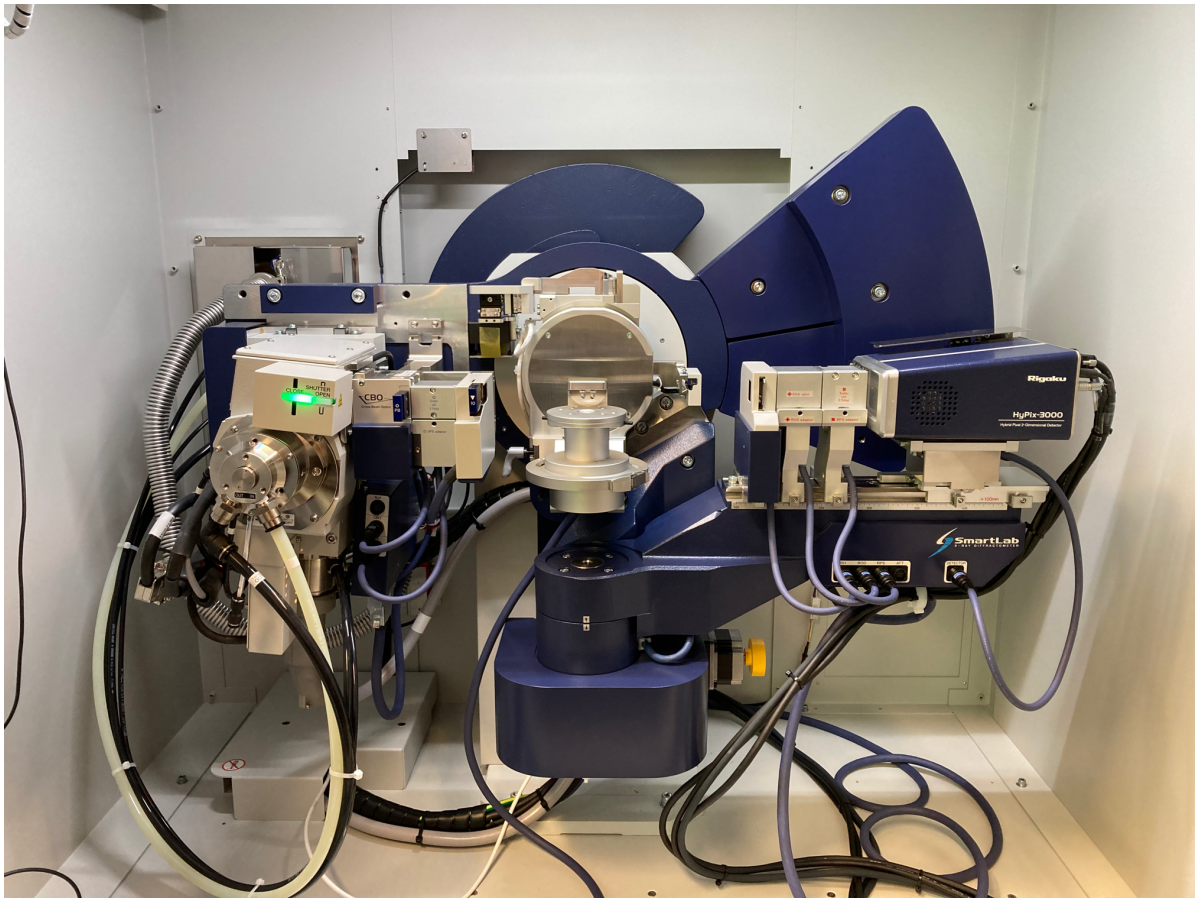
Rules and Procedures

for Users of the Rigaku Smartlab
High Resolution X-ray Diffractometer of the
Nanoparticle Process Technology Group



Short Note

Markus Winterer

Universität Duisburg-Essen, 01.09.22

Availability

The instrument is available to users outside the Winterer group upon request. Advanced support is possible in a collaborative manner (including coauthorship).

Application and Scheduling for Measurement Time

You may apply for measurements time by sending an e-mail to

nppt_smartlab@uni-due.de

In your e-mail you should describe shortly

- your samples (composition and other information available for example about phase or structure) including hazards,
- scientific goal of your experiment
- method of the Rigaku Smartlab you intend to use

We will then contact and discuss with you before scheduling your experiment.

Costs

The instrument is dedicated to academic use. Members of academic groups are encouraged to apply for measurement time and to use it for academic purposes according to the rules described in this document. Users will be asked to participate fairly in costs for maintenance and consumables.

User Modes

- **one time user / few measurements per year**: we will perform the measurement for you as a service or in collaboration depending on the complexity of the experiment
- **frequent user:** after training by an expert user, one dedicated person per scientific group will be allowed to perform measurements her- / himself
- **expert user** is a trained NPPT group member. Only expert users are allowed to train other users.

