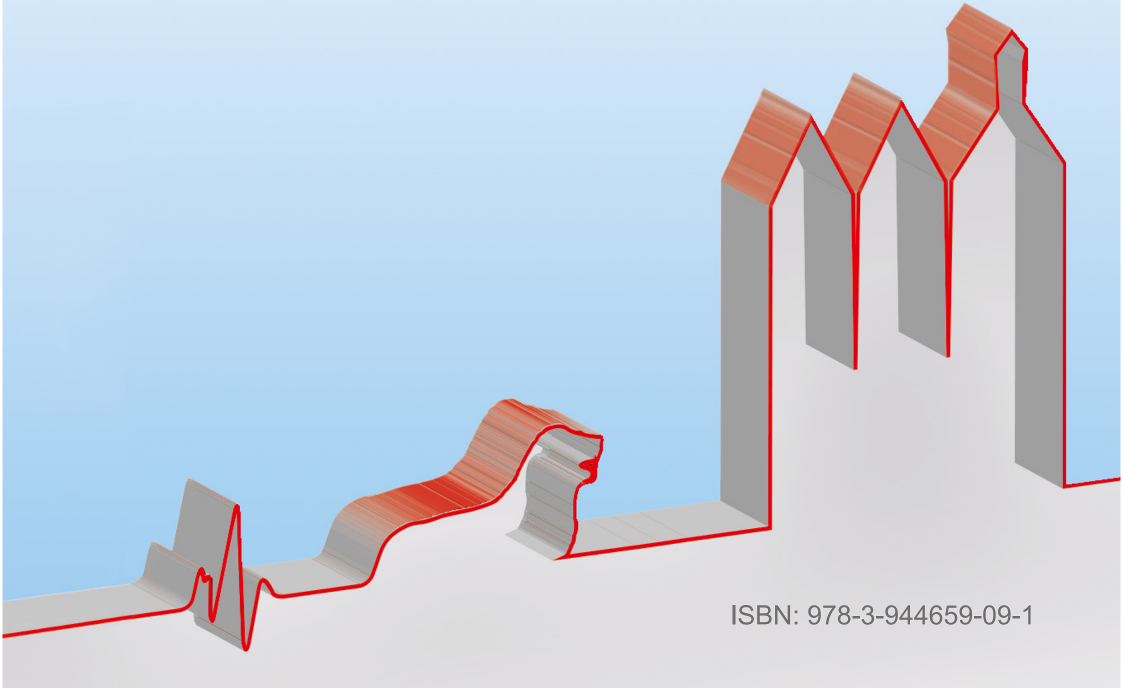
Book of Abstracts

Die 25.
Kalorimetrietage

Braunschweig, Germany
31 May – 02 June 2023



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Die 25. Kalorimetrietage

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Book of Abstracts

Die 25. Kalorimetrietage

Braunschweig, Germany
31 May – 02 June 2023

Imprint

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- Gesellschaft für Thermische Analyse e. V.
- Physikalisch-Technische Bundesanstalt

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- Dr. J. Seidel. Freiberg
- Prof. Dr. med. D. Singer, Hamburg
- Prof. Dr. G. Wolf, Freiberg

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Bundesanstalt
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Content

Greetings

Die 25. Kalorimetrietage
31 May to 02 June 2023

Dear friends and colleagues,

the National Metrology Institute of Germany (Physikalisch-Technische Bundesanstalt, PTB) and the German Society for Thermal Analysis (GEFTA) are very pleased to welcome you to the 25th Calorimetry Meeting from 31 May to 02 June 2023.

We are very excited that in time for the jubilee the conference is back in an ideal setting at PTB. Unfortunately, the 25th Calorimetry Meeting will be held for the last time at PTB in Braunschweig after ten years and five events, respectively, and will therefore be held at another location in the future.*

This conference is special because it gives biologists, chemists, engineers, physicians, and physicists the opportunity to present the importance of their work to the scientific community and at the same time learn about exciting developments outside their discipline. This will give you the opportunity to think about how your research might help in areas that you have not previously considered. We hope you can take advantage of this unique opportunity to learn more about other fields of calorimetry and thermal analysis.

Thanks to the generous support of many instrument manufacturers on site, the participants can be offered the possibility to inform themselves about instruments as well as about the latest trends in the instrument development. In addition, several representatives of the manufacturers are once again actively enriching our scientific program with their high-grade contributions.

We are also pleased that personal contact, getting to know each other and exchanging scientific ideas at the end of a day of events - in the context of a pleasant social gathering - is once again possible and may also contribute to personal enrichment.

We are very anxious to be able to present interesting scientific topics and latest findings, so that the meeting will be an enrichment for all participants. We wish everyone a good time and look forward to interesting discussions, a lively exchange, and inspiring conversations. We would also like to take this opportunity to thank the lecturers for their presentations. We will do our best to make this event an unforgettable success once again.

Stefan Sarge
Chair of the Organising Committee

Dirk Walter
Chair of GEFTA

** For the very generous organization of the always successful events in the past years, we would like to express our most sincere thanks and great appreciation to the PTB, the local organizers under the leadership of Dr. Stefan Sarge and all participants for the realization and the successful outcome.*

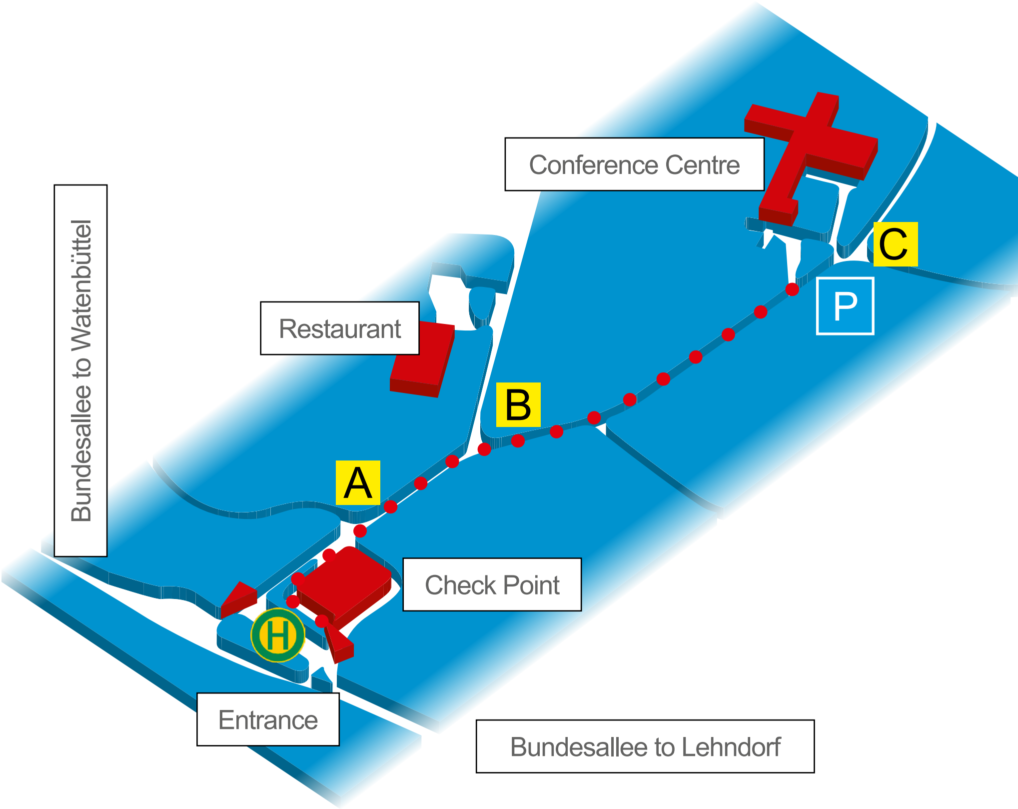
Dirk Walter

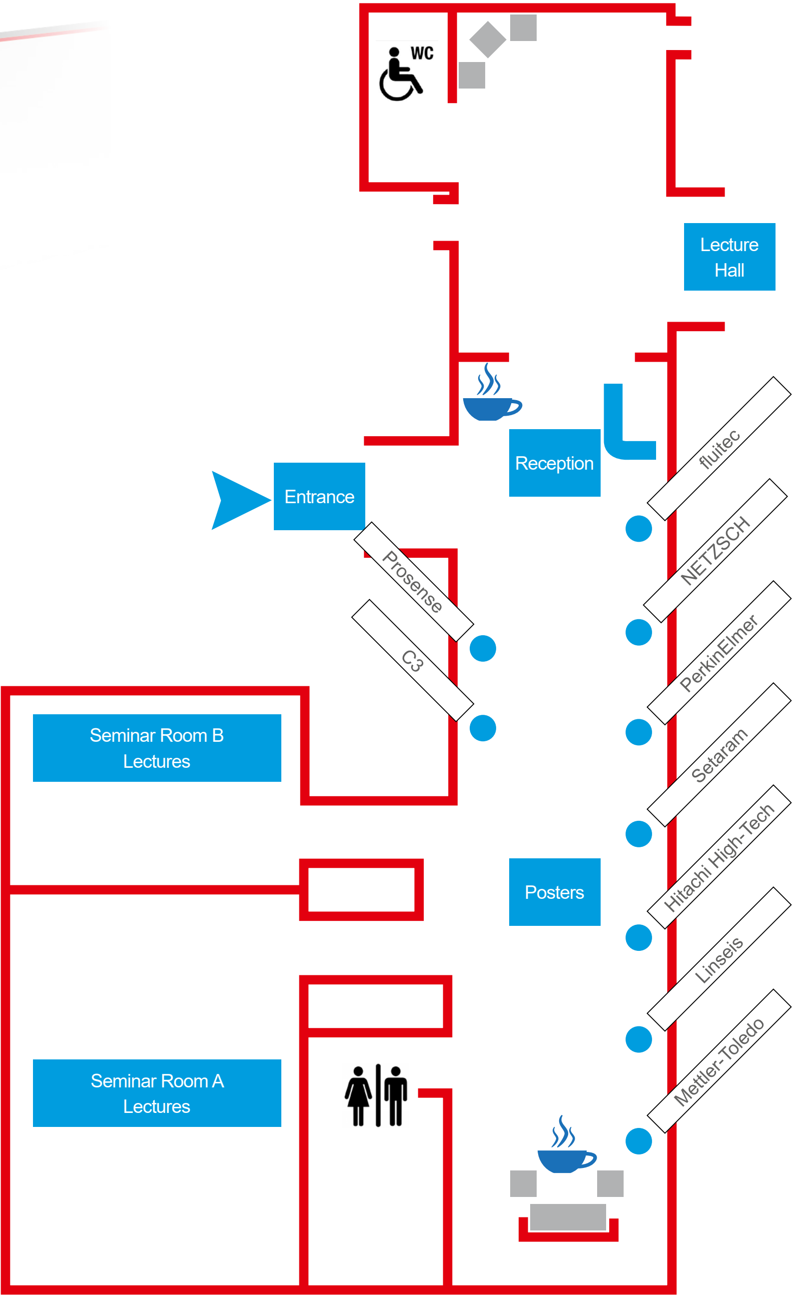
General Information and Locations

The plenary lectures and some parts of the progress reports will be given in our seminar room A. The other parts of the progress reports will be presented in seminar room B. The manufacturers' exhibition and the poster presentation are located in the foyer.

On Thursday, a buffet lunch will be served during GEFTA's annual meeting in the foyer. On the other days, PTB's canteen is happy to welcome you.

Car parking is possible on the PTB campus. A special parking area is reserved and marked for the participants of the conference. Bus 433 and bus 461 connect PTB and the city every 30 minutes.




WC
Lecture Hall
Reception
Entrance
fluitec
NETZSCH
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Linseis
Mettler-Toledo
Prosense
C3
Seminar Room B Lectures
Posters
Seminar Room A Lectures

433 Völkenrode - PTB - Hondelage

Völkenrode	6:02			18:29		
...		every 30 min				
PTB	6:10		18:10	18:37	19:07	19:37
Bundesallee	6:12		18:12	18:39	19:09	19:39
Paracelsusstraße	6:13		18:13	18:40	19:10	19:40
Pfleidererstraße	6:14		18:14	18:41	19:11	19:41
von Pawelsches Holz	6:15		18:15	18:42	19:12	19:42
Saarbrückener Straße	6:16		18:16	18:43	19:13	19:43
Saarplatz	6:17		18:17	18:44	19:14	19:44
Ottweilerstraße	6:18		18:18	18:45	19:15	19:45
Hildesheimer Straße	6:20		18:20	18:47	19:17	19:47
Rudolfplatz	6:22		18:22	18:49	19:19	19:49
Amalienplatz	6:24		18:24	18:52	19:22	19:52
Maschplatz	6:25		18:25	18:54	19:24	19:54
Hamburger Straße	6:27		18:27	18:55	19:25	19:55
Pockelsstraße	6:28		18:28	18:57	19:27	19:57
Hans-Sommer-Straße	6:30		18:30	18:58	19:28	19:58
Richard-Strauß-Weg	6:31		18:31	19:00	19:30	20:00
Beethovenstraße	6:32		18:32	19:01	19:31	20:01
Bahnhof Gliesmarode	6:33		18:33	19:02	19:32	20:02
...						
Hondelage	6:56		18:56	19:26	19:56	20:26

