

User Regulations

Core Facility

Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI)

**Faculty of Chemistry – Inorganic and Structural Chemistry
University of Bielefeld**

§1 General

The Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI) is a research infra-structure centre located at the University of Bielefeld within the Faculty of Chemistry. It is managed by Prof. Norbert W. Mitzel, Chair of Inorganic and Structural Chemistry.

§2 Mission

The Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI) has been established to provide access and service to the scientific community, for the determination of molecular structures of small molecules. This includes gaseous or vaporizable samples for gas electron diffraction work and a range of crystallization techniques for samples which are liquid at ambient temperature preventing standard crystallography methods to be applied.

GED@BI offers electron diffraction measurements of samples provided by collaborators on a state-of-the-art instrument and complete data analysis for structure determination including documentation.

GED@BI also offers in-situ crystallization of low melting liquids or gases for structure determination in the solid phase by X-ray crystallography. There is also the possibility of determination of electron density distribution of such samples.

GED@BI maintains and develops the method of gas electron diffraction further in terms of improving instrumentation and continuously improving the methods of data analysis and automation of these procedures.

GED@BI provides access to gas electron diffraction equipment and in-situ crystallization devices for scientists with specialized problems, which need to be solved in close on-site collaboration and construction of dedicated pieces of apparatus.

GED@BI provides training on the techniques to young academics and for interested collaborators.

GED@BI also offers accompanying spectroscopic measurements and quantum-chemical calculations in the context with structure determination of small molecules.

§3 Information

The Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI) runs a web-page providing information such as

- Contact details
- these User Regulations
- general Information on the method and links to relevant sources of information, in particular structural databases, technical and theoretical background, other labs and their area of specialization, publication lists of GED@BI
- technical information on sample submission and safety issues

§4 Instrumentation

The Core Facility Gas Electron Diffraction (GED@BI) operates the following instrumentation:

- Gas Electron Diffractometer KDG2-Eldigraph
 - o Updated between 2006 and 2011
 - o Image Plate Detection
 - o STAIB electron gun 100 kV, 50 μ A
 - o Nozzle for measurements at ambient temperature
 - o Nozzle for measurements up to 400°C sample temperature
 - o Nozzle with glass inlet for sensitive materials
- Fuji Image Plate Scanner BAS1800
- In-situ crystallization devices for low-melting liquids or gases and crystal structure determination with:
 - o OHCD (Optical Heating Crystallization Device, Sci-Consult)
 - o Miniaturized crystallization from solid-liquid equilibria
 - o Nonius Kappa CCD X-ray diffractometer for data collection
 - o SuperNova A (Dual) diffractometer system SuperNova Platform with Atlas CCD Nova (Cu) and Mova (Mo) X-ray Sources
- Computer-Cluster with 8 nodes and software for electron diffraction data analysis, various quantum chemical program packages and other scientific software

The instrumentation is continuously updated, improved and refined and can be adapted to specialized scientific problems.

The Core Facility Gas Electron Diffraction (GED@BI) has access to instrumentation of the Chair of Inorganic and Structural Chemistry and the Faculty of Chemistry in Bielefeld and can offer accompanying measurements, namely various NMR and IR spectrometers.

§5 Quality of Results

The Core Facility Gas Electron Diffraction (GED@BI) has internal standards of quality assurance in terms of measurement precision and data analysis. Experiments are generally directly and individually calibrated in order to guarantee the reliability of the data.

The collaborator must contribute to data quality in the sense that sample purity and stability have to be guaranteed. If this is difficult under circumstances given by the nature of the sample, the resulting limitation of reliability of results has to be jointly considered.

§6 Staff

The Core Facility Gas Electron Diffraction (GED@BI) has staff to provide the offered services:

- Management
- Permanent scientific staff securing the maintenance of expertise the continuity of operation and access and development of the method
- Permanent technical staff securing the up-to-date level of instrumentation continuity of operation
- Doctoral students and post-doctoral workers receiving training on the method and developing certain aspects of the method or determining the structures of certain classes of compounds
- Guest scientists collaborating closely on specialized problems

§7 Service

The Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI) offers the complete service for the determination of molecular structures of free molecules of gaseous or vaporizable samples. This service includes

- Guidance and advice for collaborators interested in gas electron diffraction
- Sample handling and preparation for measurements (see also technical appendix)
- Electron diffraction measurements on a state-of-the-art instrument
- Data reduction
- Definition of appropriate models and implementation of such models into data analysis software
- In-situ crystallization experiments for liquid samples to be examined by X-ray diffraction, optionally determination of experimental electron density distributions
- Calculation of supporting information for data analysis such as vibrational and centrifugal distortion corrections, thermodynamic data for conformational analysis geometrical parameters for restrained data analysis
- Data analysis, also under consideration of complementary available information
- Data preparation for storage of the structural (and other) data and submission to databases
- Documentation of the scientific results in reports or contributions to publications

These parts of work can be fully provided by the Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI), but it is also possible to arrange alternative forms of collaboration. This is in particular

- Providing access to the instrument to trained staff from collaborators in cases of long-term collaboration or special needs, e.g. the handling of explosive materials, which needs specially trained staff for safety reasons.
- Provision of training for collaborators to gain the required technical knowledge to

- perform in part or fully the experiment and data analysis procedures
- Provision of access to software for data analysis

In all cases GED@BI offers support in administrative issues and in supporting collaborators in access to the GED@BI facilities (support in transportation issues, search for accommodation if collaborators need to have physical presence at GED@BI etc.)

