of 0.2 mg/L. In conclusion, the study on the hybrid proces coagulation and co-precipitation with filtration for defluoridation of drinking water has brought a new insight on how to control the coagulation and co-precipitation process. Extension to this work by using additional parameter gives a new opportunity on its development. The investigation has also shown that the hybrids defluoridation process is considerable technique that can be applied as alternatives to the existing one. Investigation to different material of membrane will open a new challenge in its application.



26.04.2017

**Florian Uteschil**

“Drift tube ion mobility spectrometry laser induced fluorescence detection”

### Summary

The coupling of ion mobility spectrometry (IMS) and laser-induced fluorescence spectroscopy (LIF) at atmospheric pressure was realized in this work. The construction of a module based drift tube enables the use of different ionization techniques, as well as the implementation of a laser induced fluorescence detection system and a Faraday plate detector. With the analysis of the ionized dye Rhodamine 6G, the system was characterized. For this, a “homemade” electrospray ionization source was used as ion source. At a distance of approximately 9 mm from the ESI tip to the drift tube entrance, a high desolvation rate of gaseous Rhodamine 6G was observed. The length of the drift tube was 3.2 cm. The drift time resolved laser induced fluorescence analysis of ions at ambient conditions on the example of dye Rhodamine 6G was investigated in a simplified IMS drift cell of open design.

Continuous wave radiation at a wavelength of 447 nm and 462 nm was used for excitation. The maxima of the fluorescence were observed at  $\lambda = 505$  nm. The drift time resolved fluorescence emission was monitored by a spectrograph in combination with a gated intensified charge coupled device.

The dependencies of Rhodamine 6G fluorescence intensities (continuous ion flow) on the electric fields of both the ion gate field and the drift field were determined at the exit of the 32 mm long drift cell. With increasing gate voltage, the signal intensity decreased. With an electrospray flow rate of  $300 \text{ nL min}^{-1}$  and a laser diode with 462 nm used as excitation source, a gate voltages of  $\geq 50 \text{ V}$  was required to block or deflect fluorescent ions of Rhodamine 6G in the drift cell. The increase of the fluorescence intensity was proportional to the electric field strength in the range of  $510 - 638 \text{ V cm}^{-1}$ .

Drift time dependent (pulsed ion flow) LIF analysis was performed under optimized conditions (462 nm, ESI flow rate  $300 \text{ nL min}^{-1}$ , gate voltage 80 V, ICCD integration time:  $100 \mu\text{s}$ ). The drift time spectra produced with both, the Faraday plate detector and the laser induced fluorescence detector showed the same trends. Significantly, smaller FWHM were found with the LIF detector as compared to that found with the electrometer.

Based on the findings of the experiments with Rhodamine 6G, the experimental setup for polycyclic aromatic hydrocarbons (PAH) analysis was designed and developed. A unidirectional flow, closed, heated (up to  $200^\circ \text{C}$ ) drift tube (length: 16.8 cm) ion mobility spectrometer with photoionization source (VUV Kr, 10.0/10.6 eV) was implemented in a pulsed laser induced fluorescence detection system. With this setup, it was possible to investigate the dependence of the fluorescence intensity of selected PAH (continuous ion introduction) on the experimental parameters such as: PID-lamp current, ion gate voltage, electric field strength in the drift tube.

The fluorescence intensity was dependent on the PID lamp current. The rise of both the fluorescence intensity and the continuous ion current with the increase of the PID lamp current was observed. The simultaneous decrease of both the total ion current and the fluorescence intensity with increase of the ion gate voltage was monitored. The LIF signal intensities increased proportional to the drift field strength in the range of  $157 - 265 \text{ V cm}^{-1}$ . The ions generated by photoionization could be transported through the drift region and detected with the laser induced fluorescence monitoring system.



02.06.2017

**Terence Hetzel**

“Characterization of micro liquid chromatography - theoretical performance limits and practical aspects for routine analysis on the example of cytotoxic drugs”

## Summary

Miniaturized liquid chromatography (LC) is ideally suited to reduce the resource consumption without compromising data quality in the analytical laboratory. However, the applicability and handling of miniaturized LC in routine analysis is often being questioned. In addition, only little information on the theoretical performance limits of this separation technique is available. Therefore, the aim of this study was the characterization of micro-liquid chromatography (*micro-LC*) from the theoretical and practical point of view. For the evaluation the development of a fast method for the analysis of antineoplastic drugs from wipe samples using micro-LC-MS/MS was applied. The investigated analytes comprise polar as well as non-polar compounds. In addition, three critical peak pairs were included that either cannot be differentiated by mass spectrometry

or are affected by ion suppression at co-elution. Therefore, a chromatographic separation was mandatory. Micro-LC was used as separation dimension using a column with an inner diameter of 300  $\mu\text{m}$  due to the advantages of reduced resource consumption and high linear velocity leading to increased sample throughput. To achieve a chromatographic separation, a suitable chromatographic phase system was identified by principal component analysis (PCA). Afterwards, the column efficiency was investigated using van Deemter and kinetic plot analysis for isocratic and gradient elution. The results indicate a higher packing quality of the sub-2  $\mu\text{m}$  fully porous particle packed column compared to larger particle diameters of various morphology. In addition, similar values for the reduced plate height were found in micro-LC compared to conventional column inner diameters ( $\geq 2.1$  mm). The investigation of peak capacity also demonstrates the benefit of sub-2  $\mu\text{m}$  fully porous particles for gradient times between 5 s and 5 min. Further optimization of the extraction efficiency, ion source parameters and system design was done to establish the method on basis of micro-LC-MS/MS. This hyphenation technique enables to separate the target analytes within 2.5 min at a flow rate of only 25  $\mu\text{L min}^{-1}$ . Thereby the sample throughput can be increased by a factor of 2 reducing the resource consumption by 98% compared to the previously used conventional LC-MS/MS method. Method validation was accomplished to demonstrate that sensitivity and robustness of the developed method is given in a routine environment. Furthermore, it was shown that the method can be used to analyze real samples from hospital pharmacies in order to verify the reference value of 0.1  $\text{ng cm}^{-2}$  for workplace contamination of antineoplastic drugs. In summary, it was exemplarily demonstrated that micro-LC can successfully replace conventional LC-MS in targeted ultrasensitive MS analysis.



