5/6 September 2017

2nd Lab Meeting of the Young Crystallographers

STOE

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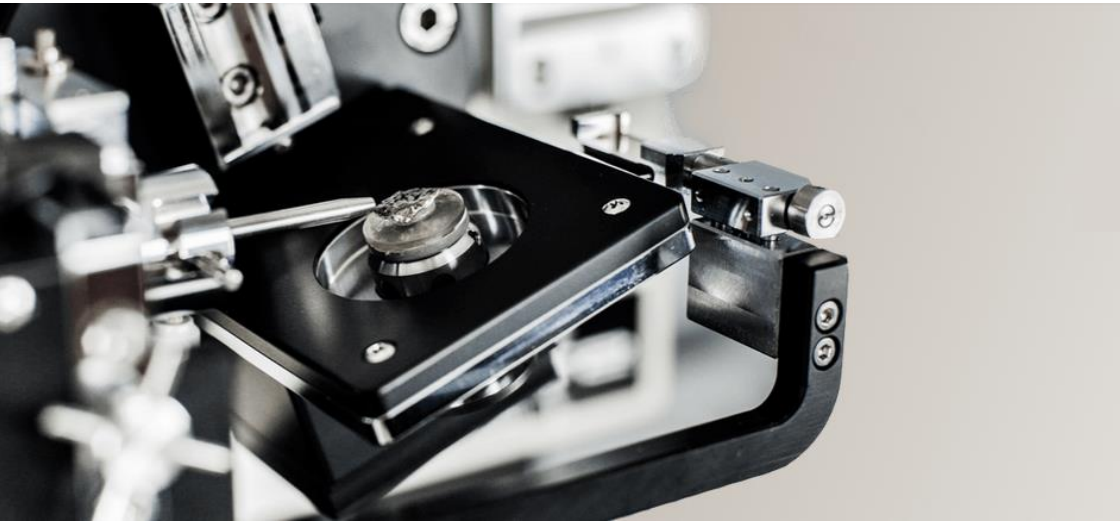

Contents

Features, dates, venue and accommodation

Dates

Tuesday, 05 September 2017 – Wednesday, 06 September 2017

Venue

Lecture Hall @ STOE & Cie GmbH, Hilpertstraße 10, 64295 Darmstadt, Germany

Accommodation

Commundo Tagungshotel Darmstadt, Hilpertstraße 27, 64295 Darmstadt, Germany

The hotel is located across the street from the venue. We have booked a single/twin room for each participant under his/her name. The assignment of your room mate can be found in the last mail. Feel free to go to the reception desk and check in before the "Welcome Reception".

Expenses

The registration fee of 49.-/79.- EUR per person, depending on booking a single or twin room, covers your hotel expenses for the night on the 05/06 September 2017 as well as all further costs (breakfast, lunch, coffee breaks, and BBQ).

Partial Reimbursement

If you have applied for a (partial) travel costs reimbursement during the registration, we will contact you during the STOE Lab Meeting in person. The travel support will be awarded in cash and you will have to acknowledge receiving it with your signature.

Welcome Note

Dear Young Crystallographers,

We are pleased to welcome you to our 2nd STOE Lab Meeting in Darmstadt this year!

This meeting is based on four pillars: networking, knowledge transfer, lightning talks, and social activities. You will get in close contact with STOE during lab tours and lectures in an open-minded and relaxed atmosphere. We will have two lectures of experienced researchers on single-crystal and powder diffraction. Furthermore, you will have the chance to present your latest research, ideas, and results throughout your lightning presentations (short talk + poster session). The meeting will offer you the chance to ask questions and connect with the experts during the sessions and especially during the lunches and our BBQ on Tuesday evening.

Mainly, this meeting is about meeting your peers – other (PhD) students and postdocs working in the field of crystallography or working with crystallography – in chemistry, physics, biology, geology, materials science, etc.: Find out more about the different aspects of your/their field(s). Get to know new methods and maybe make interesting connections to your project and gain new insights.

Learn more about working with or in crystallography in industry.

In order to profit from our exchange-based meeting format, please make sure to pitch your topic for 5 minutes, be present at your poster to discuss your research in more detail, and visit other posters in the remaining time.

We are happy to welcome Semën Gorfman (Freiburg, Germany) and Leonore Wiehl (Darmstadt, Germany) to give the lectures on diffraction. We are looking forward to listen to the STOE talks of Jens Richter and Thomas Hartmann on the company's history as well as their career and daily working schedule.

