σ — standard deviance;
I_k – peaks intensuities with $I_k \geq 10 \cdot \sigma_k$
k — a number of peaks with intensities $I_k \geq 10 \cdot \sigma_k$
I_k' – a highest peak intensity;
w_k – weight, $w_k = 1/\sigma_k + 1$

The second set of R-factors and goodness of fit are calcuated only for $|I_{obs}-I_{calc}|-\sigma_i$., i.e.,

$R'_1 = \Sigma((|I_{obs}-I_{calc}|-\sigma)/I_{obs})/N$ $R'_{1w} = \Sigma w((|I_{obs}-I_{calc}|-\sigma)/I_{obs})/N$

$R'_2 = (\Sigma((|I_{obs}-I_{calc}|-\sigma))/N)/(\Sigma I_k/k_h)$ $R'_{2w} = \Sigma(w(|I_{obs}-I_{calc}|-\sigma)/N)/(\Sigma w I_k/k_h)$

$R'_3 = (\Sigma(|I_{obs}-I_{calc}|-\sigma))/N)/I_k'$ $R'_{3w} = \Sigma(w(|I_{obs}-I_{calc}|-\sigma))/N)/(I_k'))$

Keywords: powder diffraction, Rietvld method, R-factors

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Multialiquot cell approach for the SDPD of high-symmetry compounds.

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Recently mentioned [1] inconsistency of figures of merit [2,3] when indexing high symmetry lattices turned to a conclusion the smaller cells of lower symmetry can be applied as building units when solving a structure by direct space methods. The approach is expected to decrease time necessary for simulated annealing of one structure solution and may appear particularly useful for large organic structures. The poster illustrates the approach based of example compounds with a small pyrochlore structure. The indexing program suggest several possible solutions and the correct solution of highest symmetry among them. The repetition of the same lattice described by different cells should be considered as an indication of the correct indexing solution. From the other hand, that might be random and unfruitful indexing solution if the lattice is non-primitive but is not observed among proposed cells with its primitive representation. The extension to indexing algorithms, eliminating lower symmetry cells for the same lattice described by high-symmetry cell, and the corrected figures of merit taking into account the number of equal proposal cells might be drawn as follows:

$$M'(20) = M(20)_h \cdot N_{ep}$$
$$F'(20) = F(20)_h \cdot N_{ep}$$

where $M(20)_h$ and $F(20)_h$ are M(20) and F(20) for the ***h***ighest symmetry cell

N_{ep} is the number of ***e***qual ***p***roposal cells

Then, one may start to search for the structural model applying a cell of lower/volume symmetry providing it may represent a building unit for the larger cell of higher symmetry or may asssist to find a sublattice..

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Keywords: powder diffraction, indexing, figures of merit

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DDMSuite – a powder diffraction full-profile analysis system.

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In this presentation we describe a new freeware program package *DDMSuite* designed for crystal structure analysis from powder diffraction data. The program is based on the recently proposed derivative difference minimization (DDM) method of whole-profile fitting [1] as a background-independent alternative to the conventional Rietveld refinement procedure. In this method the refinement is aimed not at minimizing the absolute difference between the observed and calculated patterns, but at minimizing the oscillations (or curvature) of the difference curve. The difference curve is considered as an estimation of background which, in the absence of crystalline admixtures, usually varies much less rapidly along the powder profile than does the diffraction pattern. The main advantage of this method is that it does not involve background line modelling thus avoiding the background-related systematic errors and allowing structure refinement with higher stability and accuracy [2]. Newly developed software implementation *DDMSuite* is intended to provide a free comprehensive use of the DDM method by means of an easy-to-use graphical user interface (GUI). The program includes both DDM and Rietveld refinement procedures for X-ray and neutron powder diffraction data, profile decomposition routines, the quantitative phase analysis and the size-strain calculations. Great effort has been made to design a versatile tree-type phases/atoms hierarchy navigation. It gives convenient means for macro-editing parameters of multiple selected phases and atoms. A dedicated dialog allows controlling the refinement process facilitated by a number of graphical tools: structure 3D-view; powder pattern plot; Fourier and Patterson mapper. The GUI has interfaces to a number of widely used crystallographic software packages (Diamond, CCDC Mercury etc.) and CIF import-export routines. Applications to various types of powder diffraction data including semi-crystalline substances such as mesostructured materials and complicated multi-phase samples will be demonstrated. The program (to date, a Microsoft Windows version) can be freely downloaded from:
http://www.icct.ru/eng/content/persons/Sol_LA/ddm.html.

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Keywords: powder diffraction software, full-profile refinement, derivative difference minimization

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On execution of Pawley method without requiring intensity constraints on overlapping reflections.

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The Pawley method and the Le Bail method are two major methods for extraction of integrated intensities from powder diffraction patterns. The advantage of the Pawley method to the Le Bail method is that it can directly obtain the intensity covariance matrix. In both methods, it is considered to be necessary to group overlapping reflections and impose linear

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

FA5-MS44-P19

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.

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

FA5-MS44-P20

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.