16.11.2017

**Alaa Salma**

“Advanced oxidation of micropollutants in water by photolytic and photocatalytic processes”

### Summary

Photochemical processes and related technologies have been often used in the 20<sup>th</sup> century for the disinfection of drinking water and wastewater (secondary and tertiary sewage effluents). Recently, direct ultraviolet (UV) photolysis, photocatalysis and advanced oxidation procedures have been widely reported as emerging methods for the removal of organic micropollutants from water. Nowadays, based on the progress made in analytical techniques, sensitivity, micropollutants such as pharmaceuticals can be determined down to  $\text{ng L}^{-1}$  scale in the aquatic environment. There is growing interest in the removal of these contaminants from water, particularly driven by the overall public concern about potential toxic effects they might induce in humans and ecosystems.

In this work, the beta-blocker nebivolol has been detected for the first time in effluent samples of 12 wastewater treatment plants (WWTPs) in Germany. The photolytic degradation of nebivolol has been investigated under three different UV sources, namely, UV-C (main emission band at 254 nm), UV-B (main emission band at 312 nm) and UV-A (main emission band at 365 nm) in different matrices: pure water, pure water in the presence of a hydroxyl radical scavenger and in wastewater. During the photodegradation study, no elimination of nebivolol was observed under UV-A radiation. In contrast, nebivolol degradation under UV-B and UV-C radiation followed pseudo first order reaction kinetics, with the highest removal rate under UV-C radiation in pure water ( $k = 7.8 \times 10^{-4} \text{ s}^{-1}$ ). Also the degradation mechanism of nebivolol under the UV-B and UV-C radiation has been studied. Three transformation products (TPs) were identified after UV-B and UV-C photolytic degradation using high resolution mass spectrometry. The TPs are formed by the substitution of the fluorine atom from the

benzopyran ring with a hydroxyl group. The biologically active part of nebivolol is still preserved in the identified TPs even after two hours of irradiation. The matrices' pH plays an important role for the elimination mechanism of the micropollutants in the environment. With regard to photolysis, the different species might have various photolytic degradation pathways, transformation products and kinetics of mechanism-based degradation. In order to demonstrate this, the influence of different pH values (3, 5, 7 and 9) on the reaction kinetics and on the degradation mechanism of ciprofloxacin by direct ultraviolet photolysis (UV-C irradiation) and photocatalysis (TiO<sub>2</sub>/UV-C) has been investigated. During the photolytic and photocatalytic degradation of ciprofloxacin, pseudo-first order kinetics were found with the highest removal rates at pH 9 ( $k_{UV \text{ and } TiO_2/UV} = 4.0 \times 10^{-4} s^{-1}$ ). 18 transformation products have been identified at different pH values (3, 5, 7 and 9). Four transformation products have been detected for the first time, two of the newly proposed structures were supported by the results obtained using deuterated ciprofloxacin.

Photolysis was studied for five further common micropollutants. A corrosion inhibitor (1*H*-benzotriazole) and four pharmaceuticals from different compound classes were included in the study: a  $\beta$ -blocker (metoprolol), an antibiotic (sulfamethoxazole), an anti-inflammatory drug (diclofenac) and an anti-epileptic agent (carbamazepine). The photodegradation was affected by the WWTP effluent matrix. Organic and inorganic substances in wastewater or natural water environments played a dual role of sensitizer and quencher in the photodegradation. In this study, photodegradation rate constants of metoprolol and carbamazepine increased in presence of WWTP effluent matrix, probably due to the presence of photosensitizer compounds. In contrast, diclofenac and sulfamethoxazole showed decreased photodegradation rate constants, due to physical or chemical quenching of the photochemical degradation intermediates by competitors. The matrix also posed non-negligible influences on benzotriazole photodegradation process. Overall, direct photolysis was demonstrated to be relevant for micropollutant abatement from the aquatic environment. However, to promote the photolysis application on a broader scale, it is essential to further understand how this process is affected by the UV sources, micropollutant structures and matrix composition.



