improvement of the refinement can be obtained by eliminating the badly fitted reflections and/or by correcting the model, which could be too inaccurate or involve too subtle features to be modelled depending on the quality of the observations processed. A successfully recognition of the actually influencing reflections is represented by some statistical estimators based on leverage analysis (Belsley et al., 1980, Merli, 2005). Among several exploitable estimators, there are some of them that seem to be particularly suitable in crystallographic calculations: i) Cook distance (Cook, 1977), ii) DFITTIS and iii) DFBEETASij (Belsley et al., 1980). In particular, estimator i) can be used to recognize an actual outlier of the refinement, whereas estimator ii) is useful to investigate the effects when a reflection is eliminated from the data set and iii) is able to recognize the variables that are mostly influenced by each reflection. The combined analysis of these diagnostics will be presented. It will be shown how successfully it yields to the recognition of a dangerous reflection and/or the inaccurate estimate of any particular variable.

Cook(1977) Technometrics, 19, 15-18

Keywords: crystallographic refinement, crystallographic statistics, leverage analysis

MS.88.3

Anisotropic displacement parameters for molecular crystals from periodic HF and DFT calculations
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Accurate X-ray diffraction experiments give information about the thermally averaged charge density in the unit cell. Investigations of the static charge density requires a proper deconvolution of thermal motion and static density. Complementary information about the thermal motion in molecular crystals is seldom available. We present an approach based on periodic HF and DFT calculations using the CRYSTAL06 code [1,2] to estimate both intramolecular and intermolecular vibrational contributions to the anisotropic displacement parameters as applied to molecular crystals. Crystalline urea has been used as case system. We investigate the role of the basis-set and the adopted DFT functional: LDA, GGA and hybrid. The effect of including an empirical dispersion-force correction to B3LYP calculations has also been considered [3]. Finally, at the B3LYP/6-31G(d,p) level, the dispersion of acoustic phonons has been estimated and included through a supercell approach. The results are compared with experimental data from spectroscopy, X-ray and neutron diffraction experiments. On the basis of previous results, work has been extended to urotropine and benzene crystals and preliminary data are reported. These estimates of atomic and molecular motion are especially important for hydrogen atoms because of their low scattering power and intense thermal motion. Recent attempts to estimate hydrogen atom motion show promising results, and we compare the CRYSTAL06 approach with the SHADÉ [4] procedure.


Keywords: molecular motion, ab-initio calculations, X-ray charge-density analysis

MS.88.4

Joint refinement of single crystal and powder data from X-ray and neutron sources
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The last version of the crystallographic software Jana - a well known tool for structure determination of aperiodic structures - has introduced the generalized data input. In the program Jana2006 data

Keywords: small molecules, cctbx crystallographic refinement, graphical user interface
from X-ray, synchrotron and neutron diffraction can be combined regardless of the powder or single crystal nature of samples and refined jointly in order to get a structure model profiting from unique features of various experimental techniques. The most obvious and widely used case is combination of neutron powder data yielding good resolution for light atoms with carefully measured X-ray single crystal data in order to improve the overall quality of the structure model. The joint refinement is also very useful for magnetic structure determination which is often done from limited neutron powder data set at very low temperature. Joint refinement with complete low temperature X-ray data opens possibility to investigate relationship between magnetic and conventional structure. As a special case refinement of multiphase single crystal data should be mentioned. In this contribution we shall summarize our experience with joint refinement gathered during the last two years. Several practical examples will be presented as well as technical aspects of the joint refinement.

Keywords: structure refinement, joint diffraction data, Jana software

**MS.88.5**

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**FIDDLE: A method for simultaneous indexing and structure solution from powder diffraction data**

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The complexity of crystal structures determined from powder diffraction data has steadily increased through further development of traditional methods for structure determination in reciprocal space and application of global optimization algorithms in direct space. The usual process for structure determination from powder diffraction data consists of the following steps: (1) indexing of the pattern, (2) space group determination, (3) structure solution, (4) structure refinement. The currently available powder methods rely on successfully passing the first step, powder indexing. Due to a number of fundamental and experimental problems, like peak broadening, the presence of impurity phases, dominant zones and geometrical ambiguities, powder indexing will remain difficult in many cases, thereby hampering the next steps in structure determination. There is no fundamental reason to separate, as is usual today, the process of unit cell determination and the process of structure solution. Structure determination from powder diffraction data can be seen as a process of global optimization of all model parameters, including the unit cell parameters. This strategy is applied in the FIDDLE program. For the simultaneous optimization of the parameters that describe a crystal structure genetic algorithms together with a pattern matching technique based on auto and cross correlation functions are used. This one-pot strategy for indexing and structure solution, as applied in FIDDLE, was successfully used for determining the unknown crystal structures of Ethinyl Estradiol anhydrate, Naloxone monohydrate and Creatine anhydrate, cases for which indexing was problematic.

Keywords: indexing, crystal structure determination X-ray powder data, optimization algorithms

**MS.89.1**

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**Experiencing space groups**

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Presented are highlights of fifty years of personal experience with space groups, enriched by their many different aspects (algebraic, geometric, arithmetic, crystallographic, computational, material oriented) and interwoven with friendly human relations, started in 1958 with Edgar Ascher at the Battelle Institute, Geneva, and kept on later from the University of Nijmegen leading eventually to higher-dimensional space groups acting on a lower-dimensional space.

Keywords: space groups, group cohomology, superspace crystallography

**MS.89.2**

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**Space groups resulting from 3D sections of (3+1)D superspace groups. Can all 3D groups be generated?**

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Using the superspace formalism for the unified description of sets of commensurate structures requires an unambiguous understanding of how symmetry of a 3-dimensional structure may be inherited from the symmetry of a higher-dimensional one. Although reducing 3D groups to 2D groups has been thoroughly described [1], the similar research for (3+1)D and 3D groups was missing. Further research on space-superspace symmetry relations is needed for the extension of the International Tables for Crystallography towards incommensurately modulated crystals and quasicrystals. We studied the hyperplane t cuts of (3+1)D symmetry elements and constructed a complete network relating (3+1)- and the corresponding 3-dimensional space groups derived by rational cuts. A complete set of data for (3+1) to 3D group relations has been obtained for the first time. The corresponding database has been established and is available via Web interface at http://superspace.epfl.ch/finder. It is particularly useful in finding common superspace ’denominators’ for series of ’composition-flexible’ structures and analysis of possible or forbidden space group sequences for phase transitions. The results answer among other questions like: can all space groups be obtained as sections of superspace groups?

Keywords: superspace approach, symmetry groups, commensurate modulation

**MS.89.3**


**Space groups, subgroups and a lot more**

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Space groups G are the fundamental tool to describe the symmetry