XAFS16 Satellite Meeting - Data acquisition, treatment, storage – quality assurance in XAFS spectroscopy

Friday 21 August 2015 - Saturday 22 August 2015

DESY Hamburg

Programme

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XDI: An XAFS Data Interchange format

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We propose a standard data format for the interchange of XAFS data. The XAFS Data Interchange (XDI) standard is meant to encapsulate a single spectrum of XAFS along with relevant metadata. With this standard, we aim to meet the following goals:

- Establish a common language for transferring data between XAS beamlines, XAS experimenters, data analysis packages, web applications, and anything else that needs to process XAS data.
- Increase the relevance and longevity of experimental data by reducing the amount of *data archeology* future interpretations of that data will require.
- Enhance the user experience by promoting interoperability among data acquisition systems, data analysis packages, and other applications.
- Provide a mechanism for extracting and preserving a single XAS-like data set from a related experiment (for example, a DAFS or inelastic scattering measurement) or from a complex data structure (for example, a database or a hierarchical data file used to store a multi-spectral data set).
- Provide a representation of an XAS spectrum suitable for deposition with a journal or in a database.

In short, we aim to share data across continents, decades, and analysis toolkits.

XDI is a text-based format with a simple syntax which clearly delineates metadata from the data table in a way that is easily interpreted both by a computer and by a human. The metadata header is inspired by the format of an electronic mail header, representing metadata names and values as an associative array. The data table is represented as columns of numbers. This format can be imported as is into most existing XAFS data analysis, spreadsheet, or data visualization programs.

XDI is implemented as a project at GitHub [1]. It includes a specification and a dictionary of common XAFS metadata. Implementations of the specification are provided in Fortran, C, Python, and Perl. The C wrapper provides functions for recognizing and importing data in the XDI format, validating the content of the data table, and validating that the metadata meets the requirements and definitions found in the XDI metadata dictionary. The Python and Perl interfaces are implemented as thin wrappers around the C interface. In this way, it will be easy to provide compliant interfaces in new languages as needed.

References

[1] https://github.com/XraySpectroscopy/XAS-Data-Interchange (accessed 26 May, 2015).

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Progress Toward Improved XAFS Data Libraries

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Libraries of XAFS Spectra have high potential value for many studies using XAFS. Since good samples for many materials of interest are challenging to obtain, and because high-quality XAFS can be challenging to measure for some systems, having readily available spectral libraries can increase the general quality of many analyses, and also provide canonical spectra for a variety of materials to better enable comparisons of beamlines, analysis methods and theoretical calculations. Though a few libraries exist and are well-used, these are all incomplete and generally represent data from only a few beamlines or a few elements. There are many challenges for creating more complete and useful libraries, including what forms of data the library should use, what metadata is important, how spectra can be organized, the criteria for adding spectra, and the method for adding and extracting spectra. These questions ultimately fall to the community - not a small group of individuals – to work out and support.

We will present design ideas and preliminary implementations of a candidate way of building and operating such libraries using relational database management systems such as PostgreSQL, MySQL, and SQLite. The approach allows a flexible approach to a rich set of metadata that can evolve. The library software defines the organization of the data and metadata (using the XDI format for guidance) and the basic ways to add spectra, categorize them with many classifications, search through libraries, and extract spectra to XDI, and possibly other formats. An important feature of the initial design is to enable not only a web-based library of “Community Approved Data”, but also group-specific libraries that might be private. Though a prototype library and interface will be shown, this is meant to prompt community-wide discussion, input, and potential collaboration for further development

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Keynote Vector Analytics of Searching Chemical Complexity from FEFF Kernel Mixture Data Base

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For new-generation EXAFS data analysis, many experimentalists will consult on recent technology trend of big data science and clouds computer utilities. To analyze EXAFS data for complex materials systems, the investigators and reviewers are doubtfully paying time-consuming efforts with accepting high difficulties in conventional methods. Data storage with fidelity was taken care of, but subsequent data analytics is not easy to go without advanced software engine and collaborated data scientists. First of all, FEFF kernel data base must be prepared over all possible chemical mixtures of simulating EXAFS signals. These matrix mixture structures must be computed beforehand in all matrix refinement processes of delicately curing numerical ill-posedness problems. Also, these keynote vectors are representatives of strong feature contents within matrix. When a set of multiple data are sent for EXAFS analysis request, clouds computers must search the simulated data over whole range of chemical models. Such comparison analytics requires the covariance evaluations between the multiple experimental data and the trial simulated data. A fast search algorithm includes matrix manipulation functions of filtering out the experimental noises over all selectable FEFF mixture kernels. Optimal regularization process was successfully achieved by our novel algorithm which has been developed for the image data keynote abstraction according to mathematics principle of numerical linear algebra. Due to high data compression rate from the 2-D FEFF kernel of (k,r) domain to the 1-D keynote vector, the fast searching engine will be plausible. We described the detailed mathematics formulations and an upcoming software development project relevant to LAPACK installation in future.