433 Hondelage - PTB - Völkenrode

Hondelage	5:36	6:06		8:05	8:35		18:35
...			every 30 min			every 30 min	
Bahnhof Gliesmarode	5:55	6:25		8:25	8:55		18:55
Beethovenstraße	5:56	6:27		8:27	8:57		18:57
Richard-Strauß-Weg	5:57	6:28		8:28	8:58		18:58
Bültenweg	5:59	6:30		8:30	9:00		19:00
Pockelsstraße	6:00	6:31		8:31	9:01		19:01
Hamburger Straße	6:02	6:34		8:34	9:04		19:04
Maschplatz	6:03	6:35		8:35	9:05		19:05
Amalienplatz	6:05	6:37		8:37	9:07		19:07
Rudolfplatz	6:06	6:39		8:39	9:09		19:09
Hildesheimer Straße	6:08	6:41		8:41	9:11		19:11
Ottweilerstraße	6:09	6:43		8:43	9:13		19:13
Saarplatz	6:10	6:44		8:44	9:14		19:14
Saarbrückener Straße	6:11	6:45		8:45	9:15		19:15
von Pawelsches Holz	6:12	6:46		8:46	9:16		19:16
Pfleidererstraße	6:13	6:47		8:47	9:17		19:17
Paracelsusstraße	6:14	6:48		8:48	9:18		19:18
Bundesallee	6:15	6:49		8:49	9:19		19:19
PTB	6:17	6:51		8:51	9:21		19:21
...							
Völkenrode		6:58		8:58			

461 PTB - Kanzlerfeld - Hauptbahnhof

PTB	6:43	7:08	7:43	every 30 min	18:43
Bundesallee	6:45	7:10	7:45		18:45
Paracelsusstraße	6:46	7:11	7:46		18:46
Pfleidererstraße	6:47	7:12	7:47		18:47
von Pawelsches Holz	6:48	7:13	7:48		18:48
Saarbrückener Straße	6:49	7:14	7:49		18:49
Saarplatz	6:50	7:15	7:50		18:50
Ottweilerstraße	6:51	7:16	7:51		18:51
Hildesheimer Straße	6:53	7:18	7:53		18:53
Rudolfplatz	6:55	7:20	7:55		18:55
Kälberwiese	6:56	7:26	7:56		18:56
Maienstraße	6:57	7:27	7:57		18:57
Madamenweg	6:58	7:28	7:58		18:58
Johannes-Selenka-Platz	6:59	7:29	7:59		18:59
Cyriaksring	7:01	7:31	8:01		19:01
Luisenstraße	7:02	7:32	8:02		19:02
Europaplatz	7:02	7:32	8:02		19:02
Am Wassertor	7:04	7:34	8:04		19:04
John-F.-Kennedy-Platz	7:06	7:36	8:06		19:06
Campestraße	7:07	7:37	8:07		19:07
Braunschweig Hauptbahnhof	7:10	7:40	8:10		19:10

461 Hauptbahnhof - Kanzlerfeld - PTB

Braunschweig Hauptbahnhof		6:10	every 30 min	18:10
Campestraße		6:11		18:11
John-F.-Kennedy-Platz		6:13		18:13
Friedrich-Wilhelm-Platz		6:15		18:15
Europaplatz		6:16		18:16
Luisenstraße		6:17		18:17
Cyriaksring		6:19		18:19
Johannes-Selenka-Platz		6:20		18:20
Madamenweg		6:21		18:21
Maienstraße		6:22		18:22
Kälberwiese		6:23		18:23
Rudolfplatz		6:24		18:24
Hildesheimer Straße		6:26		18:26
Ottweilerstraße		6:28		18:28
Saarplatz		6:29		18:29
Saarbrückener Straße		6:30		18:30
von Pawelsches Holz		6:31		18:31
Pfleidererstraße		6:32		18:32
Paracelsusstraße	5:32	6:33		18:33
Bundesallee	5:33	6:34		18:34
PTB	5:35	6:36		18:36

Conference Programme

Wednesday, 31 May 2023

Room A

	Introduction (Chair: D. Walter)
13:00	*J. Hornig, Head of Department 'Gas Flow'* Welcome address
	Hydrogen Storage (Chair: D. Walter)
13:15	*M. Heere (Braunschweig)* Sector coupling on a MW Scale or what to do with 500 kg of ‘green‘ H_2 a day?
14:00	*F. Mertens (Freiberg)* Limitations and Opportunities in Solid State Hydrogen Storage
14:45	Instrument presentation / Poster presentation / Coffee break
	Process Safety / Biocalorimetry (Chair: H. Heerklotz)
15:15	*M. Gödde (Ludwigshafen)* Assessment of Polymerizing Substances and SAPT
16:00	*A. Bartelt (München)* Biological principles of energy metabolism
16:45	*G. Makhatadze (Troy, NY)* Pressure Perturbation Calorimetry and the Effects of High Hydrostatic Pressure on the Conformational Equilibrium of Biomacromolecules
17:30	Transfer to city
19:00	Informal meeting at Rheinische Republik

Conference Programme

Thursday, 1st June 2023

Room A

	Hydrogen Storage (Chair: F. Mertens)
09:00	*R. Siewert, S.P. Verevkin, K. Müller (Rostock)* Thermochemistry of Liquid Organic Hydrogen Carrier: How Calorimetry sets the foundations for development of the technology
09:45	*T. Klapoetke (München)* New Possibilities for Hydrogen Transport
10:30	Instrument presentation / Poster presentation / Coffee break
	Hydrogen Storage (Chair: F. Mertens)
11:00	*M. Dornheim (Nottingham)* Characterisation of Reactive Hydride Composites as potential Hydrogen Storage Materials
11:45	Annual meeting of GEFTA members and lunch
	Chemical Process Safety (Chair: S. Neuenfeld)
13:10	*D.P. Rütti, F.L. Steinemann, M. Moser, A.G. Georg, D.M. Meier (Winterthur)* Simulate chemical reactions and derive kinetics from milli-scale flow calorimetry
13:30	*J. Nowottny, D. Bender, J. Franke (Frankfurt a. M.)* Heat-/Pressure-Accumulation Test - Adiabatic Calorimetry for Assessing Process Safety Relevant Parameters such as ADT$_{24h}$ and T_{exo}
13:50	*A. Zentel, D. Ohlig, J. Sartorius, J. Schröder, R.J. Blanchard, M. Gödde (Ludwigshafen)* Evaluation of apparatus effects on calorimetric data as a basis for safe operating limits in thermal process safety
14:10	*Z. Gulsoy Serif, M. Hii, D. Ohlig, M. Goedde, K. Hellgardt (London)* Flow Calorimetry: A Methodology for Determining Kinetics of Exothermic Reactions
14:30	Instrument presentation / Poster presentation / Coffee break

Conference Programme

Thursday, 1st June 2023

Room B

	New Methods (Chair: E. Füglein)
13:10	*J. Lerchner, L.H. Saccani, K.C. Bícego, G.S. Garcia, M.T. Oliveira, F. Mertens (Freiberg)* Calorimetric measurements on small tissue samples
13:30	*M. Zipf, A. Narymany Shandy, J. Manara, J. Hartmann (Würzburg)* Development of an Optical Differential Scanning Calorimetry Setup for Determining the Specific Heat Capacity at High Temperatures
13:50	*J.E.K. Schawe (Nänikon)* An Advanced Heat Flux DSC Operated in the Power Compensation Mode
14:10	*M.J. Kriegel, A. Walnsch, M. Löffler, O. Fabrichnaya, A. Leineweber (Freiberg)* Utilizing high-temperature calorimetry for a thermodynamic assessment based on the CalPhaD approach

Conference Programme

Thursday, 1st June 2023

Room A

	Thermodynamics / Kinetics (Chair: M. Gödde)
15:00	*J.E.K. Schawe (Nänikon)* On Fictive and Real Kinetics of the Glass Transition
15:20	*C.B.M. Groß (Mainz)* Determination of the specific heat capacity of melts using a tabletop DSC
15:40	Short interruption
15:45	*K. Holtappels (Berlin)* Safety aspects for hydrogen technologies
16:30	Transfer to city
18:00	Visit to Herzog Anton August Museum
19:00	Conference dinner at restaurant Al Duomo

Room B

	Thermodynamics (Chair: J. Lerchner)
15:00	*K. Burkmann, F. Habermann, J. Kraus, B. Störr, J. Seidel, R. Gumeniuk, K. Bohmhammel, J. Kortus, F. Mertens (Freiberg)* Thermodynamic Study of Yttrium Boranate - $Y(BH_4)_3$
15:20	*F. Habermann, A. Wirth, K. Burkmann, B. Störr, J. Seidel, K. Bohmhammel, R. Gumeniuk, F. Mertens (Freiberg)* Determination and Assessment of Thermodynamic Data for the Earth Alkali Metal Alanates $Mg(AlH_4)_2$, $Ca(AlH_4)_2$ and $Sr(AlH_4)_2$

Conference Programme

Friday, 2nd June 2023

Room A

	Biocalorimetry (Chair: H. Bunjes)
09:00	*W. Frieß (München)* Thermal Analysis in Formulation of Protein Drugs
09:45	*T. Rades (Copenhagen)* Investigation of pharmaceutically relevant properties of amorphous drug system with differential scanning calorimetry
10:30	Coffee Break
	Biocalorimetry (Chair: D. Singer)
11:00	*C. Fricke, T. Klee, S. Richter, S. Paufler, K. Stahr, T. Maskow (Landau)* Micro(bio-)calorimetry - Instrument and Method Development for Microbiological Drinking Water Analysis
11:20	*T. Maskow, H.L. Duong, S. Paufler, D. Schlosser (Leipzig)* Characterisation of fungal lignocellulose utilisation strategies using biocalorimetry
11:40	*J. Lerchner, L.H. Saccani, F. Mertens, K.C. Bícego (Freiberg)* Heat production of muscle tissue of tegu lizards (*Salvator merianae*)
12:00	Short interruption
	Energy Storage (Chair: D. Walter)
12:05	*M. Felderhoff (Mülheim/Ruhr)* Hydride-based thermal energy storage
12:50	*D. Walter* Farewell address

Conference Programme

Friday, 2nd June 2023

Room B

Batteries (Chair: A. Leineweber)

M. Heere (Braunschweig)

11:00 Characterization of a novel Mg-conductor using Neutron Methods

K. Böhm, P. Ganninger, F. Heeger, T. Markus, D. Henriques (Mannheim)

11:20 Possibilities and methods of Calorimetry to investigate the heat generation of silicon anodes for Li-Ion-Batteries

M. Steeb, S.M. Sarge, W. Tegethoff, J. Köhler (Braunschweig)

11:40 Thermal investigation of inhomogenities in aged batteries using an isoperibolic calorimeter

Social Programme

Wednesday, 31 May 2023

7 pm	Informal meeting at Rheinische Republik (food & drinks not included in registration fee) *Neue Straße 10, 38100 Braunschweig*

Thursday, 1st June 2023

6 pm	Guided tour through the famous Herzog Anton Ulrich-Museum *Museumstraße 1, 38100 Braunschweig*
7 pm	Conference dinner at Al Duomo *Ruhfäutchenplatz 1, 38100 Braunschweig*

List of Lectures

Bartelt, Alexander (München)
Biological principles of energy metabolism

Böhm, Kevin (Mannheim)
Possibilities and methods of Calorimetry to investigate the heat generation of silicon anodes for Li-Ion-Batteries
(K. Böhm, P. Ganninger, F. Heeger, T. Markus, D. Henriques)

Burkmann, Konrad (Freiberg)
Thermodynamic Study of Yttrium Boranate - $Y(BH_4)_3$
(K. Burkmann, F. Habermann, J. Kraus, B. Störr, J. Seidel, R. Gumeniuk, K. Bohmhammel, J. Kortus, F. Mertens)

Dornheim, Martin (Nottingham)
Characterisation of Reactive Hydride Composites as potential Hydrogen Storage Materials

Felderhoff, Michael (Mülheim/Ruhr)
Hydride-based thermal energy storage

Fricke, Christian (Landau)
Micro(bio-)calorimetry - Instrument and Method Development for Microbiological Drinking Water Analysis
(C. Fricke, T. Klee, S. Richter, S. Paufler, K. Stahr, T. Maskow)

Frieß, Wolfgang (München)
Thermal Analysis in Formulation of Protein Drugs

Gödde, Markus (Ludwigshafen)
Assessment of Polymerizing Substances and SAPT

Groß, Christoph B. M. (Mainz)
Determination of the specific heat capacity of melts using a tabletop DSC

Gulsoy Serif, Zeynep (London)
Flow Calorimetry: A Methodology for Determining Kinetics of Exothermic Reactions
(Z. Gulsoy Serif, M. Hii, D. Ohlig, M. Goedde, K. Hellgardt)

List of Lectures

Habermann, Franziska (Freiberg)
Determination and Assessment of Thermodynamic Data for the Earth Alkali Metal Alanates $Mg(AlH_4)_2$, $Ca(AlH_4)_2$ and $Sr(AlH_4)_2$
(F. Habermann, A. Wirth, K. Burkmann, B. Störr, J. Seidel, K. Bohmhammel, R. Gumeniuk, F. Mertens)

Heere, Michael (Braunschweig)
Sector coupling on a MW Scale or what to do with 500 kg of 'green' H_2 a day?