06.12.2017

**Michail Dronov**

“Concentration-Gradient-Method for improvement of precision and accuracy of isotope ratio analysis using quadrupole based inductively coupled plasma-mass spectrometry”

## Summary

Isotope ratio analysis makes it possible to approach many analytical problems concerning the identity and provenance of different samples. In recent years the number of published applications on this topic increased greatly since introduction of stable isotope analysis by means of (Laser-Ablation) Multi-Collector-inductively-coupled-plasma mass spectrometer. However, the main disadvantage of these highly precise, so-called MC-ICP-MS, devices are very high investment costs. In this context, this thesis deals with the development of a novel approach to measure and evaluate more precise and accurate isotope ratios using commonly established and widespread quadrupole based ICP-MS (ICP-QMS) as it is possible by means of the common approach. The newly developed method is based on the constant isotope ratio at changed sample concentration. By means of an appropriate measurement setting, the linear relationship can be described by a suitable regression model by means of a mutual plot of signal intensities. As a result, this method requires a modification of the measurement procedure with respect to the classical method. Instead of measuring one well defined concentration, the measurement of a concentration series of the sample has to be performed. The resulting signal

intensities of each isotope are plotted against each other in an x,y-diagram. The different concentrations of the samples correlate by a linear model. The slope of the estimated line is per definition the isotope ratio. Since both data sets are influenced by uncertainties the use of an orthogonal-distance-regression (ODR) model is required. This measurement and evaluation approach is called Concentration-Gradient-Method (CGM). One particularity of this kind of data evaluation is a significant increase of precision and accuracy of the isotope ratio determination performed with ICP-QMS. Moreover, a significant intercept is expressed by linear regression. It could be demonstrated that this intercept is a consequence of constant interference overlapping the isotope signals. Constant values that may cause such interference are signal noise and polyatomic interference of the measuring system as well as isobaric interference of the dilution medium. In the course of the optimization process, it was shown that transient measurements yield more precise results since the accuracy of the model depends on the incoming number of data points. For this reason, a program was written that greatly facilitated the very time-intensive analysis and thus allowed an enormous time-saving. The enhanced precision and accuracy by means of CGM could be demonstrated in comparative measurements using defined isotope standard-reference-solutions, which were measured under the condition of very poor signal-noise-ratios (S/N). A significant improvement of the accuracy could be achieved by means of CGM. In reproducibility test the newly developed CGM achieved a recovery rate of 103 % independent of S/N, while the classical measurement approach showed a deterioration of the recovery rate up to 123 %. In addition, only the use of CGM enabled plausible measurements of the Sulphur isotope ratio even without using the collision/reaction cell of the ICP-QMS in gypsum samples of different origins. The massive interference of  $^{16}\text{O}_2$  on the signals of  $^{32}\text{S}$  were compensated by the intercept of the regression line, so that no further signal treatment was necessary. An further important application of isotope ratio analysis is the radiogenic Sr isotopic ratio determination for the identification of the origin of diverse food products to identify counterfeit products. However, this determination requires a complex sample preparation in the form of Rb separation by means of fractionated ion exchange chromatography to avoid isobaric interferences. Within the scope of this thesis, an alternative sample preparation procedure was developed based on the classical  $\text{SrCO}_3$  precipitation reaction. By this approach a two to five times better separation of Rb and Sr was achieved in mineral water and wine samples than in the standard approach. In combination with the CGM, mineral water and wine samples could be characterised by their Sr isotope ratio depending on their origin and varietal. The here developed cost-efficient and very effective sample preparation procedure increase the importance of isotope analysis by means of the widespread and in the routine analytic established ICP-QMS to a certain extent. Finally, a simple and effective modification of the CGM was developed using common Continuous-Flow-Analysis (CFA) techniques to allow a continuous change of a defined start concentration of a sample. The aim of this modification was a reduction of sample volume and maximisation of cost-efficiency due to minimisation of sample preparation steps that also cause an avoidance of errors or contaminations by the reduction of pipetting steps. The continuous dilution method yielded three times more accurate results of the isotope ratio determination of different standard-reference-solutions with respect to previously described discontinuous CGM. The improvement was mainly caused by avoiding plasma disruption by air bubbles. Interference experiments additionally showed an influence on the precision, due to the decreased S/N, but the accuracy was still not affected (99.8 % WFR). In summary, the approach of continuous dilution is a simple and powerful modification of the here developed CGM.

## Master Theses

### **Jennifer Becker**

“Chromium (VI) elimination in raw water treatment with activated carbon and superparamagnetic iron oxide nanoparticles”  
(at Stadtwerke Düsseldorf, Host Supervisor: Dr. Norbert Konradt)

### **Johanna Buss**

“Chlorine dioxide in wastewater treatment – pollutant degradation and by-product formation”

### **Lokman Coban**

“Development of introduction/separation system for atmospheric pressure ion mobility spectrometry”

### **Matin Funck**

“Sorption Experiments on Carbon Nanomaterials with Reversed Phase Liquid Chromatography”

### **Lotta Hohrenk**

“Development and validation of a suspect-target method for the screening of micropollutants and transformation products in the aquatic environment”

### **Sidra Ilyas**

“Removal and biodegradation of micropollutants (biocides) from Wastewater effluent using biofilm reactors to study the influence of organic carbon in the filter medias”  
(at Aarhus University, Host supervisor: Prof. Kai Bester)

### **Wiebke Kaziur**

“Liquid Phase Microextraction (LPME) optimization for the extraction of Aromatic Amines in urine”

### **Lukas Lesmeister**

“Optimising the potential of Sand Filters and Retention soil filters for the attenuation of organic Micropollutants”  
(at TZW Karlsruhe, Host supervisors: Dr. Karsten Nödler, Dr. Marco Scheurer)

### **Christian Timon Orths**

“Influence of organic ligands on kinetics and OH-yield of the Fenton-Reaction”

### **Jan Peters**

“Electrochemical characterization and degradation of organic compounds with Boron-doped Diamond Electrodes”

### **Nan Sun**

“Determination of the H<sub>2</sub>O<sub>2</sub>-activation of persulfate in homogeneous and heterogeneous Fenton/Fenton-like systems and investigation of its reaction mechanism”  
(at Harbin Institute of Technology, Host Supervisor: Prof. Jun Ma)