Training Requirements for frequent users before independent operation of the instrument

- training by expert users (as defined above)
- radiation safety training (by the radiation safety officer of NPPT: Prof. Dr. Frank Schmidt, from January 2023: M. Eng. Maximilian Stepponat)
- compliance with NPPT laboratory rules (see 'Laborordnung' document at the NPPT secretariate)

Measurements

- The instrument should be used with care. Please contact the supporting scientist or engineer immediately / first if questions or problems arise.
- Frequent users of powder diffraction are kindly asked to order their own sample holders/ capillaries from Rigaku Rigaku Europe SE, Hugenottenallee 167, 63263 Neu-Isenburg.
- Please note information about your measurement in the **NPPT Smartlab Logbook:**

		User	**Sample**	**Instrumental parameters**				
Date	**time**	**Name and group**	**Phase / Chemistry**	**Anode voltage and current**	**anode vacuum**	**cooling water *T* and dV/dt**	**measurement mode**	**comment**

Data Transfer

- The instrument computer should only be used for measurements, not for data storage. Please transfer your data after measurement to the XRD Rackstation dedicated for data storage of NPPT Smartlab data. It is reachable within the uni-due network. For users of the NPPT Smartlab, a user account for the XRD Rackstation will be created. Log-in details will be sent subsequently via e-mail.

Data Analysis

- Data analysis may be performed with your favorite software or with software provided by Rigaku. The Rigaku software including access to the crystallographic data bases runs on Windows. Software download, installation instructions as well as software license (1 out of ten) are described in the following:
  - Obtain the IP address of your Windows computer, for example using the following steps
    - click on 'Windows-Symbol'
    - click on 'Einstellungen'
    - click on 'Netzwerk und Internet'
    - click on 'Ethernet'
    - double click on 'Netzwerk'
    - 'IPv4-Adresse' is the information needed
  - Write an e-mail to 'nppt_smartlab(at)uni-due.de' with the request for the XRD-software (SmartLab Studio II) including your IPv4 address
  - Download the required installation files from the XRD Rackstation (data server)
  - Install SmartLab Studio II on your Windows computer
    - Open the 'Launcher' file
    - click on 'Install SmartLab Studio II'
    - Choose 'For Analysis ONLY' → 'Next'
    - Select 'Auto'
    - After the installation click 'Back'
    - and 'Exit'
  - Open the 'CodeMeter Control Center' software
    - Click the 'WebAdmin' button

    - Your default browser is launched
    - select ‘Configuration’
    - and ‘Add new server’
    - Enter the IP address 134.91.120.49
    - click ‘Add’
    - click ‘Apply’
- Upon starting the software ‘SmartLab Studio II’ you will be informed that there is no license available for the database. This information can safely be ignored!

Support

- Scientific support is provided by Dr. Martin A. Schroer martin.schroer@uni-due.de
- Technical Support is provided by M. Eng. Maximilian Stepponat (0203-37 9-3420); maximilian.stepponat@uni.due.de

Funding

The instrument is funded through the DFG (German Research Foundation) instrument proposal INST 20876/395-1 FUGG project number 450350889 and the state of North Rhine-Westfalia, Germany.

Acknowledgements

If you publish or present data collected with NPPT’s Rigaku Smartlab High Resolution X-ray diffractometer, please acknowledge the funding and any non-collaborative support as:

‘The X-ray data were collected using the Rigaku Smartlab High Resolution X-ray diffractometer of the Nanoparticle Processing Technology (NPPT) group at the University Duisburg-Essen. The instrument is funded through the DFG (German Research Foundation) Instrument proposal INST 20876/395-1 FUGG project number 450350889 and the state of North Rhine-Westfalia, Germany. We would like to thank N.N.** for assistance in using the instrument.’

** please include the name of the scientist supporting you.

Appendix: Specifications of the Rigaku Smartlab High Resolution X-ray Diffractometer of the Nanoparticle Process Technology Group

Instrument

The Rigaku Smartlab High Resolution X-ray diffractometer (launch 2018; installed at NPPT in 5/2022) is equipped with

Source

9 kW rotating anode X-ray generator using a Cu-Anode

Optics

- Cross beam optics (CBO) for divergent and parallel beam operation
- CBO-μ-optics for spots size of ~ 100 μm: converts line to point focus while conserving high beam intensity
- Ge(220) and Ge(400) double monochromators for high angular resolution measurements
- Ge(220) and Ge(400) double analysator crystals
- collimators for beam shaping

Sample stages & measurement modes

- X-Y- and RxRy-sample stages
- theta-theta goniometer with in-plane goniometer arm
- multifunctional Euler cradle and rotation stage
- sample stage for micro-XRD (including mapping)
- sample changer with 10 positions including sample rotation (in reflection mode)
- different sample holders for powder samples including Si single crystals (zero background)
- components for SAXS, 2D-SAXS/WAXS, U-SAXS and GI-SAXS
- capillary spinner

Detectors

- Hypix-3000-2D detector for 0D-, 1D- and 2D mode measurements
- observation camera (including mapping)

DAQ, Analysis & Accessories

- control computer
- control and analysis software
- integrated crystallographic data base: COD & ICDD-PDF-2
- calibration samples

Science

Typical sample types and structural information obtainable are

- nanoscaled powders and solids (crystal-, microstructure, phase analysis, and texture)
- thin (polycrystalline and epitaxial) films (crystal-, microstructure, phase analysis, and texture),
- stress, thickness, roughness, and coherence
- colloidal nanoparticles (microstructure).

Methods and measurement modes available:

- X-ray powder diffraction (XRD)
- microdiffraction, including mapping
- Small- and wide angle scattering in transmission (2D: SAXS, WAXS; 0D: SAXS, USAXS)
- Small- and wide angle scattering in grazing incidence (GISAXS, GIWAXS)
- Texture determination (pole figures, ODF)
- high resolutions diffraction
  - stress determination
  - reciprocal space map (RSM)
  - high resolution rocking curves (HRRC)
  - reflectometry (XRR)