Heere, Michael (Braunschweig)
Characterization of a novel Mg-conductor using Neutron Methods

Holtappels, Kai (Berlin)
Safety aspects for hydrogen technologies

Klapoetke, Thomas M. (München)
New Possibilities for Hydrogen Transport

Kriegel, Mario J. (Freiberg)
Utilizing high-temperature calorimetry for a thermodynamic assessment based on the CalPhaD approach
(M.J. Kriegel, A. Walnsch, M. Löffler, O. Fabrichnaya, A. Leineweber)

Lerchner, Johannes (Freiberg)
Calorimetric measurements on small tissue samples
(J. Lerchner, L.H. Saccani, K.C. Bícego, G.S. Garcia, M.T. Oliveira, F. Mertens)

Lerchner, Johannes (Freiberg)
Heat production of muscle tissue of tegu lizards (*Salvator merianae*)
(J. Lerchner, L.H. Saccani, F. Mertens, K.C. Bícego)

Makhatadze, George (Troy, NY)
Pressure Perturbation Calorimetry and the Effects of High Hydrostatic Pressure on the Conformational Equilibrium of Biomacromolecules

Maskow, Thomas (Leipzig)
Characterisation of fungal lignocellulose utilisation strategies using biocalorimetry
(T. Maskow, H.L. Duong, S. Paufler, D. Schlosser)

Mertens, Florian (Freiberg)
Limitations and Opportunities in Solid State Hydrogen Storage

List of Lectures

Müller, Karsten (Rostock)

Thermochemistry of Liquid Organic Hydrogen Carrier: How Calorimetry sets the foundations for development of the technology

(R. Siewert, S.P. Verevkin, K. Müller)

Narymany Shandy, Amir (Würzburg)

Development of an Optical Differential Scanning Calorimetry Setup for Determining the Specific Heat Capacity at High Temperatures

(M. Zipf, A. Narymany Shandy, J. Manara, J. Hartmann)

Nowottny, Jonas (Frankfurt a. M.)

Heat-/Pressure-Accumulation Test - Adiabatic Calorimetry for Assessing Process Safety Relevant Parameters such as ADT_{24h} and T_{exo}

(J. Nowottny, D. Bender, J. Franke)

Rades, Thomas (Copenhagen)

Investigation of pharmaceutically relevant properties of amorphous drug system with differential scanning calorimetry

Rütti, David (Winterthur)

Simulate chemical reactions and derive kinetics from milli-scale flow calorimetry

(D.P. Rütti, F.L. Steinemann, M. Moser, A.G. Georg, D.M. Meier)

Schawe, Jürgen (Nänikon)

An Advanced Heat Flux DSC Operated in the Power Compensation Mode

Schawe, Jürgen (Nänikon)

On Fictive and Real Kinetics of the Glass Transition

Steeb, Michael (Braunschweig)

Thermal investigation of inhomogenities in aged batteries using an isoperibolic calorimeter

(M. Steeb, S.M. Sarge, W. Tegethoff, J. Köhler)

Zentel, Adrian/Ohlig, Dominik (Ludwigshafen)

Evaluation of apparatus effects on calorimetric data as a basis for safe operating limits in thermal process safety

(A. Zentel, D. Ohlig, J. Sartorius, J. Schröder, R.J. Blanchard, M. Gödde)

Abstracts of Lectures

Biological principles of energy metabolism

Alexander Bartelt

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I will discuss mammalian energy metabolism, which is classically considered as a balance of energy intake, absorption, storage, and expenditure. Nowadays, the genetic makeup of humans exposed to environments rich in high caloric, ultra-processed foods has led to a rise in obesity, as every second person in Germany is overweight. In my presentation. I will highlight how we use indirect calorimetry to study biological principles of energy expenditure in preclinical animal models, including the molecular basis of adaptive thermogenesis and exercise.

Kevin Böhm

Possibilities and methods of Calorimetry to investigate the heat generation of silicon anodes for Li-Ion-Batteries

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The utilization of silicon-based anodes presents a promising solution to address the increasing demand for high energy density lithium-ion batteries. However, a major challenge associated with this type of anode is its volume expansion, which can reach up to 400%, leading to rapid capacity deterioration of the battery. To mitigate this issue, one strategy is to develop anodes that combine carbon and silicon nanoparticles, which has been shown to enhance the cycling stability of the anode. Despite its crucial role in ensuring safety, the thermal characteristics of such anodes remain poorly understood and require further investigation.

The calorimetry offers a variety of methods to tackle this lack of information. The decomposition of battery materials and the investigation of the thermal runaway allow the determination of the critical temperatures and the developed heat during this process. Moreover, the heat generation during charging and discharging and the cell performance depending on temperature can be determined without a destruction of the cells.

In this work, a comparison of crystalline and amorphous silicon anodes is conducted. Our inhouse battery production enables the manufacturing of 2032 coin cells consisting of a GF/A separator (260 µm), EC/DEC (50:50, 1M $LiPF_6$) electrolyte, Si/C- and Li-electrodes. We can customize the silicon structure, the coating thickness, additives and particle size to create a variety of testing scenarios.

In this study, a DSC-like method using a multi-module calorimeter from NETZSCH (Germany), equipped with a coin-cell module, was employed to measure heat flow in-situ during the formation and cycling of silicon-based anodes. Furthermore, changes in the signal resulting from the aging of the anodes were determined.

The goal of this research is to investigate the material properties of heat generation during formation and cycling. Additionally, the study aims to identify any differences between crystalline and amorphous starting materials in terms of their SEI-formation, cyclical or thermal stability. The data generated from this study is used to illustrate the effect of the anode structure on the thermal performance and stability of silicon-based Li-Ion batteries.

Kevin Böhm (cont.)

This research is part of the BMBF-NanoMatFutur research group Kems4Bats, which combines established and novel experimental methods to comprehend heat and gas evolution in terms of materials properties. (Visit www.kems4bats.de for more information on the research group.)

Konrad Burkmann (cont.)

Thermodynamic Study of Yttrium Boranate - $Y(BH_4)_3$

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The storage of hydrogen is a necessary step to achieve the transition of the german energy sector from fossil fuels to renewable energy sources [1]. Transition metal boranates may contribute to reach this goal. Yttrium boranate, $Y(BH_4)_3$, is such a candidate. Several investigations concerning the decomposition of this boranate can be found in the literature, but there is no agreement on the final decomposition products [2]. In addition, no investigations concerning the thermodynamic data of the compound exist, despite their necessity for the detail understanding of the decomposition reactions. Therefore, we conducted a calorimetric study to provide these data and employed them in thermodynamic equilibrium calculations to study the decomposition and the rehydrogenation behaviour of Yttrium boranate.

Because there are several problems by using a wet chemical route to produce $Y(BH_4)_3$, we applied a metathesis synthesis by ball milling of YCl_3 with three equivalents of $LiBH_4$ adapted from [3]. The obtained mixture of $Y(BH_4)_3$ with three equivalents of LiCl was used for all further investigations.

The heat capacity of the compound was measured between 2 K and 300 K using a Quantum Design Physical Property Measurements System and between 280 K and 380 K using a Setaram DSC-111. The Debye temperature and absolute entropy at 298.15 K were determined from the low temperature measurements. TG-DSC-MS measurements and Hess law were employed to determine the enthalpy of formation at 298.15 K, which is comparable to the respective value estimated from DFT calculations [4].

In addition, there is no consent in the literature on the final decomposition products. Therefore, thermodynamic equilibrium calculations were used to shed light on the thermodynamic favoured decomposition

Konrad Burkmann (cont.)

reactions. An evaluation of possible decomposition reactions to various products (YH_2, YH_3, B, H_2, YB_2, YB_4, YB_6) was carried using data form our own experiments, own DFT calculations and the literature.

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Martin Dornheim

Characterisation of Reactive Hydride Composites as potential Hydrogen Storage Materials

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Hydride-based thermal energy storage

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Reversible thermochemical reactions are suitable for storing thermal energy due to their high energy density compared to sensible or latent heat storage. In thermochemical heat storage, a thermochemical substance is converted into two other components with the addition of heat. Both can be stored separately. Recombination of the components restores the original thermochemical substance, releasing the same amount of heat that was previously stored.

For continuous power generation with solar thermal power plants and for the use of waste heat from industrial processes, the storage of heat at a temperature level between 400 and 800 °C is essential. High-temperature metal hydrides offer high heat storage capacities in this temperature range. Based on Mg and Ca compounds, these hydrides are in principle low-cost materials with excellent cycling stability. Since high-temperature metal hydrides can be used both as hydrogen storage and as heat storage, they are an ideal link to combine hydrogen and heat applications in different sectors.

Over the years, we have shown the use of high temperature metal hydrides in several demonstration projects. In our current project we have demonstrated the functionality of this storage materials in a technically relevant size of 350 kg MgH_2 storage material (250 kWh_{th}).

Christian Fricke

Micro(bio-)calorimetry - Instrument and Method Development for Microbiological Drinking Water Analysis

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Drinking water is one of the most valuable resources on our planet and forms the fundament for a healthy life. Lack of hygiene and poor maintenance threaten the cleanliness and quality of drinking water, especially from a microbiological point of view. Pathogenic microorganisms like coliform bacteria, Legionella species and faecal germs pose an immediate threat to our health. For more than 100 years, microbiological drinking water analysis has been based on the simple but time-consuming enumeration of colony-forming units on agar plates. A defined sample volume is applied directly to a solid culture medium (0.5 to 1 mL), or a pre-enrichment step is performed using membrane filtration (50 to 100 mL) and the culture is then incubated. The analytical procedure is precisely prescribed by various ISO standards and requires between 2 (*P. aeruginosa*) and 10 days (*L. pneumophila*), depending on the pathogen.

Although there are already many applications of calorimetric detection of pathogens, these are often limited to bacteriological studies of body fluids [1,2]. Interestingly, applications for microcalorimetry in the context of detecting microbial contaminations in drinking water are rare [3]. In addition, the microcalorimeters currently available are not specifically designed for microbiological analyses or are limited to the use of conventional cultivation techniques, which are defined in the ISO standards.

In order to overcome the current instrument limitations and application gaps in calorimetric analysis of drinking water samples, this work is based on three main research issues: (i) the systematic investigation of conventional cultivation techniques in the context of calorimetric detection of microorganisms, (ii) the evaluation of selective culture media for calorimetric detection of *Legionella pneumophila*, and

Christian Fricke (cont.)

(iii) the development and testing of an early-engineer micro(bio-)calorimeter, specifically designed for microbiological analysis.

Our results demonstrate that (i) conventional cultivation techniques are transferable to calorimetric detection [4], although commercial microcalorimeters have some limitations, (ii) target bacteria-oriented calorimetric detection via selective media can be a promising approach in drinking water analysis, and (iii) the novel micro(bio-)calorimeter has been successfully tested on reference strains and cooling tower samples.

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Wolfgang Frieß

Thermal Analysis in Formulation of Protein Drugs

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Protein drugs, specifically monoclonal antibodies (mAbs), are at the forefront of modern therapy. Their potential is demonstrated by the fact that 5 of the 10 best selling drugs worldwide are mAbs. All of them are given parenterally as aqueous solutions. Correspondingly they are either formulated as aqueous solutions or come as lyophilisates which are reconstituted prior to application. Proteins show inherently low stability in solution; the instability pathways can be of chemical, colloidal or conformational nature.

DSC can be used to characterize the conformational stability of proteins, to some extent at domain level. The application for mAbs is however limited due to a) the typically irreversible character of the unfolding process and b) the high melting temperature (T_m) well above the intended storage temperature, mostly refrigerated. Still, in a few cases a correlation between storage stability and T_m has been concluded. In formulation development DSC is frequently flanked or substituted by other analytical methods with nDSF probably being the most frequently used. nDSF utilizes the intrinsic protein fluorescence or the fluorescence of an extrinsic dye which can bind to hydrophobic sites upon heating the sample. It requires only few microliters. Furthermore, light scattering techniques are used to complement as both unfolding and aggregation can be detected. New instruments combine fluorescence and light scattering in well plate or capillary stacks format. This allows high throughput screening in early lead candidate selection and in formulation development. A case study of the use of the thermal analysis for formulation development is presented.

In the context of protein formulation development DSC is also used to analyze the glass transition temperature of the maximally freeze concentrated solution (T_g'). T_g' reflects a critical temperature during lyophilization which should not be exceeded during primary drying in order to avoid collapse. We used DSC to analyze the T_g' of pure protein solutions using highly concentrated sucrose mixtures. In various cases, collapse temperature (T_c) analysis by freeze-drying microscopy has to be used for further understanding of the formulation behavior in the course of primary drying. Furthermore the T_g' is of importance in case of long term frozen storage of bulk drug substance. Thus, thermal analysis is of high importance in early development of protein drugs enabling both high throughput screening and deeper scientific characterization.

Markus Gödde

Assessment of Polymerizing Substances and Self Accelerating Polymerization Temperature (SAPT)

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Christoph B. M. Groß

Determination of the specific heat capacity of melts using a tabletop DSC

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The specific heat capacity of solids can be easily determined by DSC following a standardised procedure [1]. First, the instrument is calibrated for heat flow detection by recording the background-corrected heat flow signal resulting from applying a constant temperature ramp to a reference sample of well-known heat capacity. Second, the heat-flow calibration is used to determine the specific heat capacity of a sample from the background-corrected heat-flow curve derived by applying the same temperature ramp on a test specimen of known mass.

Differences in the thermophysical and geometrical properties of sample and reference sample, such as thermal conductivity, thermal contact resistivity and sample geometry, affect the response of the DSC instrument. To overcome these issues, isotherms are required before and after the heating ramp that allow for mathematical corrections of small interferences.

Melting of samples results in a displacement of the sample in the crucible including changes in sample geometry as well as thermal contact resistivities. These changes often result in apparent measurement artifacts which cannot be corrected by standardised methods.

A method is presented that allows for the correction of measurement artifacts due to sample melting which meets the following targets:

- The approach must result in reproducible results.
- The approach must not require further information about sample properties.
- The experimental effort should not exceed the effort for the standard procedure.
- Incorporation into commercial DSC software should be easily possible.

