Jana2006 - overview

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Praha

What the program offers?

Structural model:

- Solution and refinement of regular, disordered, twinned, modulated, composite and magnetic structures
- For description of the regular structure it allows using of anharmonic ADPs up to sixth order, multipole parameters forcharge density studies and magnetic moments for magnetic structures
- For modulated and composite structures it allows modulations of all structural parameters except multipoles
- Structural parameters are automatically restricted on the base of site symmetry

What the program offers?

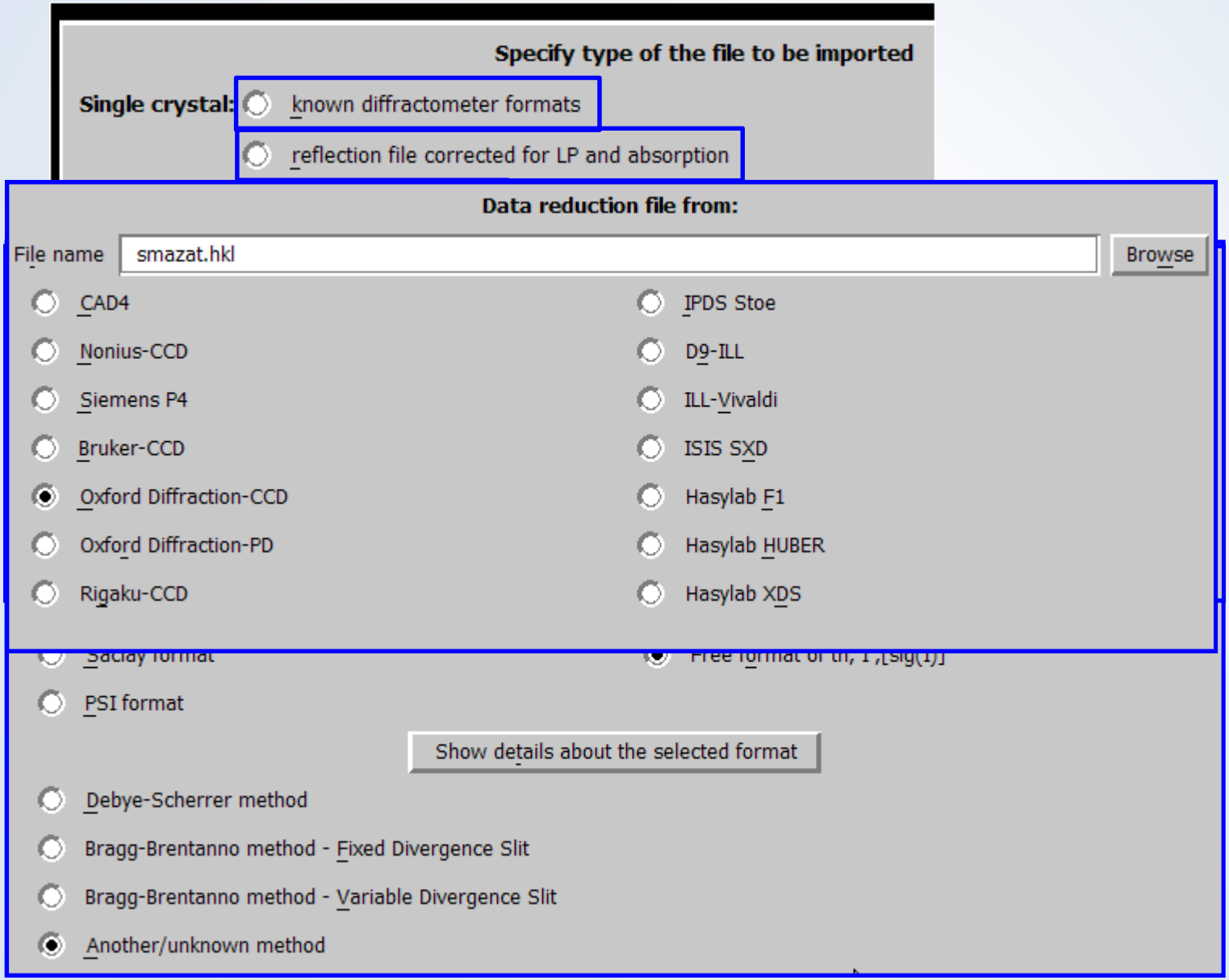
Data:

- The program can handle data single crystal and powder data from X-ray, neutron (including TOF) and electron diffraction
- Several data sets can be combined together in a joint refinement
- For X-ray data most of corrections (absorption, scaling) of integrated data can be made during data import when necessary
- Extinction correction as developed by Becker & Coppens
- Data merging and symmetry analysis is made for regular and modulated crystals

ЯÉCIPROCS
Grenoble 2015

Where to start?

FYZIKÁLNÍ ÚSTAV
Akademie věd ČR, v.v.i.



Data input

X-rays **OR** neutrons **OR** electrons

Powder data of one or more phases

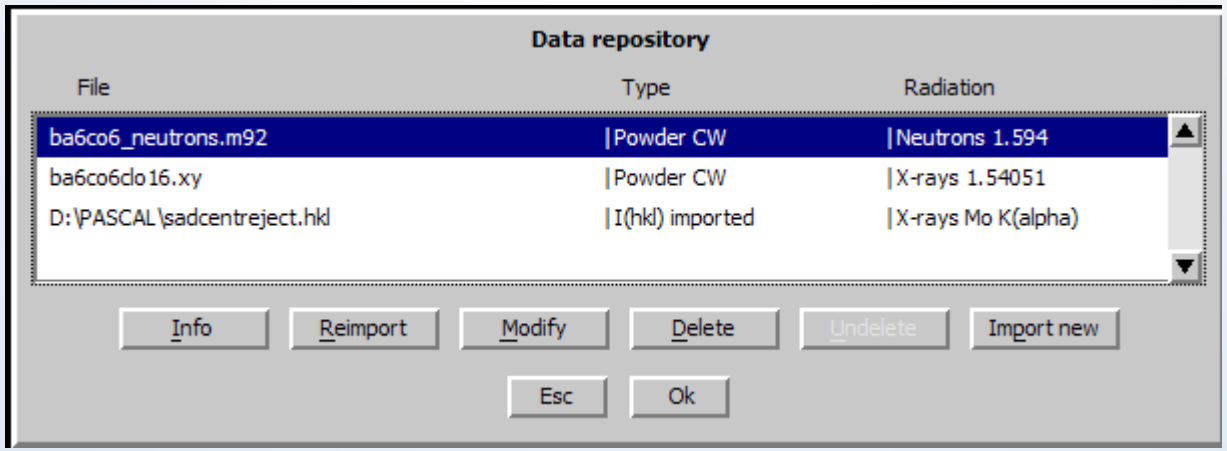
OR

Single crystal data of one or more domains

Import Wizard

Format conversion, cell transformation, sorting reflections to twin domains

Data Repository



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Program Scheme

Reading of one or more data sets

M95 + M50

Determining symmetry, merging symmetry equivalent reflections, absorption correction
Refinement of powder profile parameters

M90, M41

Solution (by calling external programs)

M40

Refinement
Transformation
Introduction of twinning
Change of symmetry

Plotting, geometry parameters, Fourier maps ….

M95	data repository
M90	refinement reflection file
M50	basic crystal information, form factors, program options
M40	structure model

Anharmonic ADP

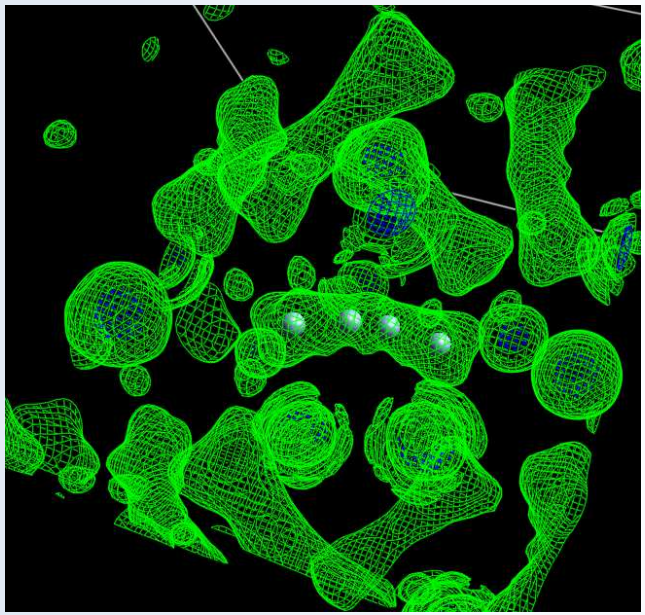
ADP tensors of the second order (harmonic ADP, “temperature parameters”) need not fully describe distribution of atoms in the crystal. Then the Gram-Charlie expansion can be used:

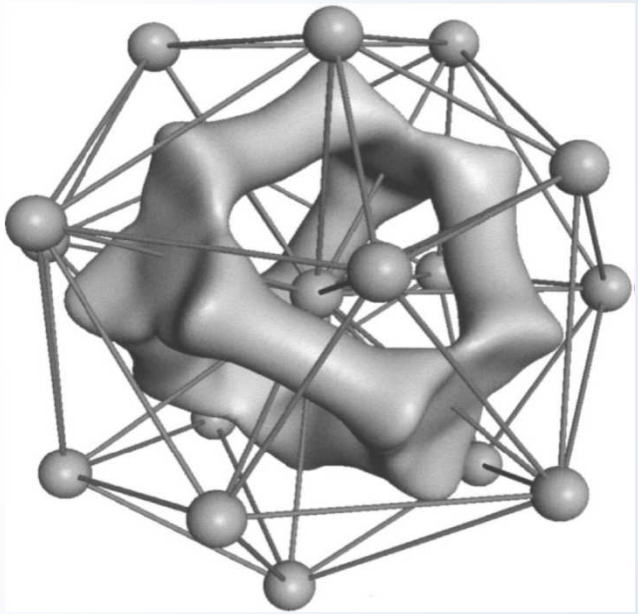
$$F(\mathbf{h}) = \sum_{\nu=1}^{n} f_\nu(|\mathbf{h}|)\exp(2\pi i \mathbf{r}_\nu \cdot \mathbf{h})\exp(-\beta^{ij} h_i h_j)\cdot$$
$$\left(1 - iC^{ijk} h_i h_j h_k + D^{ijkl} h_i h_j h_k h_l + iE^{ijklm} h_i h_j h_k h_l h_m \ldots\right)$$

$C^{ijk}, D^{ijkl}, E^{ijklm}, \ldots$ – contravariant components of symmetric tensor of order 3,4,5,6 are used

Anharmonic ADP

Ionic conductor Ag_8TiSe_6 – argyrodite
E.Gaudin, V.Petříček, F.Boucher, F.Taulelle & M.Evain, ActaCryst (2000), B56, 972-979.

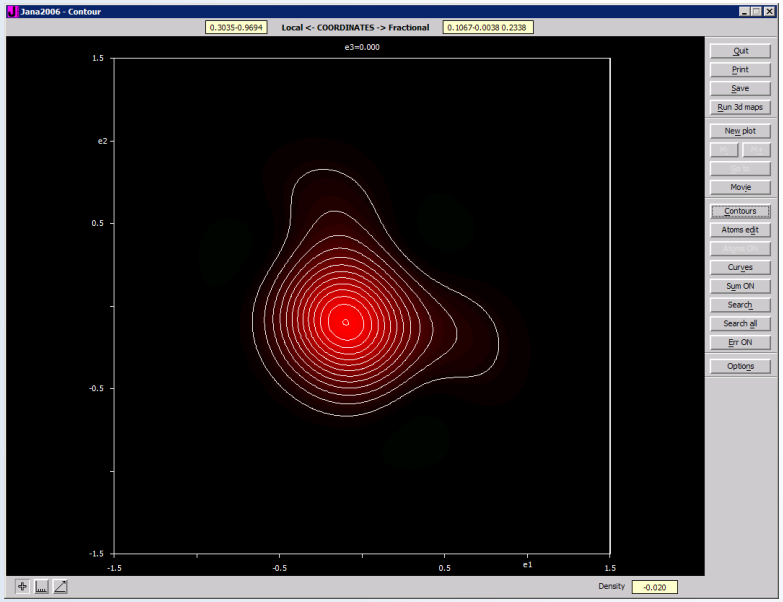


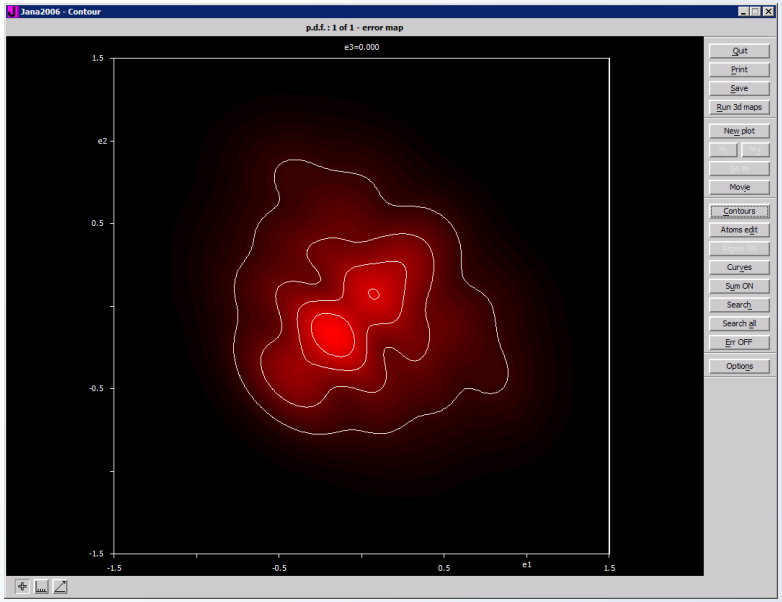


Jana calculates j.p.d.f. maps and then calls 3d visualization program.

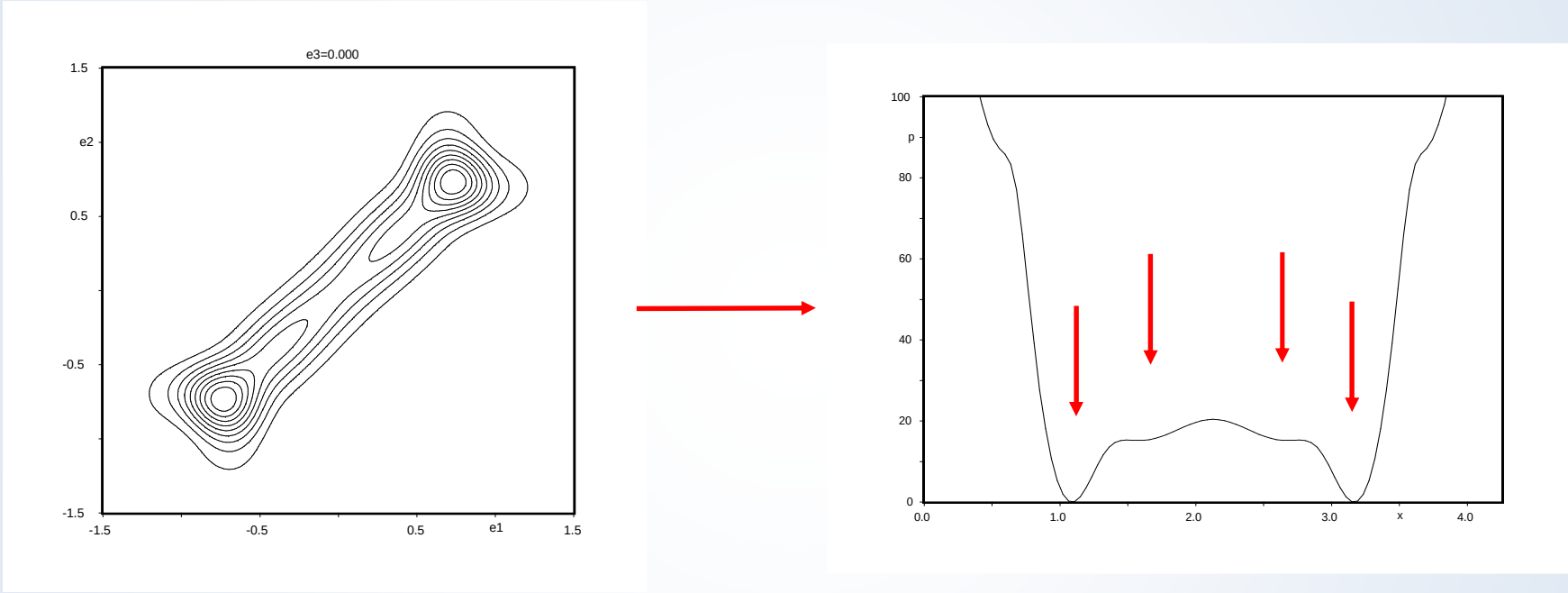
Anharmonic ADP

Except of p.d.f. and j.p.d.f. map Jana can calculate also so called error map showing “map uncertainty” as follows from covariation matrix from the refinement. For its calculation the Monte-Carlo technique has been implemented (Kuhs, W.F. (1992). *Acta Cryst*. A**48**, 80-98):





Anharmonic ADP



[1,1,0] section through j.p.d.f.