We are hoping to see you again in March 2018 for the Annual DGK Meeting in Essen (Germany, 05 – 08 March 2018) where we will organize dedicated Lightning Session(s) again. Furthermore, the 3rd Meeting of the Young Crystallographers will be hosted in Aachen (Germany) in September 2018. The planning and organisation are ongoing.

So please stay tuned, we will keep you posted: http://dgk-home.de/aks/jkyc/

We wish you a very pleasant time! Please do not hesitate to ask us if you have any questions.

Best wishes,

Melanie & Khai

Organizers

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We gratefully acknowledge the financial and organizational support provided by STOE & Cie GmbH and DECTRIS AG during the organisation of this meeting. We would especially like to thank Christine Heil and Martin Fark (STOE) as well as Sarah Diallo (DECTRIS) for their help. Furthermore, we thank the German Crystallographic Society (DGK) and Fonds der Chemischen Industrie (FCI) for their financial support.

Deutsche Gesellschaft für Kristallographie

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Schedule

- Presentation = lightning talk **and** poster presentation
- Lightning talk = "appetizer" for the poster, **strictly** limited to 5 min, without Q&A
  Please prepare a few slides in PPT(X) or PDF format for your talk and give it to us on an USB stick just before your Lightning Talks Session. We will provide a computer, beamer, and laser pointer.
- Poster presentation: DIN A0 (841 × 1189 mm), portrait format, not laminated

Tuesday, 05 September 2017

13:00 – 13:45	Welcome reception
13:45 – 14:00	Welcome note
14:00 – 14:30	STOE talk 1 – Dipl.-Ing. Jens Richter
14:30 – 15:30	Academic talk 1 – Dr. Semën Gorfman
15:30 – 16:00	Coffee break
16:00 – 17:00	STOE lab tour/training 1
17:00 – 18:00	Lightning talks session 1
18:00 – 19:00	Poster session 1
19:00 – 22:00	Barbecue @ STOE

Wednesday, 06 September 2017

09:00 – 09:30	STOE talk 2 – Dr.-Ing. Thomas Hartmann
09:30 – 10:30	Academic talk 2 – Dr. Leonore Wiehl
10:30 – 11:00	Coffee Break
11:00 – 12:00	Lightning talks session 2
12:00 – 13:30	Lunch @ Commundo Tagungshotel
13:30 – 14:30	Poster session 2
14:30 – 15:30	STOE lab tour/training 2
15:30 – 16:00	Closing remarks

Academic talks

Academic talk 1 – How X-ray crystallography helps to understand the materials functions

Semën Gorfman (gorfman@physik.uni-siegen.de)

Albert-Ludwigs-Universität Freiburg, Crystallography, Freiburg (Germany)

The recent advances in the crystallographic instrumentation extend the realm of traditional X-ray crystallography and push our science beyond the state-of-the-art. The interdisciplinary nature of crystallography becomes more important than ever. The aim of this talk is to introduce the crystallography of materials. I will show how the new generation of synchrotrons, fast area detectors, computational resources and time-resolved data-acquisition systems help to investigate real crystalline materials in action. I will demonstrate the mechanism of piezoelectricity in Perovskite based ferroelectrics and discuss the interplay between atomic and domain structures.

Academic talk 2 – X-Ray Diffraction under High Pressure

Leonore Wiehl (wiehl@materials.tu-darmstadt.de)

Technical University of Darmstadt, Disperse Solids, Darmstadt (Germany)

Knowledge of the crystal structure (and its evolution at extreme conditions) is an essential prerequisite for understanding physical properties of materials and for predicting properties of functional materials. The talk will give an overview of X-ray diffraction experiments, using synchrotron or laboratory sources (also some electron diffraction) on single-crystal and powder samples in-situ at high-pressure in a diamond anvil cell or ex-situ on samples recovered from HP-HT experiments using a multianvil press. A short look back to the roots will cover the growth of large single crystals and the investigation of elastic properties as well as phase transitions of inorganic and metal-organic compounds.