[1] DIN 51007:2019-04, Thermal analysis - Differential thermal analysis (DTA) and differential scanning calorimetry (DSC) - General Principles

Zeynep Gulsoy Serif

Flow Calorimetry: A Methodology for Determining Kinetics of Exothermic Reactions

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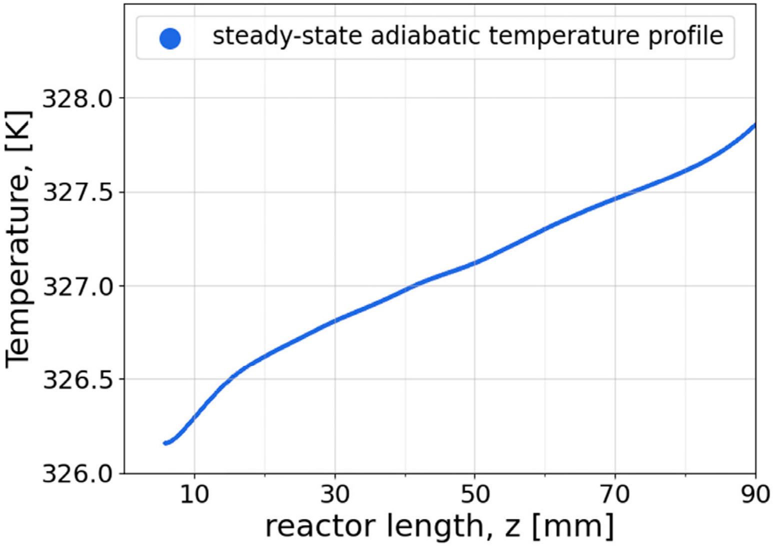
The kinetics of exothermic reactions are an important aspect of chemical engineering, as they determine the rate at which heat is released during the reaction. Industrial processes should always prioritise safety, particularly for fast and highly exothermic reactions containing toxic reactants as these pose significant risks. Hence, accurately measuring the kinetics and heat release can play a vital role in understanding and controlling the risks involved, whilst also leading to improved process efficiency.

Due to a number of unique features of continuous flow reactors, flow calorimetry can provide an intrinsically safer way to study the kinetics of exothermic reactions, particularly when spatial temperature distributions are measured under steady-state conditions. By monitoring the temperature as a function of reactor length, and fitting the data to appropriate kinetic models, kinetic information like the activation energy, pre-exponential factor, and rate constant of the reaction can be determined. Furthermore, real-time monitoring of process conditions and control of potential hotspots via time-dependent spatially resolved temperature profile is another significant strength of flow calorimetry.

In this work, an optical isoperibolic flow calorimeter, designed and built at Imperial College London, was used to validate the methodology for kinetic studies. Hydrolysis of acetic anhydride reaction was performed as a model reaction in flow calorimetry and the collected data was evaluated by kinetic modelling to extract the activation energy and pre-exponential factor. The graph below displays an experimental adiabatic temperature profile as a function of reactor length. After the heat loss to the environment was corrected, steady-state adiabatic temperature profiles this graph was obtained. Steady-state adiabatic temperature profiles were then evaluated using kinetic models. In this presentation, we will explain our setup and how we conducted the case study, along with the comparison of our results with literature.

Zeynep Gulsoy Serif (cont.)



Franziska Habermann

Determination and Assessment of Thermodynamic Data for the Earth Alkali Metal Alanates $Mg(AlH_4)_2$, $Ca(AlH_4)_2$ and $Sr(AlH_4)_2$

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The suitability of complex hydrides such as alanates for solid-state hydrogen storage applications, especially regarding the transition to a renewable energy-based economy, is being widely discussed. Although the alanates of the earth alkali metals Mg, Ca and Sr were already investigated to some extent in that regard, hardly any reliable thermodynamic data exists [1].

For instance, until now no heat capacity functions and absolute entropies have been reported. In order to measure the heat capacity functions of $Mg(AlH_4)_2$, $Ca(AlH_4)_2$ and $Sr(AlH_4)_2$ in the temperature range from 2 K to 360 K, the alanates had first to be synthesised as they are not commercially available. Since the synthesis of the pure compounds proved to be challenging because of their metastability, mixtures of the composition $M(AlH_4)_2 + 2\ M'Cl$ (M = Mg, Ca, Sr; M' = Li, Na) were used instead. Those mixtures were prepared by ball milling via metathesis reactions. The heat capacities of the alanates themselves were then obtained by subtracting the ones of the by-product from those measured.

Heat capacity measurements from 2 K to 298 K were carried out on a Physical Property Measurement System (PPMS, Quantum Design, USA). The measurement was performed using the heat capacity option based on a relaxation technique. By means of a DSC-111 (SETARAM) the heat capacity between 283 K and 360 K was measured employing a C_p-by-step technique.

The absolute entropies of the alanates were derived from the respective measured heat capacity functions.

While theoretically computed enthalpies of formation have been reported for $Mg(AlH_4)_2$ [2] and $Ca(AlH_4)_2$ [3], no value exists for $Sr(AlH_4)_2$. $Mg(AlH_4)_2$ is the only one of the three alanates for which a enthalpy of formation was derived from the dehydrogenation enthalpy in the literature [4]. Nevertheless, as the heat capacity function of $Mg(AlH_4)_2$ was not known then, the temperature dependence of the enthalpy of formation could not have been taken into account and therefore the published value has to be regarded as an estimate. To assess the values reported in the

Franziska Habermann (cont.)

literature and to determine one for $Sr(AlH_4)_2$, we computed the enthalpies of formation of the earth alkali metal alanates using their heat capacity functions and dehydrogenation enthalpies obtained by integrating the corresponding heat effects determined by DSC measurements.

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Michael Heere

Characterization of a novel Mg-conductor using Neutron Methods

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Neutrons are a unique probe for non-destructive structural studies of energy materials, especially for future investigation and development of highly conductive solid state Mg electrolyte, neutrons are one of the main requirements for successful post-Li battery research. Recently, a new compound synthesized from γ-$Mg(BH_4)_2$ and ethylenediamine ($C_2H_8N_2$, abbreviation "en") was reported to have an exceptionally high magnesium ion conductivity of up to 6 x 10^{-5} S cm^{-1} at 70 °C in the solid state. In our work, the structure of this new compound has been solved and shows a different ratio of the precursors, γ-$Mg(BH_4)_2$: $[Mg(en)_3(BH_4)_2]$, while initially reported was 2:1. A new ratio of precursors will increase the ionic conductivity, simply because there is less unreacted γ-$Mg(BH_4)_2$. High resolution neutron powder diffraction data was previously collected at the NOVA beamline at J-Parc Spallation Source, Japan, and shows a very good correlation with the proposed model. Conductivity measurements will be presented as well as quasi elastic neutron scattering (QENS) experiments.

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Michael Heere

Sector coupling on a MW Scale or what to do with 500 kg of ‘green‘ H_2 a day?

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The integration of renewable energy sources has created new opportunities for energy storage and utilization, particularly through the use of metal hydrides. This paper will explore the feasibility of sector coupling on a MW scale using a 1 MW electrolyzer and the world's largest (land-based) metal hydride store being built at TU Braunschweig, Germany, with a capacity to produce and store 500 kg of hydrogen a day, respectively. The focus of the „Demonstration Lab“ will be on the application of metal hydrides in the energy sector, specifically in the context of sector coupling. The results will provide insights into the technical and economic viability of using metal hydrides to store and utilize surplus energy, particularly in terms of their performance, durability, and safety. The study will also explore the potential of metal hydrides to enhance energy security, reduce greenhouse gas emissions, and promote the growth of a low-carbon economy. The findings will be of particular interest to those involved in the development of metal hydrides for energy storage and sector coupling and are viable for different research fields, such as fuel cell systems and powertrains.

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Kai Holtappels

Safety aspects for hydrogen technologies

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Hydrogen is a central component of the energy transition and the European Green Deal for a climate-neutral Europe. To be able to achieve the targets defined by 2045, the EU and the German government have drafted a framework for action and are making long-term investments in research, development and the rapid implementation of innovative hydrogen technologies.

The conditions for a successful energy transition and the economic use of green hydrogen as a clean energy carrier are Hydrogen readiness and a rapid market ramp-up, as well as the establishment of the necessary value chains in the national and European framework. Reliable quality and safety standards are the prerequisite for ensuring safety of supply and environmental compatibility and for creating trust in these technologies.

BAM has bundled its expertise in the field of hydrogen technologies into a competence center H2Safety@BAM to create confidence in the technology and to support the hydrogen strategies of the German government and the EU. The topic of hydrogen has been current at BAM for more than one hundred years and is also currently the focus of numerous research and cooperation activities. The competence centre H2Safety@BAM and its vision of “We build trust in hydrogen technologies” will be presented with its many safety-related topics, which can be found throughout the entire hydrogen value chain. Two deep dives will present the results on projects dealing with the design-to-cost issue of classical pressurized gas storage containments and the consequences of the release of liquid hydrogen, which will be roughly compared to the release of ammonia.

Thomas M. Klapötke

New Possibilities for Hydrogen Transport

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Most countries are relying on being able to use hydrogen technology within the energy transition. This should make "green" hydrogen marketable and enable its industrial production, transportability and usability. The most sensible place to produce green hydrogen is where there is enough renewable energy available to operate water electrolysis, for example, Egypt, Algeria and Morocco. Depending on the amount of hydrogen to be transported, different transport methods make sense. For large volumes over short distances, hydrogen pipelines are the best option. However, for medium and longer distances, other methods could be used. This means that hydrogen can be transported under high pressure, liquefied, bound to a carrier liquid, or in the form of hydrogen derivatives. However, each method has its advantages and disadvantages. For transport over longer distances, a chemically bound form such as ammonia (NH_3) or borazane (H_3B-NH_3) seems to make the most sense. In this presentation, the advantages of borazane over ammonia will be discussed in this context, and solutions to possible problems will be evaluated.

Mario J. Kriegel

Utilizing high-temperature calorimetry for a thermodynamic assessment based on the CalPhaD approach

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The investigation of phase diagrams making use of the CalPhaD (Calculation of Phase Diagrams) approach is one of the central research interests in our group. This method can be applied to develop new metallic or oxide materials with promising or improved properties. The CalPhaD method can help to reduce the number of experiments that are necessary for a conventional alloy development, because it identifies contradictions among different sets of experimental and theoretical data, and indicates the most crucial experiments that are needed to study the respective system. This approach takes into account all available experimental information simultaneously to derive a Gibbs energy function for each phase of the system, which will be used then to calculate phase diagrams. The usage of only experimental phase equilibrium data, however, can easily yield CalPhaD descriptions predicting thermodynamic quantities outside the uncertainty limits of calorimetric data and can result in poor extrapolations to higher-order systems. Thus, the modelling of the phase diagrams should be based on both, the measurement of thermodynamic properties and phase equilibrium data, in order to derive a consistent database. In this contribution, the application of different calorimetric and thermoanalytical methods as a basis for the modelling of metallic and oxide system using CalPhaD is illustrated.

Iron-based shape memory alloys (SMAs) have gained significant attention from researchers in recent years due to their numerous advantages over conventional NiTi-based alloys, especially in fields like damping in civil infrastructure and actuation for aerospace applications. These advantages include lower material costs, ease of manufacturing due to good cold workability, and the possibility of applying steel-based processing procedures. A thermodynamic description of the Al–Fe–Mn–Ni system was developed with a specific focus on the target composition of 43.5Fe-34Mn-15Al-7.5Ni (at.%). This description aimed to explore new compositional regions relevant for Fe-based SMAs and to adjust necessary heat-treatment procedures. To achieve this, complementary investigation methods were used to experimentally study the quaternary Fe–Mn–Al–Ni system. The heat capacities of intermetallic phases were measured using the classical three-step method in the Pegasus 404C heat flux DSC device (NETZSCH-Gerätebau GmbH, Germany) and the power-compensated DSC 8000 (Perkin

Elmer, Inc., USA). Enthalpy increment measurements were also performed using the MHTC-96 calorimeter (SETARAM, France) in transposed drop calorimetry mode. Phase transition temperatures were determined through heating in the aforementioned heat-flux DSC devices, and the results were used as a basis for modeling this quaternary system.

Ceramic materials based on yttria, zirconia and alumina are of interest for many applications. Exemplarily, Y_2O_3-stabilized zirconia co-doped by rare earth elements are of interest for application as oxygen sensors, solid states electrolytes and thermal barrier coatings (TBC). Simultaneous co-doping of ZrO_2 by Y_2O_3 and Ta_2O_5 (at the level of 9-11 mol. % of each) makes the tetragonal phase non-transformable to monoclinic phase up to 1500 °C. Moreover, this material presents interests as possible TBC due to an increase of stability and durability of coating. The orthorhombic compound $Ta_2Zr_6O_{17}$ presents interest for structural applications. Materials based on the ZrO_2–TiO_2 system are of interest due to their applications in electrical and optical devices such as capacitors, piezoelectric sensors, ultrasonic motors, and microwave dielectric resonators. In order to develop consistent thermodynamic databases for these ceramic systems, the phase relations were studied and thermal quantities such as heat capacities and formation enthalpies were investigated experimentally. The high temperature heat capacities of ceramic compounds were measured applying the classical three-step method between 220 and 570 K using power-compensated (Perkin Elmer, DSC 8000) and between 330 to 1473 K using heat-flux (NETZSCH, Pegasus 404C) DSC devices. The data on heat capacities in the mentioned temperature ranges were fitted using the Maier-Kelley equation to provide $c_P(T)$ in the temperature range between 298 and 1400 K. The heat capacities were measured for $Eu_2Zr_2O_7$, $Yb_3Al_5O_{12}$, $EuAlO_3$, $Eu_4Al_2O_9$, $ZrTiO_4$ and $Ta_2Zr_6O_{17}$ (Fig. 1) compounds. The formation enthalpy of the $ZrTiO_4$ was determined using the AlexSys 800 calorimeter (SETARAM) and the 'drop solution' method. The temperatures of the invariant reactions taking place in the systems ZrO_2–Eu_2O_3, Yb_2O_3–Al_2O_3, Eu_2O_3–Al_2O_3, ZrO_2–TiO_2 and ZrO_2–Ta_2O_5 were determined using a differential thermal analysis device (SETARAM, Evolution 2400, DTA-TG). Based on experimental and literature data thermodynamic descriptions were developed for further use in higher order systems.