Potential curve

Chance to localize possible positions of atoms after phase transition

Charge density studies

N.K.Hansen & P.Coppens, *Acta Cryst.* (1978). A**34**, 909-921.
Their formalism is used in the MOLLY, XD and also in Jana2000/Jana2006

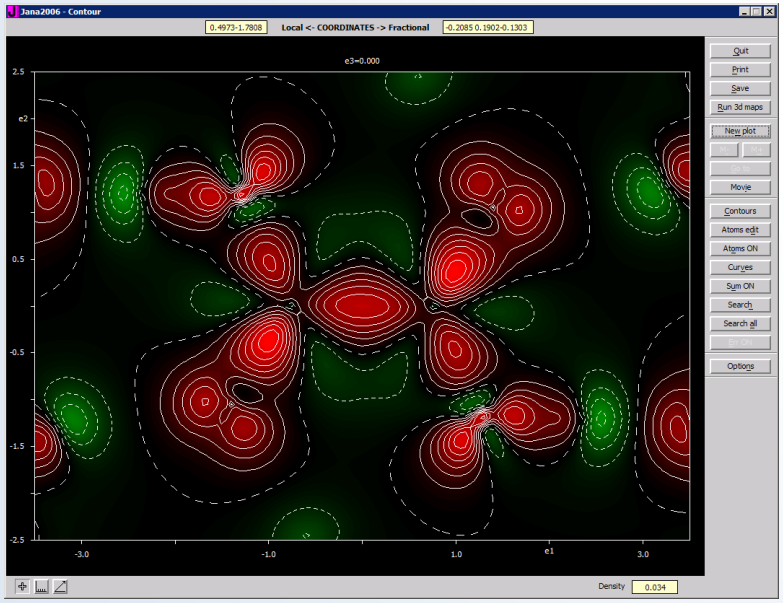
$$\rho_k(\mathbf{r}) = \rho_{k\,core}(r) + P_{k\,valence}\,\kappa^3\,\rho_{k\,valence}(\kappa_k\,r) + \sum_{l}\sum_{m=-l}^{l} P_{k\,l\,m}\;\kappa_k'^{\,3}\,R_{k\,l}(\kappa_k'\,r)\,y_{l\,m}(\mathbf{r}/r)$$

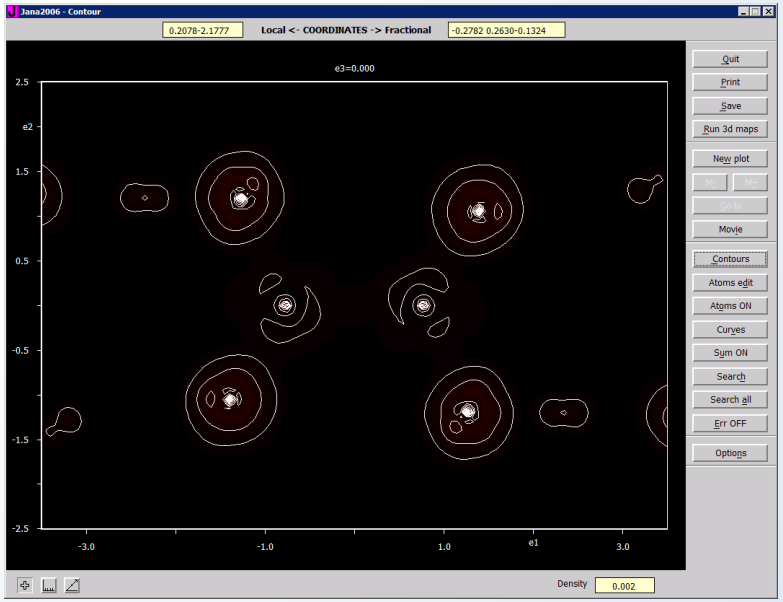
The first term is a local density connected with core electrons and the second one with valence electrons. Both have still a spherical symmetry. The second term allows spherical redistribution of valence electrons by modifying of two adjustable parameters - $P_{k,valence}$ and κ .

The third term represents an ashperical term with several adjustable parameters - P_{klm} and κ_k' .

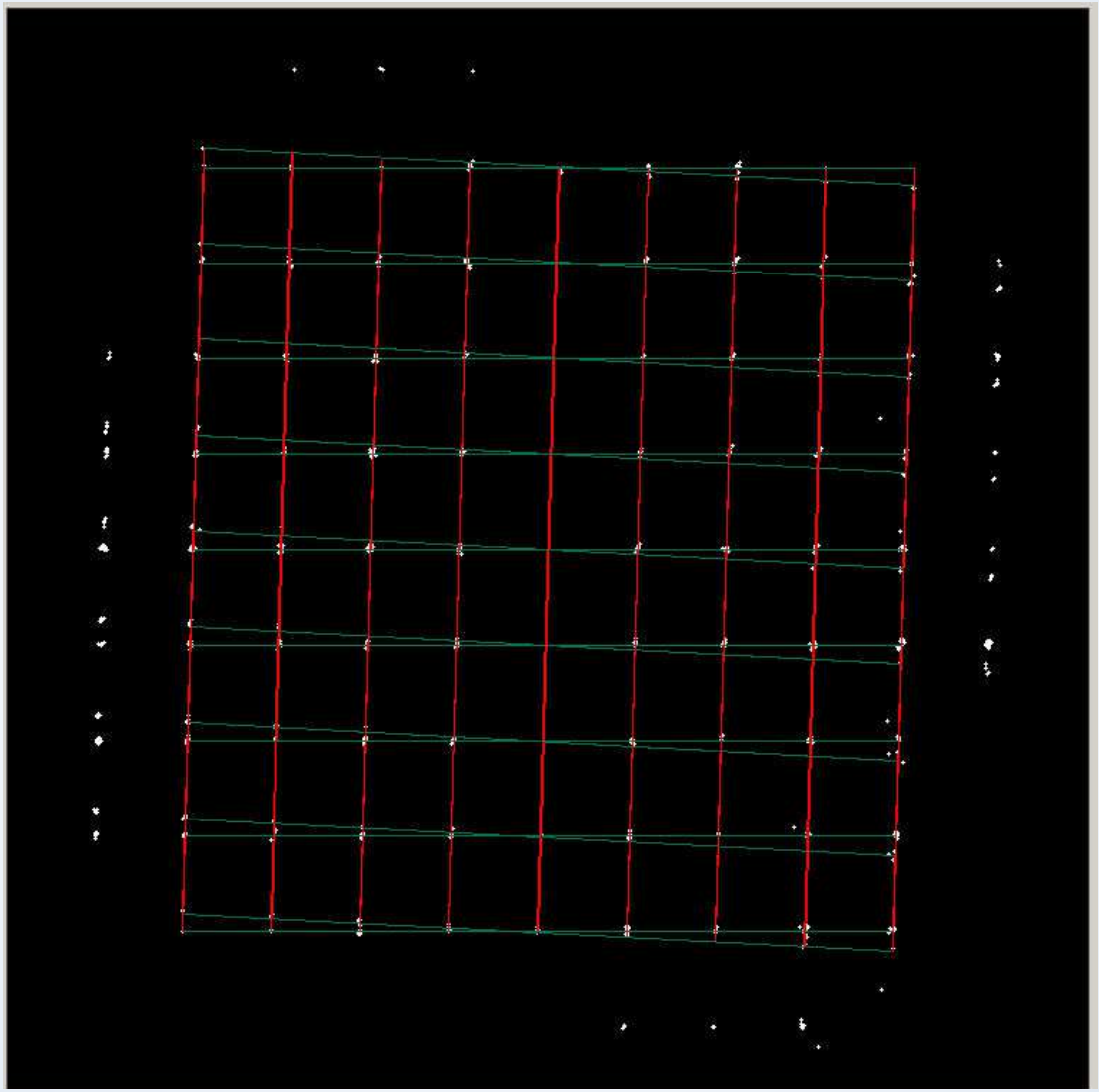
Charge density studies

Similarly as for anharmonic ADP refinement the charge density maps calculated in the direct space can be compared with the error map as follows from the Monte Carlo simulation.

