constraints on intensities of overlapping reflections for stable calculation. However, it seems not to have been clearly mentioned that the covariance matrix obtained in any least squares algorithm loses some amount of information by imposing artificial constraints on fitting parameters. Furthermore, in such cases, the definition of the number of independent reflections in a powder diffraction pattern is ambiguous [1, 2]. These statistical quantities are required in next step of ab-initio powder structure determination [1, 3] and play an important role particularly when a lot of reflections in a wide range are dealt simultaneously. Therefore, there is no doubt about the advantages to execute the Pawley method without requiring any intensity constraints. The robust nonlinear least squares algorithm in Z-Rietveld makes it possible. As a result, we can obtain an intensity covariance matrix that represents correlations among intensities accurately. By analyzing the covariance matrix with singular value decomposition, the number of independent reflections in a powder diffraction pattern is naturally defined. We can also establish a method to gain an objective function that is considered to be optimal for ab-initio powder structure determination, using a theory on the numerical errors of singular value decomposition. In EPDIC-12, results obtained by Z-Rietveld in the Pawley method are presented. We also introduce a method to gain the theoretically optimal objective function using singular value decomposition and results when it is utilized for ab-initio powder structure determination. Finally, we introduce Z-Rietveld briefly; Z-Rietveld is a multi-platform Rietveld software developed for users of the Japan Proton Accelerator Research Complex (J-PARC). It can execute the Pawley method and the Rietveld analysis, including multiphase analysis and multi-source analysis using powder diffraction patterns from laboratory X-rays, spallation neutrons and reactor neutrons [4].

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Keywords: singular value decomposition, Pawley method, ab-initio powder structure determination

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Automated Single Crystal Structure Determination – A Tool for Synthetic Chemists? Bernd Hinrichsen[a], Martin Adam[a], Michael Carr[b], Dieter Schollmeyer[c], [a]*Bruker AXS GmbH, Karlsruhe, Germany,* [b]*Bruker UK Ltd, Coventry, United Kingdom,* [c]*Johannes Gutenberg University, Institute of Organic Chemistry, Mainz, Germany*
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During recent years large improvements in software functionality and its ease-of-use have made single crystal X-ray structure determination easier than ever. These days most structures can be measured, processed, solved and refined using well selected defaults with no or little crystallographic knowledge. Recently, microfocus sources and CCD detectors both air-cooled, have entered the marketplace. Combining these innovations with an automated sample loader and an intelligent graphical user interface allows for the design of a table top single crystal diffractometer, which only requires a standard single phase power connection and no cooling water at all. An instrument taking advantage from these software and hardware developments would enable synthetic chemists or pharmacists to perform a complete single crystal structure analysis almost next to the reaction flask.
However, such an instrument might not provide the entirely correct structure and/or the data quality might not match established scientific standards. In order to elucidate the capabilities of the instrument we have measured a number of real-life samples[1] using the SMART X2S automated instrument (Figure 1). The results of a comparison with more conventional, currently installed systems are favourable towards the SMART X2S and will be presented. Potential limitations will be addressed and their solutions will be discussed.

Figure 1: SMART X2S table top SC-XRD solution.

Keywords: Automation in Chemistry, Microfocus source, Table top solution

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Recent Developments in CrysAlis[Pro]. Oliver Presly, Alexandra Griffin, Zoltan Gal. *Agilent Technologies XRD (formerly Oxford Diffraction).*
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CrysAlis[Pro] is the software used to collect and reduce data for Agilent Technologies XRD systems (*formerly Oxford Diffraction*). It is important for the software to look and function equally as well as the diffraction hardware in order to achieve the best results. Encouraging feedback from users allows the provision of a continually evolving program and new features and bug fixes are frequently implemented according to the community's requirements. The presentation will highlight several examples of user-inspired software tools, alongside the addition of new utilities for protein screening, simplistic multi-temperature and wavelength experiment strategies and extended options for high pressure data collection and reduction. The updates are presented here with a discussion of how the new tools may be implemented to improve data quality.

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New developments in structure analysis of powders using preferred orientation. <u>Jürgen Grässlin</u>[a], Lynne B. McCusker[a], Christian Baerlocher[a], Fabia Gozzo[b], Bernd Schmitt[b], Luca Lutterotti[c] , [a]*Laboratory of Crystallography, ETH Zurich, Switzerland,* [b]*Swiss Light Source, PSI, Villigen, Switzerland,* [c]*Department of Materials Engineering and Industrial Technology, University of Trento, Italy*
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In recent years, the reflection overlap problem, which is the primary hindrance to structure determination from powder diffraction data, has been addressed by exploiting preferred orientation. The changes in the diffraction pattern intensities as a function of sample orientation (tilt χ and rotation ϕ) that are observed for a textured sample in an X-ray beam, can be used to resolve the relative intensities of reflections that overlap in a conventional powder diffraction pattern. It has been shown that this principle can indeed be exploited to obtain more single-crystal-like data from a polycrystalline material [1]. The availability of a new version of the 1-dimensional Si-microstrip detector Mythen II on the Materials Science Beamline at SLS [2] and new features in the data analysis software *Maud* [3] have made it possible to improve the quality of extracted reflection intensities significantly. Textured samples of a zirconium phosphate framework material (ZrPOF-pyr) with a known crystal structure [4] and of an ammonium niobium silicate with an unknown structure were measured recently using the new experimental setup. The measurements consist of two steps. First, 302 orientations are measured quickly to determine the orientation of the crystallites in the sample. Then a few selected orientations are measured with better counting statistics for the intensity extraction. Data analysis using *Maud* also involves two steps. First, the intensities of a few non-overlapping reflections as a function of sample orientation (302 patterns) are used to generate pole figures (PFs) for these reflections and to determine the orientation distribution function (ODF). The ODF is then used to calculate the pole figure values for all reflections in the pattern, and these are applied in a joint refinement of the patterns collected with better counting statistics to obtain a single set of single-crystal-like reflection intensities. Analysis of the ZrPOF-pyr data shows that indeed more reliable intensities can be derived for reflections that overlap in a conventional powder diffraction pattern. However, the few orientations corresponding to main zone axes (e.g. *h00, 0k0, 00l, hh0, h0h, 0kk*), which were measured longer for intensity extraction, do not contain sufficient information to resolve all overlapping reflections. Consequently, a new strategy for selecting an optimized set of sample orientations for longer data collection has been developed. All PFs are calculated from the ODF, and from these, difference PFs are calculated for overlapping reflections. Those orientations that represent maxima in the PFs (i.e. above a given threshold value) and maximum differences for overlapping reflections (texture contrast) are selected. This new approach will be tested during our next experimental sessions in May and June 2010.

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