### **Maryam Taghavi**

“In-vitro metabolites profiling of Escherichia coli and Pseudomonas aeruginosa biofilm by use of Thin Film Microextraction”

### **Abd Tavazoei**

“Effect of pressure on analysis of aromatic compound with High Field Asymmetric Waveform Ion Mobility Spectrometry (FAIMS) regarding two difference ionization sources”



**Jens Terhalle**

“Chlorine dioxide in wastewater treatment –potential and by-products-“

**He Wang**

“Degradation of PAHs by aerobic and/or anaerobic bacteria in the presence or absence of biosurfactants”

(at Helmholtz Zentrum München, Host Supervisor: Dr. Sviatlana Marozava; Home Supervisor: Maik Jochmann)

**Vanessa Wirzberger**

“Detection of microplastics in Arctic deep-sea sediments by uFTIR spectroscopy”

(at Alfred-Wegener-Institute on Helgoland, Host supervisor: Dr. Gunnar Gerdts)

**Shengwei Xian**

“Quantification of long chain fatty acid by GCxGC/qMS and filamentous bacteria by real-time PCR for avoiding bulking and foaming based on a pre-flotation or precipitation system”

(Home Supervisor: Prof. Martin Denecke, Host Supervisor: Maik Jochmann)

## Bachelor Theses

**Michelle Diederichs**

„Bestimmung von 17 $\alpha$ -Ethinylestradiol, 17 $\beta$ -Estradiol und Estron in Oberflächenwasserproben mittels LC-MS/MS und Festphasenanreicherung“

(at IWW Water Centre, Host Supervisor: Dr. Peter Balsaa)

**Lena Enns**

„Ramanspektroskopie als Detektionsmethode für Chipbasierte Trenntechniken“

(at University of Leipzig, Host Supervisor: Prof. Dr. Detlev Belder)

**Dominik Hennecke**

„Strategien zur Vermeidung und Verminderung von Sulfat in Werksabwasser“

(Home Supervisor: Dr. Maik Jochmann, Host Supervisor: Dr. Uwe Müller)

**Mischa Jütte**

„Abbau von Bisphenol S mit Hilfe der Fenton-Reaktion“

**Felix Niemann**

“Optimization and development of PAL SPME Arrow based methods for GC-IRMS analysis of BTEXs and LMW PAHs in groundwater”

**Mona Rahmatullah**

“On the complete biodegradation of dissolved organic carbon in a drinking water treatment system”

(Host Supervisor: Dr. Maik Jochmann; Home Supervisor: Prof. Rainer Meckenstock)

**Philipp Witzel**

„Einsatz von Passivsammlern zur Beprobung von Oberflächengewässern auf Pestizide“

(at LANUV NRW, Host Supervisor: Dr. Harald Rahm)

# Publications

## Peer-reviewed Journals

1. H. L. Wiegand, C. T. Orths, K. Kerpen, H. V. Lutze, T. C. Schmidt:  
Investigation of the iron-peroxo-complex in the Fenton reaction: Kinetic indication, decay kinetics and hydroxyl radical yields  
*Environ. Sci. Technol.* **51** (2017), 14321–14329  
DOI: 10.1021/acs.est.7b03706
2. G. Renner, J. Schram, T. C. Schmidt:  
A New Chemometric Approach for Automatic Identification of Microplastics from Environmental Compartments Based on FT-IR Spectroscopy  
*Anal. Chem.* **89** (2017) 12045–12053  
DOI: 10.1021/acs.analchem.7b02472
3. F. Itzel, L. Gehrmann, H. Bielak, P. Ebersbach, A. Boergers, H. Herbst, C. Maus, A. Simon, E. Dopp, M. Hammers-Wirtz, T. C. Schmidt, J. Tuerk:  
Investigation of full-scale ozonation at a municipal wastewater treatment plant using a toxicity-based evaluation concept  
*J. Toxicol. Environ. Health A* **80** (2017), 1242-1258  
DOI: 10.1080/15287394.2017.1369663
4. M. Schulte, M. A. Jochmann, T. Gehrke, A. Thom, T. Ricken, M. Denecke, T. C. Schmidt:  
Characterization of methane oxidation in a simulated landfill cover system by comparing molecular and stable isotope mass balances  
*Waste Managem.* **69** (2017), 281-288  
DOI: 10.1016/j.wasman.2017.07.032
5. X. L. Osorio Barajas, M. A. Jochmann, T. Hüffer, B. Schilling, T. C. Schmidt:  
Sorbent Material Characterization Using In-tube Extraction Needles as Inverse Gas Chromatography Column  
*J. Sep. Sci.* **40** (2017), 2390-2397  
DOI: 10.1002/jssc.201601198
6. J. Leonhardt, J. Haun, T. C. Schmidt, T. Teutenberg:  
The Benefits of Coupling Miniaturized Comprehensive 2D LC with Hybrid High-Resolution Mass Spectrometry  
*LC-GC Europe* (2017), 264-270
7. S. Willach, H. V. Lutze, K. Eckey, K. Löppenberg, M. Lüling, J. Terhalle, J.-B. Wolbert, M. A. Jochmann, U. Karst, T. C. Schmidt:  
Degradation of sulfamethoxazole using ozone and chlorine dioxide - compound-specific stable isotope analysis, transformation product analysis and mechanistic aspects  
*Water Res.* **122** (2017), 280-289  
DOI: 10.1016/j.watres.2017.06.001
8. A. A. Deeb, S. Stephan, O. J. Schmitz, T. C. Schmidt:  
Suspect screening of micropollutants and their transformation products in advanced wastewater treatment  
*Sci. Tot. Environ.* **601-602** (2017), 1247-1253  
DOI: 10.1016/j.scitotenv.2017.05.271
9. M. Nestola, T. C. Schmidt:  
Determination of mineral oil aromatic hydrocarbons (MOAH) in edible oils and fats by online liquid chromatography–gas chromatography–flame ionization detection (LC-GC-FID) –