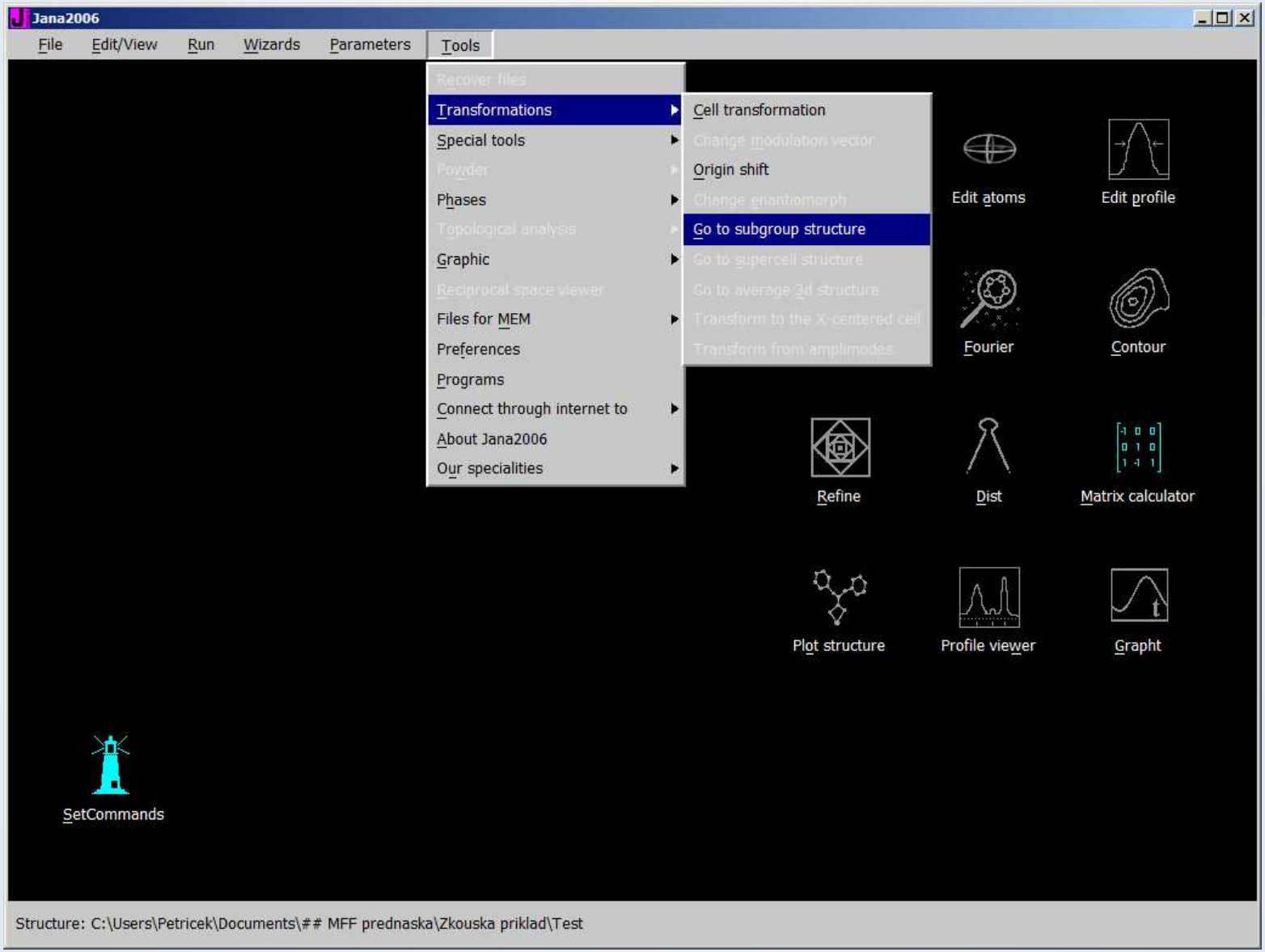




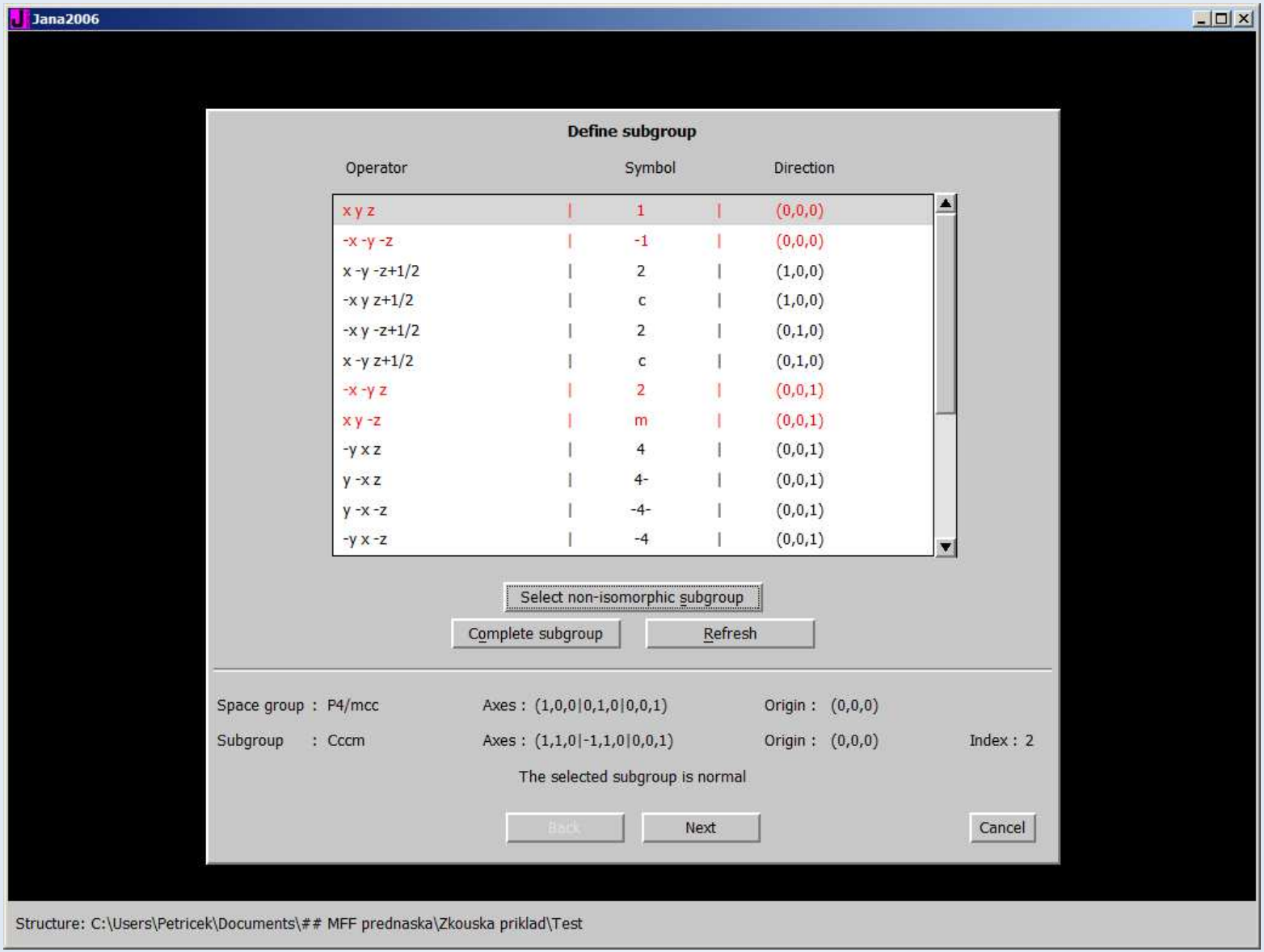
Twins



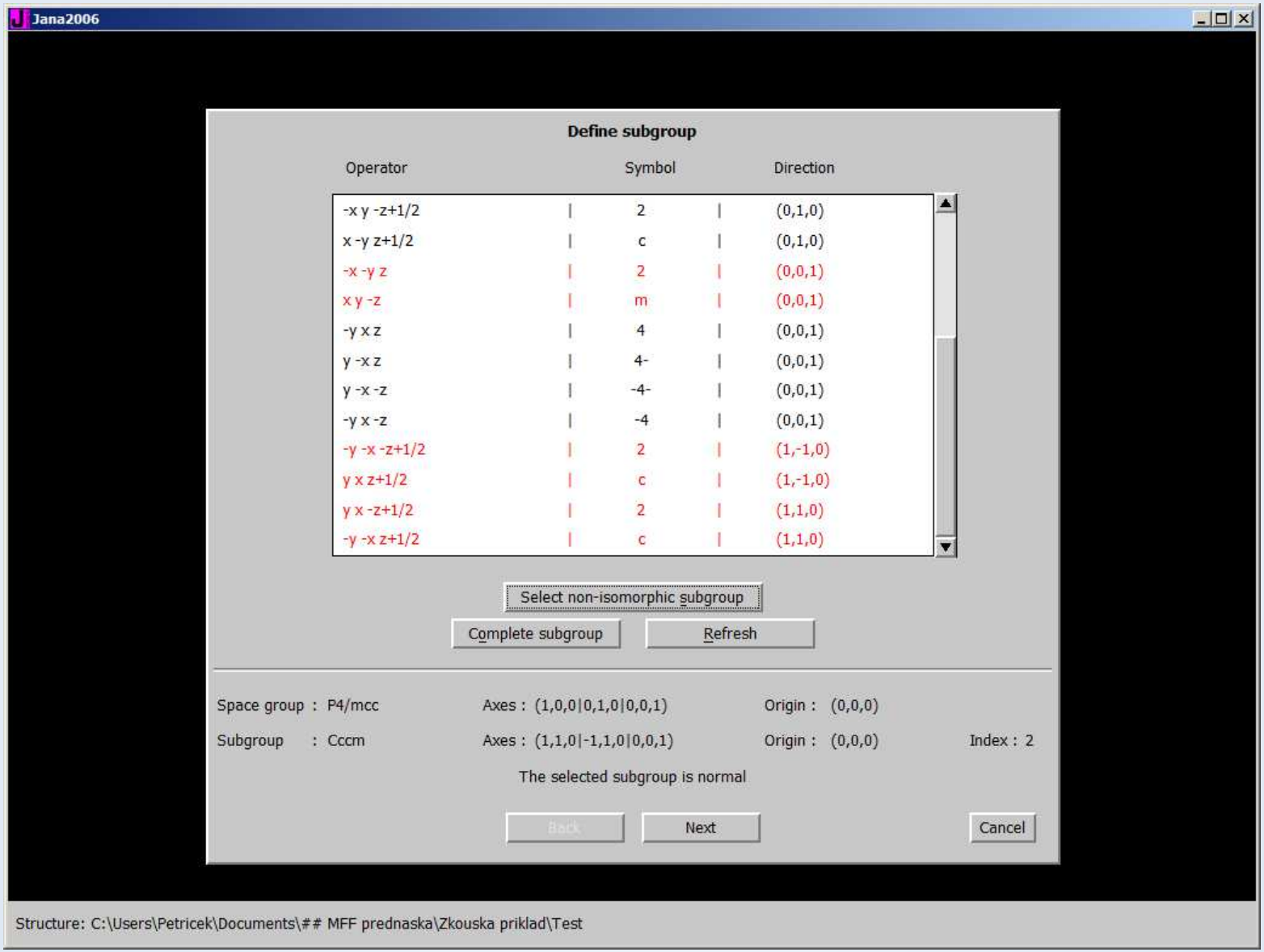
Twins


Jana2006
File
Edit/View
Run
Wizards
Parameters
Tools
Recover files
Transformations
Special tools
Powder
Phases
Topological analysis
Graphic
Reciprocal space viewer
Files for MEM
Preferences
Programs
Connect through internet to
About Jana2006
Our specialities
Cell transformation
Change modulation vector
Origin shift
Change enantiomorph
Go to subgroup structure
Go to supercell structure
Go to average 3d structure
Transform to the X-centered cell
Transform from amplimodes
Edit atoms
Edit profile
Fourier
Contour
Refine
Dist
Matrix calculator
Plot structure
Profile viewer
Grapht
SetCommands
Structure: C:\Users\Petricek\Documents\## MFF prednaska\Zkouska priklad\Test

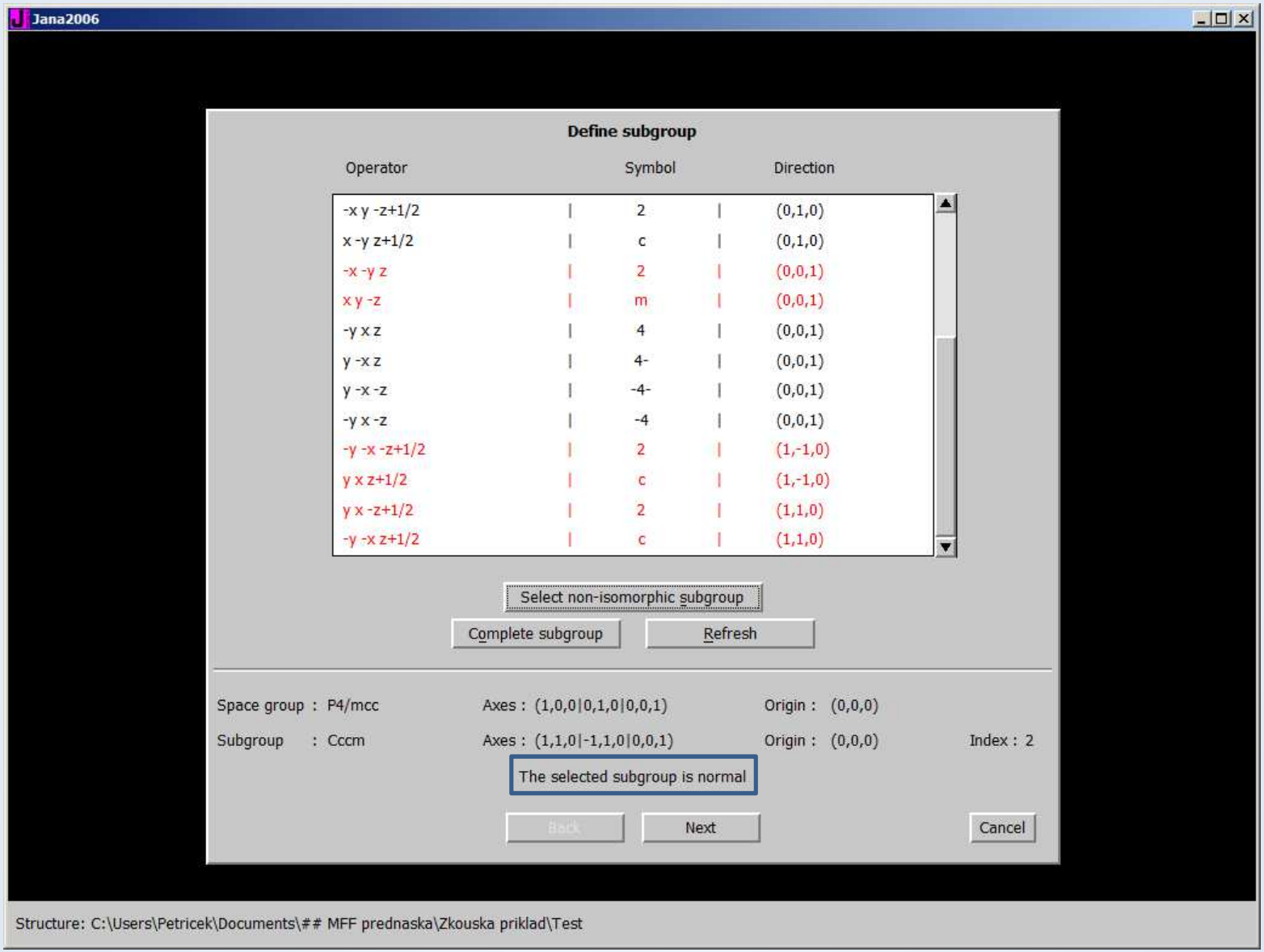
Twins


Jana2006
Define subgroup
Operator
Symbol
Direction
x y z | 1 | (0,0,0)
-x -y -z | -1 | (0,0,0)
x -y -z+1/2 | 2 | (1,0,0)
-x y z+1/2 | c | (1,0,0)
-x y -z+1/2 | 2 | (0,1,0)
x -y z+1/2 | c | (0,1,0)
-x -y z | 2 | (0,0,1)
x y -z | m | (0,0,1)
-y x z | 4 | (0,0,1)
y -x z | 4- | (0,0,1)
y -x -z | -4- | (0,0,1)
-y x -z | -4 | (0,0,1)
Select non-isomorphic subgroup
Complete subgroup
Refresh
Space group : P4/mcc
Axes : (1,0,0|0,1,0|0,0,1)
Origin : (0,0,0)
Subgroup : Cccm
Axes : (1,1,0|-1,1,0|0,0,1)
Origin : (0,0,0)
Index : 2
The selected subgroup is normal
Back
Next
Cancel
Structure: C:\Users\Petricek\Documents\## MFF prednaska\Zkouska priklad\Test

Twins


Jana2006
Define subgroup
Operator
Symbol
Direction
-x y -z+1/2 | 2 | (0,1,0)
x -y z+1/2 | c | (0,1,0)
-x -y z | 2 | (0,0,1)
x y -z | m | (0,0,1)
-y x z | 4 | (0,0,1)
y -x z | 4- | (0,0,1)
y -x -z | -4- | (0,0,1)
-y x -z | -4 | (0,0,1)
-y -x -z+1/2 | 2 | (1,-1,0)
y x z+1/2 | c | (1,-1,0)
y x -z+1/2 | 2 | (1,1,0)
-y -x z+1/2 | c | (1,1,0)
Select non-isomorphic subgroup
Complete subgroup
Refresh
Space group : P4/mcc Axes : (1,0,0|0,1,0|0,0,1) Origin : (0,0,0)
Subgroup : Cccm Axes : (1,1,0|-1,1,0|0,0,1) Origin : (0,0,0) Index : 2
The selected subgroup is normal
Back
Next
Cancel
Structure: C:\Users\Petricek\Documents\## MFF prednaska\Zkouska priklad\Test

Twins


Jana2006
Define subgroup
Operator
Symbol
Direction
-x y -z+1/2 | 2 | (0,1,0)
x -y z+1/2 | c | (0,1,0)
-x -y z | 2 | (0,0,1)
x y -z | m | (0,0,1)
-y x z | 4 | (0,0,1)
y -x z | 4- | (0,0,1)
y -x -z | -4- | (0,0,1)
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Complete subgroup
Refresh
Space group : P4/mcc
Axes : (1,0,0|0,1,0|0,0,1)
Origin : (0,0,0)
Subgroup : Cccm
Axes : (1,1,0|-1,1,0|0,0,1)
Origin : (0,0,0)
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The selected subgroup is normal
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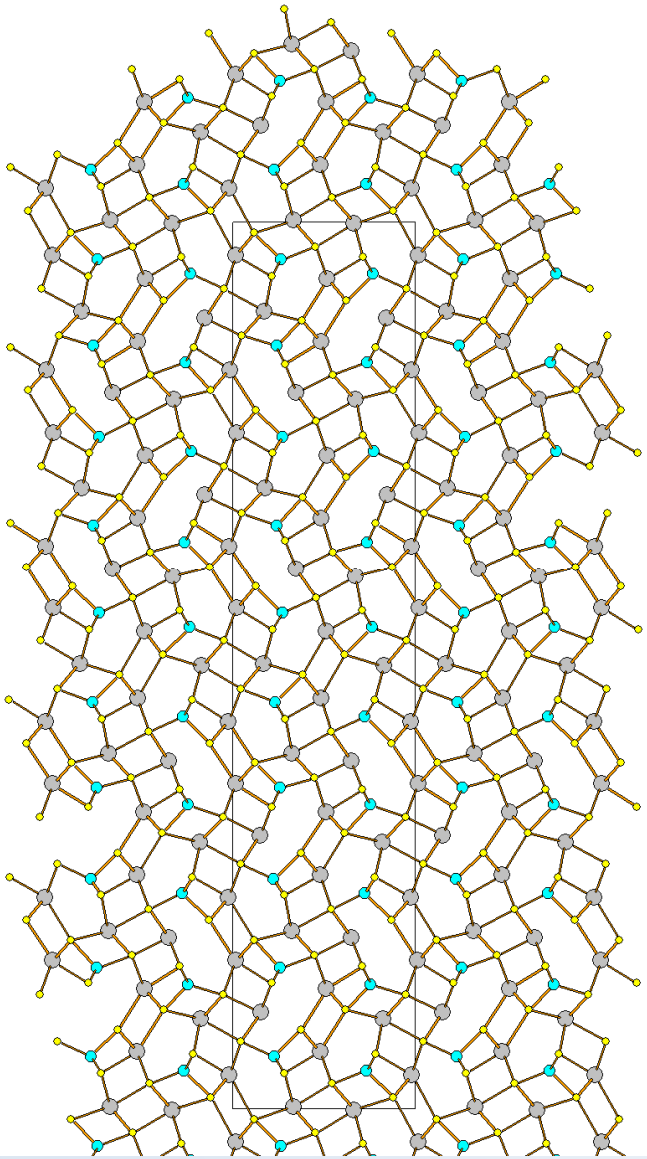
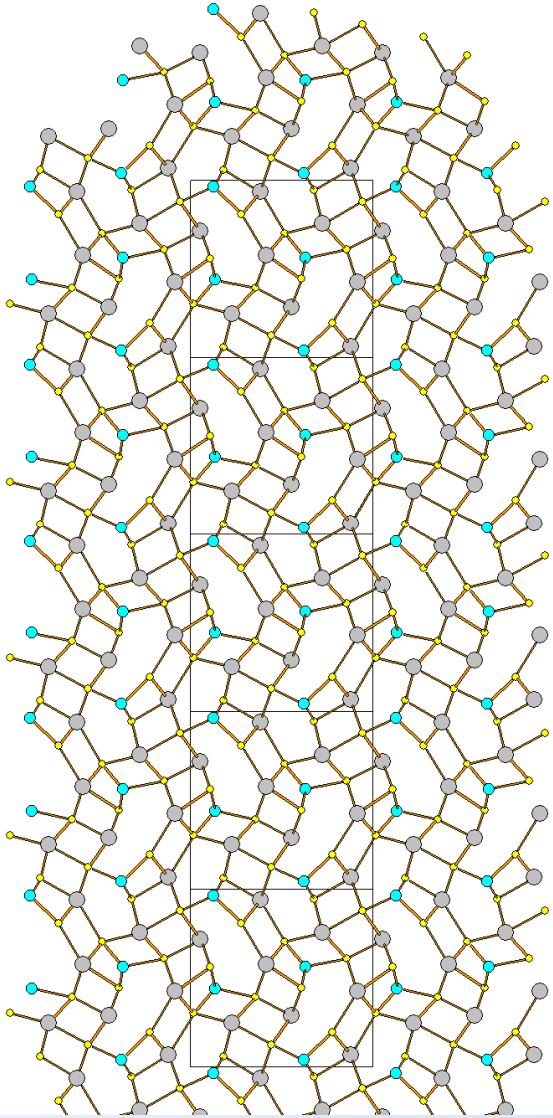
Structure: C:\Users\Petricek\Documents\## MFF prednaska\Zkouska priklad\Test