Mario J. Kriegel (cont.)

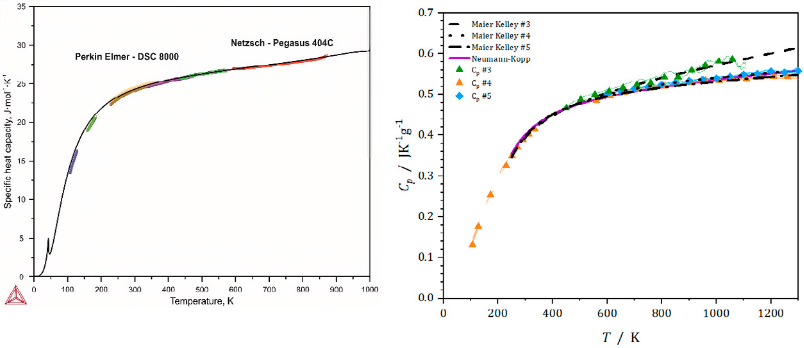

Fig. 1: Heat capacity measurements of the intermetallic Ni_3Al phase (left), and the ceramic compound $Ta_2Zr_6O_{17}$ (right) [1].

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Johannes Lerchner

Heat production of muscle tissue of tegu lizards (*Salvator merianae*)

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In general, small ectothermic vertebrates are incapable of endogenously sustaining a body temperature substantially above ambient temperature. However, contrary to prevailing notions, some ectotherms can engage in facultative endothermy. For example, during the reproductive season, tegus lizards have an enhanced capacity to augment heat production and heat conservation [1]. In this case, an elevated body temperature is sustained mainly through enhanced metabolic rates in muscle tissue. Goal of the study was the identification of the heat contributions from different sources inside the muscle cells of tegus lizards.

Direct calorimetry is a valuable tool if anaerobic processes have to be studied. This is the case if heat contributions from reactions catalyzed by SERCA enzymes have to be analyzed. SERCA is an enzyme in the membrane of the sarcoplasmatic reticulum (SR), an organelle which plays an important role in the regulation of the Ca^{2+} concentration inside the muscle cell. SERCA acts as calcium pump when the Ca^{2+} concentration has to be decreased in the cytosol in order to force the relaxation of the muscle fibers. The ATP hydrolysis reaction is the driving force for the transport of Ca^{2+} from the cytosol into the SR against the concentration gradient. However, ATP hydrolysis can be uncoupled from the calcium ion transport and the evolved heat serves as contribution to the thermogenesis of the organism.

To determine the contribution of SERCA to the overall heat production, oxidative phosphorylation was blocked and the effect of the SERCA inhibitor thapsigargin was analyzed by measuring the heat production of small pieces of muscle tissue obtained from biopsy using a flow-through chip calorimeter. Results obtained at different states of animal's activity are compared.

Keywords: Endothermy, Tegus lizard, SERCA, chip calorimetry

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Johannes Lerchner

Calorimetric measurements on small tissue samples

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If heat from anoxic processes significantly contributes to the overall heat production of living matter, calorimetry becomes a valuable tool in addition to respirometry. This is particularly relevant in the study of specific metabolic processes that run completely anaerobically, such as the enzyme-catalyzed hydrolysis of ATP in muscle cells, one of the processes that is important for the thermogenesis in mammals.

When studying the energy metabolism of mammals, the potentially time-dependent physiological state of the organism has to be taken into account. Sampling at different times and without significantly affecting the subject is therefore often necessary. Therefore, only little material, usually obtained by biopsy, is available for measurements in this case. This is the situation where chip calorimetry proves beneficial.

In order to be able to measure the heat production in small tissue samples but also in single multi-cellular organisms, the Freiberg flow-through chip calorimeter [1] was equipped with a special sample transport technique that allows reliable positioning of tissue samples in the measuring chamber of the calorimeter. Using this technique, samples of amounts in the range of 2 - 5 mg can be measured within 6 min after insertion into the measuring channel.

Selected results from our studies on muscle tissue of Tegus lizards, brown adipose tissue (BAT) and on larvae of *Drosophilia* flies will be presented.

Keywords: Chip calorimetry, Tegus lizards, *Drosophilia*, brown adipose tissue

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George Makhatadze

Pressure Perturbation Calorimetry and the Effects of High Hydrostatic Pressure on the Conformational Equilibrium of Biomacromolecules

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Temperature and pressure, *T* and *P*, are important environmental variables that play an essential role in the life and death of organisms on Earth. From a practical perspective, the sensitivity of microorganisms to *P* and *T* has led to significant progress in civilization through the ability to provide food safety via canning, pasteurization, and pascalization processes. From a molecular perspective, the physicochemical mechanisms that control survival are the effects of pressure and temperature on biomacromolecules (proteins, DNA, RNA, lipid membranes). Biomacromolecules adopt a unique three-dimensional structure (native or folded state) that is required for their biological function. The stability of this structure is very important for the function, and thus biomacromolecules need to evolve to remain folded under living conditions. Increasing pressure, much like increasing temperature, perturbs the thermodynamic equilibrium between the native folded state and the denatured unfolded state. In thermodynamic terms, stability is defined by the Gibbs energy, Δ*G*, which is a function of both temperature and pressure and is related to the population of the native, *N*, and unfolded, *U*, states as shown in Equation (1).

$$\Delta G(T,P) = -RT\ln\left(K_{\mathrm{eq}}(T,P)\right) = -RT\ln([U]/[N]) \qquad (1)$$

where K_{eq} is equilibrium constant. The pressure dependence of Δ*G* is defined by the volume changes between the unfolded and native states, Δ*V*:

$$\Delta V = \left(\frac{\partial \Delta G}{\partial P}\right)_T \qquad (2)$$

Thus, the response of the system to changes in pressure is driven by Le Chatelier's principle. If Δ*V* is positive, the stability will increase with an increase in temperature, and thus the thermodynamic equilibrium will be shifted towards the native state. If Δ*V* is negative, an increase in pressure will shift the equilibrium towards the unfolded state, and thus at some pressure, all molecules are expected to be in the unfolded state. The method of choice to experimentally measure Δ*V* is pressure perturbation calorimetry (PPC). I will discuss the principles of PPC and highlight some important findings using this calorimetric method. Finally, I will also discuss novel

George Makhatadze (cont.)

computational methods to predict the values of ΔV from the knowledge of three-dimensional coordinates of proteins and nucleic acids.

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Thomas Maskow

Characterisation of fungal lignocellulose utilisation strategies using biocalorimetry

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Fungi are important primary destructors of lignocelluloses of plant biomasses and thus play a key role in the global carbon cycle [1]. At the same time, they are also considered as promising biocatalysts with considerable potential for various (environmental) biotechnological applications. These include, for example, the conversion of lignocellulosic by-products from agriculture and forestry into valuable materials using biorefineries, as well as the potential treatment and detoxification of various wastes and environmental pollutants [2-4].

This raises the question of whether non-invasive metabolic heat flux analysis can be used to determine functional traits in free-living saprotrophic decomposer fungi and to predict fungal influences on ecosystems processes. To answer such questions, the growth of seven fungal species, including ascomycetes, basidiomycetes, and zygomycetes, was studied in a standardized laboratory environment on wheat straw as a globally relevant lignocellulosic substrate. Our study demonstrates that biocalorimetry can provide various growth-related parameters of fungal activity. These include apparent maximum growth rates (AMGR), cultivation times until the observable onset of fungal growth at AMGR (tAMGR), and heat yield coefficients ($Y_{Q/X}$); the latter indicating the degree of resource investment in fungal biomass relative to other functional properties. Particularly exciting is the quotient obtained from AMGR and tAMGR, which we refer to as competitive growth potential (CGP). All these parameters seem to be suitable to link the fungal potential for biomass production with the corresponding ecological strategy in resource utilization. Therefore, they may be considered as fungal life history traits. A close relationship observed between CGP and $Y_{Q/X}$ values suggests an interpretation in terms of fungal life history strategies [5].

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Florian Mertens

Limitations and Opportunities in Solid State Hydrogen Storage

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Thermochemistry of Liquid Organic Hydrogen Carrier: How Calorimetry sets the foundations for development of the technology

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Liquid Organic Hydrogen Carrier (LOHC) are an attractive option for the storage of hydrogen. Hydrogen is stored by the reversible hydrogenation of an aromatic substance (the hydrogen lean form), forming the corresponding saturated derivative (the hydrogen-rich form). The covalent bond enables storage at ambient conditions in a safe and dense form. Hydrogen release is done catalytically in an endothermal dehydrogenation reaction.

Several substances have been proposed as LOHC materials. For the selection of suited candidates, caloric properties are of utmost importance. In theory, all unsaturated components could be utilized. However, for reasonable conditions in the hydrogen release reactor, it is important to find substances with enthalpies of reaction of about +40 kJ mol(H_2)$^{-1}$ for the dehydrogenation. Yet, most materials have enthalpy values higher than this (often between +55 and +70 kJ mol(H_2)$^{-1}$).

Systematic search for suited carrier materials therefore requires consistent data sets regarding enthalpies of formation. Particularly data for the hydrogenated forms are often not available or the quality of the respective data is questionable. Hence, LOHC research is highly depending on an improved data base for caloric properties of potential LOHC candidates.

Experimental data still play a vital role in this regard and are crucial for obtaining reliable data. However, nowadays predictive methods, such as quantum-chemistry based approaches, are becoming increasingly more relevant. They do not only provide a shortcut method for obtaining data experimentally not available, but can also help to validate experimental data for LOHC materials and extend their range of applicability.

Amir Narymany Shandy

Development of an Optical Differential Scanning Calorimetry Setup for Determining the Specific Heat Capacity at High Temperatures

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Modern material science is of high importance for further development in many fields. The increase of the process temperature of thermal energy conversion processes and therefore the development of highly temperature stable materials is one of the most important issues on the way to higher efficiencies. This, however, requires the development of materials, which are more stable towards high temperature areas. One example for such successful material development are thermal barrier coatings (TBC) used for gas turbine blades. In some cases under operating conditions, the temperature of the TBCs is far above the melting point of the protected body material. In additive manufacturing techniques as laser sintering material is locally molten by a laser beam. Hereby, an increase of precision and the enhancement of process control are aims of further development.

In order to achieve improved process control, the reliable prediction of the heat conductive behavior of different materials at harsh operation conditions and therefore the exact knowledge of material parameters at high temperatures above 1000 °C and up to 2000 °C is needed. In this regard, the two material properties thermal diffusivity and the specific heat capacity are important. For the determination of the thermal diffusivity even at high temperatures above 2000 °C, the laser flash method can be used as a verified measurement method. The specific heat capacity at temperatures below 1000 °C can already be measured by differential scanning calorimetry (DSC). Today, in most DSC setups thermocouples are utilized for the necessary temperature measurement. However, up to now, thermocouples are not suitable for higher temperatures. For this reason, a reliable measurement method to provide verified information about the specific heat-capacity of material samples at temperatures above 1000 °C is still needed.

To resolve this lack of information in future, within the project “Optical Differential Scanning Calorimetry For Modern Material Science At High Temperatures (OptiMa)” it is intended to develop a DSC

Amir Narymany Shandy (cont.)

method for measurements at temperatures between 1000 °C and 2000 °C. In this new built DSC setup (Fig. 1), several optical detectors like radiation thermometers and thermographic cameras will be tested and analysed in order to gather the required temperature information without fault-prone thermocouples. Simultaneously, different methods of sample heating will be tested e.g. inductive heating and laser-heating. Furthermore, it is planned to utilize the triple-cell sample holder design, which reduces the number of necessary measurement cycles [1, 2].

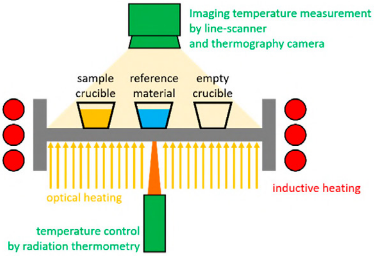

Fig. 1. Principle drawing of the new DSC setup with optical temperature measurement for determination of the specific heat capacity at temperatures between 1000 °C and 2000 °C.

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Heat-/Pressure-Accumulation Test - Adiabatic Calorimetry for Assessing Process Safety Relevant Parameters such as ADT_{24h} and T_{exo}

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Process Safety has been, is and will be a key factor while planning and enduringly carrying out chemical reactions on an industrial scale. Besides the reaction kinetics, a crucial parameter for a safety assessment of a chemical reaction is the heat balance which includes the system's cooling capacity. The heat removal capacity is worse for industrial-scale reactors due to the high volume to surface ratio. Therefore, from a safety point of view and for assessing even high scale-up systems the worst case and adiabatic conditions are most often assumed to account for the high volume to surface ratio.

Assessment of the criticality of a synthesis reaction during a cooling failure scenario and a possible transition to a decomposition reaction and a thermal runaway, experiments and the determination of safety-relevant parameters is needed. One of those parameters is the temperature for safe handling, the T_{exo}, which indicates a temperature up to which a decomposition reaction does not occur or is still controllable. The T_{exo} and decomposition behaviour on industrial scale can be determined in the adiabatic heat-storage test and carried out according to the test setup, according to Grewer and Klais [3] and VDI-Guideline 2263 Page 1. This setup consists of a pressure vessel with a volume of approximately 0.75 l. A Dewar vessel is placed in this autoclave. The Dewar vessel isolates its content thermally from its surroundings by a double-walled vacuum jacket with a reflective coating. The autoclave is closed, placed in an oven and heated. After a reaction starts in the vessel, the oven's temperature is regulated to follow the sample temperature and establish quasi-adiabatic conditions. Additionally, the pressure in the headspace is measured and recorded.

