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 (Belsey 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) DFFTIS and iii) DFBETASij (Belsey 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.

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## MS.88.2

### Small-molecule refinement using the computational crystallography toolbox (cctbx) with Olex2

Horst Puschmann, Luc Bourhis, Oleg Dolomanov
Durham University, Chemistry, South Road, Durham, Durham, DH1 3LE, UK, E-mail：horst.puschmann@gmail.com

In recent years, the cctbx[1] (including the macromolecular toolbox, mmtbx) has firmly established itself as a highly flexible, comprehensive and all-encompassing programming tool for macromolecular structure solution and refinement. The program suite PHENIX[2] is based on this framework, and is the most visible part of this long-standing and well-managed framework. As part of an effort to modernise computational tools for small-molecule crystallography, we have been working in collaboration with the cctbx. Our aim is to extend the functionality currently offered by the cctbx to include small molecule solution and refinement. This has resulted in the creation of the Small Molecule Toolbox (smtbx) which is now included with the cctbx framework. In order to make these tools accessible to those chemists and crystallographers who are working with small molecules, the cctbx/smtbx functionality can be accessed via Olex2[2] - a modern Graphical User Interface which is designed to make working with crystal structures as easy as possible for all groups of users. In this presentation we shall elaborate on the areas of the cctbx where modifications and extensions were necessary to achieve `small-molecule functionality' and will outline how we manage the collaboration between the various contributors (in the hope that many may join!). We will also demonstrate how this functionality is accessed from the end-user's point of view. Examples of how structure solution/refinements achieved using the smtbx compare to other solution/refinement programs will be presented.

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[1] cctbx: http://cctbx.sourceforge.net/

[2] PHENIX:http://www.phenix-online.org/

[3] Olex2 http://sourceforge.net/projects/olex2 and http://www.olex2.org

## MS.88.3

### Anisotropic displacement parameters for molecular crystals from periodic HF and DFT calculations

Anders O. Madsen[1,4], Bartolomeo Civalleri[2], Fabien Pascale[3], Roberto Dovesi[2], Sine Larsen[1,4]
[1]University of Copenhagen, Department of Chemistry, Universitetsparken 5, Copenhagen, DK-2100, Denmark, [2]Department of Chemistry IFM and NIS centre of excellence, University of Torino, Italy, [3]Laboratoire de Cristallographie et Modelisation des Materiaux Mineraux et Biologiques, University of Nancy, France, [4]European Synchrotron Radiation Facility, Grenoble, France, E-mail：madsen@kemi.ku.dk

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 SHADE [4] procedure.

[1] R. Dovesi et al. CRYSTAL06 User's Manual, University of Torino, 2006. (http://www.crystal.unito.it).

[2] F. Pascale et al. J. Comput. Chem. 25 888 (2004).

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## MS.88.4

### Joint refinement of single crystal and powder data from X-ray and neutron sources

Michal Dusek, Vaclav Petricek
Institute of Physics, Na Slovance 2, Prague, 18221, Czech Republic, E-mail：dusek@fzu.cz

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

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

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

Rene De Gelder, Carmen Guguta, Jan M.M. Smits
Radboud University Nijmegen, Molecular Materials, Heyendaalseweg 135, Nijmegen, Gelderland, 6525 AJ, The Netherlands, E-mail：r.degelder@science.ru.nl

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

**Experiencing space groups**

Aloysio Janner
Radboud University Nijmegen, Theoretical Physics, Heyendaalseweg 135, Nijmegen, Nederland, NL-6525 AJ, The Netherlands, E-mail：A.Janner@science.ru.nl

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

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

Ivan Orlov, Lukas Palatinus, Gervais Chapuis
Ecole Polytechnique Federale de Lausanne, Laboratoire de Cristallographie, EPFL, BSP - Dorigny, Lausanne, Vaud, 1015, Switzerland, E-mail：ivan.orlov@epfl.ch

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**

Peter Zeiner
Bielefeld University, Faculty of Mathematics, Postfach 100131, Bielefeld, NRW, 33501, Germany, E-mail：pzeiner@math.uni-bielefeld.de

Space groups G are the fundamental tool to describe the symmetry