- Evaluation of automated removal strategies for biogenic olefins  
*J. Chromatogr. A* **1505** (2017), 69-76  
 DOI: 10.1016/j.chroma.2017.05.035
10. P. Kosse, M. Lübken, T. C. Schmidt, M. Wichern:  
 Quantification of nitrous oxide in wastewater based on salt-induced stripping  
*Sci. Tot. Environ.* **601-602** (2017), 83-88  
 DOI: 10.1016/j.scitotenv.2017.05.053
  11. A. Fischbacher, C. von Sonntag, T. C. Schmidt:  
 Hydroxyl radical yields in the Fenton process under various pH, ligand concentrations and hydrogen Peroxide/Fe(II) ratios  
*Chemosphere* **182** (2017), 738-744  
 DOI: 10.1016/j.chemosphere.2017.05.039
  12. X. L. Osorio Barajas, T. Hüffer, P. Mettig, B. Schilling, M. A. Jochmann, T. C. Schmidt:  
 Investigation of carbon based-nanomaterials as sorbents for headspace in-tube extraction of polycyclic aromatic hydrocarbons  
*Anal. Bioanal. Chem.* **409** (2017), 3861-3870
  13. F. Metzelder, T. C. Schmidt:  
 Environmental conditions influencing sorption of inorganic anions to multi-walled carbon nanotubes studied by column chromatography  
*Environ. Sci. Technol.* **51** (2017), 4928-4935  
 DOI 10.1021/acs.est.6b06386
  14. M. Nachev, M. A. Jochmann, F. Walter, J. B. Wolbert, S. M. Schulte, T. C. Schmidt, B. Sures:  
 Understanding trophic interactions in host-parasite associations using stable isotopes of carbon and nitrogen  
*Parasites & Vectors* **10:90** (2017), 1-9  
 DOI 10.1186/s13071-017-2030-y
  15. A. Salma, H. V. Lutze, T. C. Schmidt, J. Tuerk:  
 Photolytic degradation of the  $\beta$ -blocker Nebivolol in aqueous solution  
*Water Res.* **116** (2017), 211-219  
 DOI 10.1016/j.watres.2017.03.029
  16. T. Hetzel, C. Blaesing, M. Jaeger, T. Teutenberg, T. C Schmidt:  
 Characterization of peak capacity of microbore liquid chromatography columns using gradient kinetic plots  
*J. Chromatogr. A* **1485** (2017), 62–69  
 DOI 10.1016/j.chroma.2017.01.018
  17. A. R. Ribeiro, T. C. Schmidt:  
 Determination of acid dissociation constants (pKa) of cephalosporin antibiotics: computational and experimental approaches  
*Chemosphere* **169** (2017), 524-533  
 DOI 10.1016/j.chemosphere.2016.11.097
  18. S. M. Seyed Khademi, M. Tabrizchi, U. Telgheder, Y. Valadbeigi, V. Ilbeigi:  
 Determination of MTBE in drinking water using corona discharge ion mobility spectrometry  
*Intern. J. Ion Mobility Spectrom.* **20** (2017), 15-21  
 DOI:10.1007/s12127-017-0217-8
  19. S. Waclawek, H. V. Lutze, K. Grübel, V. V.T. Padil, M. Černík, D. D. Dionysiou:  
 Chemistry of persulfates in water and wastewater treatment: A review  
*Chem. Eng. J.* **330** (2017), 44–62  
 DOI:10.1016/j.cej.2017.07.132

20. A. Kuklya, T. Reinecke, F. Uteschil, K. Kerpen, S. Zimmermann, U. Telgheder:  
X-ray ionization differential ion mobility spectrometry  
*Talanta* **162** (2017), 159–166  
DOI:10.1016/j.talanta.2016.10.024
21. F. Uteschil, A. Kuklya, K. Kerpen, R. Marks, U. Telgheder:  
Time-of-flight ion mobility spectrometry in combination with laser-induced fluorescence  
detection system  
*Anal. Bioanal. Chem.* **409** (2017), 6279–6286  
DOI:10.1007/s00216-017-0584-3
22. R. Rodriguez-Maecker, E. Vyhmeister, S. Meisen, A. Rosales, A. Kuklya, U. Telgheder:  
Identification of terpenes and essential oils by means of Static Headspace Gas Chromatography  
- Ion Mobility Spectrometry  
*Anal. Bioanal. Chem.* **409** (2017), 6595-603  
DOI:10.1007/s00216-017-0613-2
23. X. Dong, M. A. Jochmann, M. Elsner, A. H. Meyer, L. E. Bäcker, M. Rahmatullah, D. Schunk, G.  
Lens, R. U. Meckenstock:  
Monitoring Microbial Mineralization Using Reverse Stable Isotope Labeling Analysis by Mid-  
Infrared Laser Spectroscopy  
*Environ. Sci. Technol.* **51** (2017), 11876-11883  
DOI:10.1021/acs.est.7b02909