Such an adiabatic experiment yields the time-dependent temperature and pressure curve of the decomposition, which is converted in gas production- and heat production rates. From the heat production rate, the activation energy and, additionally, the self-accelerating decomposition temperature SADT (for monomers, the SAPT as polymerizing temperature) can be received according to the UN-Transportbook, UN-Test H.2 [1]. The SADT is an essential parameter for transport classifications.

From a process safety point of view, the most relevant parameter received from an

Jonas Nowottny (cont.)

adiabatic heat-/pressure accumulation test is the ADT_{24h} or adiabatic induction time 24 hours before the highest temperature increasing rate (the runaway). Sometimes, this parameter is known as the time to maximum rate (TMR). Adding a safety margin of 10 K to the determined ADT_{24h}, the T_{exo} can be obtained. It is one of the most relevant safety parameters for assessing a sample's thermal stability and evaluating a safety concept for a process.

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Thomas Rades

Investigation of pharmaceutically relevant properties of amorphous drug systems with differential scanning calorimetry

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Amorphous drugs and drug delivery systems offer an important solution to the pressing problem of poor water solubility of most low molecular weight drugs. Without a sufficient aqueous solubility, oral drug delivery, the most important route of administration, will not be possible or at least remain suboptimal. In an amorphous form the solid drug will have a much improved apparent solubility, but this advantage comes with a risk of recrystallization of the drug during manufacturing, storage and administration. This makes the development of amorphous drugs and drug delivery systems challenging. In this talk we will discuss investigations on use of thermal techniques (in combination of diffractometric and spectroscopic techniques) in the structure elucidation and stability of amorphous drugs and drug delivery systems, such as amorphous solid dispersions (drug polymer systems) and co-amorphous systems (drug low molecular weight co-former combinations).

David Rütti

Simulate chemical reactions and derive kinetics from milli-scale flow calorimetry

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Introduction

Continuous reaction technology has many advantages over traditional batch reactors including higher productivity, stable product quality and increased process safety. In particular, the determination of the enthalpy and kinetics of the reaction is a fundamental part of the safety assessment of a process. The closer this determination is to the industrial process, the more robust the safety data will be. It is well known that significant deviations occur for non-selective reactions [1]. Recently, we have shown that the enthalpy of reaction can be measured in the continuous flow calorimeter without extensive calibration and that the accuracy of the measurements is highly dependent on the process parameters applied, in particular the flow rate [2,3]. In fact, the resolution of the sensors can be increased as desired with different flow rates and the obtained data becomes highly reliable.

In contrast to the well-established calorimetry, obtaining kinetic data directly from a polytropic temperature profile is a relatively new discipline. To our knowledge, this could be addressed by two means; segment wise analysis as reported by Frede *et al.* [4], and a temperature profile fitting approach reported by Imamura *et al*. [5].

In this work, we present a third approach by combining the temperature profile fitting approach with various reaction temperatures to improve the data quality and reduce uncertainty. The oxidation of sodium thiosulfate with hydrogen peroxide (Eq. 1) served as model reaction and was carried out in a milli-scale continuous flow calorimeter (Contiplant, Fluitec, Switzerland), that is scalable to production scale [2].

$$\begin{aligned}&2\,\mathrm{Na_2S_2O_3} + 4\,\mathrm{H_2O_2}\\&\rightarrow \mathrm{Na_2S_3O_6} + \mathrm{Na_2SO_4} + 4\,\mathrm{H_2O}\end{aligned} \qquad (1)$$

Results and Discussion

The quality of the determination could be improved by adapting the modelling described by Stegehake [6]. Therefore, the oxidation reaction was performed at 20 and 60 °C and different flow rates. For each

David Rütti (cont.)

temperature profile, several pairs of E_a and k_0 were evaluated that best fit with the measured temperature profile (Fig. 1). The here presented software allows to freely choose reaction kinetics model. For this evaluation, a second order reaction model was assumed (Eq. 2).

$$-r_{\mathrm{Na2S2O3}} = k \cdot c^{1}_{\mathrm{Na2S2O3}} \cdot c^{1}_{\mathrm{H2O2}} \qquad (2)$$

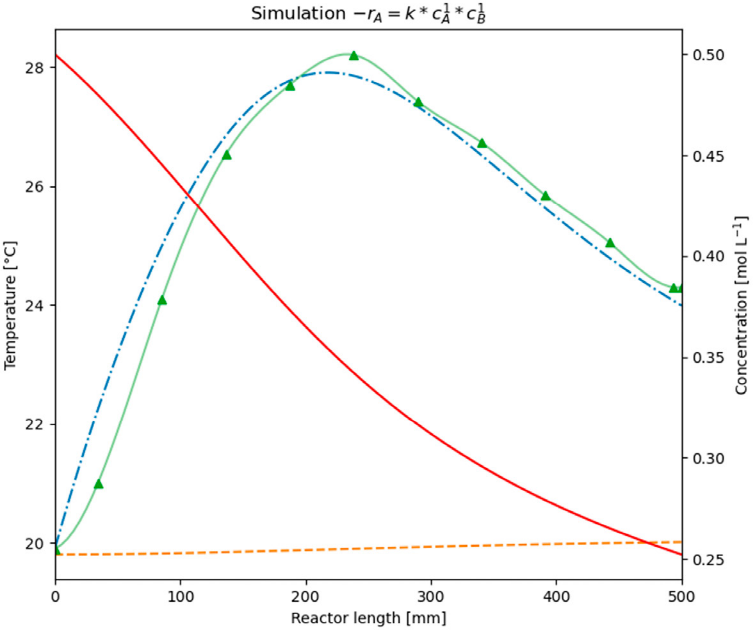

Fig. 1: Simulation of the product temperature (blue dash-dotted), heat transfer medium temperature (orange dashed) and $Na_2S_2O_3$ concentration (red line) of the progress of the chemical reaction (1) and comparison with measured data (green triangles). Heat of reaction, kinetics model and kinetics parameters could be freely selected.

According to Stegehake, there are many pairs that fit well, so different E_a have been fixed and k_0 fitted. These pairs correlate in a linear regression but with different slopes for each temperature. The intersection of all correlations represents the true kinetic parameters (Fig. 2).

David Rütti (cont.)

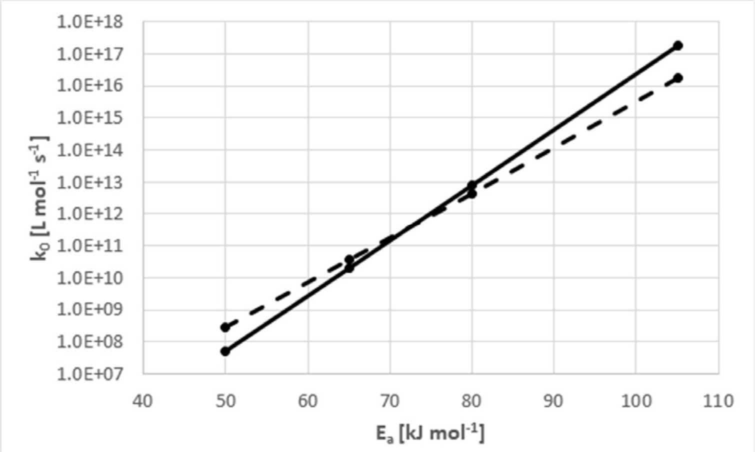

Fig. 2: Determination of activation energy E_a and initial reaction rate constant k_0. Flow rate was 40 ml min^{-1}. 20°C (solid line); 60°C (dashed line).

The obtained value of E_a and k_0 are therefore 73.1 kJ mol^{-1} and 5.1×10^{11} L mol^{-1} s^{-1}, respectively. These values fit well with literature references [4,7–9].

Keywords: Continuous flow calorimetry, Flow reactor calorimeter, Heat of reaction, Safety, Process development, Scale-up

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Jürgen Schawe

An Advanced Heat Flux DSC Operated in the Power Compensation Mode

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The instrument presented by Boersma in 1955 [1] can be considered the starting point for the development of disk type heat flux differential scanning calorimeters (DSC). In this type of DSC, a single furnace contains thermocouples or heat flow sensors with positions for refence and sample. This type of DSC has been further developed by commercial suppliers and is widely used. One of the advantages of this type of DSC is the high sensitivity and robustness.

The alternative power compensated DSC technique was first developed first by O'Neil in 1964 [2]. In this approach, the power required for compensate for heat is measured. The measurement system consists of two small furnaces installed in a cooled metal block. This concept is used for conventional DSC and fast DSC using chip calorimeters (Flash DSC).

An advantage is the relatively short signal time constant, τ, which is given not only by the heat transfer conditions in furnace and sample, but also by the parameter of the controller of the difference temperature.

The new DSC type presented is based on a conventional heat flux DSC, which has been extended with additional heating elements and temperature sensors for power compensation. This new DSC type was developed to combine the robustness of the heat flux DSC with the signal time constant of a power compensated DSC. The new developed instrument has electrical power calibration for outstanding accuracy, small time constant for high resolution, improved sensitivity and excellent baseline stability.

The performance of this device is demonstrated using heat capacity measurements and fast transformations as examples.

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Jürgen Schawe

On Fictive and Real Kinetics of the Glass Transition

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The apparent activation energy, E_a, of the glass transition and the dynamic fragility index, *m*, are characteristic properties of the molecular (or atomic) interactions in supercooled melts of glass-forming materials. These properties are related to the glass-forming ability, the kinetics of relaxation in the glassy state and the stability of glasses.

These parameters can be determined using differential scanning calorimetry (DSC) by measuring the cooling rate dependence of the glass transition. Such measurements are associated with two major problems. The first is due to the influence of the thermal inertia on the measured curves, and the second is linked to the stability of the materials. There are organic compounds that cannot cooled from the melt due to stability limitations. Such glasses are produced using techniques like lyophilization or spray drying. Glasses-forming metal alloys may have a high tendency to crystallize. For such materials, glasses are produced using fast cooling techniques such as splat quenching or melt spinning. In such cases, many authors attempt to derive kinetic data of the glass transition from the apparent heating rate dependence of the glass transition.

This contribution discusses the determination of kinetic parameters from cooling and heating measurements using DSC and fast DSC (FDSC) at consideration the influence of the thermal inertia and its correction. It is shown that the use of heating measurements cannot provide meaningful kinetic data because the apparent heating rate dependence of the glass transition is essentially due to thermal inertia.

The materials of investigation cover a wide range of glass formers. They are a soda-lime silica glass, a palladium based metallic glass forming alloy and a non-crystallizing polymer (polystyrene).

Michael Steeb

Thermal investigation of inhomogenities in aged batteries using an isoperibolic calorimeter

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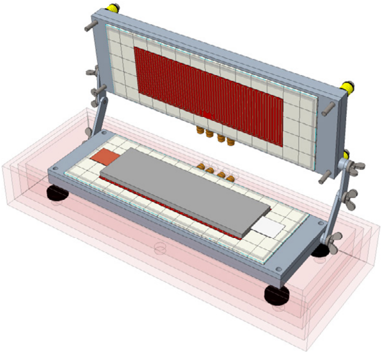
The energy dissipation and the corresponding heat release within a lithium-ion battery cell is an important indicator regarding the overall state of health (SoH). In this context, inhomogeneities resulting from the layered internal structure and the complex production chain of the cell play a crucial role as they lead to additional stress. While in new pristine cells these inhomogeneities are typically small, they increase with aging [1] evolving to distinct non-uniform heat release distribution so called “hotspots”.

In this work, an isoperibolic calorimeter (see figure) as an extension of [2] is built to measure the heat release of large format pouch cells. It uses spatially distributed thermoelectric generators (TEGs) based on the Seebeck effect to accurately determine not only the total heat release of the cell but also the local heat flow across the outer surface of the pouch cell.

Based on this experimental data, the thermal uniformity of the cell can be used as an additional quantity for aging characterisation. This allows insights of the underlying degradation modes extending the information derived from the most commonly used electrochemical methods like electrochemical-impedance-spectroscopy (EIS) and differential-voltage-analysis (DVA).



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Adrian Zentel / Dominik Ohlig

Evaluation of apparatus effects on calorimetric data as a basis for safe operating limits in thermal process safety

Adrian Zentel[1], Dominik Ohlig[1], Jana Sartorius[1], Johannes Schröder[1],
Robert John Blanchard[1], Markus Gödde[2]

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Process safety in chemical industry acts within the area of conflict between economic and safety requirements. Sufficient safety measures are required in order to reduce risk to an acceptable level, protecting human life, the environment, and surrounding assets. But on the other hand, overly conservative safety measures can be adversely affect the economic aspects of the process. Therefore, the better we can characterize the safe operating window of a chemical process the better we are able to apply suitable measures to fulfill both safety and economic requirements.

The assessment of thermal process safety is essentially based on various calorimetric methods. When setting permissible safe operating limits, the measurement uncertainty, detection sensitivity and other factors that may influence the validity of the methods must always be identified and, if necessary, considered as an additional safety margin. In the case of simple screening methods, the required safety margin is correspondingly greater. If higher-quality methods with more precise knowledge of the boundary conditions and influencing factors are used, it is possible to get closer to the permissible limits while maintaining the same level of safety [1]. Using the simple example of evaluating the stability limit of reactants and reaction products, the differences can be shown:

The required limiting safety temperature can be estimated by a simple rule of thumb like the 100 K-rule related to the DSC onset or derived more precisely by thermos kinetic modelling of DSC-curves or via adiabatic calorimetry. Here, high quality data is required. In general, experimental data is affected by several apparatus effects as mentioned before.