Twins



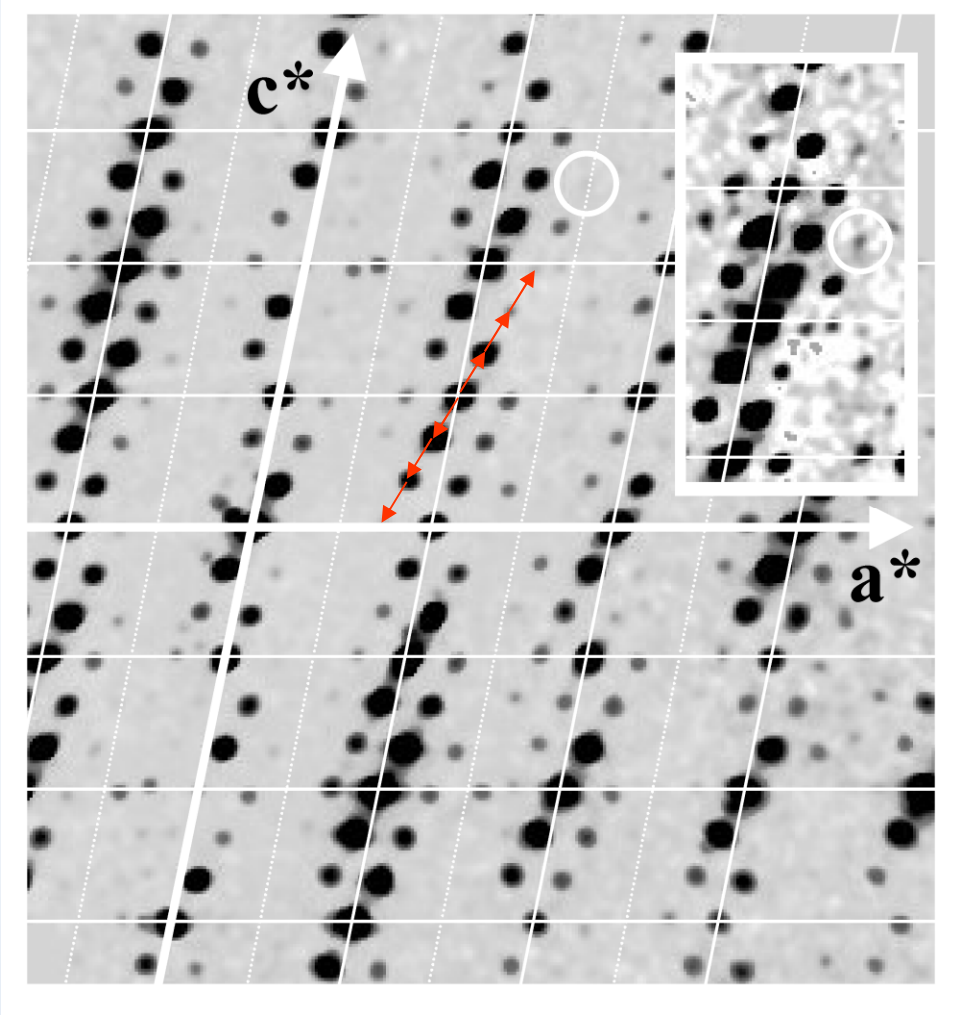
View along **a**
◀Lindströmite

View along **a**
Krupkaite ▶



Modulated structures

Diffraction pattern



Example – Na_2CO_3

Modulated structures

- One or more additional (*modulation)* vectors, have to be added to the reciprocal base in order to index all diffraction spots.
- Diffraction pattern does not have anymore 3d lattice character ⇔ the basic property of crystal 3d translation symmetry is violated but in a specific regular way.
- In the classical definition the object is not crystal → A new definition of crystal is based on its diffraction pattern
- Modulations can considerably change physical properties of the crystal → challenge for structure analysis to say more

Modulated structures

Superspace theory

Ted Janssen

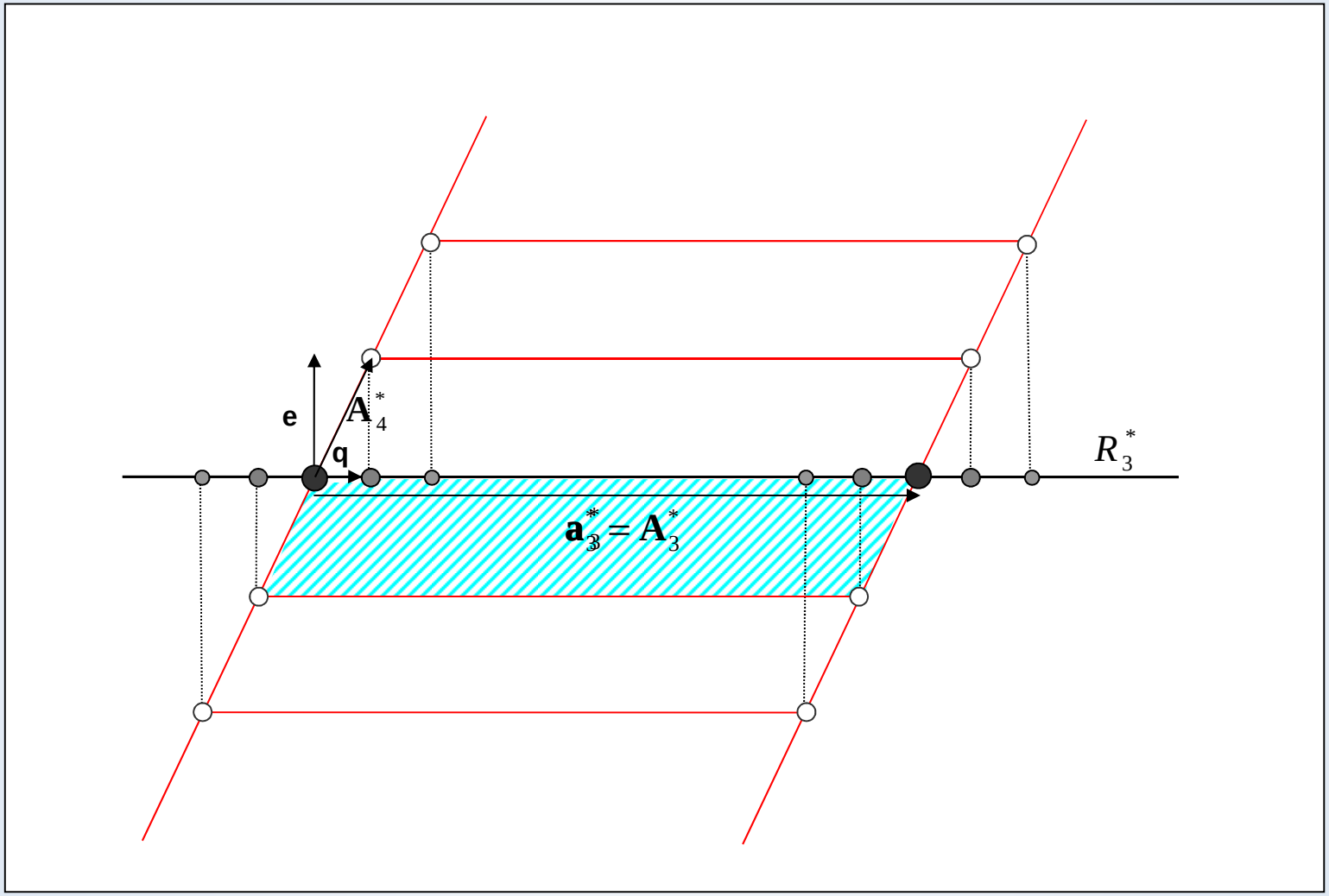
Peter M. de Wolff

Aloysio Janner

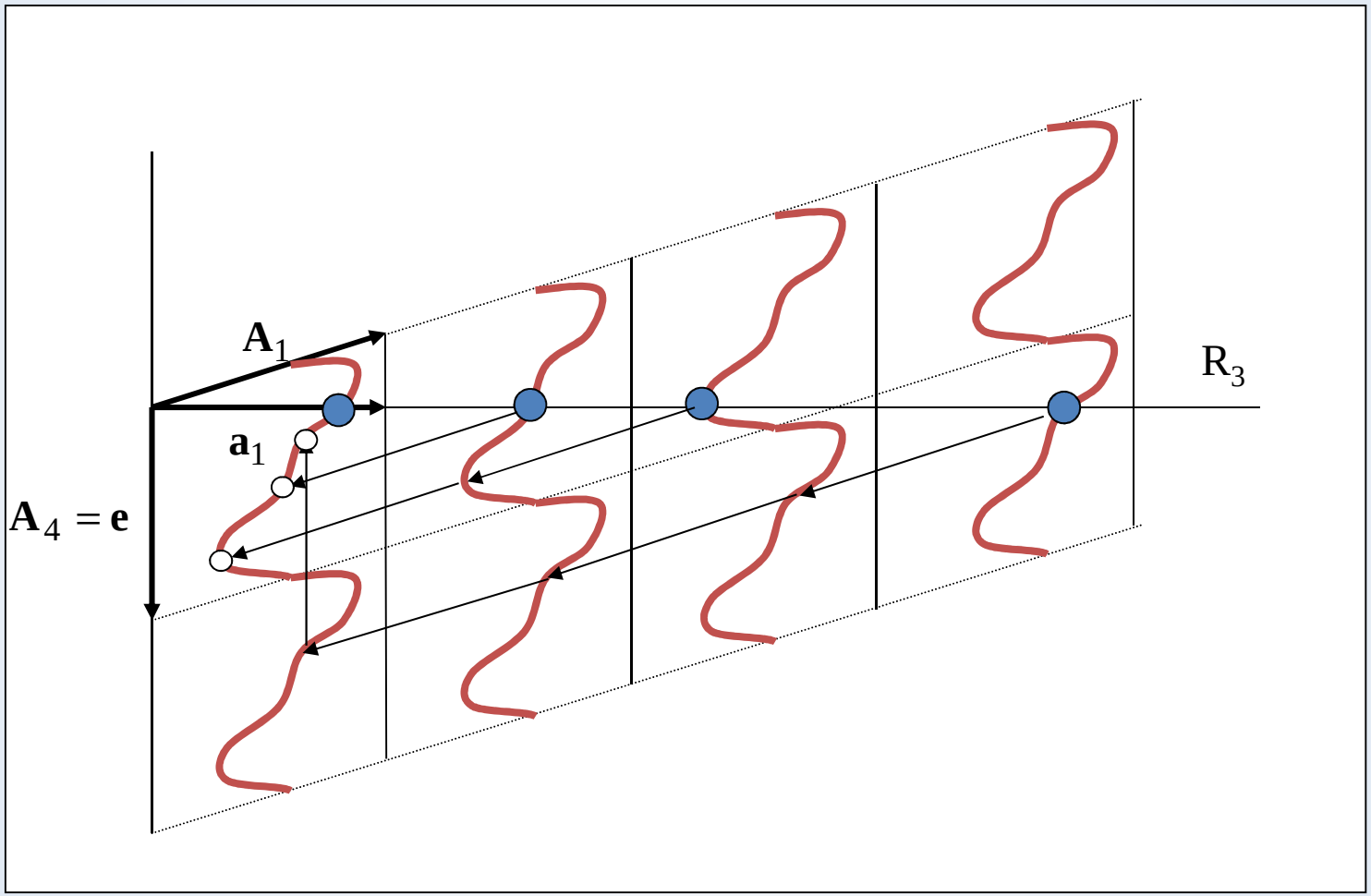
Aminoff prize in 1998

Ewald prize in 2014

Modulated structures



Modulated structures



Modulated structures

Symmetry in the superspace

$$\tilde{\rho}(\mathbf{r}) = \tilde{\rho}(\mathbf{r} + \sum_{i=1}^{3+d} n_i \mathbf{A}_i)$$

$\tilde{\rho}(\hat{S}\mathbf{r}) = \tilde{\rho}(\mathbf{r})$	$\hat{S}\mathbf{r} \cdot \hat{S}\mathbf{r} = \mathbf{r} \cdot \mathbf{r}$	$\hat{S} = (\mathbf{R}, \mathbf{t})$
↓	↓	↓
basic property	unitary operator	matrix representation

Trivial symmetry operator - translation symmetry :

$$\mathbf{R} = \mathbf{E}, \quad \mathbf{t} = \sum_{i=1}^{3+d} n_i \mathbf{A}_i$$

Modulated structures

General symmetry element: $\mathbf{R} = \left(\left[\begin{array}{c|c} \Gamma_E & 0 \\ \hline \Gamma_M & \Gamma_I \end{array} \right], \left[\begin{array}{c} \mathbf{s}_E \\ \hline \mathbf{s}_I \end{array} \right] \right)$

1. From the basic symmetry as determined from main reflections
2. Internal space cannot be mixed up with external one
3. Form the metric properties (unitary conditions) → $\mathbf{q}\Gamma_E - \Gamma_I\mathbf{q} = \Gamma_M$

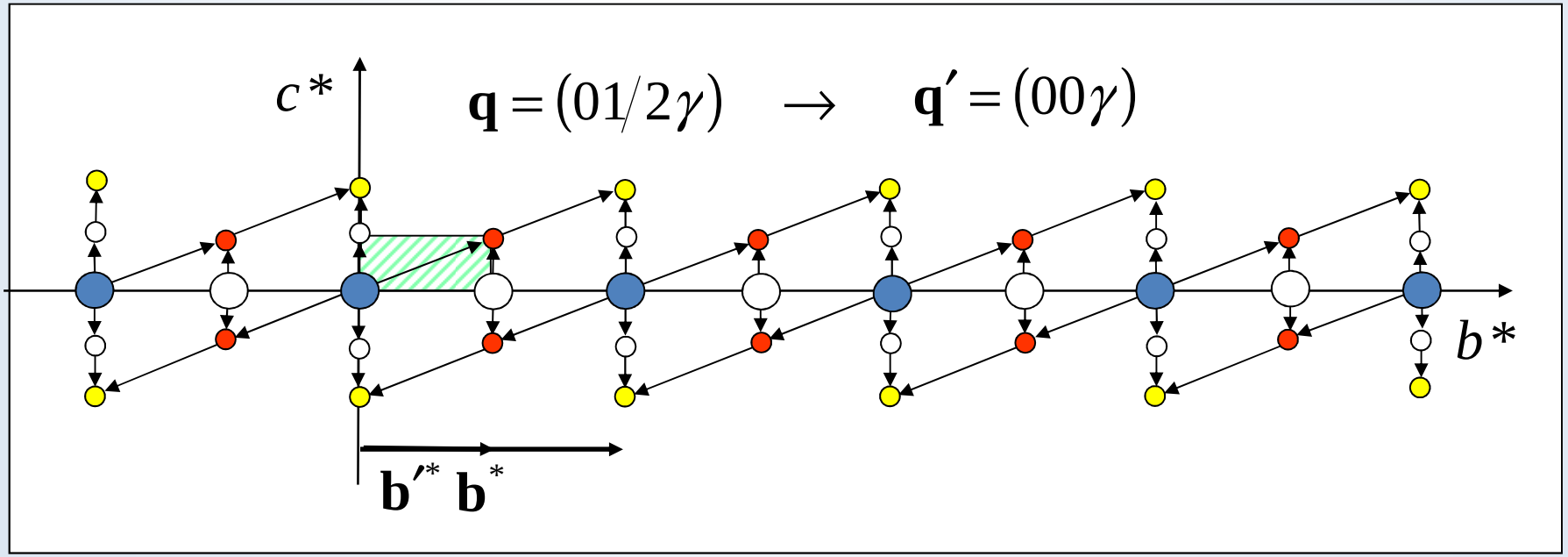
The only new information is the intrinsic part of the 4th component of the translation vector which, analogically to 3d symmetry, affects systematic extinctions of reflections (here satellites). It expressed how the modulation wave is shifted the internal space.

Modulated structures

Super-space group symbols

Example: $Pmna(01/2\gamma)s00$

The rational part of the modulation vector represents an additional centring. It is much more convenient to use the centred cell instead of the explicit use of the rational part of the modulation vector.