## Invited Book Chapters

- A. Fischbacher, H. V. Lutze, T. C. Schmidt:  
Ozone/H<sub>2</sub>O<sub>2</sub> and ozone/UV processes, in:  
M. Stefan (Ed.):  
Advanced Oxidation Processes for Water Treatment: Fundamentals and Applications  
IWA Publishing, 2017, pp 163-194  
ISBN 9781780407180
- G. Renner, T. C. Schmidt, J. Schram:  
Characterization and Quantification of Microplastics by Infrared Spectroscopy  
in:  
T. A. P. Rocha-Santos, A. C. Duarte (Ed.): Characterization and Analysis of Microplastics,  
*Comprehensive Analytical Chemistry* **75** (ISBN: 9780444638984)  
Elsevier, Amsterdam, 2017, pp 67-118  
DOI: 10.1016/bs.coac.2016.10.006

## Books

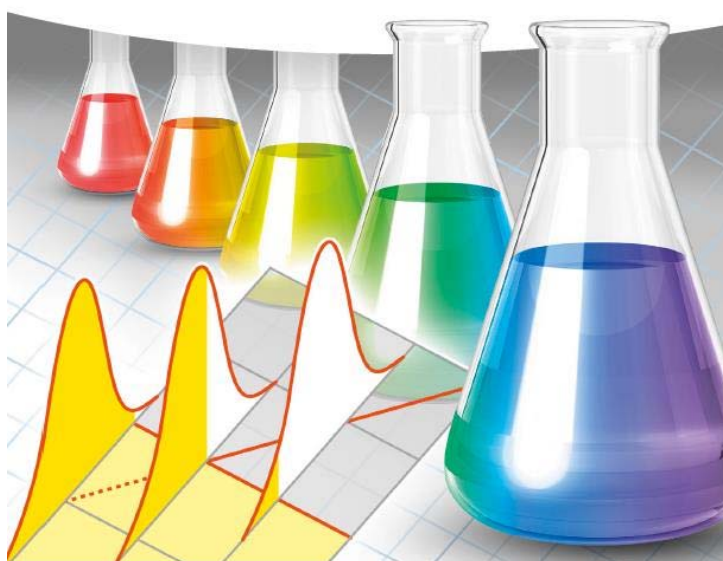
WILEY-VCH

Georg Schwedt, Torsten C. Schmidt  
und Oliver J. Schmitz

# Analytische Chemie

Grundlagen, Methoden und Praxis

Dritte, überarbeitete und aktualisierte Auflage



Die verschiedenen Bücher von Prof. Georg Schwedt zur analytischen Chemie gelten bereits als Klassiker. Dazu trägt das Konzept der Verbindung von Grundlagen, Methoden und Praxis bei, aber auch die hochwertige graphische Gestaltung. Abbildungen aus „dem Schwedt“ finden sich sicherlich in allen Vorlesungen zur analytischen Chemie wieder. Das fundamentale Lehrbuch „Analytische Chemie“ ist im Dezember 2016 nun in der dritten Auflage bei Wiley-VCH erschienen. Alle relevanten Aspekte der Analytischen Chemie werden in diesem Lehrbuch, das gleichzeitig auch als Referenz für Praktiker dient, umfassend und klar auf den Punkt gebracht. Ab dieser Auflage wird das Lehrbuch nicht mehr von Prof. Schwedt alleine verantwortet, sondern unter Beteiligung der beiden Professoren für analytische Chemie an der Universität Duisburg-Essen, Oliver Schmitz und Torsten Schmidt. Die beiden haben in dieser Auflage Kapitel inhaltlich neu gestaltet, für die infolge der methodischen und technologischen Entwicklungen eine Aktualisierung erforderlich war. So wurden von Oliver Schmitz hauptsächlich die Kapitel über Qualitätssicherung, Massenspektrometrie, chromatographische Trennverfahren und elektrophoretische Trennverfahren überarbeitet. Torsten Schmidt hat vor allem das Kapitel zur Probenvorbereitung aktualisiert und das Grundlagenkapitel ergänzt.

ISBN: 978-3-527-34082-8

Die verschiedenen Bücher von Prof. Georg Schwedt zur analytischen Chemie gelten bereits als Klassiker. Dazu trägt das Konzept der Verbindung von Grundlagen, Methoden und Praxis bei, aber auch die hochwertige graphische Gestaltung. Abbildungen aus „dem Schwedt“ finden sich sicherlich in allen Vorlesungen zur analytischen Chemie wieder. Das fundamentale Lehrbuch „Analytische Chemie“ ist im Dezember 2016 nun in der dritten Auflage bei Wiley-VCH erschienen. Alle relevanten Aspekte der Analytischen Chemie werden in diesem Lehrbuch, das gleichzeitig auch als Referenz für Praktiker dient, umfassend und klar auf den Punkt gebracht. Ab dieser Auflage wird das Lehrbuch nicht mehr von Prof. Schwedt alleine verantwortet, sondern unter Beteiligung der beiden Professoren für analytische Chemie an der Universität Duisburg-Essen, Oliver Schmitz und Torsten Schmidt. Die beiden haben in dieser Auflage Kapitel inhaltlich neu gestaltet, für die infolge der

# Conferences and Meetings

## Organization

### **7th Spring School "Industrial Analytical Chemistry", Essen, March 12 to 24, 2017**

For the seventh time the spring school "Industrial Analytical Chemistry" was organized by Prof. Schmidt, Prof. Schmitz and the Industry forum of GDCh under the supervision of Dr. Arlt held at the University of Duisburg – Essen. The aim of this 10-day block course was to acquaint students in chemistry master's programs with analytical methods and issues that are of outstanding importance in industrial analysis. Speakers from the chemical-pharmaceutical as well as from the manufacturing industry introduced in-depth analytical topics from the fields of methodology, process analytics, quality assurance and management as well as entrepreneurial action, social skills and successful application to industrial companies and reported on their daily work. The participants trained their social skills while cooking together and during social gathering in Unperfekthaus in Essen.