STOE talks

STOE – Your Partner in X-Ray Diffraction.
Get inside: https://youtu.be/vYOhUi4CA5k

New developments in single crystal and powder diffractometry

- The STOE scientists will introduce the latest projects in the field of single crystal and powder diffraction, such as high end sources, latest detectors and accessories for non-ambient sample investigations.
- Workshop on "how to apply successfully for a job opportunity at the university or in the industry"

STOE talk 1 – Single crystal X-ray diffraction

Jens Richter (richter@stoe.com)

STOE and Cie, Co-CEO & CTO*, Darmstadt*

STOE talk 2 – Powder X-ray diffraction

Thomas Hartmann (Hartmann@stoe.com)

STOE and Cie, Sales and Powder, Darmstadt

Lightning Presentations – Tuesday (Time Schedule)

1	17:00 –17:05	Melanie Nentwich	Structure variations in RSi_2 and R_2TSi_3 silicides
2	17:05 –17:10	Dennis Wiedemann	Ion Diffusion in $BaLiX_3$ (X = F, H, D): A Neutron-Diffraction Study
3	17:10 –17:15	Hassan Osseili	Chemoselective Hydroboration Catalysis by $[HBPh_3]^-$ Anion
4	17:15 –17:20	Rüdiger W. Seidel	The crystal & molecular structure of adiponitrile
5	17:20 –17:25	Asmaa Loutati	Synthesis, X-ray diffraction, Rietveld refinement of magnetic nanoparticles
6	17:25 –17:30	Marius Kremer	Building crystalline ladders with a ditopic ligand
7	17:30 –17:35	Linda Hollenbeck	Crystal growth of magnetoelectrics by chemical transport reaction (CTR)
8	17:35 –17:40	Sophie-Charlotte Lappe	Time- and temperature-dependent Curie temperature variations in titanomagnetites
9	17:40 –17:45	Aziz Kheireddine	Structural & vibrational study of diphenyl-hydrazine dihydrogenophosphate (single crystal)
10	17:45 –17:50	Wilhelm M. Hützler	Uncovering a new synthon for application in crystal engineering: the triply hydrogen-bonded ADA-DAD N-H···S/N-H···N/N-H···O synthon
11	17:50 –17:55	Falk Meutzner	Assessing Al-ion conductors from sulphides and selenides
12	17:55 –18:00	Laura Ritterbach	Kinetics of gypsum phase ($CaSO_4 * xH_2O$) phase transitions

Lightning Presentations – Tuesday (Abstracts)

1 *Structure variations in RSi_2 and R_2TSi_3 silicides*

Melanie Nentwich (melanie.nentwich@physik.tu-freiberg.de)
TU Bergakademie Freiberg, Institute für Experimentelle Physik

The RSi_2 and R_2TSi_3 silicides exhibit two different structure types (hexagonal AlB_2 and tetragonal $ThSi_2$) with many variations according to local symmetry. We present an overview of these structures in a Bärnighausen diagram and present correlations between different physical properties as lattice parameters and electronics.

2 *Ion Diffusion in $BaLiX_3$ (X = F, H, D): A Neutron-Diffraction Study*

Dennis Wiedemann (dennis.wiedemann@chem.tu-berlin.de)
Technische Universität Berlin, Institut für Chemie

Fluoride and hydride perovskites are discussed for application in opto-/piezoelectronics and energy storage. We have studied powders and a single crystal of the inverse perovskites $BaLiX_3$ (X = F, H, D) using high-temperature neutron diffraction. Preliminary results on ionic diffusion pathways in these materials will be presented.

3 *Chemoselective Hydroboration Catalysis by $[HBPh_3]^-$ Anion*

Hassan Osseili (hassan.osseili@ac.rwth-aachen.de)
RWTH Aachen University

The little known hydridotriphenylborate $[HBPh_3]^-$ is exploited in hydroboration catalysis. Therefor the isolation of the catalysts and intermediates confirmed by X-ray crystallography is crucial and helps to understand the catalytic cycle.

4 *The crystal and molecular structure of adiponitrile*

Rüdiger W. Seidel (ruediger.seidel@rub.de)
Martin-Luther-Universität Halle-Wittenberg, Institut für Pharmazie

Adiponitrile (hexanedinitrile, $NC-CH_2-CH_2-CH_2-CH_2-CN$), an important precursor in the synthesis of the polyamide Nylon 66, is a colorless, viscous liquid at ambient conditions. We have determined its crystal structure at 100 K by *in situ* cryo-crystallography. In the crystal, the molecule unexpectedly exhibits a centrosymmetric gauche-anti-gauche conformation of the C-C-C-C skeleton.

