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for developing new horizons for RIs

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Contents

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Report on the 2nd International Workshop

1 Abstract

X-ray Absorption Spectroscopy (XAS) is one of the most widespread techniques at synchrotron X-ray facilities with applications across many scientific disciplines with an increased interest by industry, most notably in catalysis, battery research, and material science. This class of experiments requires complex sample environments for *in-situ* and *in operando* studies, a high level of automation, mail-in services, remote access capacities, and automated data analysis. We will develop a *Conceptual Design Report* (CDR) for a generic automated XAS beamline for catalysis research that describes the principles of all the above requirements. After successfully creating a Conceptual Design Report during the last year, we now organized a 2nd workshop in order to discuss the CDR and possible realization schemes with a large user/customer community.

2 Motivation for Automated X-ray Absorption Spectroscopy for Catalysis

Catalysis is one of the key technologies for tackling major challenges within the current energy policy targeting energy sovereignty, reduction of the dependence on fossil and nuclear fuels, and increased use of sustainable and renewable fuels. In general, investigating a catalyst implies analyzing the relationships between the activity of a functioning catalyst under realistic conditions and its synthesis route as well as structure before, during, and after the catalysis [1]. The investigation of catalytic processes facilitates efficient conversion processes among all kinds of application areas:

- Energy (distribution and storage of chemical energy carriers) [2, 3],
- Green Chemistry (closing the carbon cycle) [4, 5],
- Earth & Environment (bio-catalysis as part of biotechnology) [6, 7], and
- Air & Space Transportation (synthetic fuels) [8, 9].

The technically most important elements for catalytic experiments are the transition metals and lanthanides. In order to study the underlying processes and, thus, changes in short and long range order within the (crystal) structure, spectroscopic experiments are perfectly suited. ***X-ray Absorption Spectroscopy*** (XAS) analyses the energy dependent intensity changes in the vicinity of an absorption edge. The local atomic environment determines significant details of the energy dependent intensity, especially for transitions concerning the inner most shells (K and L edges), see Fig. 1.

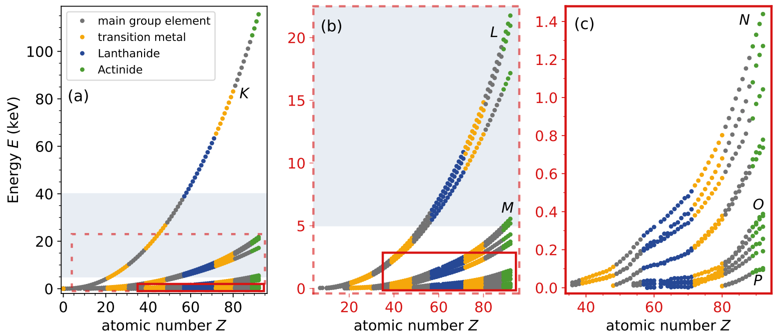

Figure 1: Absorption edge energies in dependence of the elements [10]. The gray area highlights the energy range covered by a typical hard X-ray spectroscopy beamline.

As briefly mentioned, catalytic experiments typically depend on numerous factors. Currently, the complete experimental coverage of this enormous parameter space is highly time consuming and requires a vast degree of manual work. An increase of **automatization** will be accompanied by reduced effort and time consumption for exploring the parameter space as well as the standardization of data management (FAIR principles [11]). The principles feature the machine-accessibility, which, on the one hand, facilitates the structured analysis of own data, and on the other hand the possibility to compare with data of other scientists.

Based on those highly standardized data, **machine learning** could not only reduce the reasonable parameter space while measuring, but also improve data quality by improving measurement conditions during operation. In turn, this will enhance the data quality and the sample throughput.

In general, these improvements will increase the attractiveness to industry and non-expert users and, thus, speed-up the innovative process within Germany. Further, an increase of remote and mail-in experiments will be facilitated.

3 Preparation of the 2nd Workshop

The 2nd workshop was again organized within the framework of the *DESY Photon Science Users' Meeting* (22nd to 26th January 2024) in order to share the results of the CDR published within Deliverable D4.23. By experience from the 1st workshop [12], a large part of the LEAPS community is regularly participating in this meeting making it the perfect platform to share the results.

In order to make the best use of possible synergy effects, the 2nd workshop was created in collaboration with the organizers of the regular XAS satellite workshop (focusing on beamlines P64 and P65) as well as the ROCK-IT project [13],

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which has a highly similar scope to the current part of task 4.6. The ROCK-IT project focuses on evaluating and developing all necessary tools for the automation and remote-access, also of more complicated *in-situ* and *operando* XAS experiments. Thus, the workshop will be held in two parts in the dedicated satellite meetings "XAFS at P64/65 and perspectives for future PETRA III and IV beamlines" and "ROCK-IT Outreach Meeting & Workshop".