The event was completed by two excursions. The group visited thyssenkrupp Steel Europe AG in Duisburg, where they had a factory tour of one of Europe's largest steel mills combined with a visit of the central analytics. There they had a guided tour to get an insight into the work of an analytical chemist in the steel industry. The second excursion went to Chemiepark Marl, Evonik. There, a factory tour and a guided tour through the analytical department were organized.





**Annual Meeting of the Water Chemistry Society, Donaueschingen, May 22 to 24, 2017**

(for more details check <http://www.wasserchemische-gesellschaft.de/de/wasser-2017>)

Prof. Schmidt as president of the German Water Chemistry Society, Claudia Gehrke as secretary of the society and Dr. Lutze and his team of IAC members organized for the fifth time the annual meeting of the society, this year in Donaueschingen, a small town hosting the symbolic spring of the Danube river at the south end of the Black Forest. More than 290 scientists gathered and discussed for three days 29 oral and 90 poster presentations. Dr.-Ing. Maximilian Huber, TU Munich was awarded with the Willy-Hager-Preis for his PhD thesis "Development and Evaluation of an Assessment Method for Decentralized Stormwater Treatment Systems for Runoff from Traffic Areas". The PhD prize of the Water Chemistry Society was awarded to Dr. Allan Philippe, University Koblenz-Landau, for his thesis "Hydrodynamic Chromatography for Studying Interactions between Colloids and Dissolved Organic Matter in the Environment". Dr.-Ing. Aki Sebastian Ruhl, TU Berlin, received the society award for his outstanding research and contributions to the society. Finally, Dr. Hans-Jürgen Pluta of the Federal Environmental Agency, Berlin, got the society's badge of honour for his contributions in its board and in particular his engagement in establishing funding for a standardization expert within the Water Chemistry Society.



## Invited Lectures

T. C. Schmidt, M. A. Jochmann, L. Zhang, D. M. Kujawinski, J.-B. Wolbert, E. Federherr, S. Willach, D. Köster, N. Stojanovic:

Was uns stabile Isotopen über die Welt erzählen: Von grauer Vorzeit bis zu aktuellen Dopingsündern  
GDCh-Lecture, Aalen, December 19, 2017

T. C. Schmidt:

What shall we measure tomorrow? Needs and trends in future water analysis  
Yazd, 2<sup>nd</sup> International and 2th National Conference on Environmental Health and Sustainable Development, November 28, 2017

T. C. Schmidt, F. Metzelder, T. Hüffer, S. Endo, Y. Qian, E. Bi :

Towards a mechanistic understanding of sorption processes in water on a molecular level  
Graduate program Urban Water Interfaces Summer School 2017, Kremmen, September 05, 2017

T. C. Schmidt:

By failing to prepare, we are preparing to fail - Trends in der Probenvorbereitung für organische Spurenstoffe

Merck, Darmstadt, August 21, 2017

T. C. Schmidt:

What shall we measure tomorrow? Needs and trends in future water analysis  
Colloquium Engler-Bunte-Institut, Karlsruhe, July 14, 2017

T. C. Schmidt:

What shall we measure tomorrow? Needs and trends in future water analysis  
TUM Water Lecture Series, Munich, June 07, 2017

T. C. Schmidt:

Trends in Sample Preparation: Smaller, faster, more reliable, more sensitive  
Dow Chemicals, Stade, May 11, 2017

T. C. Schmidt, P. Balsaa, A. Simon, U. Borchers, V. Hinnenkamp:

Non-Target-Analytik und toxikologische Charakterisierung, zukunftsweisende Kombination für eine effiziente Rohwasserüberwachung

50. Essener Tagung für Wasser- und Abfallwirtschaft, March 23, 2017



## Institute Colloquium

04.12.2017	<p><b>Dr. Axel Boddenberg, Saltigo</b></p> <p>Analytischer Rundum-Service unter den Herausforderungen des Custom Manufacturing</p>
20.11.2017	<p><b>Prof. Dr. Michael Rychlik, TU Munich</b></p> <p>Quantifizierung und Tracing von bioaktiven Lebensmittelinhaltsstoffen durch Stabile Isotope</p>
23.10.2017	<p><b>Dr. Tobias Licha, University of Goettingen</b></p> <p>New perspectives arising from the application of organic molecules as indicators in the aquatic environment: How to teach an old dog a new trick</p>
03.07.2017	<p><b>Prof. Dr. Nils Schebb, University of Wuppertal</b></p> <p>Quantifizierung von Eicosanoiden und anderen Oxylipinen in biologischen Proben mittel LC-MS: Analytische Herausforderungen und Lösungsstrategien</p>
21.06.2017	<p><b>GDCh-Kolloquium</b></p> <p><b>Prof. Dr. Ralf Zimmermann, University of Rostock</b></p> <p>Combustion, crude oil, coffee roasting and aerosols: Analysis of complex materials and thermal processes by photo ionisation mass spectrometry (PIMS)</p>
29.05.2017	<p><b>Prof. Dr. Jens Brockmeyer, University of Stuttgart</b></p> <p>What makes an allergen? Characterization of heterogeneity and gastrointestinal metabolism of food allergens using mass spectrometry</p>
15.05.2017	<p><b>Prof. Dr. Erich Leitner, TU Graz</b></p> <p>Geruch und Fehlgeruch in Lebensmitteln und Bedarfsgegenständen</p>
24.04.2017	<p><b>Dr. Jörg Flock, ThyssenKrupp Steel</b></p> <p>Analytische Chemie für den Werkstoff Stahl - Dienstleistungen für Entwicklung und Produktion</p>
25.01.2017	<p><b>GDCh-Kolloquium</b></p> <p><b>Frank Matysik, U Regensburg</b></p> <p>Elektrochemie – flexible Partnerin im Ensemble instrumenteller Analyseverfahren</p>
23.01.2017	<p><b>Alejandro Cifuentes, U Madrid</b></p> <p>Omics Technologies, Food and Health: Foodomics</p>