5 *Synthesis, X-ray diffraction, Rietveld refinement of magnetic nanoparticles*

Asmaa Loutati (loutati.asmaa@gmail.com)
Laboratoire de Physico-chimie des matériaux appliqués LPCMA

When magnetic nanoparticles (MNPs) are single-domain and magnetically independent, their magnetic properties and the conditions to optimize their efficiency in magnetic hyperthermia applications are now well-understood. However, the influence of magnetic interactions on magnetic hyperthermia properties is still unclear. Here, we report hyperthermia and high-frequency hysteresis loop measurements on a model system consisting of MNPs with the same size but a varying anisotropy, which is an interesting way to tune the relative strength of magnetic interactions. A clear correlation between the MNP anisotropy and the squareness of their hysteresis loop in colloidal solution is observed. Increase of magnetic hyperthermia efficiency due to dipolar interactions in low-anisotropy magnetic nanoparticles. In this paper we report the synthesis, structural, X-ray diffraction, Rietveld refinement of magnetic nanoparticles which represent promising magnetic properties.

6 *Building crystalline ladders with a ditopic ligand*

Marius Kremer (marius.kremer@ac.rwth-aachen.de)
RWTH Aachen University

A ditopic ligand, an organic molecule with two different coordination sites exhibiting different chemical behavior, was used to connect two different metal cations, Al(III) and Fe(III), with Zn(II) halides, leading to six new coordination polymers. These metal-organic frameworks (MOFs) form in a one-dimensional "ladder" conformation, with nearly isomorphous structures between all six compounds, and contain large amounts of disordered solvent molecules located mostly in the cavities inside the ladder. Crystallization is possible from multiple different solvents, suggesting that the solvents in the cavities can be interchanged without altering the MOF structure.

7 *Crystal growth of magnetoelectrics by chemical transport reaction (CTR)*

Linda Hollenbeck (linda.hollenbeck@uni-koeln.de)
Section Crystallography, University of Cologne

The present work concentrates on the crystal growth and design of multiferroic and/or magnetoelectric materials. Since these properties are very sensitive to the chemical composition of the investigated material, the influence and control of doping shall be studied and understood. Successfully grown crystals shall be characterized by different methods like X-ray diffraction, thermoanalysis and optical spectroscopy.

8 *Time- and temperature-dependent Curie temperature variations in titanomagnetites*

Sophie-Charlotte Lappe (lappe@ifk.rwth-aachen.de)
RWTH Aachen University

Titanomagnetites are some of the most important natural magnetic minerals, which are widely utilized in paleomagnetic research. Recent studies discovered that their Curie temperature not only, as previously assumed, depends on their composition but is also strongly affected by their thermal history. Understanding the complex mechanism causing the variations in the Curie temperature is hugely important as it may have major effects on paleomagnetic measurements.

9 *Structural and vibrational study of diphenylhydrazine dihydrogenophosphate single crystal DPHDP $(C_6H_9N_2)_2H_2P_2O_7$*

Aziz Kheireddine (kheireddine.aziz@gmail.com)
Laboratory of Chemistry & Physics of Materials - University of Hassan II of Casablanca

Chemical preparation is reported for diphenylhydrazine dihydrogenophosphate single crystal DPHDP $(C_6H_9N_2)_2H_2P_2O_7$. Crystallographic characterization for DPHDP. Vibrational studies for DPHDP

10 *Uncovering a new synthon for application in crystal engineering: the triply hydrogen-bonded ADA–DAD N—H···S/N—H···N/N—H···O synthon*

Wilhelm Maximilian Hützler (huetzler@chemie.uni-frankfurt.de)
Goethe-University Frankfurt am Main, Institute of Organic Chemistry and Chemical Biology

The results of an extensive co-crystallization study of derivatives of 2-thiouracil and uracil with coformers containing the 2,4-diaminopyrimidine substructure, respectively, are presented. Thus, in combination with searches of the Cambridge Structural Database, it is shown that the triply hydrogen-bonded ADA–DAD N—H···S/N—H···N/N—H···O synthon is a robust synthon for application in crystal engineering.

11 *Assessing Al-ion conductors from sulphides and selenides*

Falk Meutzner (falk.meutzner@physik.tu-freiberg.de)
TU Bergakademie Freiberg, Institut für Experimentelle Physik

Due to its high charge, aluminium is an interesting material for electrochemical energy storage applications which shall enable the growing penetration of renewables into the electric grid. Up to this point, no strong evidence for working materials, *e. g.* electrodes and solid electrolytes, was presented in literature that would eventually render this kind of system possible. We will present a search for interesting materials containing either sulphur or selenium that is based on different theoretical evaluation tools: Voronoi-Dirichlet partitioning, Bond-Valence Site energy and density functional theory calculations.