In many cases the usage of non-deconvoluted DSC-raw data for thermo kinetic modelling leads to conservative adiabatic induction periods resulting in a limited operation window. To enlarge the permissible safe operation windows and therefore increase the cost efficiency of a chemical process a fundamental knowledge of the apparatus effects on experimental data is essential. Furthermore, this knowledge is useful for efficient experiment design in adiabatic calorimetry within the context of an industrial laboratory environment. In

Adrian Zentel / Dominik Ohlig (cont.)

the following presentation the influence of apparatus parameters on experimental data and derived safety limits by thermo kinetic modelling is evaluated in the context of thermal process safety. The authors are going to present results of an internal method sensitivity study with focus on DSC and adiabatic calorimetry.

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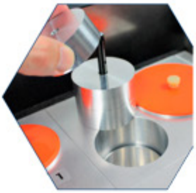

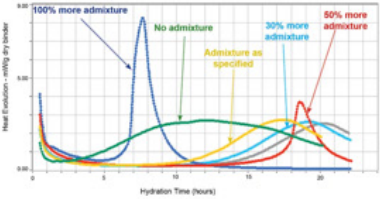
100% more admixture
No admixture
Admixture as specified
30% more admixture
50% more admixture
Heat Evolution - mW/g dry binder
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15
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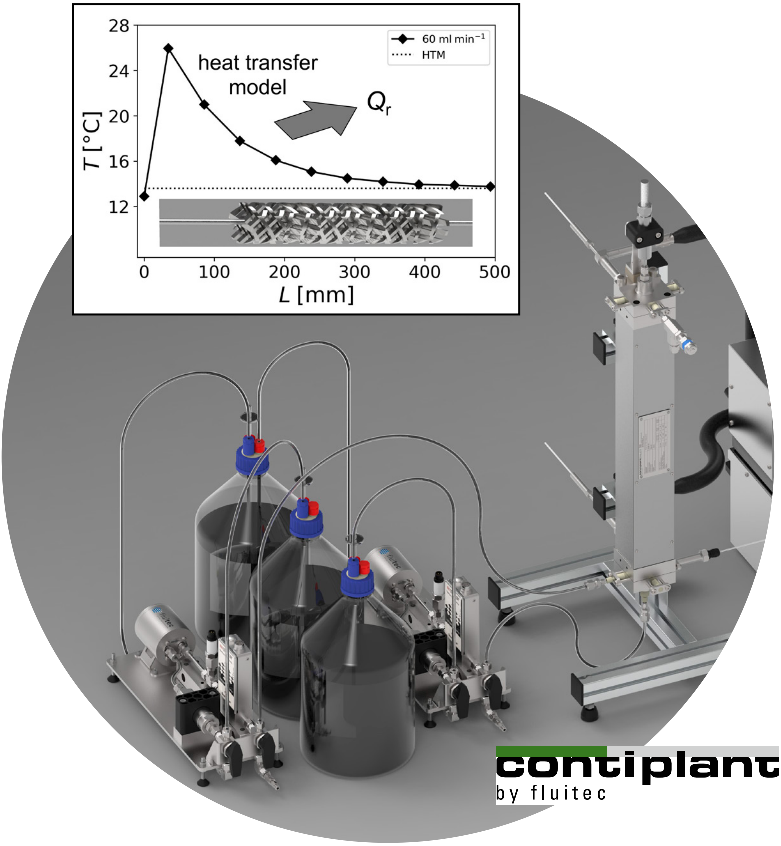
Flow Calorimetry



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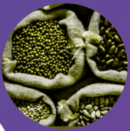


LI-ION
ELECTRIC VEHICLE BATTERY

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Relating Surface Entropy to the Heat of Intrusion into Macroscopic Pores by Scanning Transitiometry

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Die 25. Kalorimetrietage

Abstracts of Posters

Katharina Beck

Binding of antimicrobial peptides alters the thermotropic behaviour of model membranes

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The constantly growing antibiotic resistance emphasises the importance of alternatives to classical antibiotics, such as membrane-active antimicrobial peptides (AMPs). We examine the mechanism of action of the antimicrobial trivalent cyclic hexapeptide cR3W3. More precisely, we investigate the effects of the peptide on binary model membranes containing anionic and zwitterionic phospholipids. We use cardiolipin or phosphatidylglycerol as anionic and phosphatidylethanolamine or phosphatidylcholine as zwitterionic lipids, respectively.

Isothermal titration calorimetry (ITC) measurements show binding selectivity for negatively charged membranes over zwitterionic membranes. This agrees with the observed selectivity of the peptide for bacteria over mammalian cells. Differential scanning calorimetry (DSC) and Laurdan fluorescence spectroscopy reveal the influence of cR3W3 on the thermotropic membrane behaviour, such as lipid chain melting. Our findings enable the detection of electrostatic lipid clustering in saturated and unsaturated lipid membranes. We use the self-quenching dye Calcein to quantify vesicle membrane leakage induced by the peptide. On closer inspection, leakage turns out to be mainly caused by leaky fusion. Yet, the biological relevance of this mechanism should be carefully assessed.

In conclusion, binding of cR3W3 to model membranes induces various effects: electrostatic lipid clustering, membrane fusion, vesicle aggregation, and vesicle leakage.

Konrad Burkmann

Thermodynamic Study of Zirconium and Hafnium Boranate - $Zr(BH_4)_4$ and $Hf(BH_4)_4$

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The storage of hydrogen is a necessary step to achieve the transition of the German energy sector from fossil fuels to renewable energy sources [1]. Transition metal boranates may contribute to reach this goal. A concept to reduce the often very high decomposition temperatures of boranates is that of thermodynamic tuning using mixtures with other (complex) hydrides [2,3]. In respect to the rehydrogenation and dehydrogenation of complex hydrides in general, Zr and Hf compounds are known to exhibit catalytic acivity [4]. Combing these thermodynamic and catalytic aspects, $Zr(BH_4)_4$ and $Hf(BH_4)_4$ may be interesting candidates to design valuable hydrogen storage systems. Unfortunately, no thermodynamic data are available for these materials, which are prerequisit for thermodynamic calculations. Therefore, we conducted a calorimetric and DFT study to provide them.

$Zr(BH_4)_4$ and $Hf(BH_4)_4$ have been synthesised by solid state metathesis [5] instead by wet chemistry. The high vapor pressure and low boiling point of both compounds [6] allows to separate easily the product by destillation from the by-product.

The heat capacities of the compounds were measured between 5 °C and 35 °C using a Setaram DSC-111. TG-DSC-MS measurements and Hess law were employed to determine the enthalpy of formation at 298.15 K. Furthermore, DFT calculations were performed to obtain the standard entropy at 298.15 K of both compounds. Finally, our data were optimised by the CALPHAD method based on experimental vapor pressure measurements [6] and own measurements of the enthalpy of fusion as well as the temperature of fusion. With these data it is possible to perform thermodynamic calculations in regard to the decomposition behaviour of both compounds.

Konrad Burkmann (cont.)

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Miroslaw Chorążewski

Using Scanning Transitiometry to Investigate the PVT and Thermal Properties of Flexible Systems

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Scanning transitiometry is a further development of PVT high-pressure calorimetry, which was constructed for measurements of heat effects associated with variations of the state variables (pressure, volume, temperature). This makes it one of the most suitable techniques to study intrusion of liquid water into a hydrophobic surface. In this case we demonstrate Scanning Transitiometry to investigate liquid-intrusion and extrusion into and from a flexible metal organic framework Cu_2(3,3’,5,5’-tetraethyl-4,4’-biprazolate). The processes were shown not only driven by pressure but also by changing the temperature. This mechanism has the potential to be exploited for energy storage mechanism and actuator functions [1].

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Christian Fricke

Design and application of a novel calorespirometer for measuring soil microbial activity

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Calorespirometry combines calorimetric and respirometric measurements, allowing the simultaneous measurement of the total metabolic activity (heat production rate, R_q) of microorganisms in soils and the metabolic processes during which CO_2 is respired (CO_2 formation rate, R_{CO2}) in the soil. Using this method, it is possible to study the turnover of substrates and soil organic matter at the microbial level. By measuring R_q [in W] and R_{CO2} [in mol·s^{-1}], soil-relevant physicochemical quantities can be determined. The current calorespirometric method assumes a purely calorimetric approach, with R_{CO2} calculated from heat flow data.

The calorimetric sample consists of small (≤ 20 mL), airtight ampoule systems filled with the soil sample (≤ 5 g) and a vial filled with NaOH solution (CO_2 trap). The total heat production rate is the sum of the metabolic heat in the soil and the reaction in the CO_2 trap, thus R_{CO2} can be derived from the difference [1]. One major limitation of these measurements is that two separate experiments are necessary: (i) only soil sample and (ii) soil sample + CO_2 trap (vial filled with NaOH). Due to the small size and heterogeneity of soil samples, different effects in R_q cannot be attributed to the additional heat caused by the CO_2 trap and complicate an interpretation of R_q [2]. In addition, the small, airtight ampoule system quickly leads to oxygen starvation and saturation of the CO_2 trap, which on the one hand drastically changes the metabolic response of the microbial community in the soil sample and on the other hand requires frequent change of the CO_2 trap (disturbance of the calorimetric measurement).

In order to overcome these issues, we present in this work an early-engineered, multichannel (up to 18 measuring units) calorespirometer, which addresses the spatial issue of current measurements. The novel calorespirometer has a conductometric measuring cell filled with KOH solution (CO_2 trap, respirometer unit) positioned on the lid of the measuring channel. The measuring channel is placed on a heat sink, which is in contact with the heat flow sensor on which the open sample vessel (> 20 mL) with the soil sample (10 g) is placed (calorimetric unit). Consequently, the heat release is independent of the CO_2 trapping.

Christian Fricke (cont.)

In addition, oxygen depletion and saturation of the CO_2 trap do not play a role due to the large headspace (> 100 mL) of the experimental setup and a large amount of KOH solution (15 mL).

The development process of this novel calorespirometer is supported by state-of-the-art numerical simulations based on the finite-element method. Such simulations allow predictions about the sensitivity and performance of the instrument. The applicability was already successfully demonstrated on soil samples spiked with glucose as substrate and numerical solutions were validated by experimental data obtained from the custom-built multichannel calorespirometer under laboratory conditions.

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Franziska Habermann

Study of the Influence of NaCl and LiCl on the Decomposition of $Sr(AlH_4)_2$

Franziska Habermann, Konrad Burkmann, Bianca Störr, Jürgen Seidel,
Klaus Bohmhammel, Florian Mertens

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The suitability of complex hydrides such as alanates for solid-state hydrogen storage applications, especially regarding the transition to a renewable energy-based economy, is being widely discussed. Advances in the field of mechanochemistry allowed the preparation of alanates also by ball milling. In order to characterise the alanates themselves, often simply the obtained mixtures consisting of the alanate and the by-product were investigated [1]. For that purpose, the by-product is assumed to be inert. We reviewed the validity of this assumption by investigating the decomposition behaviour of $Sr(AlH_4)_2$ + 2 NaCl and $Sr(AlH_4)_2$ + 2 LiCl mixtures, synthesised by ball milling.

TG-DSC measurements (Sensys DSC, SETARAM) were used for a general characterisation of the thermal dehydrogenation of the different mixtures. We found the by-product to not affect the decomposition of $Sr(AlH_4)_2$ itself, but that of $SrAlH_5$. To investigate the influence of LiCl and NaCl on the dehydrogenation pathway of $SrAlH_5$ in more detail, the decomposition products were analysed by means of XRD and the feasibility of possible decomposition reactions was assessed from a thermodynamic point of view.

While NaCl does not affect the decomposition pathway, LiCl alters the dehydrogenation reactions by acting as a reactant. Instead of decomposing directly to SrH_2 and $SrAl_4$ (R1), $SrAlH_5$ dehydrogenates in two separate steps when LiCl is present in the sample. First SrHCl and Al (R2) and then $SrAl_4$ (R3) is formed.

$$SrAlH_5 + Al \rightarrow 0.5\ SrAl_4 + 0.5\ SrH_2 + 2\ H_2 \tag{R1}$$

$$SrAlH_5 + LiCl \rightarrow SrHCl + Al + LiH + 1.5\ H_2 \tag{R2}$$

$$0.5\ SrHCl + 2\ Al + 0.5\ LiH \rightarrow 0.5\ SrAl_4 + 0.5\ LiCl + 0.5\ H_2 \tag{R3}$$

The different effects of LiCl and NaCl correspond to the differing stability of LiH ($\Delta_f H°(298) = -90.5$ kJ/mol [2]) and NaH ($\Delta_f H°(298) = -56.4$ kJ/mol [2]). Due to the high stability of LiH its formation is thermodynamically favoured and thus the addition of LiCl changes the decomposition reactions of $Sr(AlH_4)_2$.

Franziska Habermann (cont.)

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Alexander Lowe

Relating Surface Entropy to the Heat of Intrusion into Macroscopic Pores by Scanning Transitiometry

Alexander Lowe

Institute of Chemistry, University of Silesia, ul. Szkolna 9, 40-006 Katowice, Poland

Peter Schlosser

Establishing a phase diagram of polyoxyethylene (40) stearate and ibuprofen

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Since newly developed active pharmaceutical ingredients (API) are often poorly water-soluble, it may be required to formulate them in a way that enables appropriate dissolution properties. One way to increase the dissolution rate is the development of eutectics. A frequently chosen excipient for this attempt is the water-soluble polymer polyethylene glycol. During recrystallization, melts of a eutectic composition of polyethylene glycol and API build a specific structure containing finely dispersed crystals of API and polyethylene glycol. Upon contact with water, the recrystallized melts (eutectics) release the API as micronized crystals with increased dissolution rate caused by a higher specific surface area than in the conventional drug powder [1]. In contrast, melts containing the API above the eutectic concentration recrystallize building segregates of API with a much larger particle size. Hence, in order to obtain micronized particles, it is essential to get knowledge of the eutectic point of the composition of API and excipient.