Solution of modulated structures

Modulations can affect any of structural parameters - positions, occupancy, ADP's. The most crucial for solution is positional modulation:

$$\mathbf{r}[\mathbf{q}.(\mathbf{r}_0+\mathbf{n})+t]=\mathbf{r}_0+\mathbf{u}[\mathbf{q}.(\mathbf{r}_0+\mathbf{n})+t]$$

Small displacements (weak satellites)

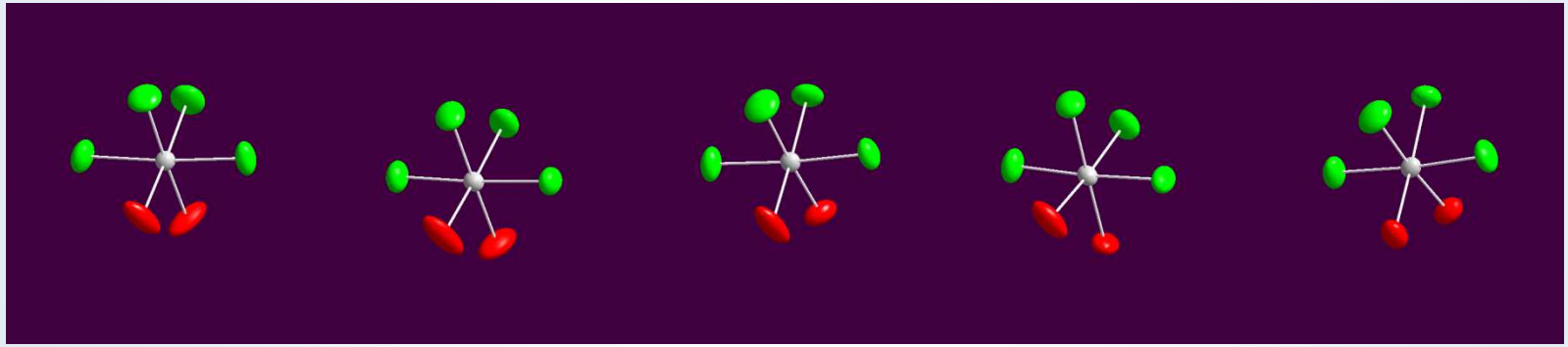
The average structure can be solved by a standard way (direct methods, heavy atom methods) from main reflections. The modulations can be found directly from subsequent refinements. As a starting values for modulations we can use just a small starting values.

Recommended strategy - refine first modulations of atoms exhibiting some anomalies - large ADP's, split positions.

Modulated structures

Example: J. Peterková, M. Dušek V. Petříček & J. Loub, (1998). *Acta Cryst.* B**54**, 809.

$AsKF_4(OH)_2$ – potassium dihydroxotetrafluoroarsenate
the anion $AsF_4(OH)_2$ strongly modulated



Modulated structures

Charge flipping method

A new technique applicable to regular and modulated structures,

For regular structures:

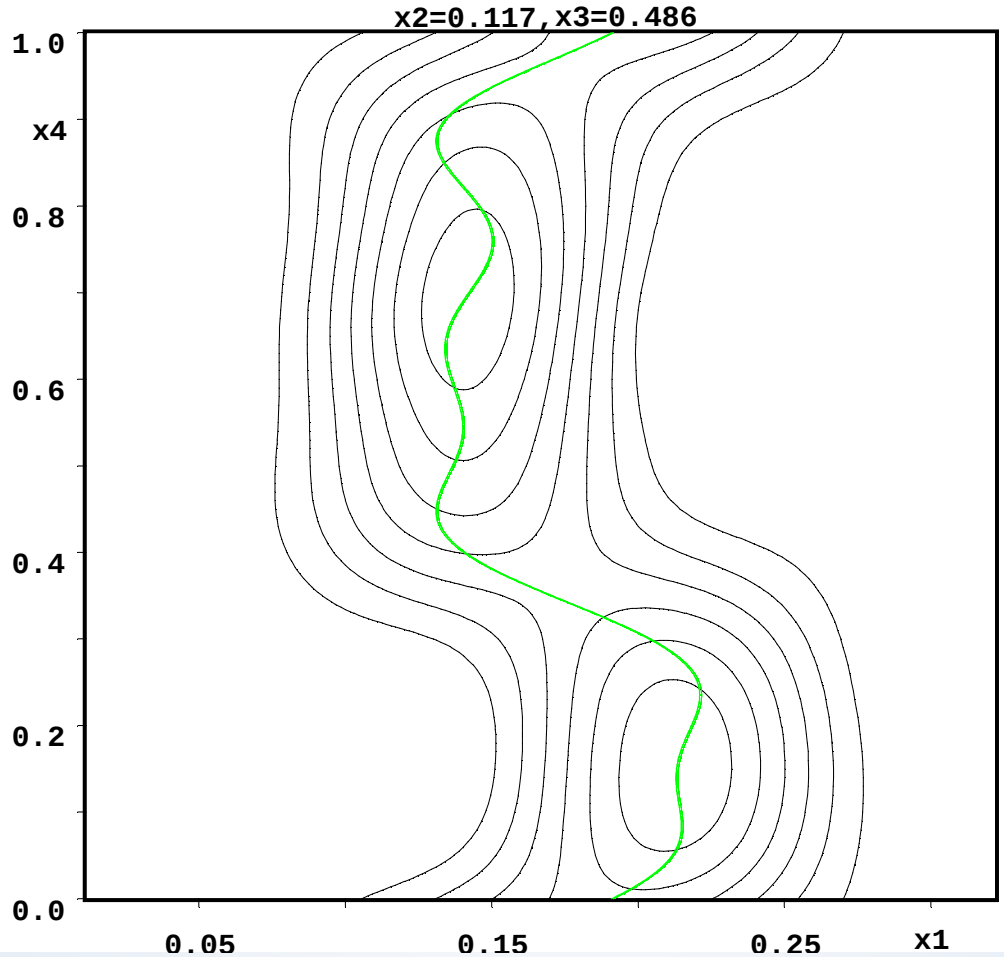
G. Oszlányi and A. Süto, (2004). Acta Cryst. **A60**, 134.

For modulated structures:

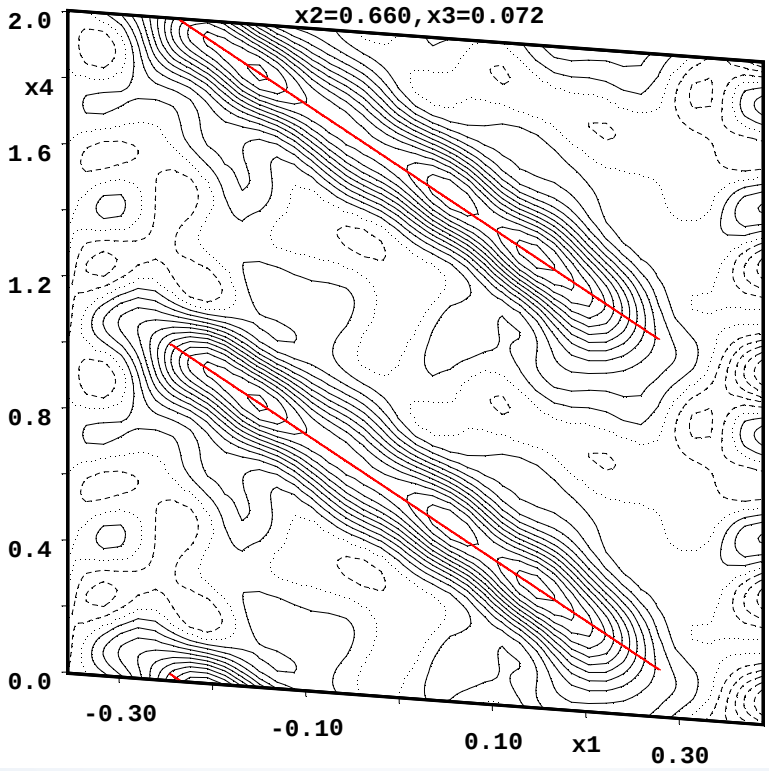
L. Palatinus (2004). *Acta Cryst.* **A60**, 604.
L. Palatinus and G.Chapuis, (2007). *J. Appl. Cryst.* **40**, 786.

It represents a small revolution in the structure analysis of modulated crystals. It is applicable even for strongly modulated crystals having discontinuously modulated occupancies or positions.

Modulated structures



Modulated structures

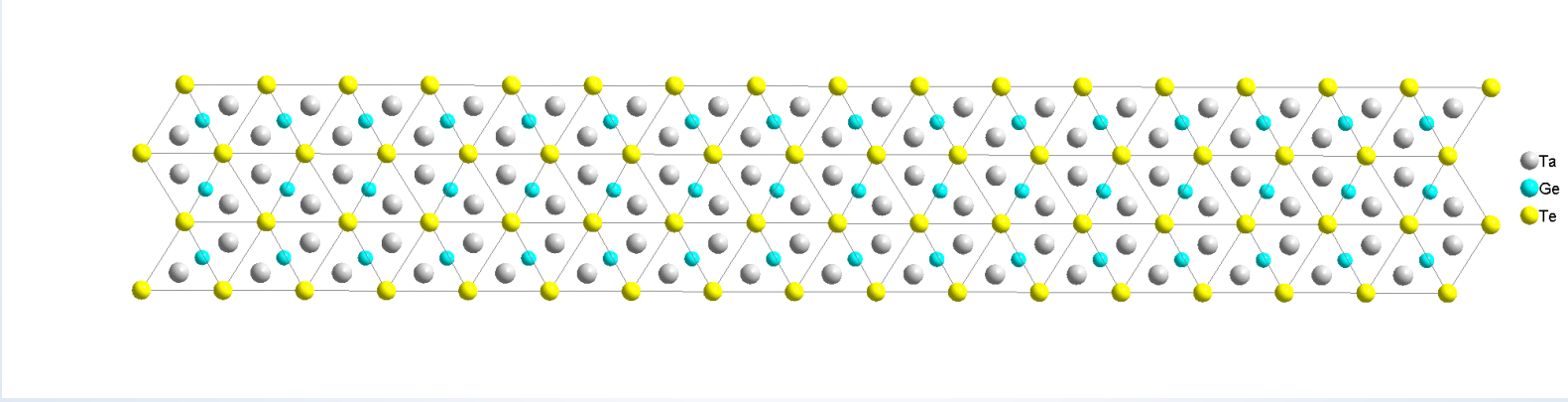


Modulated structures

The modulation curves and corresponding density maps play a crucial role during the solution and refinement process. But a fine presentation should be made in the 3d real space. Several cells and more different sections are to be presented to see various configurations in the modulated crystal:

The same example as for the crenel function - $TaGe_{0.354}Te$ – F. Boucher, M.Evain & V. Petříček, (1996). *Acta Cryst.*, B**52**, 100:

Average structure



Modulated structures

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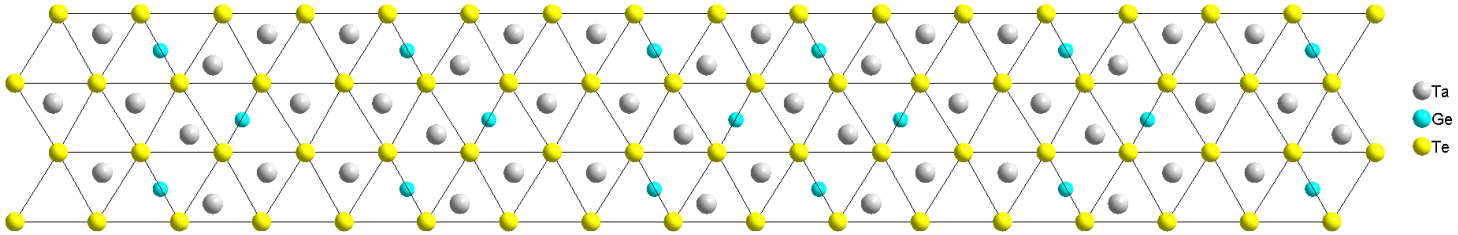
Only occupational modulation



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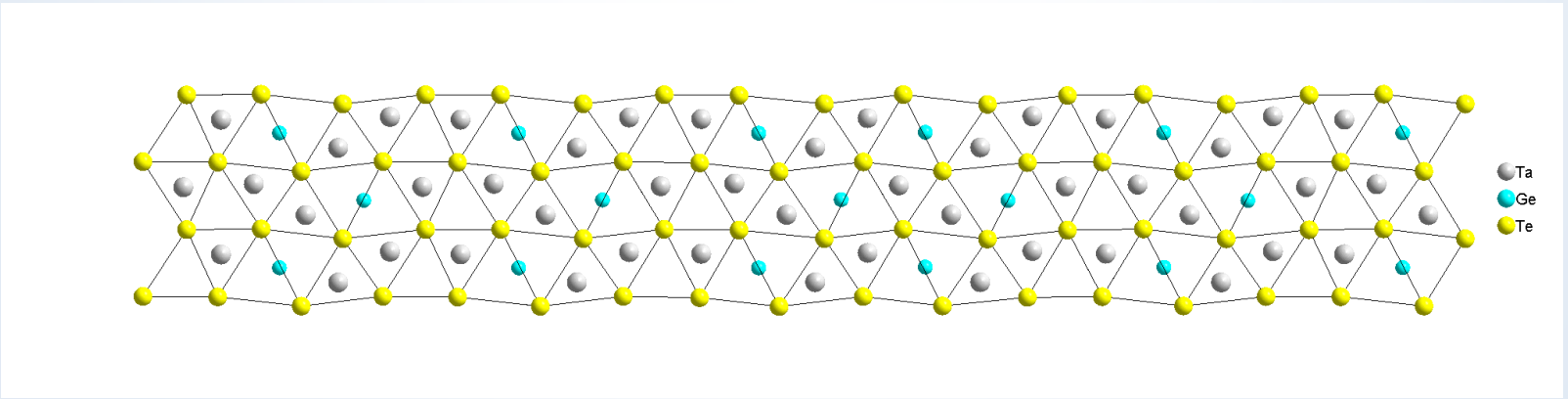
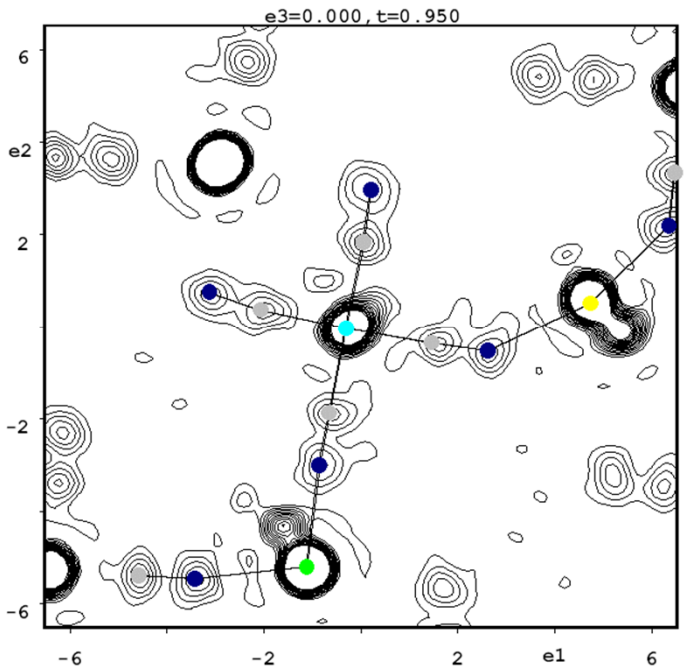
Final result