12 *Kinetics of gypsum phase ($CaSO_4$ * xH_2O) phase transitions*

Laura Ritterbach (laura.ritterbach@uni-koeln.de)
Section Crystallography, University of Cologne

In the atacama desert (Chile), $CaSO_4$ * xH_2O phases are abundant as the so-called gypsum crust. The transition between the different phases (gyp, bas, anh) depending on water activity, temperature and additive salts, as present in the desert, shall be studied with X-ray diffraction and Raman spectroscopy. Numerical modelling is intended to support laboratory work.

Lightning Presentations – Wednesday (Time Schedule)

1	11:00 –11:05	Khai-Nghi Truong	A "novel" ditopic substituted-acetylacetone ligand: Related to 3-(4-pyridyl)-acetylacetone but much more flexible
2	11:05 –11:10	Antje Hirsch	Monazite solid solutions – Model systems for ceramic radioactive waste ceramics
3	11:10 –11:15	Olufunso O. Abosede	Powder X-ray diffraction structural characterization of cis-[Co(phendione)$_2$Cl$_2$]
4	11:15 –11:20	Nils Nöthling	Two wavelengths in one source: The new high performance In- and Ga-MetalJet in Mülheim
5	11:20 –11:25	Thomas Köhler	Quantification of hydrogen in Li metal oxides
6	11:25 –11:30	Eike Markus Langer	Structural and Raman spectroscopic analysis of novel phases obtained in the Np oxo-selenate/oxo-selenite systems
7	11:30 –11:35	Uchenne P. Ogodo	Synthesis & characterization of some transition metal complexes of biologically relevant ligands
8	11:35 –11:40	Michael Haiduk	Lead & Lead-Free Halide Perovskite Solar Cells
9	11:40 –11:45	Jonas Werner	Synthesis & characterization of titanomagnetites
10	11:45 –11:50	David Mayer	Multiphoton Absorption in Metal-Organic Frameworks
11	11:50 –11:55	Alexander Pöthig	SC-XRD in Supramolecular Organometallics
12	11:55 –12:00	Tina Weigel	Determination of structure-property-relations from single crystal X-ray diffraction data

Lightning Presentations – Wednesday (Abstracts)

1 *A "novel" ditopic substituted-acetylacetone ligand: Related to 3-(4-pyridyl)-acetylacetone but much more flexible*

Khai-Nghi Truong (khai.truong@ac.rwth-aachen.de)
RWTH Aachen University

So-called "ditopic" ligands can coordinate to two different cations at the same time and are useful to design new materials acting as linkers between those cations. Research on N-donor functionalized derivatives of pentane-2,4-dione are of our interest. Here, we report the synthesis of one rather unexplored ligand, the structural characterization and its fascinating coordination behavior towards various metal salts.

2 *Monazite solid solutions – Model systems for ceramic radioactive waste ceramics*

Antje Hirsch (hirsch@ifk.rwth-aachen.de)
RWTH Aachen University

Monazite ceramics are promising nuclear waste forms for radioactive elements such as Np, Am, and Cm. Various solid solutions were prepared in forms of powders, single crystals, and ceramics. Different experimental techniques were combined to get an insight into the behavior of structural, vibrational, thermochemical, and physical properties of monazite.

3 *Powder X-ray diffraction structural characterization of cis-[Co(phendione)$_2$Cl$_2$]*

Olufunso Olumide Abosede (abosedeoo@fuotuoke.edu.ng)
Federal University Otuoke

This presentation reports the synthesis and powder X-ray diffraction structural characterization of cis-[CoII(κ2N,N'-1,10-phenanthroline-5,6-dione)$_2$Cl$_2$]. The antibacterial activity of this complex against Bacillus subtilis, Staphylococcus epidermidis, Staphylococcus aureus, and Klebsiella pneumonia is also presented.

4 *Two wavelengths in one source: The new high performance In- and Ga-MetalJet in Mülheim*

Nils Nöthling (noethling@mpi-muelheim.mpg.de)
Max-Planck-Institut für Kohlenforschung, Chemische Kristallographie und Elektronenmikroskopie

In this work, we report the first diffraction experiments using the In-rich ExAlloy I2 in our MetalJetD^{2+} X-ray source. The synchrotron-like photon flux densities of both radiation types has been investigated using a calibrated Si-pin-Diode. These results and our planned experimental setup for single crystal diffraction and electron density studies will be discussed.