4 Conclusion

All parts of the workshop attracted many interested participants, both on-site as well on-line. A total of 52 people participated in the P64/P65 satellite meeting (19 of them online) and 55 people in the ROCK-IT session (21 online). A large number of participants than usual joined the meeting online, mainly due to the public transport strikes.

Task 4.6 member M. Nentwich presented the results of the CDR concerning the future of the P65 beamline during the satellite workshop about beamlines P64 and P65 as well as during the Poster Session, see Fig. 2. In contrast, the ROCK-IT meeting focused more on the automation side and user requirements. This outreach meeting was also a great opportunity to start a collaboration between ROCK-IT and the EURIZON member ELI ERIC [15]. This possible collaboration became obvious during the Final Meeting of EURIZON when the progress of the individual task was presented and Dr. Anna Zymaková from the Department of Structural Dynamics at ELI ERIC asked for more detailed information about the automation status and possible knowledge transfer to her spectroscopy beamline.

Figure 2: Melanie Nentwich discussing her Poster [14] during the Poster Session of the DESY Users' Meeting. Foto by Marta Mayer, DESY.

During all three discussion opportunities during the *Users' Meeting* many aspects of the Conceptual Design report were discussed with a large user/customer community including various LEAPS members, *e. g.* DESY, HZB, and HZDR. These discussion covered both the *x-ray tracing* (xrt) results for P65 as well as the flow chart for XAS beamline automation. The discussion added valuable ideas to further improve presented results in the framework of various projects as the future continuation of the EURIZON project, ROCK-IT, and PETRA IV. These ideas include some aspects concerning the flow chart, but also user feedback on the priority and impact of automation.

The discussions about the flow chart included the hierarchical structuring in phases, processes, tasks, and sub-tasks was discussed. Possible phases and processes have already been specified; a new version of the flow chart is in progress, see Fig. 3. In addition, a further control step was proposed that checks if the test conditions can be met with the current setup, including checking whether

- all the required gases are available,
- all the required samples are in the magazine, and
- the connected devices can supply the required temperatures, gas flows, etc.

The user feedback included improvements on the currently existing user interfaces as well as comments on the automation with respect to its impact and its prioritization. The user community emphasized the wish for a well organized and clearly structured graphical user interface. So far, the considerations about automation only included functionality, but not simple and intuitive application, which are important aspects for new users. The community also stressed a clear prioritization of the safety aspects including a control of the manual entries by the machine also comprising the exception handling that was already discussed in the Conceptual Design Report of Deliverable D4.23. The users see the largest impact of automation in the accessibility of the synchrotron facility in general and the individual analysis method (here XAS) in particular for non-expert users. This does not only include the conduction of the experiment, but also the data analysis. Therefore, a strong and reliable online data analysis with accompanied storage of the reduced data also has a large priority. The already active user community highlighted their wish to interact with the experiment control and to make changes on the schedule if needed. Therefore, the control system should not only accept changes on the schedule from the machine learning tool included in the online analysis, but also from a human user.

In summary, the Conceptual Design Report was very positively received by the community and constructive suggestions for improvement were added in detail. Especially the ROCK-IT project will strongly benefit from these recommendations.

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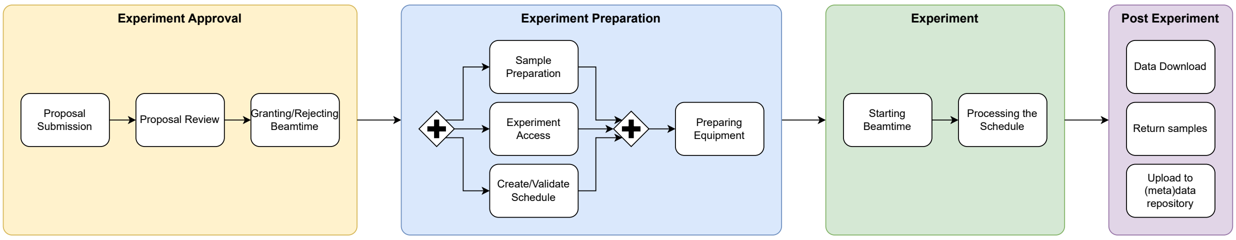
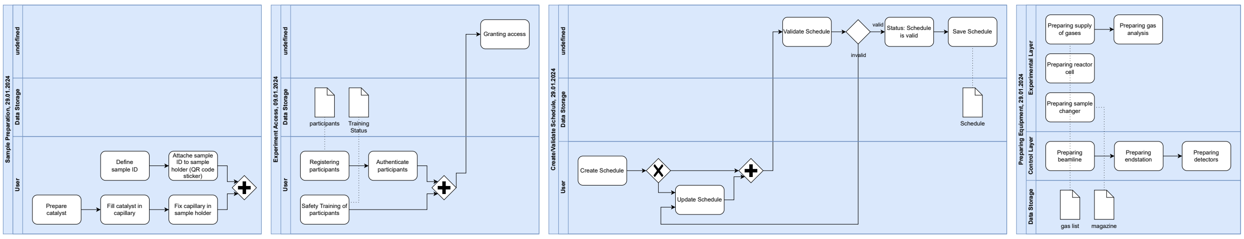