Evaluation of spectra recorded at the two EXAFS beamlines at Indus-2 synchrotron

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Generally, two types of beamline set-ups are commonly employed for recording XAFS spectra using synchrotron radiation. In the first type, a double crystal monochromator (DCM) is used and in the second type, a bent crystal polychromator is used. Two such beamlines have been developed at the 2.5 GeV Indus-2 synchrotron source at Raja Ramanna Center for Advanced Technology (RRCAT), Indore (India). The first type of beamline is named BL-9 and the second type of beamline is named BL-8. BL-8 beamline has been in use for about seven years, while BL-9 beamline has been commissioned recently. Hence, we thought it proper to do evaluation of the spectra recorded on these two beamlines.

The BL-8 beamline has a 460 mm long Si (111) crystal having *2d* value equal to 6.2709 Å mounted on an elliptical bender, which can bend the crystal to take shape of an ellipse. The polychromatic beam is focused at one of the foci of the ellipse where the sample is kept. The source is kept at the other foci of the ellipse. The radiation transmitted through the sample is detected by a position sensitive CCD detector. A plane mirror is then used before the detector to cut-off the higher harmonics from the radiation diffracted by the Si crystal.

The key component of the BL-9 beamline is a Double Crystal Monochromator (DCM). The beamline uses a 1.3 m long meridional cylindrical mirror with a radius of curvature of 11.2 Kms coated with Rh/Pt as a collimator. This mirror is followed by the DCM which consists of two Si (111) crystals with *2d* = 6.2709 Å and narrow Darwin width (~ 6 arc sec). The second crystal of the DCM is a sagittal cylinder with radius of curvature in the range 1.28-12.91 m which provides horizontal focusing to the beam. On this beamline, it is also possible to carry out measurement in fluorescence mode.

In order to evaluate the spectra obtained from these beamlines, EXAFS spectra at the Cu K-edge have been recorded in several copper complexes at both the beamlines. It has been found that the range of the EXAFS data is more in BL-9 than BL-8. However, the bond lengths obtained from EXAFS data analysis are comparable for both the beamlines. Due to larger damping of EXAFS oscillations at higher energies in BL-8, higher disorder is obtained in BL-8. The resolution in the pre-edge and XANES region has been found to be more in BL-9. The advantage of BL-8 is that spectra can be recorded very quickly (~ 300 ms) and hence beamtime is easily available. Further, BL-8 is useful for performing in-situ and time resolved studies. In BL-9, the time taken to record the spectrum is quiet large (~40 minutes) and hence it takes longer to get access to this beamline.

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Quality Improvements for Fluorescence Detected XAFS Spectra

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Methods for the quantification of statistically valid measures of the uncertainties associated with X-ray absorption fine structure (XAFS) data obtained from dilute solutions using fluorescence measurements are developed. Several systematics (*e.g.* self-absorption) associated in fluorescence detection cannot yet be corrected automatically. Multielement fluorescence detectors provide a set of absolute spectra depending on the geometry of the detector channels with different apparent sensitivities. Error analysis performing the characterization and correction of systematics (self-absorption, energy calibration, photoreduction, air path attenuation *etc.*) can measure statistical accuracy. Normalization of intensities I_0 and I_f is crucial as is the experimental geometry. Experimental data obtained from 10 mM solutions of the organometallic compound ferrocene, $Fe(C_5H_5)_2$, are analysed within this framework and, following correction for various electronic and geometrical factors, give robust estimates of the standard errors of the individual measurements. The reliability of the refinement statistics of standard current XAFS structure approaches that do not include propagation of experimental uncertainties to assess subtle structural distortions is assessed in terms of refinements obtained for the staggered and eclipsed conformations of the C_5H_5 rings of ferrocene. Incorporation of experimental uncertainties into an IFEFFIT-like analysis yield refinement statistics for the staggered and eclipsed forms of ferrocene which show a far more realistic preference for the eclipsed form which accurately reflects the reliability of the analysis. Moreover, the more strongly founded estimates of the refined parameter uncertainties allow more direct comparison with those obtained by other techniques. These XAFS-based estimates of the bond distances have accuracies comparable with those obtained using single-crystal diffraction techniques and are superior in terms of their use in comparisons of experimental and computed structures [1,2].