Modulated structures

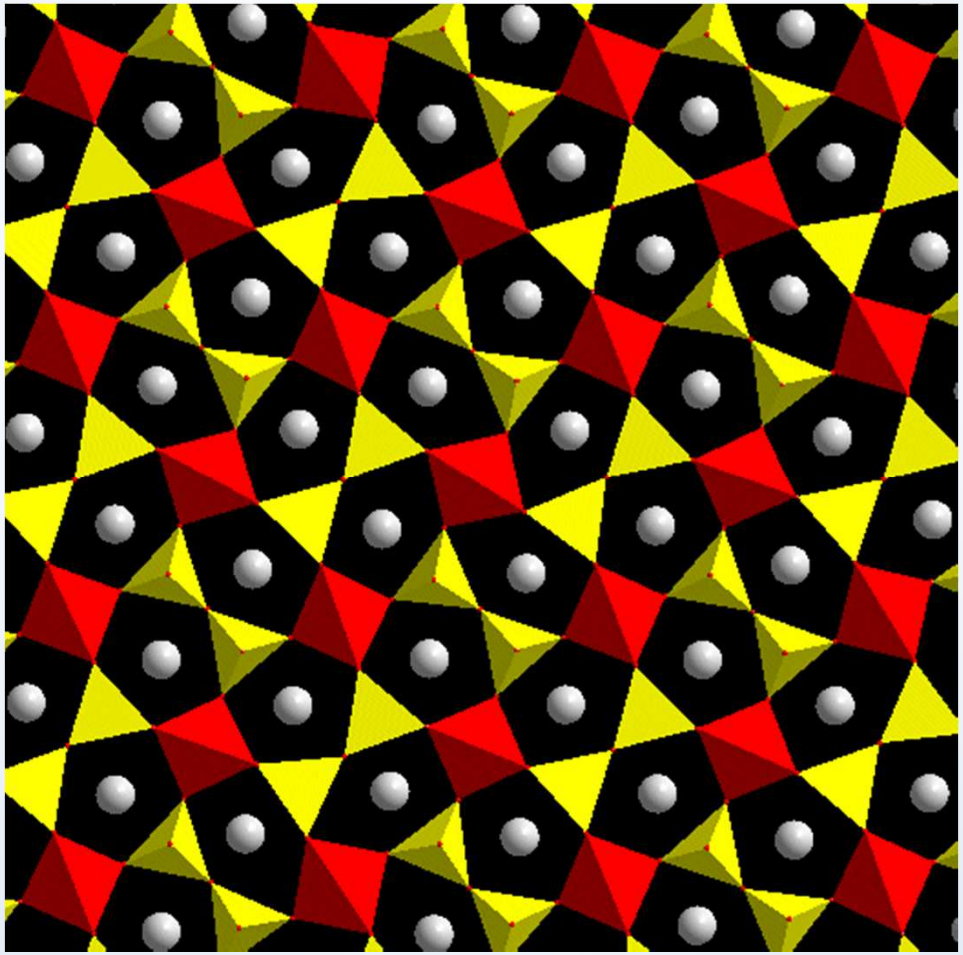
Another way how to present different situations in the crystal is an animation technique.



here combined with the density map.

Modulated structures

L. Bindi, P. Bonazzi, M. Dušek, V. Petříček and G. Chapuis, *Acta Cryst.* (2001). B**57**, 739-746.



Magnetic option

Shubnikov space groups

- Magnetic moment – axial vector
- Any symmetry operation can be combined with “time inversion” - θ, which inverts the magnetic moment

Symmetry operation: $\widehat{\boldsymbol{S}} = (\boldsymbol{R}, \theta|\boldsymbol{s})$

$\boldsymbol{R}$ rotational part (proper or improper)
θ time inversion
$\boldsymbol{s}$ translational part

$$\boldsymbol{r_j} = \boldsymbol{R}\boldsymbol{r}_i + \boldsymbol{s} \qquad \boldsymbol{M_j} = \theta \det(\boldsymbol{R})\boldsymbol{R}\boldsymbol{M_i}$$

Set of symmetry operators of the magnetic crystal → Shubnikov space group.

Magnetic option

Shubnikov space groups

Type I : F – Fedorov space groups
All symmetry operation have θ=1 → magnetic ordering allowed, magnetic reflections could be detected
e.g. *Pmna*

Type II: $F + F.1'$ gray space groups → no magnetic ordering allowed, no magnetic reflections could be detected
e.g. *Pmna1'*

Type III: $\mathrm{D} + (F - D).1'$ where D is equi-translational subgroup of F of the order 2
half of symmetry elements have θ=1, half θ=-1. All translations have θ=1.
e.g. *Pm'n'a*

Type IV: $\mathrm{D} + (F - D).1'$ where D is equi-class subgroup of F of the order 2
There translation vectors having θ=-1. Reflections violating the original translation symmetry will appear as pure magnetic reflections – ordering effect is directly detectable.
e.g. $P_a m'n'a$ (BNS) alternatively $P_{2c} m'mn$ (OG)

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Magnetic option

Shubnikov space groups

Representation analysis:

Representation	Dimension	Shubnikov space group	Axes	Origin shift	
Tau1	1	P[c]-6m2	(1,0,0|0,1,0|0,0,2)	(0,0,0)	Details
Tau2	1	P[c]-6c2	(1,0,0|0,1,0|0,0,2)	(0,0,0)	Details
Tau3	1	P[c]-6c2	(1,0,0|0,1,0|0,0,2)	(0,0,1/4)	Details
Tau4	1	P[c]-6m2	(1,0,0|0,1,0|0,0,2)	(0,0,1/4)	Details
Tau5	2	P[b]m	(0,1,0|0,0,2|1,0,0)	(0,1/4,0)	Details
Tau6	2	P[b]m	(0,1,0|0,0,2|1,0,0)	(0,0,0)	Details

Display representations

Back | Next | Cancel

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Magnetic option

Shubnikov space groups

List of kernels and epikernels:

Shubnikov space group	Axes	Origin shift	Representation
P[c]-6m2	(1,0,0|0,1,0|0,0,2)	(0,0,0)	Tau1
P[c]-6c2	(1,0,0|0,1,0|0,0,2)	(0,0,0)	Tau2
P[c]-6c2	(1,0,0|0,1,0|0,0,2)	(0,0,1/4)	Tau3
P[c]-6m2	(1,0,0|0,1,0|0,0,2)	(0,0,1/4)	Tau4
A[a]ma2	(0,0,2|1,1,0|-1,1,0)	(0,0,0)	Tau5
A[a]mm2	(0,0,2|1,1,0|-1,1,0)	(1/4,0,0)	Tau5
A[a]mm2	(0,0,2|1,1,0|-1,1,0)	(0,0,0)	Tau6
A[a]ma2	(0,0,2|1,1,0|-1,1,0)	(1/4,0,0)	Tau6
P[b]m	(0,1,0|0,0,2|1,0,0)	(0,1/4,0)	Tau5
P[b]m	(0,1,0|0,0,2|1,0,0)	(0,0,0)	Tau6

Show details

Select from above kernels/epikernels one representative of a family of Shubnikov space groups for testing.

Back Next Cancel

Magnetic option

Shubnikov superspace groups

The superspace approach developed by de Wollf, P.M., Janssen T. & Janner, A. (1981). *Acta Cryst*., A**37**, 625-636 can be used to describe periodical perturbations, commensurate or incommensurate with the basic 3 dimensional translation symmetry.

For magnetic structures :
Petříček V., Fuksa J. and Dušek M. *Acta Cryst*. (2010). A**66**, 649-655.
Perez-Mato J.M., Ribeiro J.L., Petříček V. and Aroyo, M.I. *J.Phys.: Condens. Matter* (2012). **24**, 163201.

$$\boldsymbol{M}_i = \boldsymbol{M}_{i,0} + \sum_m \boldsymbol{M}_{i,ms} \sin 2\pi m \boldsymbol{k}\boldsymbol{r}_0 + \sum_m \boldsymbol{M}_{i,mc} \cos 2\pi m \boldsymbol{k}\boldsymbol{r}_0$$

Magnetic option

Shubnikov superspace groups

Superspace symmetry operation:

$$\widehat{\boldsymbol{S}} = \left(\begin{bmatrix} \boldsymbol{R}_E & 0 \\ \boldsymbol{R}_M & \boldsymbol{R}_I \end{bmatrix}, \theta | \boldsymbol{s} \right)$$

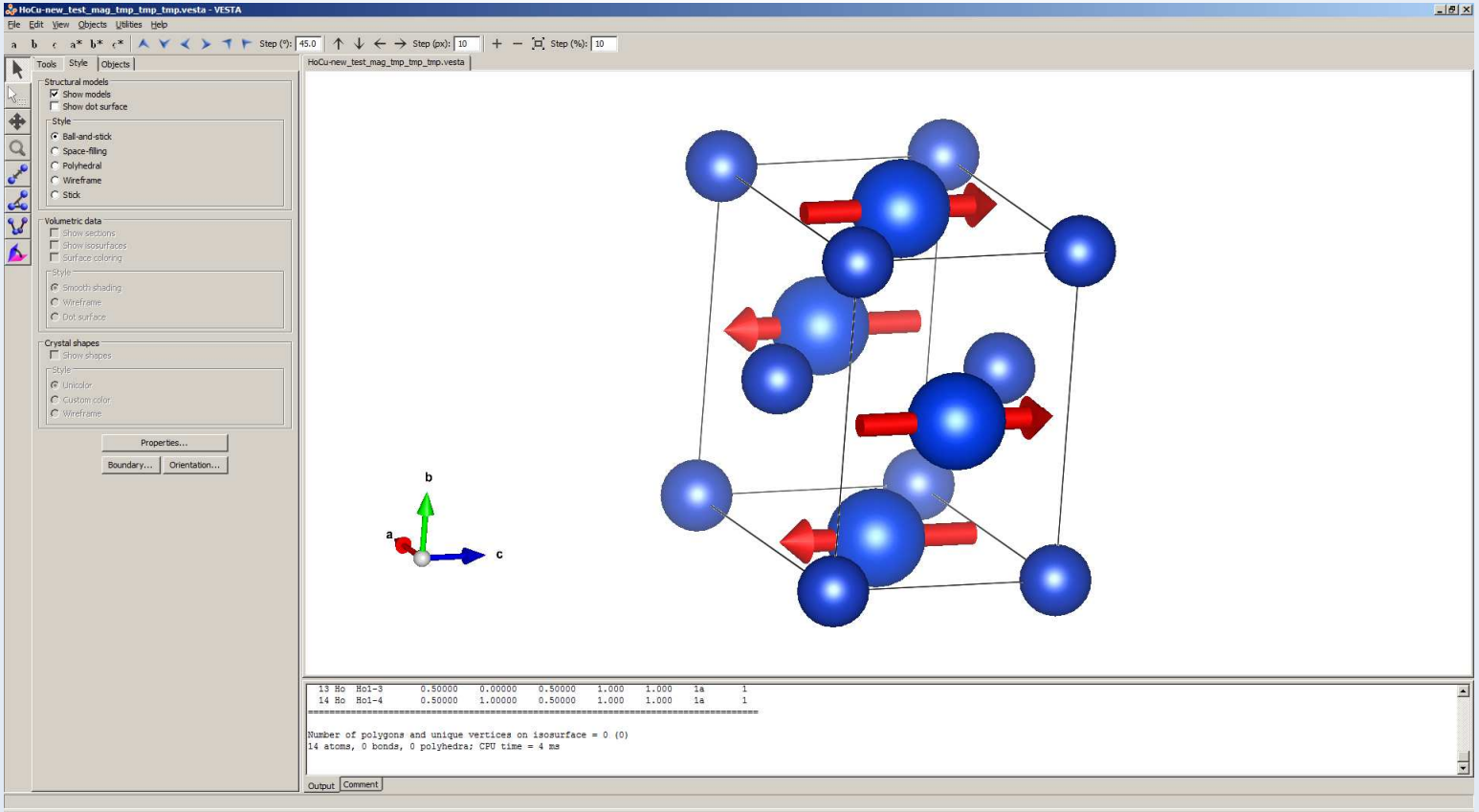
Where $\boldsymbol{R}_E$, $\boldsymbol{R}_M$ and $\boldsymbol{R}_I$ are external 3×3, mixed 1×3, internal 1×1 block matrices and the time inversion sign, respectively. The right-upper 3×1 block is a zero column.

non-zero matrix blocks are related: $\boldsymbol{R}_M = \boldsymbol{k}\boldsymbol{R}_E - \boldsymbol{R}_I\boldsymbol{k}$

Time inversion of two types: $1' \colon 0 = (\boldsymbol{E}, -1|0,0,0,0)$ and $1' \colon \mathrm{s} = (\boldsymbol{E}, -1|0,0,0,1/2)$

Magnetic option

Visualization of magnetic structure
with VESTA program (Koichi Momma and Fujio Izumi)



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Magnetic option

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ISODISTORT – H.T. Stokes, B. J. Campbell and D. M. Hatch

ISODISTORT: distortion

Space Group: 194 P6_3/mmc D6h-4, Lattice parameters: a=5.63760, b=5.63760, c=24.15760, alpha=90.00000, beta=90.00000, gamma=120.00000
Default space-group preferences: monoclinic axes a(b)c, monoclinic cell choice 1, orthorhombic axes abc, origin choice 2, hexagonal axes, SSG standard setting
Ba1 4f (1/3,2/3,z), z=-0.04041, Ba2 4f (1/3,2/3,z), z=0.36991, Ba3 2b (0,0,1/4), Co4 2a (0,0,0), Co5 4e (0,0,z), z=0.39525, Co6 4f (1/3,2/3,z), z=-0.17818, Cl1 2c (1/3,2/3,1/4), O2 12k (x,2x,z), x=0.16063, z=-0.14926, O3 12k (x,2x,z), x=-0.14584, z=-0.04948, O4 2d (1/3,2/3,3/4)
Include magnetic Co distortions
k point: GM, k16 (0,0,0)
IR: mGM2+, mk16t3
P1 (a) 194.270 P6_3/mm'c', basis={(0,-1,0),(1,1,0),(0,0,1)}, origin=(0,0,0), s=1, i=2, k-active= (0,0,0)
Lattice parameters of undistorted supercell: a=5.63760, b=5.63760, c=24.15760, alpha=90.00000, beta=90.00000, gamma=120.00000

○ View distortion ○ View diffraction ◉ CIF file ○ Distortion file ○ Domains ○ Primary order parameters ○ Modes details ○ Complete modes details ○ TOPAS.STR ○ IR matrices OK

Enter mode and strain amplitudes:

P6_3/mmc[0,0,0]mGM2+ (a) 194.270 P6_3/mm'c', basis={(0,-1,0),(1,1,0),(0,0,1)}, origin=(0,0,0), s=1 i=2, ferromagnetic, k-active= (0,0,0)

0.1 [Co4:a]A2g(a)

0.1 [Co5:e]A2(a)

0.1 [Co6:f]A2(a)

P6_3/mmc[0,0,0]GM1+ (a) 194.264 P6_3/mmc s=1 i=1

Parameters:
"View distortion":
Atomic radius: 0.4 Angstroms
Maximum bond length: 1.91 Angstroms
Length of magnetic moment vectors: 0.5 Angstroms/magneton
Applet width: 1024 pixels
Supercell enlargement: 1 x 1 x 1
"View distortion" and "View diffraction":
Maximum displacement per mode: 1.0 Angstroms
Maximum strain per mode: 0.1
Maximum occupancy per mode: 1.0
Maximum magnetic moment per mode: 4.0 magnetons
Include strain modes in TOPAS.STR: ○yes ◉no

☐ Use alternate (possibly nonstandard) setting in CIF file
Basis vectors of subgroup lattice relative to parent:
a' = a + b + c
b' = a + b + c
c' = a + b + c
Origin of subgroup relative to parent: a + b + c

Password for beta version:

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Magnetic option

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Bilbao Crystallographic Server – M. Aroyo, J.M. Pérez-Mato

Sections

- Retrieval Tools
- Magnetic Space Groups
- Group-Subgroup
- Representations
- Solid State
- Structure Utilities
- Subperiodic
- Incommensurate Structures Database
- Raman and Hyper-Raman scattering

- Contact us
- About us
- Links
- Publications
- How to cite the server
- Tutorials

Material from the ITOnline Workshop (September 2011)

Material from the school on the server (June 2009)

MAXSUB	Maximal Subgroups of Space Groups
SERIES	Series of Maximal Isomorphic Subgroups of Space Groups
WYCKSETS	Equivalent Sets of Wyckoff Positions
NORMALIZER	Normalizers of Space Groups
KVEC	The k-vector types and Brillouin zones of Space Groups
SYMMETRY OPERATIONS	Geometric interpretation of matrix column representations of symmetry operations