(a) Phases and processes.



(b) Processes and tasks of the experiment preparation phase.

Figure 3: Preliminary flow chart of the phases, processes, and tasks of the experiment preparation phase of an *in-situ* XAS measurement.

5 Acknowledgements

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References

[1] DECHEMA e. V. Homepage of NFDI (Nationale Forschungsdateninfrastruktur) for Catalysis related Science. https://nfdi4cat.org, 6 2022.

[2] M. Wu, G. Zhang, H. Tong, X. Liu, L. Du, N. Chen, J. Wang, T. Sun, T. Regier, and Sun. S. Cobalt (II) oxide nanosheets with rich oxygen vacancies as highly efficient bifunctional catalysts for ultra-stable rechargeable Zn-air flow battery. *Nano Energy*, 79:105409, 2021. 10.1016/j.nanoen.2020.105409.

[3] E. Marini, D. O. De Souza, G. Aquilanti, M. Liebert, F. Rossi, and B. Bozzini. Operando XAS of a Bifunctional Gas Diffusion Electrode for Zn-Air Batteries under Realistic Application Conditions. *Applied Sciences*, 11:11672, 2021. 10.3390/app112411672.

[4] B. Yang, H. Li, Z. Zhang, K. Xiao, M. Yang, F. Zhang, M. Wang, X. Guo, Q. Li, W. Fu, R. Si, L. Wang, and H. Chen. Nickel dual-atom catalysts for the selective electrocatalytic debromination of tribromoacetic acid as a green chemistry process. *Chemical Engineering Journal*, 427:131719, 2022. 10.1016/j.cej.2021.131719.

[5] M. Guo, M. J. Gray, H. Job, C. Alvarez-Vasco, S. Subramaniam, X. Zhang, L. Kovarik, V. Murugesan, S. Phillips, and K. K. Ramasamy. Uncovering the active sites and demonstrating stable catalyst for the cost-effective conversion of ethanol to 1-butanol. *Green Chemistry*, 23:8030, 2021. 10.1039/D1GC01979A.

[6] Y. Injongkol, P. Khemthong, N. Yodsin, Y. Wongnongwa, N. Sosa, S. Youngjan, T. Butburee, B. Rungtaweevoranit, S. Kiatphuengporn, J. Wittayakun, F. Roessner, and S. Jungsuttiwong. Combined in situ XAS and DFT studies on the role of Pt in zeolite-supported metal catalysts for selective n-hexane isomerization. *Fuel*, 314:123099, 2022. 10.1016/j.fuel.2021.123099.

[7] L. J. A. Macedo, A. A. E. Santo, G. C. Sedenho, A. Hassan, R. M. Iost, G. T. Feliciano, and F. N. Crespilho. Three-dimensional catalysis and the efficient bioelectrocatalysis beyond surface chemistry. *Journal of Catalysis*, 401:200, 2021. 10.1016/j.jcat.2021.07.022.

[8] M. Chen, D. A: Cullen, S. Karakalos, X. Lu, J. Cui, A. J. Kropf, H. Mistry, K. He, D. J. Myers, and G. Wu. Single Atomic Iron Site Catalysts via Benign Aqueous Synthesis for Durability Improvement in Proton Exchange Membrane Fuel Cells. *Journal of the Electrochemical Society*, 168:044501, 2021. 10.1149/1945-7111/abf014.

[9] J. Kang, Q.-Y. Fan, W. Zhou, Q. Zhang, S. He, L. Yue, Y. Tang, L. Nguyen, X. Yu, Y. You, H. Chang, X. Liu, L. Chen, Y. Liu, F. Tao, J. CHeng, and Y. Wang. Iridium boosts the selectivity and stability of cobalt catalysts for syngas to liquid fuels. *Chem*, 8:1050, 2022. 10.1016/j.chempr.2021.12.016.