With the intention of later developing self-dispersible pellets by extrusion-spheronization, the water-soluble surfactant polyoxyethylene (40) stearate (Myrj™ S40) and the poorly water-soluble model substance ibuprofen were selected in the current study to form a eutectic. Myrj™ S40 not only forms a eutectic mixture with ibuprofen but also may improve its solubilization in water upon dissolution. Using recrystallized melts of mixtures of these two substances, a phase diagram was created by differential scanning calorimetry and polarization microscopy. Depending on the mass ratio of API and excipient, the recrystallized formulations exhibited a significant melting point depression. The eutectic point was found to be around 30 wt.% ibuprofen and around 39 °C. With regard to the impact of the formation of the eutectic on the behavior to be expected upon dissolution, the particle size of ibuprofen crystals released from the prepared formulations in contact with water was estimated by polarization microscopy. The aqueous

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dispersion of the eutectic composition exhibited ibuprofen crystals around 12 µm in length and 6 µm in width.

The combination of DSC and hot stage polarization microscopy proved to be a good approach to determine the eutectic composition of ibuprofen and Myrj^{TM} S40. Beyond its importance concerning the dissolution behavior, knowledge about the eutectic behavior is also of great interest with regard to the processing of these compositions by extrusion-spheronization, since the processing of low melting substances such as Myrj$_{TM}$ S40 by extrusion-spheronization is very challenging due to their sensitivity to temperature [2].

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Tanja Stahl

Determination of the measurement accuracy of reaction calorimetric investigations under difficult reaction conditions for the heat flow balance

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The determination of safety-related parameters with the help of reaction calorimetric investigations represents an important basis for the safe scale-up of chemical processes. The accuracy of reaction calorimetric measurements can be influenced by various parameters and lead to measurement tolerances. The aim of the tests carried out is to investigate the influences on the heat balance of the RC1e calorimeter caused by the viscosity of the reaction mixture, reactions with superimposed heating phases, as well as boiling effects occurring during a reaction process, which can be caused by the formation of low boilers. For the investigations, reaction mechanisms known from literature are used and the experiments are carried out under the influence of the described parameters. The results obtained are evaluated and analysed accordingly in order to determine the measurement tolerance of reaction calorimetric investigations under difficult reaction conditions for the heat flow balance.

The tests carried out show that the reaction calorimetry is significantly influenced from viscosities of 500 mPa s and that no reliable results can be obtained from viscosities of 16 600 mPa s in RC1e. Superimposed heating ramps with a final temperature at least 50 K below the boiling point of the reaction mixture give a slightly conservative result. Heating ramps that end above the boiling temperature of the reaction mixture cannot be investigated by reaction calorimetry, even with pressurisation to suppress the boiling effects that set in. For reactions with constant reflux conditions, a too low reaction enthalpy is determined. Based on the series of experiments carried out on reactions with initiating reflux, several methods have been worked out that can be used to measure these reaction systems.

Oyunbileg Sukhbat

Influence of poloxamer type on the structural changes upon autoclaving of trimyristin nanoemulsions: A DSC study

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The development of new drugs often involves poorly water-soluble compounds. In such cases, lipid-based nanoparticles can provide a suitable formulation option [1]. Drug delivery systems composed of lipid nanoparticles have particle sizes that typically range from 1 to 500 nm. Because of their nanoscale size, these drug delivery systems are suitable for intravenous administration [2]. One such system are lipid nanoemulsions, which are composed of a matrix lipid dispersed in an aqueous phase with an emulsifier. Trimyristin (TM) was used as the lipid matrix for our study. It is a saturated triglyceride of myristic acid with a melting point of around 56 °C. It forms a special type of colloidal lipid nanoemulsions, in which the disperse phase is a supercooled melt [3]. Different types of poloxamers (polyoxyethylene-polyoxypropylene block copolymers, Pol) were used as emulsifiers in our study. TM nanoemulsions were prepared by high-pressure homogenization. For parenteral formulations, sterility is required, and lipid nanoemulsions are usually sterilized by autoclaving. As has been observed previously, autoclaving of Pol 188-stabilized dispersions can lead to an increase in particle size and a change in particle size distribution [4].

This study aimed to examine the effect of autoclaving on the change in particle size and particle size distribution of the emulsions stabilized with different poloxamers. Additionally, the concentration of free poloxamer in the aqueous phase was of interest. The particle size (z-average diameter) and polydispersity index (PdI) of the emulsions was measured by photon correlation spectroscopy (PCS) before and after autoclaving. The z-average diameter had increased and the PdI decreased for all samples after autoclaving. Upon heating in DSC, the melting curves of non-autoclaved TM nanoemulsions exhibited broad and

Oyunbileg Sukhbat (cont.)

jagged peaks. This peak broadening, combined with multiple separated melting events and a shift towards lower melting temperatures, indicates the presence of small particles in the respective TM nanoemulsion [4]. After autoclaving, melting occurred in a single sharp event, indicating a narrow particle size distribution. Thus, the DSC results confirmed a growth in particle size and a decrease in polydispersity during autoclaving, supporting the results obtained by PCS. The concentration of free poloxamer in the aqueous phase was quantified by measuring the refraction index in the ultrafiltrate of the emulsions. After autoclaving, the free poloxamer concentration of all TM nanoemulsions had increased significantly. Larger particles have a lower specific surface area. It can be assumed that excess emulsifier was detached from the particle surface and resulted in an increased concentration of free emulsifier in the aqueous phase. Depending on the type of poloxamer used as emulsifier, changes in the crystallization behavior of the TM nanoemulsions were observed after autoclaving. The crystallization temperature of TM nanoemulsions stabilized with poloxamers with a concentration-dependent adsorption behavior shifted to lower temperatures. In contrast, the use of poloxamers with a less concentration-dependent adsorption behavior displayed a slight increase in the crystallization temperature.

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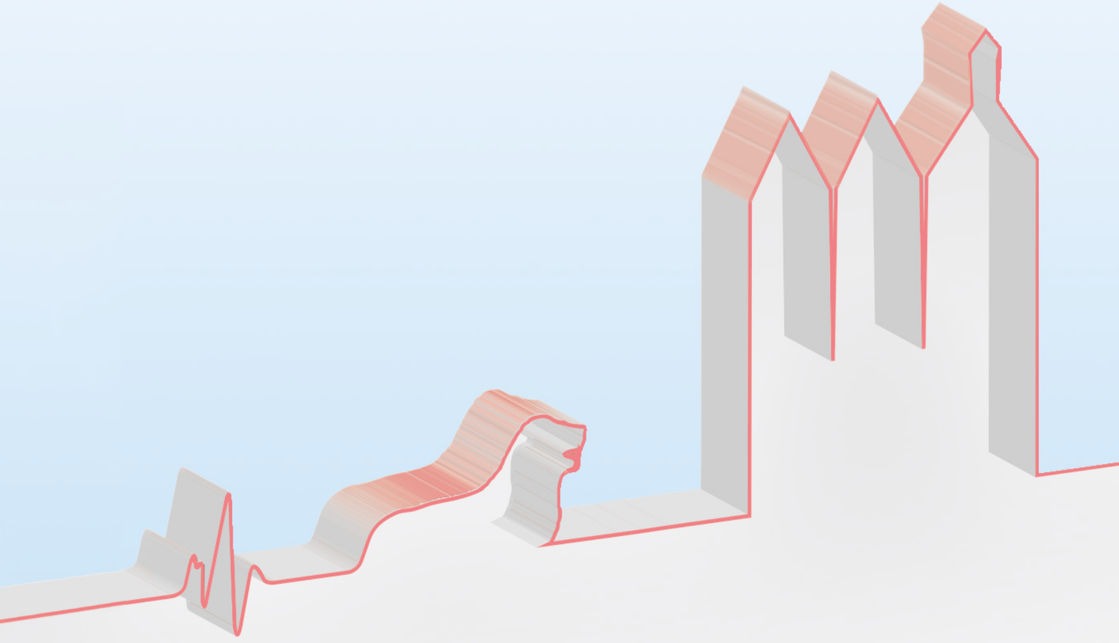

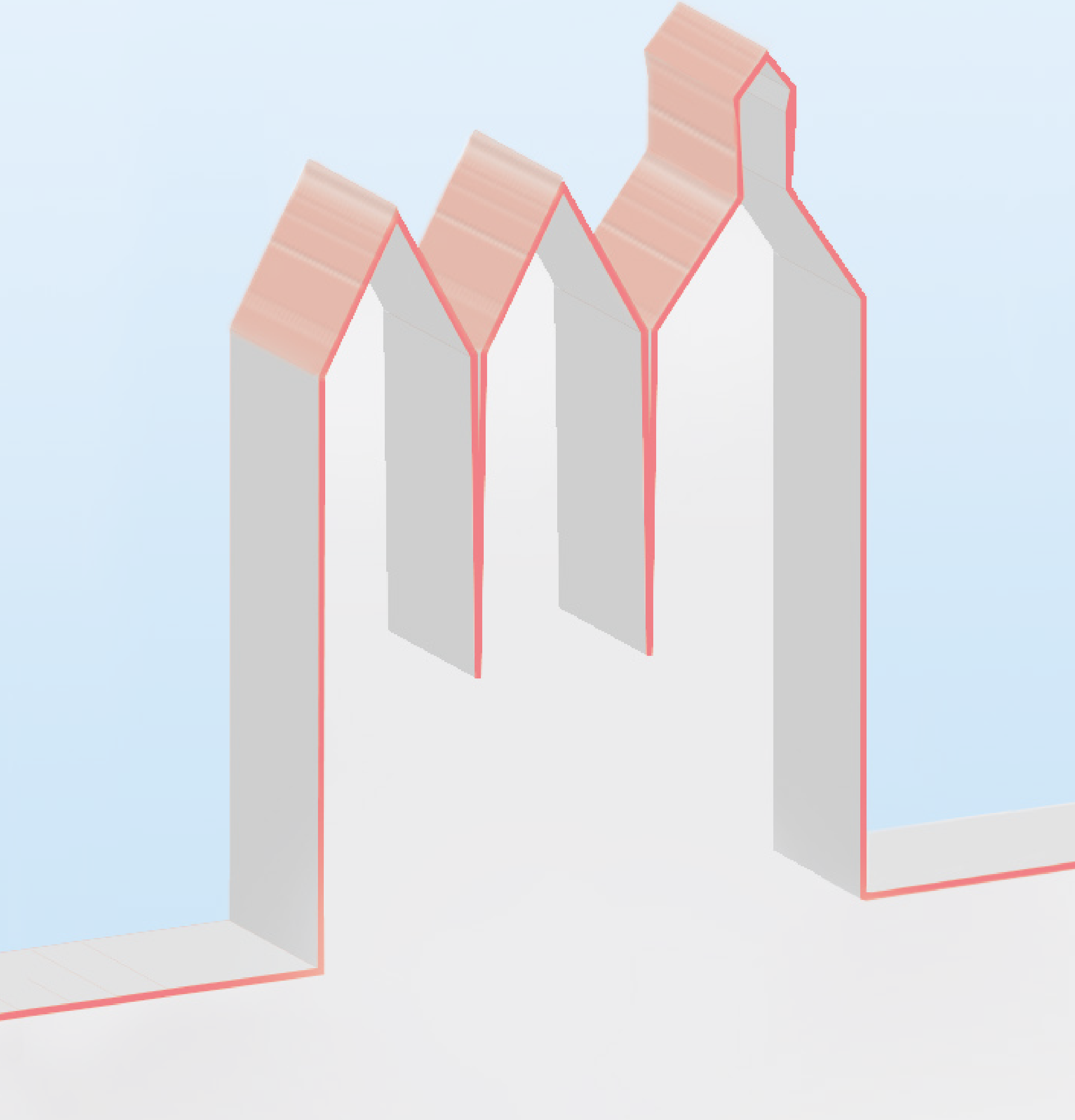

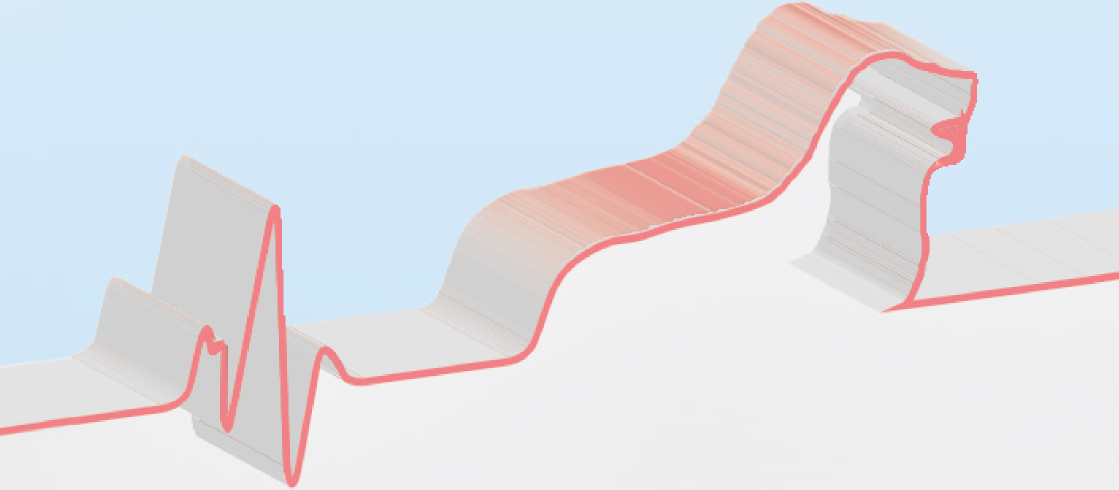

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