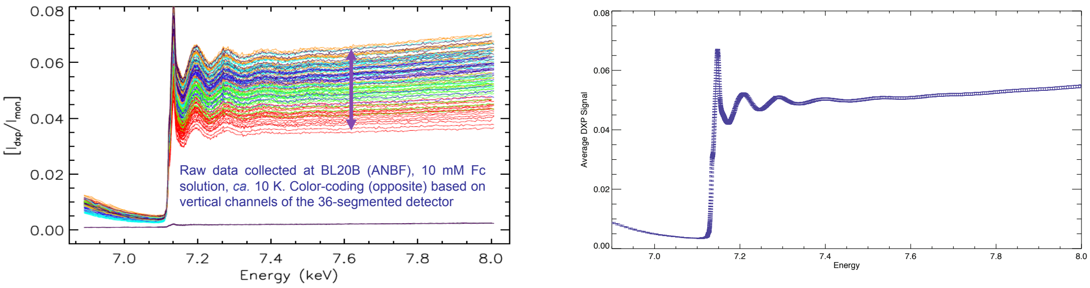

Figure 1: a) Raw variation of spectral response function from multiple pixels b) Standard error after correction for self-absorption and pixel variation.

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XAFS data acquisition with 2D-detectors: Transmission mode XAFS of inhomogeneous samples and grazing incidence EXAFS spectroscopy

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The data quality of transmission mode EXAFS experiments rely on the homogeneity of the investigated samples in the region where the X-ray beam transmits through the sample. Lateral thickness variations, especially holes or inhomogeneous sample compositions may lead to severe distortions of the EXAFS spectra. For example, the amplitude of the EXAFS oscillations is significantly damped compared to a perfect sample, and the determined coordination numbers and Debye-Waller factors are erroneous [1]. While a careful sample preparation may prevent such artifacts in the case of ex-situ experiments, there are many situations where the sample considerably changes in the case of in-situ studies, for example in the case of catalysts in a reacting environment [2]. Instead of an ionization chamber that measures the average absorption through the sample, the use of a 2D-area pixel detector which provides local absorption spectra for each position within the sample may be favourable. Recent experiments have demonstrated the capabilities of such a setup for in-situ experiments of battery materials, where local charging and discharging occurs [3]. In this contribution, we will present transmission mode EXAFS data of inhomogeneous model samples measured with a large, unfocussed beam of a wiggler source in order to evaluate the data quality offered by pixel detectors for real, inhomogeneous samples.

In a second series of experiments, we will also address grazing incidence X-ray absorption spectroscopy, where the X-rays impinge on the surface of the sample under a small incident angle $\phi_{i.}$ Besides the specular reflected beam with identical incident and exit angles $\phi_i = \phi_{e,}$ a second distinct scattering peak (Yoneda-peak) occurs if the exit angle is equal to the position of the critical angle ϕ_c of the scattering material [4]. From related EXAFS experiments, a depth profiling of the sample is feasible, and structural information of the surface and buried layers may be obtained [5, 6]. If ionization chambers are used as detectors, however, the EXAFS has to be measured sequentially for the different scattering angles, which makes Yoneda-XAFS experiments extremely time-consuming. Using a 2D-area detector, the entire scattering pattern is acquired simultaneously for each energy in the EXAFS scan, and a wealth of information is available in a moderate time. Here we will discuss the feasibility of Yoneda-XAFS measurements for the structural investigation of multi-layered samples.