[10] A. C. Thompson, D. Lindau, I. Attwood, Y. Liu, E. Gullikson, P. Pianetta, M. Howells, A. Robinson, K.-J. Kim, J. Scofield, J. Kirz, J. Underwood, J. Kortright, G. Williams, and H. Winick. *X-Ray Data Booklet*. Lawrence Berkeley National Laboratory, 2009.

[11] Go FAIR. Homepage of Go FAIR. https://www.go-fair.org/, 1 2022.

[12] Deutsches Elektronen-Synchrotron DESY. DESY Photon Science Users' Meeting 2023 – Overview Satellite Meetings. https://indico.desy.de/event/36974/page/4290-overview-satellite-meetings, 01 2023.

[13] Deutsches Elektronen-Synchrotron DESY. ROCK-IT Homepage. https://www.rock-it-project.de/project/about/, 1 2024.

[14] M. Nentwich, O. Seeck, M. Krisch, and E. Welter. Analysing the mirror placement of the future PETRA IV AppAnaXAFS beamline with x-ray tracing, 1 2024. Poster 258, https://indico.desy.de/event/41899/attachments/87868/117547/BoA_V4.pdf.

[15] ELI ERIC. Homepage of Extreme Light Infrastructure (ELI). https://eli-laser.eu/, 1 2024.

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Supplement

SATELLITE WORKSHOP - Photon Science

XAFS at P64/65 and perspectives for future PETRA III and IV beamlines

Thursday, 25 January 2024

The XAFS satellite meeting will place emphasis on assessing the status of the operational PETRA III XAFS beamlines, P64/65, future developments for the P63 beamline and the status of planning for future PETRA IV beamlines. A number of showcases of recent work conducted by diverse user groups will give insight into the performance of the current beamlines at PETRA III. Additionally, the current planning status for XAFS beamlines at PETRA IV and a preview of the upcoming XAFS/XRD beamline, P63 will be presented. A separate workshop on Wednesday will focus on the automation of operando (XAFS-) experiments which is currently developed in the context of the ROCK-IT project.

Organisers: E. Welter, A. Kalinko, W. Caliebe Contact: wolfgang.caliebe@desy.de

PROGRAMME

		Chair: M. Naumova
9:00	Introduction	
9:05	Status and Future of P65	E. Welter, M. Nentwich
9:25	Status and Future of P64	A. Kalinko
9:45	Status and Future of P63	W. Caliebe
10:05	Valence-to-Core XES spectroscopy of Ge complexes in aqueous fluids up to 400°C	A. Loges
10:25	Coffee	
10:45	From solid solutions to high-entropy materials: probing local structure in tungstates using X-ray absorption spectroscopy and reverse Monte Carlo simulations	A. Kuzmin
11:05	Peculiar bond length dependence and its impact on the band gap bowing in (Ag,Cu)(In,Ga)Se2 thin film alloys	H. Falk
11:25	X-ray spectroscopy in P64/P65 beamlines for chemical research	M. Nowakowski
11:45	Tracking and steering the dynamic structures of single atom catalysts for electrocatalytic CO2 reduction reaction using operando XAS.	J. Timoshenko
12:05	UV to mid-IR Pump-Probe Setup at P64 for Transient X-Ray Absorption Spectroscopy of Electronic and Structural Degrees of Freedom	T. Glier

SATELLITE WORKSHOP - Photon Science

ROCK-IT Outreach Meeting

Wednesday, 24 January 2024

ROCK-IT aims to develop all necessary tools for the automation and remote control of in-situ and operando catalysis experiments. These include remote access for experimental users, experiment automatization, data management, and near real-time analysis and optimization using machine learning (ML).

The workshop will start with a brief introduction to the ROCK-IT project. Afterwards, we will engage the user community to discuss and gather valuable feedback about the following topics:

- Graphical user interface (GUI) design of the user experience
- GUI / Experiment control / Experiment automation
- Data analysis
- Remote access

The workshop is designed to be highly interactive, prioritizing active engagement from the user community to communicate their preferences and requirements.

ROCK-IT (**R**emote, **O**perando **C**ontrolled, **K**nowledge-driven, and **IT**-based) is a Helmholtz collaboration project between DESY, KIT, HZDR, and HZB.

Organisers: Zeynep Isil ISIK DURSUN, Mehdi KAZEMI Contact: zeynep.isik.dursun@desy.de

PROGRAMME

13:00	**Welcome**
13:05	**Introduction to ROCK-IT and Meeting Objectives** (Edgar Weckert - Director in Charge of Photon Science, DESY)
13:30	**Outreach Agenda and Opening Questions**
14:00	**Coffee Break and Discussions**
14:40	**User Impressions and Discussions**
16:00	**Coffee Break**
16:15-16:30	**Conclusion**

Figure 4: Agendas of the two parts of the 2nd Workshop.

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