## Teaching

At IAC we are involved in teaching mostly in the Bachelor and Master program “Water Science” that is a unique science-based curriculum with a focus on chemistry, analytics and microbiology (see details at <https://www.uni-due.de/water-science/>). All courses are also optional for students in the Bachelor and Master program “Chemistry”, some are also offered as elective courses for chemistry students studying towards a teacher’s degree and for students of the related Master programs “Environmental Toxicology” (offered in the faculty of biology) and “Management and Technology of Water and Wastewater – MTW3” (offered in the faculty of engineering).

### Summer term

Lecture and Tutorial “Water Chemistry” (B.Sc. Water Science, in German)

Lecture “Water – The Lecture” (B.Sc. Water Science, in German)

Excursion to Wastewater Treatment Plant Duisburg-Kaßlerfeld (B.Sc. Water Science, in German)

Lecture and Tutorial “Oxidative Processes” (M.Sc. Water Science, in English)

Lecture and Tutorial “Stable Isotope Analysis” (M.Sc. Water Science, in English)

Laboratory Practical “Stable Isotope Analysis” (M.Sc. Water Science, in English)

Lecture and Tutorial “Quality Management” (M.Sc. Water Science, in English)

Laboratory Practical “Environmental Analytics” (M.Sc. Environmental Toxicology, in English)

Excursion to the Alfred-Wegener-Institute and Climate House, Bremerhaven (M.Sc. Water Science, in English)

Excursion to the Institute for Reference Materials and Measurements, Geel (B) (M.Sc. Water Science, in English)

### Winter term

Lecture and Tutorial “Water Analysis” (B.Sc. Water Science, in German)

Laboratory Practical “Analytical Chemistry” (B.Sc. Water Science, in German)

Laboratory Practical “Water Chemistry and Analysis” (B.Sc. Water Science, in German)

Lecture and Tutorial “Water Chemistry” (M.Sc. Water Science and Environmental Toxicology, in English)

Lecture and Tutorial “Chemometrics and Statistics” (M.Sc. Water Science, in English)

Individual Practical Projects “Analytical Chemistry” (M.Sc. Water Science, in English)

Excursion to the Drinking Water Treatment Plant incl. Ultrafiltration, Roetgen (M.Sc. Water Science, in English)

## Outreach

### **UNIKids, 08.03.2017 „Wasser baut Brücken, aber hat es auch ein Gedächtnis?“**

600 children between the ages of 8-12 years got a blue T-shirt matching the theme and looked a bit like drops of water. Excited, they followed the lecture by Torsten Schmidt, Ursula Telgheder and Claudia Ullrich on the subject of water for an hour under the title "Water builds bridges, but does it also have a memory?". With illustrative examples, film clips and impressive experiments, the properties of water and their effects on humans and the environment were imparted. The children were particularly impressed by the water bridge, which arises between two vessels when a very high voltage is applied, and the flashes, which are clearly visible when the lecture hall is darkened. In the second part, the children learned why there is so much hocus-pocus in the water community, e.g. the alleged water memory. A simple example showed them how to expose nonsense with scientific methodology. The children had a lot of fun with the lecture, but the team was also enthusiastic about the active listeners.



### **S.U.N.I SommerUni in Natur- & Ingenieurwissenschaften, 24.08.2017 „Der Blick ins Wasser“**

S.U.N.I. is a unique platform for young people to support their study choice orientation in the natural and engineering sciences. By attending impressive experiments and presentations, the participants have the opportunity to experience the university's everyday life in special events and get to know the fields of work in science and technology in order to receive support for their study and career development process. The event “A glance at the water” includes sampling procedure at a small lake with subsequent analysis and water treatment of the samples in the lab. The participants learn fundamental strategies of chemical analysis and treatment techniques.



### **Trial course of Studies (Probestudium Chemie), 12.12.2017 “Water Science – Zur Struktur des Wassermoleküls und den Folgen “**

In addition to the early studies, pupils from upper classes have the opportunity to gain in-depth insights into the study areas of natural sciences, engineering and (economic) computer science, to speak with teachers, to explore the facilities of the University of Duisburg-Essen. Within the program of the Faculty of Chemistry, Dr. Ursula Telgheder and Claudia Ullrich presented an experimental lecture about the special properties of water. At first glance, the water molecule has a very simple structure. And yet it is something very special. Physicochemical: water is different as all structurally related compounds. That's why life, as we know it, makes it possible. Meaning: Water is involved in all biological and very many non-biological processes in our environment. It is an ideal solvent for many components, therefore the chemistry of and in aqueous systems is particularly diverse. The lecture went on from the special properties of water to water purification illustrated with simple experiments.

## Miscellaneous

### Births



Jarik Zscheppank, born on June 24, 2017



Ida Ananias, born on October 16, 2017

### Weddings



Gerd und Monika Fischer, October 24, 2017