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A versatile capillary flow cell for operando XAS and XRD: a case study on PdO-Co_3O_4 for CO oxidation

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The improvement of the activity of catalysts requires understanding the nature of the active sites, the reaction and deactivation mechanisms. However, the majority of current studies are based only on ex situ analyses of the catalysts and on kinetic experiments. Very little is known about structure and chemistry of catalysts under reaction conditions. Therefore, an in-depth investigation of the structural and electronic changes of catalysts occurring under catalytic conditions is crucial. Both X-ray diffraction (XRD) and X-ray absorption spectroscopy (XAS) are important techniques that are commonly used to determine the geometric and electronic structure of catalysts. The success of the in situ/operando XRD and XAS experiments strongly depends on the reaction cell. Therefore, a lot of attention is paid for the development and optimization of the reaction cells for XRD and XAS. Various sample-environment/reaction cells have been proposed for XRD and XAS experiments. However, these designs are focused mainly on the narrow and specific types of experiments and do not enable to use the same cell for both, XRD and XAS. Further development and optimization of the reaction cells that allow recording XAS spectra both in the transmission and fluorescence mode and X-ray diffraction/total scattering using the same cell is of great scientific interest.

Herein, we present a versatile capillary flow cell that offers a greater capability to perform X-ray absorption and X-ray diffraction/total scattering studies of catalysts under operando conditions (Figure 1). The design of this cell is based on the design by Peter Chupas et al [1]. We made further modification of the cell that allows using this cell for both XAS and XRD experiments with capillaries in the range from 0.3 mm to 3 mm in a flow of reaction gases up to 800 °C.

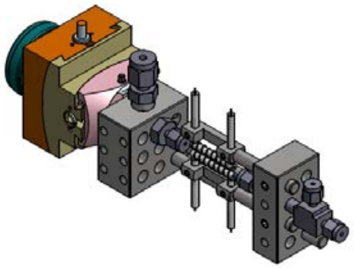

Figure 1: The flow-cell/furnace for operando XRD and XAS experiments

Constructed cell was used for studying operando the electronic and geometric structure changes of PdO-Co_3O_4 CO oxidation catalysts in SuperXAS (SLS, Villigen, Switzerland) (3 mm quartz capillary) using a quick scanning monochromator. Full Pd K edge (24350 eV) in quickEXAFS spectra were recorded in transmission mode with a 250 ms time resolution. The complementary X-ray total scattering experiments for studying the local/intermediate-range structure and defect structure of PdO-Co_3O_4 catalysts using the same cell (0.8 mm quartz capillary) were performed in P02.1 (PETRA III, Hamburg, Germany) with 60 keV energy resulting in Q_{max} of 22 Å^{-1}, PerkinElmer XRD1621 area detector and a time resolution of 30 s. For all measurements a quadrupole mass spectrometer was used to monitor the educts and products concentration.

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ROCK: the new QUICK-EXAFS beamline at SOLEIL

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X-ray Absorption Spectroscopy is a well-known "workhorse" technique for *in situ* materials characterization like catalysts and batteries. With the advent of 3rd generation synchrotron radiation facilities allowing a sub-second time resolution in the monitoring of kinetics, the experimentalist can now access to a deeper and more accurate temporal description of the chemical species involved in such processes. *Operando* investigation of materials requires the development of suitable tools and methods to simultaneously assess to the evolution of local order of the materials and to its catalytic or electrochemical properties. The investment in such technical or methodological skills leads today to the worldwide building of *dedicated* X-ray Absorption beamlines for catalysis. The SOLEIL's ROCK beamline, recently funded by the French *Agence Nationale de la Recherche* in the framework of the national "*Grand Emprûnt*", is one of these dedicated infrastructures for the *operando* characterization of materials by time-resolved Quick-EXAFS [1]. In this presentation, the performances and capabilities of the ROCK beamline [2] will be presented. With the huge amount of data collected each minute, new tools must be provided to the users for data handling. Chemometric tools such as Multivariate Curve Regression with Alternating Least Square (MCR-ALS) fitting emerge as a new powerful method for getting more information from XAS spectra of evolving mixtures upon reaction [3]. Examples of the use of the MCR-ALS method on the investigation of heterogeneous catalysts will be given to discuss its strength and limitation.

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QUATI: time-resolved XAS beamline at SIRIUS

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X-ray Absorption Spectroscopy, providing structural and chemical information about a selected element, is a very powerful technique for establishing structure-property relationships in materials. With the high photon flux delivered by 3rd generation synchrotron facilities, time-resolved X-ray absorption spectroscopic studies of dynamic processes can be investigated over a wide variety of areas (*e.g.* in catalysis or energy storage/conversion).