§8 Costs

The Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI) is a research infra-structure for fundamental research, primarily intended to provide structural determination service to academic institutions, but is accessible to all potential users including industry. GED@BI is operated as a non-profit financed research infra-structure unit on the basis of full costing.

It is expected that collaborators contribute to the costs involved in this service in a way that the centre can maintain its operation ability and development. To achieve this the following rules apply.

- As a general rule, collaborators will compensate / contribute to the costs either directly (in particular applicable to industrial users) or in a form refinanced by the user by including the costs into their own research applications for project funding; costs depend on the complexity of the system to be analyzed and finally on the time spent on the individual problem. Costs will be calculated according to the EU state aid framework and are composed of instrument cost, personnel expenses and overheads. As first crude estimates, for electron diffraction a range between EUR 7000 to 20000 is to be expected for structure determinations, depending on complexity (this covers staff costs, instrument time and consumables plus overhead). In very complicated cases such costs can be exceeded. A more precise estimate, based on an expert's opinion, will be provided by the centre on request. In-situ crystallization experiments including measurements will approximately cause costs between 1000 and 5000 EUR. (Amounts are sums estimated in July 2012 and will be adapted from time to time).
- for larger series of measurements or projects including a bigger part of gas diffraction collaborators are encouraged to apply jointly with GED@BI for third-party project funding
- small initializing projects will be (pre)financed by GED@BI, if joint application for funding is subsequently intended; this is to foster further collaboration and subsequent acquisition of external funding.

§9 Priority

By the nature of the gas-diffraction experiment and analysis dictates that it can be a time-consuming task and collaborators have to allow for that. As a rule of thumb, analysis of the data for simple structure can be done within weeks, while complicated cases will sometimes take considerably longer. The success of in-situ crystallization experiments cannot be predicted exactly, and they are also time-consuming. For this reason they have also to be prioritized.

- prioritizing the individual structure determination projects with the permanent staff involved is generally done in the order of request
- structure determination projects with additional externally funded staff are independent on the above queuing have their own priority rules
- exceptions will be decided by the management of GED@BI and are only on scientifically based reasons, e.g. prioritizing a measurement because the sample is unstable.

§10 Safety

Safety during experiments is of highest priority. Therefore collaborators have to provide any known information on safe handling of the compounds.

This applies in particular to

- o toxicity
- o fire-hazard if pyrophoric or inflammable
- o explosion hazard also in combination with the necessity of heating
- o material compatibility with the materials the instrument is made of
- o decomposition under various conditions like
 - air-contact
 - temperature
 - light etc.
- o safe disposal of residual sample or unsuitable samples
- o as part of the sample deposits on the inner parts of the instrument during the experiment and measures have to be considered how to clean these without danger to the health of the experimenting persons
- o safety in transport of the samples to the GED@BI lab
- o safety in transport of the samples if they have to be returned

§11 Good scientific practice

Both, collaborators and staff of GED@BI, commit themselves to obey the rules of good scientific practice as are outlined in the DFG document “Safeguarding good scientific practice”

(accessible under:

http://www.dfg.de/download/pdf/dfg_im_profil/reden_stellungnahmen/download/empfehlung_wiss_praxis_0198.pdf.)

This applies in particular to the following points:

- storage of primary data and subsequent data analysis
- consistently questioning the findings
- practising strict honesty with regard to the contributions of partners, competitors
- confidentiality of information on sample material and data unless otherwise agreed one
- the obligation to publish the results in time, unless other (legal) reasons apply
- the obligation of respecting rights in the treatment of intellectual property

The high scientific input in data analysis of gas electron diffraction will in general justify co-authorship of those persons involved in this process. The management of GED@BI will take care that the number of persons involved in experiment and data analysis will be restricted to the scientifically justified number. There will be no co-authorship rights for those, involved in purely technical standard procedures, but instead the right to be mentioned in acknowledgements of publications (e.g. "We would like to acknowledge the assistance of the Core Facility Gas Electron Diffraction and Small Molecule Structures Centre, Bielefeld (GED@BI), Germany"). Any deviations from these rules have to find the agreement of all persons involved, those who provide the sample and those who do structural analyses.

Bielefeld, July 10th 2012



Prof. Norbert W. Mitzel