Magnetic Space Groups

MGENPOS	General Positions of Magnetic Space Groups
MWYCKPOS	Wyckoff Positions of Magnetic Space Groups
MAGNEXT	Extinction Rules of Magnetic Space Groups
MAXMAGN	Maximal magnetic space groups for a given space group and a propagation vector

Group - Subgroup Relations of Space Groups

SUBGROUPGRAPH	Lattice of Maximal Subgroups
HERMANN	Distribution of subgroups in conjugated classes
COSETS	Coset decomposition for a group-subgroup pair
WYCKSPLIT	The splitting of the Wyckoff Positions
MINSUP	Minimal Supergroups of Space Groups
SUPERGROUPS	Supergroups of Space Groups
CELLSUB	List of subgroups for a given k-index.
CELLSUPER	List of supergroups for a given k-index.
NONCHAR	Non Characteristic orbits.
COMMONSUBS	Common Subgroups of Space Groups
COMMONSUPER	Common Supergroups of Two Space Groups

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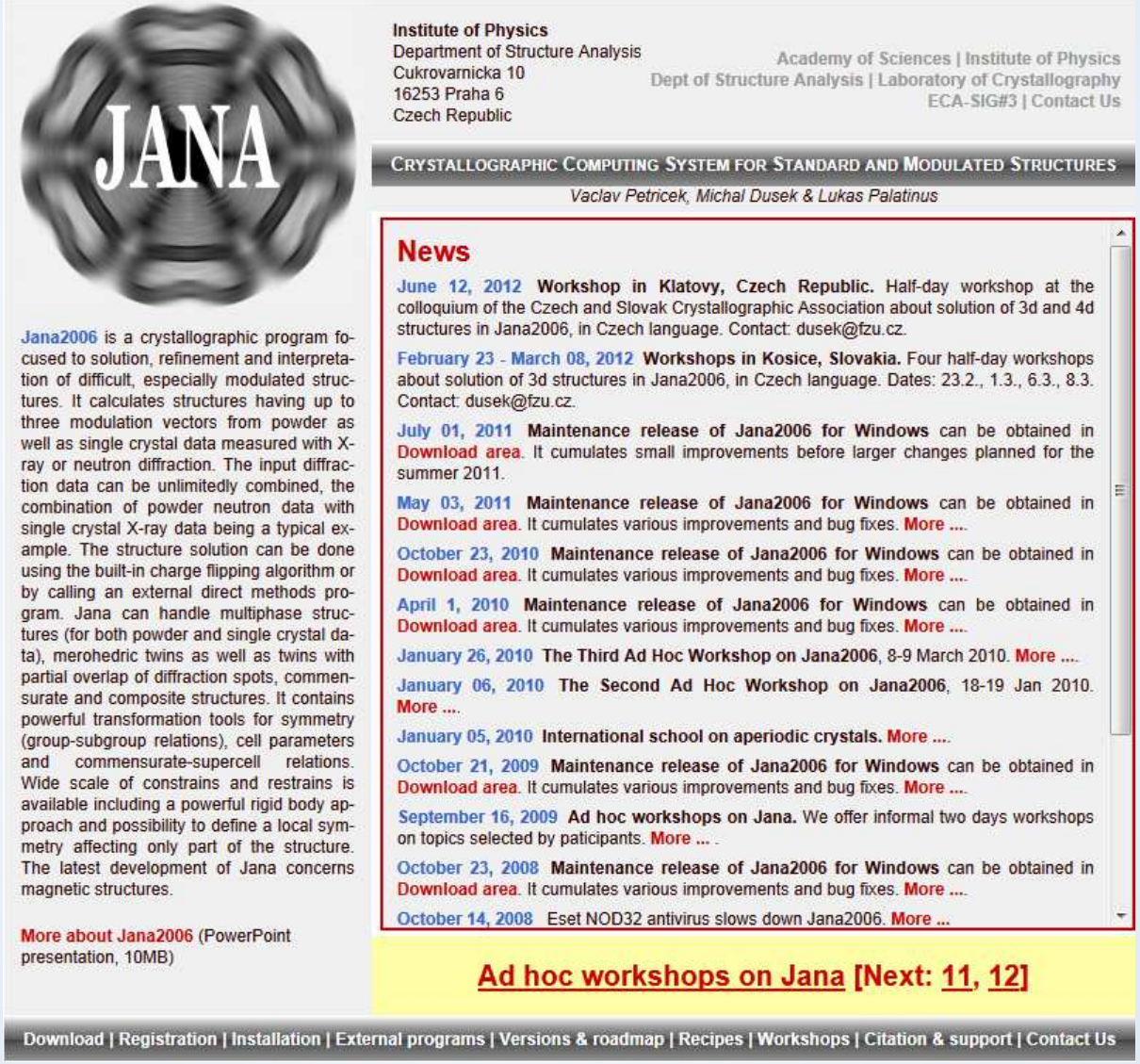
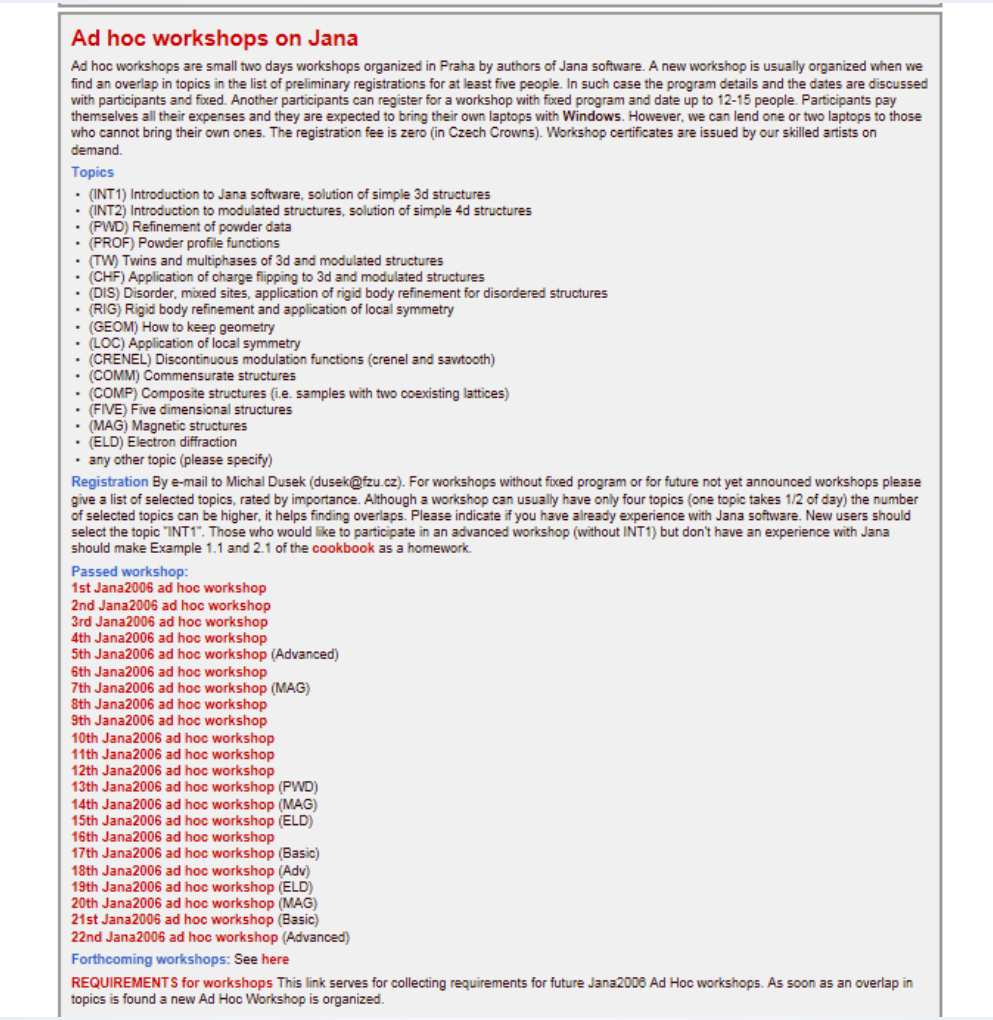
User support



Ad hoc workshops on Jana



Ad hoc workshops on Jana

Ad hoc workshops are small two days workshops organized in Praha by authors of Jana software. A new workshop is usually organized when we find an overlap in topics in the list of preliminary registrations for at least five people. In such case the program details and the dates are discussed with participants and fixed. Another participants can register for a workshop with fixed program and date up to 12-15 people. Participants pay themselves all their expenses and they are expected to bring their own laptops with **Windows**. However, we can lend one or two laptops to those who cannot bring their own ones. The registration fee is zero (in Czech Crowns). Workshop certificates are issued by our skilled artists on demand.

Topics

- (INT1) Introduction to Jana software, solution of simple 3d structures
- (INT2) Introduction to modulated structures, solution of simple 4d structures
- (PWD) Refinement of powder data
- (PROF) Powder profile functions
- (TW) Twins and multiphases of 3d and modulated structures
- (CHF) Application of charge flipping to 3d and modulated structures
- (DIS) Disorder, mixed sites, application of rigid body refinement for disordered structures
- (RIG) Rigid body refinement and application of local symmetry
- (GEOM) How to keep geometry
- (LOC) Application of local symmetry
- (CRENEL) Discontinuous modulation functions (crenel and sawtooth)
- (COMM) Commensurate structures
- (COMP) Composite structures (i.e. samples with two coexisting lattices)
- (FIVE) Five dimensional structures
- (MAG) Magnetic structures
- (ELD) Electron diffraction
- any other topic (please specify)

Registration By e-mail to Michal Dusek (dusek@fzu.cz). For workshops without fixed program or for future not yet announced workshops please give a list of selected topics, rated by importance. Although a workshop can usually have only four topics (one topic takes 1/2 of day) the number of selected topics can be higher, it helps finding overlaps. Please indicate if you have already experience with Jana software. New users should select the topic "INT1". Those who would like to participate in an advanced workshop (without INT1) but don't have an experience with Jana should make Example 1.1 and 2.1 of the cookbook as a homework.

Passed workshop:
1st Jana2006 ad hoc workshop
2nd Jana2006 ad hoc workshop
3rd Jana2006 ad hoc workshop
4th Jana2006 ad hoc workshop
5th Jana2006 ad hoc workshop (Advanced)
6th Jana2006 ad hoc workshop
7th Jana2006 ad hoc workshop (MAG)
8th Jana2006 ad hoc workshop
9th Jana2006 ad hoc workshop
10th Jana2006 ad hoc workshop
11th Jana2006 ad hoc workshop
12th Jana2006 ad hoc workshop
13th Jana2006 ad hoc workshop (PWD)
14th Jana2006 ad hoc workshop (MAG)
15th Jana2006 ad hoc workshop (ELD)
16th Jana2006 ad hoc workshop
17th Jana2006 ad hoc workshop (Basic)
18th Jana2006 ad hoc workshop (Adv)
19th Jana2006 ad hoc workshop (ELD)
20th Jana2006 ad hoc workshop (MAG)
21st Jana2006 ad hoc workshop (Basic)
22nd Jana2006 ad hoc workshop (Advanced)

Forthcoming workshops: See here

REQUIREMENTS for workshops This link serves for collecting requirements for future Jana2006 Ad Hoc workshops. As soon as an overlap in topics is found a new Ad Hoc Workshop is organized.

For more details: http://jana.fzu.cz/
Praha – Prague (Czech Republic)

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Jana cookbook

FYZIKÁLNÍ ÚSTAV
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Acknowledgements

The program is freeware: result of scientific research made with a close collaboration with Jana users

Work is continuously supported by Academy of Sciences of the Czech Republic and (occasionally) by Grant agency of the Czech Republic

Questions???

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End of the first part

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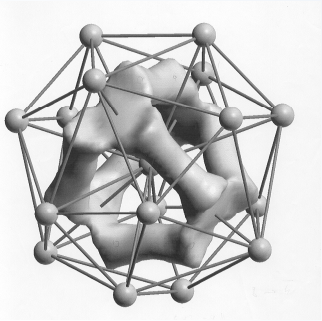
Jana2006 – powder option

Václav Petříček, Jan Rohlíček and Michal Dušek
Institute of Physics
Academy of Sciences of the Czech Republic
Praha

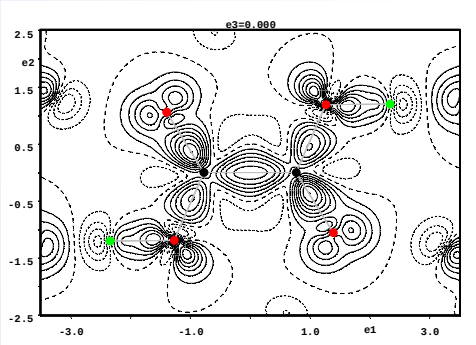
Functionality for powders

Jana2006 is a refinement program originally developed for single crystal data. Except for the refinement of basic structural parameters it allows also:

ADP parameters till 6th order – **ionic conductors, positional disorder**

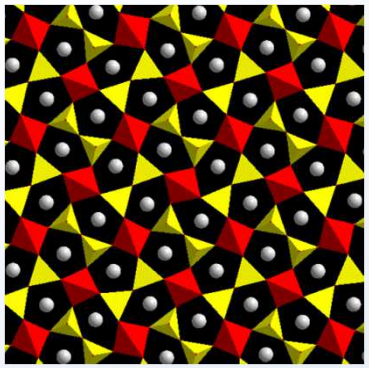


E.Gaudin, F.Boucher, V.Petříček, F.Taulelle and M.Evain, Acta Cryst. (2000), **B56, 402-408.**



Charge density studies - multipole refinement

Refinement of **modulated** and **composite** structures

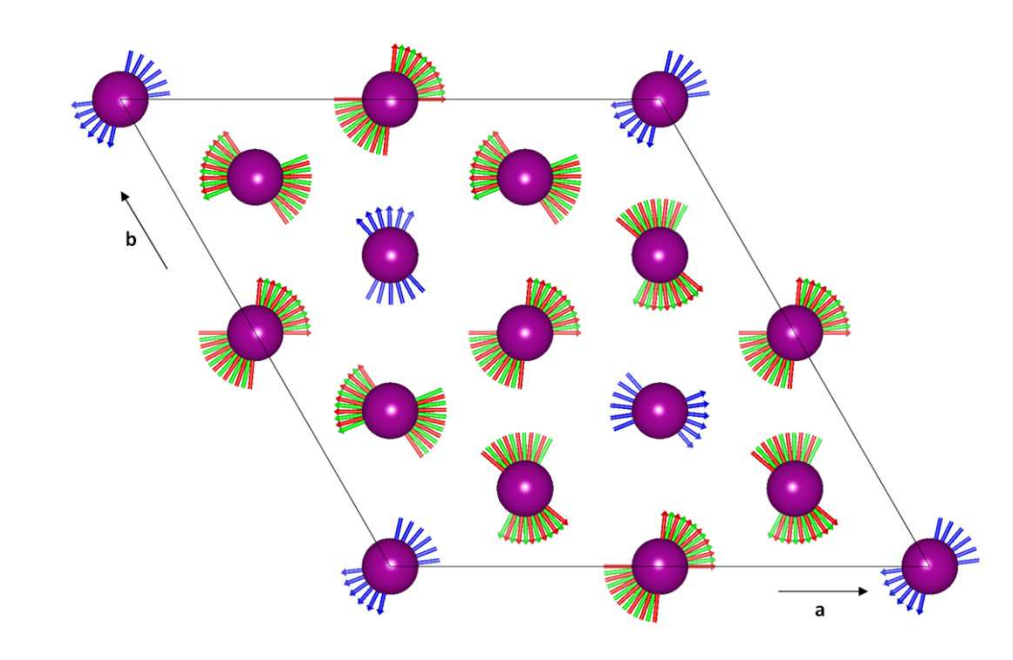


Melilite – Bindi L., Bonazzi P., Dušek M., V.Petříček and Chapuis G., (2001), Acta