The new Brazilian SIRIUS storage ring is designed to have the lowest emittance. The QUATI beamline dedicated to time-resolved XAS measurements, will be located on a 2T superbend dipole. Targeted for quick-XAS studies with various sample environments (gas/liquid flow, high/low temperature, high pressure…), complementary techniques such as Raman, IR spectroscopy, mass spectrometry or XRD will be available to unravel reaction intermediate species.

With a photon flux superior to 10^{12} ph/s, time-resolved XAS measurement in the sub-second regime will be achievable with a Frahm's type Quick-XAS monochromator [1] in the energy range of 5-45 keV. Installed on a tilt table, the channel-cut monochromator is continuously moved during one acquisition while the data are collected on the fly [2].

Last but not least, measurements in quick-XAS mod will rapidly engender a large amount of data that cannot be treated in a conventional manner. New strategies have to be developed and made available for users.

In this contribution, the design of the new QUATI beamline will be presented. The chemometric method with multivariate-curve resolution methodology, which will be directly implemented on the beamline for the users, will be presented through examples in catalysis revealing reaction intermediates [3].

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High throughput *in situ* XAS data acquisitions in Li-ion batteries in one go – multiple sample and edge measurements

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Characterization techniques like XRD and XAS are often carried out under *in situ* conditions on Li-ion batteries, in order to investigate the electrochemical mechanism and structural changes of the electrodes occurring during the electrochemical cycling process. However, electrochemical cycling process of Li-ion batteries electrode materials can take several hours. Moreover, time resolution requirement for the investigation are often in several minute regime. By using multiple sample changer, high throughputs measurements involving several samples are possible. Our group has effectively tested these multiple sample (up to 4 cells) measurements at diffraction beamline PO2.1 at PETRA [1]. More recently *in situ* XAS measurements on Li-ion batteries were also performed in multiple sample mode (up to 2 cells) as well as edges at XAS beamline ANKA [2,3]. The challenges and the information content related to multiple sample as well as multiple edge measurements will be discussed. Moreover, with the new two beamline P64 and P65 at PETRA insight, an efficient and optimized Li-ion battery sample changer need to be developed in future.

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High level of automation is essential for high quality XAFS data

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Over the last 11 years of operation at the ANKA-XAS beamline it became obvious, that the quality and the stability of the beamline operation (scientific users and industry) strongly depends on the level of automation. Therefor we made an ambitious automation upgrade program on hardware and software.

After a hardware upgrade the beamline has now a fast sample holder exchange option based on a zero-clamp system. Not only the sample holder on top of the motor stages can be exchanged by the system but also the sample stages (Huber goniometer set-up & Hexapod) below. The automatic detection of the sample holders system ensures, that the motors are switch of at the driver-layer and can't be moved with any client. The reliability of this system is within 5 µm and allows extremely fast sample exchange and re-measurements of formerly measured samples without any new alignment.

The data visualization and graphical control interface is separated into independent Applications based on IgorPro [1]. While the former is used as a powerful data display with minimal integrated data evaluation (eg. normalization of data), the later offers a graphical user interface with an integrated task list manager. Both communicate via Rato with spec and can directly access any of the Tango-based components due to the IgorPro-Tango-binding supplied by Soleil.

Unique features of the graphical control are:

- An automated naming scheme of beamline- and sample-alignment scans is integrated to distinguish different type of scans. Planning for the integration of meta-data into the HDF5 is done.
- Display of shutter states is also available. Automated reaction based on shutter status changes due to storage ring operation is currently in implementation (eg. PI-regulation of piezo system). The software ensures, that shutters is opened before measurement
- Display of selected - and for the user important - motor positions and states.
- The software includes a task-list manager, which enables step-wise and highly parallel interaction with the beamline to maximize the used beamtime. It enables the user to add motor movements or scans to the list, based and during the ongoing scans or movements.
- A notification manager is integrated into the software to send out SMS messages in case any of the scan takes longer than expected.
- With a table-based interface it is possible to generate complex scan scenario with several sample, absorption edges, detector set-ups and scan-types with different pre-sets.

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XAFS and X-ray Microprobe Data Collection with Larch, Epics, and PostgreSQL

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Data collection, reduction, and visualization tools are critical for reliably generating quality data during any synchrotron experiment. As beamlines and experiments mature to include more diverse kinds of measurements the needs for data collection software also expands. Important criteria for such software include: guiding novices through basic measurements and adjusting allowed beamline settings, providing a macro system for unattended collection, enabling tight integration and feedback between data visualization and collection of both X-ray and ancillary data, allowing experts to customize and program data collection, assisting management of complex data sets and metadata, and supporting remote monitoring and the possibility of remote control with security.