5 *Quantification of hydrogen in lithium metal oxides*

Thomas Köhler (thomas.koehler@physik.tu-freiberg.de)
TU Bergakademie Freiberg, Institut für Experimentelle Physik

Hydrogen is a naturally impurity in all lithium tantalate crystals related to the growth conditions of the Czochralski method. In this study, normally pure congruent and hydrogen doped lithium tantalate crystals were analyzed by nuclear magnetic resonance (NMR) and elastic recoil detection analysis with a time-of-flight detector (TOF-ERDA) regarding their composition and hydrogen content. For a hydrogen occupation site model, the theoretical chemical shifts were calculated by density functional theory (DFT) and compared to the solid state 1H MAS NMR experiment.

6 *Structural and Raman spectroscopic analysis of novel phases obtained in the Np oxo-selenate/oxo-selenite systems*

Eike Markus Langer (e.langer@fz-juelich.de)
Forschungszentrum Jülich, IEK-6, Solid State Chemistry of Actinides Working Group

The investigation of the Np oxo-selenate/oxo-selenite systems in acidic media has resulted in unexpected redox behavior. Hereby the first Np(VI) selenates to date were found. Five novel phases obtained within this scarcely investigated system have been observed and structurally characterized by single crystal X-ray diffraction as well as spectroscopically by Raman measurements.

7 *Synthesis and characterization of some transition metal complexes of biologically relevant ligands*

Uchenne Patrick Ogodo (smartologyuche@gmail.com)
Federal University Otuoke, Department of Chemistry, Bioinorganic Chemistry Group

Transition metal complexes of some biologically relevant ligands have been synthesized and structurally characterized. The comparison of the solvent-free synthesis and solvent-based synthesis will be presented together with the structures of the complexes.

8 *Lead and Lead-Free Halide Perovskite Solar Cells*

Michael Haiduk (mhaiduk@uni-koeln.de)
University of Cologne, Department of Chemistry, Institute of Inorganic and Materials Chemistry

Atomic layer deposition (ALD) becomes attractive for organometal halide perovskite solar cells (PSCs) due to their unique possibility to generate pinhole-free, continuous and dense films with precise thickness control. First results will be presented.

9 *Synthesis and characterization of titanomagnetites*

Jonas Werner (jonas.werner@rwth-aachen.de)
RWTH Aachen University

Many paleomagnetic studies on natural samples depend on the magnetic properties of titanomagnetites. Recently, it was discovered that their Curie temperature is a function not only of their composition but also reflects their thermal history. Studying synthetic samples may provide a way to gain further insight into the complex relationship between the magnetic properties and the thermal background.

10 *Multiphoton Absorption in Metal-Organic Frameworks*

David Mayer (david.mayer@tum.de)
Technische Universität München, Fakultät für Chemie

Solid state hybrid materials, namely metal-organic frameworks and coordination polymers appear to be highly promising novel non-linear optical materials, especially in the field of multi-photon absorption. We study these properties by using a design based approach guiding the MOF crystal engineering to access new materials which are likely to outperform any known solid-state material in this field, so far.

11 *SC-XRD in Supramolecular Organometallics*

Alexander Pöthig (alexander.poethig@tum.de)
Technische Universität München, Catalysis Research Center

Discreet complex organometallic scaffolds can be used in supramolecular applications. Examples of the determination of their structure by means of SC-XRD (including host-guest interactions) are presented and discussed.

12 *Determination of structure-property-relations from single crystal X-ray diffraction data*

Tina Weigel (tina.weigel@physik.tu-freiberg.de)
TU Bergakademie Freiberg, Institut für Experimentelle Physik

For the optimal determination of structure-property-relations, *e. g.* pyroelectricity, is a high-quality structural characterization in combination with a measurement of physical properties necessary. This work shows an approach, where single crystal X-ray diffraction data are used for estimation of material properties without additional measurements. Therefore, in a temperature range from 80 K to 400 K structural parameters are determined and used for the estimation of temperature depending material parameters, which show a high comparability with theoretical and experimental data.

List of Participants

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AK Junge Kristallographen
http://dgk-home.de/aks/jkyc/darmstadt-2017/