I will show the data collection system developed for the X-ray fluorescence microprobe and spectroscopy beamline at GSECARS, sector 13 of the Advanced Photon Source. Central to this system is a PostgreSQL relational database management system used as a centralized store for all configuration information and metadata about the instrumentation that also provides a complete history of data collection steps. The database management system is inherently client/server based, and so also provides a natural communication and security layer between processes. This naturally means that clients such as the GUIs normally used to collect data are separated from the collection "server" process that interacts with the underlying Epics beamline control system. The server acts as a simple process the Larch framework, interpreting and executing commands written by clients into the database as Larch commands. This approach allows robust and isolated testing of data collection and beamline configuration primitive commands. Complex scripts built from these primitives can easily be written, modified, and run in sequences by the end-user. Example GUIs and visualization tools will be shown for X-ray fluorescence mapping and XAFS, and the interaction of these with data collection will be described.

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The data acquisition, analysis and storage system at the Spectroscopy Village beamlines, Diamond Light Source

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Diamond Light Source has a portfolio of six beamlines fully or partially dedicated to X-ray Absorption Spectroscopy. The beamlines, grouped in the Spectroscopy Village, cover an energy range spanning from the soft X-rays (at 240 eV) up to the hard X-ray regime (35 keV). Experimental techniques and acquisition system vary from conventional scanning quick-EXAFS, to time resolved dispersive acquisition, to microfocus 2d mapping, to tomographic experiments. We will present the present status of the acquisition system, the online data reduction and data storage strategies adopted at Diamond.

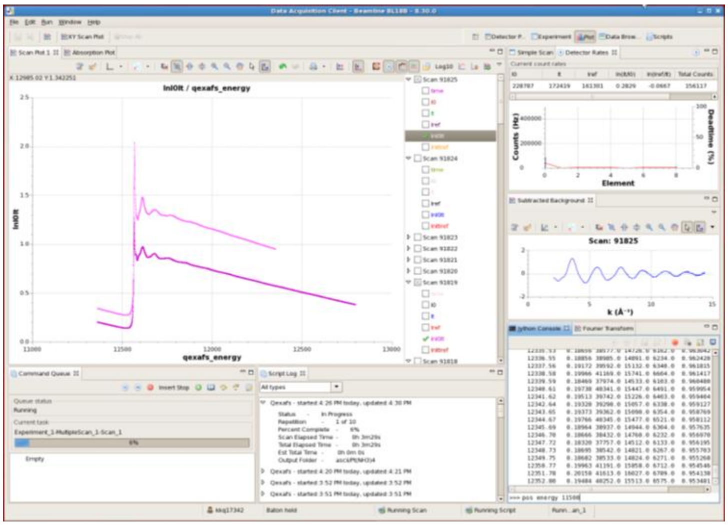

Figure 1: GDA (the Generic data Acquisition system) with online data analysis working on B18-Core XAS beamline

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GLP, ISO 900X, certification and EXAFS in a research environment?

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In analytical chemistry it is universally accepted that formalised quality assurance schemes are a necessary prerequisite for reliable analytical results. The analytical result must be independent from the place and time where and when a sample was investigated. This is obvious for legal maximum or minimum permissible values but is of similar importance for the control of production processes or to ensure that a product has warranted properties when it is sold to a customer. In the late 90s of the last century many (international) organisations started to develop guidelines for reliable analytical work. Keywords are ISO/IEC 17025, ISO 9000 and 9001 and the OECD's Good Laboratory Practice (GLP) requirements.

In a research environment, here a laboratory where EXAFS spectra are measured, similar standards are requested (Good Scientific Practice) and often achieved but usually not bindingly codified. Moreover it might be impractical to follow strict operation procedures in a research environment where new problems often require innovative solutions.

For analytical laboratories in Europe the Eurachem together with the CITAC (Co-operation on international Traceability in analytical Chemistry) has published several guidelines for different aspects of quality assurance in analytical laboratories [1]. The intention of the talk is to try to transfer the guidelines for analytical laboratories, especially those of the guide for *Quality assurance for Research and Development and Non-routine Analysis* [2] to EXAFS applications.

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