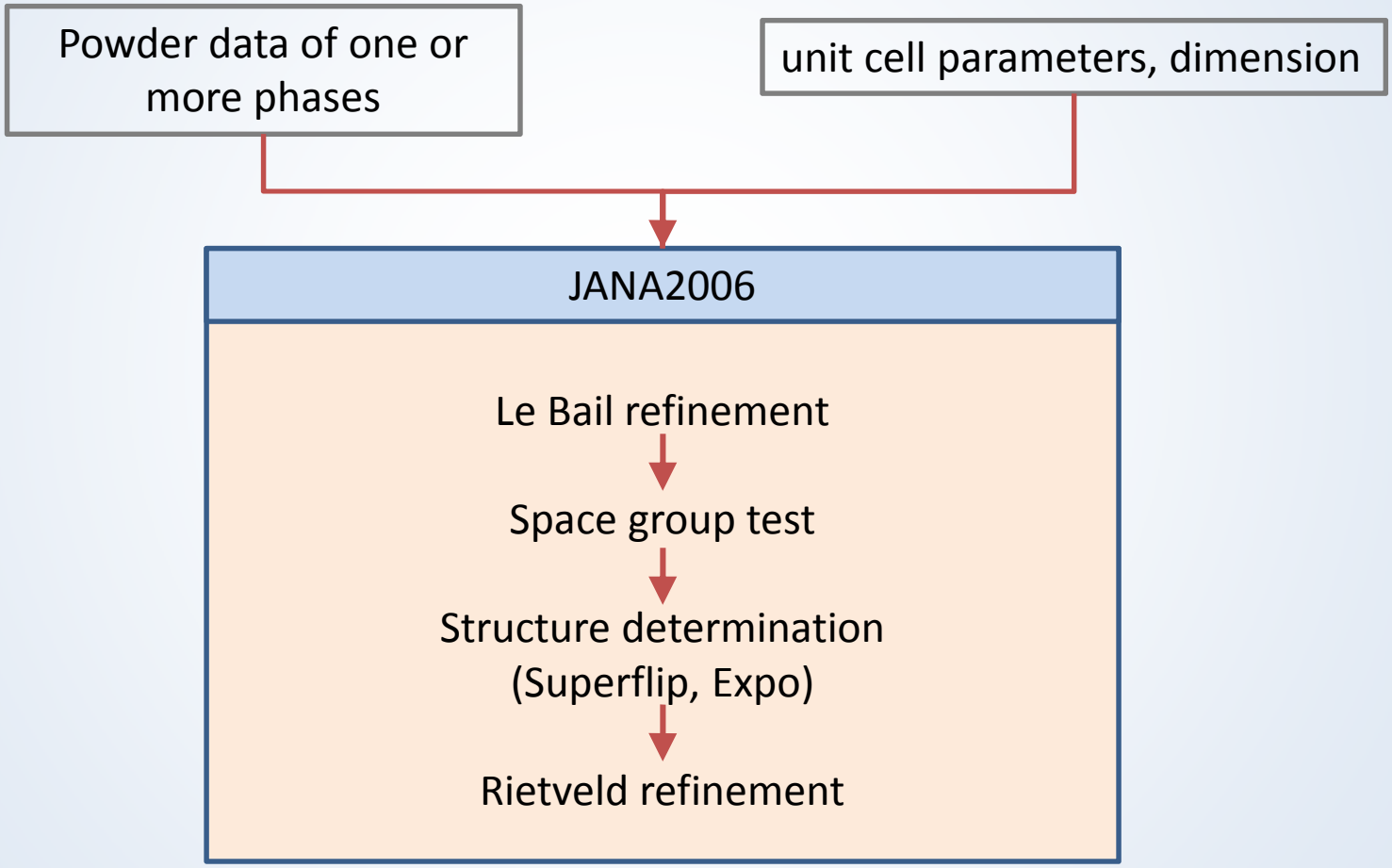
Cryst., **B57,** 739-746.

Functionality for powders

magnetic structures



Functionality for powders



Functionality for powders

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Le Bail refinement

Separate intensities from powder pattern
in Jana2006 activated by
„Make only profile matching“

Structure solution

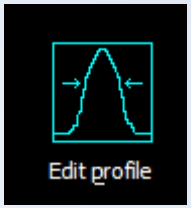
Use separated intensities to solve the crystal structure

Rietveld refinement

Refine the model from the structure solution process by using powder data

Jana2006 tools for powders

„Edit Profile“:



Profile functions: Gauss, Lorentz and pseudo-Voigt

Background corrections: Legendre, Chebyshev polynomials, “cos” functions

Peak asymmetry: Simpson, Berar-Baldinozzi, Finger-Cox-Jephcoat

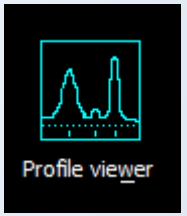
fundamental approach (Cheary-Coelho)

Preferred orientation: March-Dollase, Sasa-Uda

Roughness: Pitchke-Hermann-Matter, Suorti

Anisotropic line-broadening according to Stephens modified by Leineweber and Petricek to include modulated phases

„Profile Viewer“:



Edit manual background

checking calculated and measured data

The powder diffraction option (Rietveld refinement + leBail technique) was implemented in **2001**: Dušek,M., Petříček,V., Wunschel,M., Dinnebier,R.,E. & Van Smaalen,S. (2001). J. Appl. Cryst. 34, 398-404.

Powder profiles in Jana2006

Gaussian: $$G(b_G, x) = \frac{1}{\sqrt{2\pi b_G}} \exp\left(-x^2 / 2b_G^2\right)$$

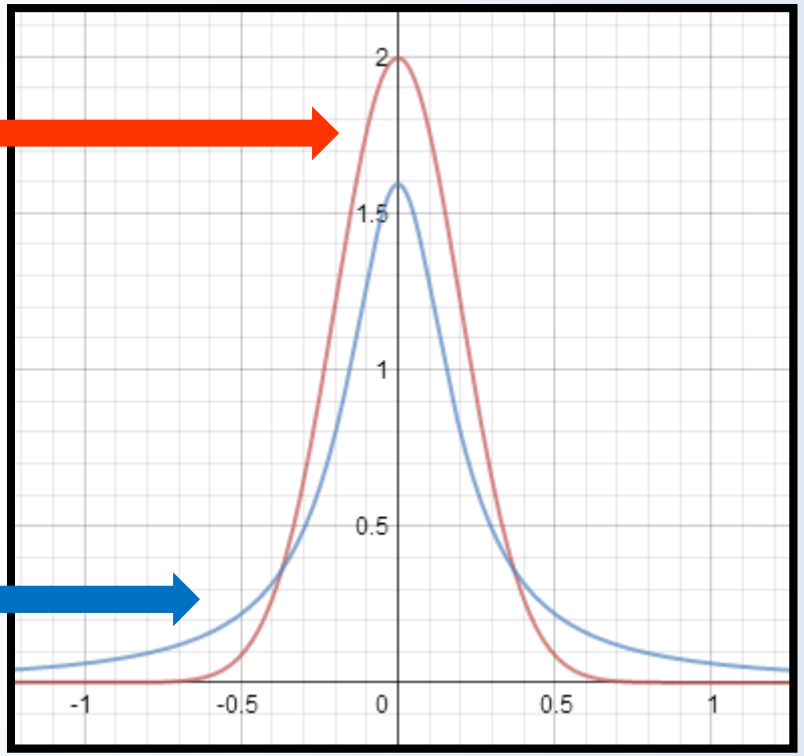
$$b_G^2 = \frac{H_G^2}{8 \ln 2}$$

where H_G is Full-Width at Half-Maximum

Lorentzian: $$L(b_L, x) = \frac{2}{\pi b_L} \frac{1}{1 + (2x/b_L)^2}$$

$$b_L = H_L$$

where H_L is Full-Width at Half-Maximum



Powder profiles in Jana2006

Voigt function: $$V(b_G, b_L, x) = \int_{-\infty}^{+\infty} G(b_G, x') L(b_L, x - x') dx'$$

is a convolution of Gaussian and Lorentzian function. For powder profile we use a simpler analytical approximation of the Voigt function called pseudo-Voigt function:

$$pV(H, x) = \eta L(H, x) + (1 - \eta) G(H, x)$$

the parameters η and H are functions of H_G and H_L

$$H^5 = H_G^5 + 2.69269 H_G^4 H_L + 2.42843 H_G^3 H_L^2 + 4.47163 H_G^2 H_L^3 + 0.07842 H_G H_L^4 + H_L^5$$

$$\eta = 1.36603 \frac{H_L}{H} - 0.47719 \left(\frac{H_L}{H} \right)^2 + 0.11116 \left(\frac{H_L}{H} \right)^3$$

Powder profiles in Jana2006

The diffraction line broadening induced by the sample is usually divided into to crystalline size effect T and microstrain Y. In a first approximation they have the following angular dependence:

$$H_L = b_L = X_L/\cos\theta + Y_L \tan\theta \qquad X_L = 180\lambda/\pi T$$

Similar equations are valid for Gaussian distribution but then an additional factor $8\ln 2$ is to be applied to get b_G from H_G.

$$H_G^2 = 8\ln 2 b_G^2 = X_G^2/\cos^2\theta + Y_G^2 \tan^2\theta$$

These ideal equations are valid only if broadening , induced by experimental conditions are neglected. This means we can use the refined values to make conclusions about crystalline size and microstrain only if some type of fundamental approach or correction for experimental broadening is used.

But usually we are using an additional terms to take into account other experimental effects and then direct interpretation of refined parameters is not possible.

Powder profiles in Jana2006

For the Gaussian term the formula Cagliotti, Pauletti & Ricci, 1958 (Nucl.Instrum., **3**, 223) is used:

$$b_G^2 = U\tan^2\theta + V\tan\theta + W + \frac{P}{\cos^2\theta}$$

In the original formula only three first terms were used. The last one was introduced later as a Scherrer term and it is connected with “crystalline size”. But from the fact that:

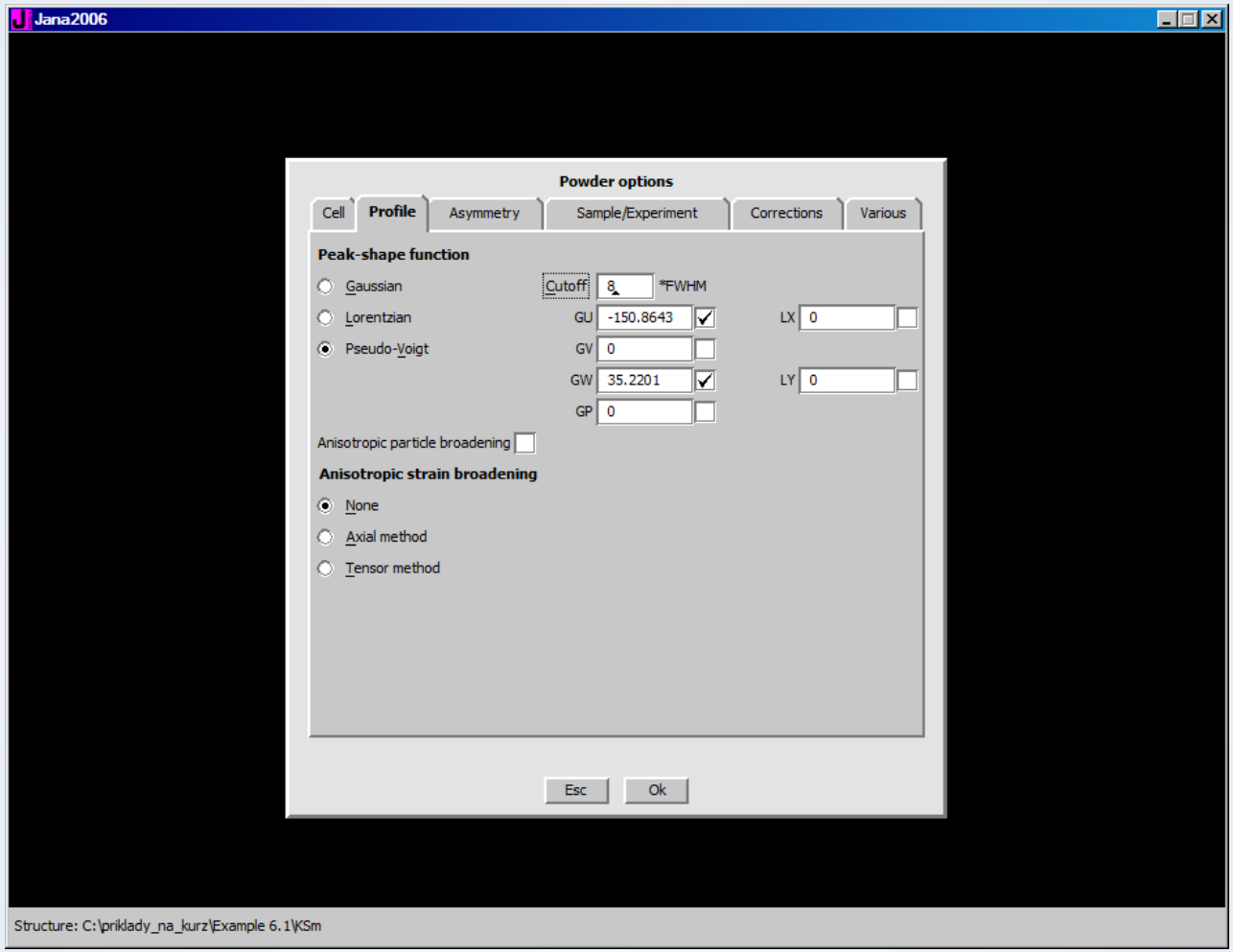
$$\tan^2\theta + 1 = \frac{1}{\cos^2\theta}$$

follows that only two of three coefficients U, W, P can be refined simultaneously.

For the Lorentzian part we use the same terms as for pure crystal broadening but, as mentioned above, they cannot be used directly to find sample characteristics.

$$b_L = X_L/\cos\theta + Y_L\tan\theta$$

Powder profiles in Jana2006



The angular parameters (LX, LY,…) are measured in 0.01 deg, the squared ones in 0.0001 deg^2.

Anisotropic broadening

This phenomenological model is based on a general tensor expression in which the anisotropic strain is described by a symmetrical 4th order tensor:

$$\sigma^2(hkl) = D^{ijmn} h_i h_j h_m h_n = \sum_{H,K,L} S_{HKL} h^H k^K l^L$$

The first term is just a general tensor expression where Landau summation convention is used and which allows a simple derivation of symmetry restrictions similar to those for 4th order ADP parameters. The second term has an explicit form as used by Stephens in which summation is restricted to $H + K + L = 4$

This term is used to modify equations for b_G and b_L

For modulated structures this method has been generalized by A.Leineweber and V.Petříček, (2007). J.Appl.Cryst. ,**40**, 1027-1034.

Peak asymmetry in Jana2006

Simpson's method – Peak is combined with several shifted peaks having the identical shape according to the formula:

$$P_{corr}[2\theta] = \frac{1}{6n}\sum_{i=1}^{2n+1} k_i P\left[2\theta + a\left(\frac{i-1}{2n}\right)^2 \cot 2\theta\right]$$

$$k_i = 1 \quad \text{for} \quad i = 1 \text{ or } i = 2n+1$$
$$k_i = 4 \quad \text{for} \quad i = 2m$$
$$k_i = 2 \quad \text{for} \quad i = 2m+1 \quad m \neq 0 \quad m \neq n$$

a is the only parameter to be refined

Peak asymmetry in Jana2006

Berar-Baldinozzi method

$$P_{corr}(2\theta) = P(2\theta)\left(1 + \frac{p_1 F_1(z) + p_2 F_2(z)}{\tan\theta} + \frac{p_3 F_1(z) + p_4 F_2(z)}{\tan 2\theta}\right)$$

where $F_1(z) = H_1(2z)\exp(-z^2)$ $\quad z = \dfrac{\theta - \theta_0}{FWHM}$

$F_2(z) = H_3(2z)\exp(-z^2)$

$H_n(z)$ stand for Hermit polynomials

Peak asymmetry in Jana2006

By axial divergence – according to Finger, Cox and Jephcoat, (1994) J.Appl.Cryst. **27**, 892-900.

$$H/L \quad S/L$$

These parameters are strongly correlated. Our recommendation is to estimate their ratio and keep it as a restriction during the refinement.

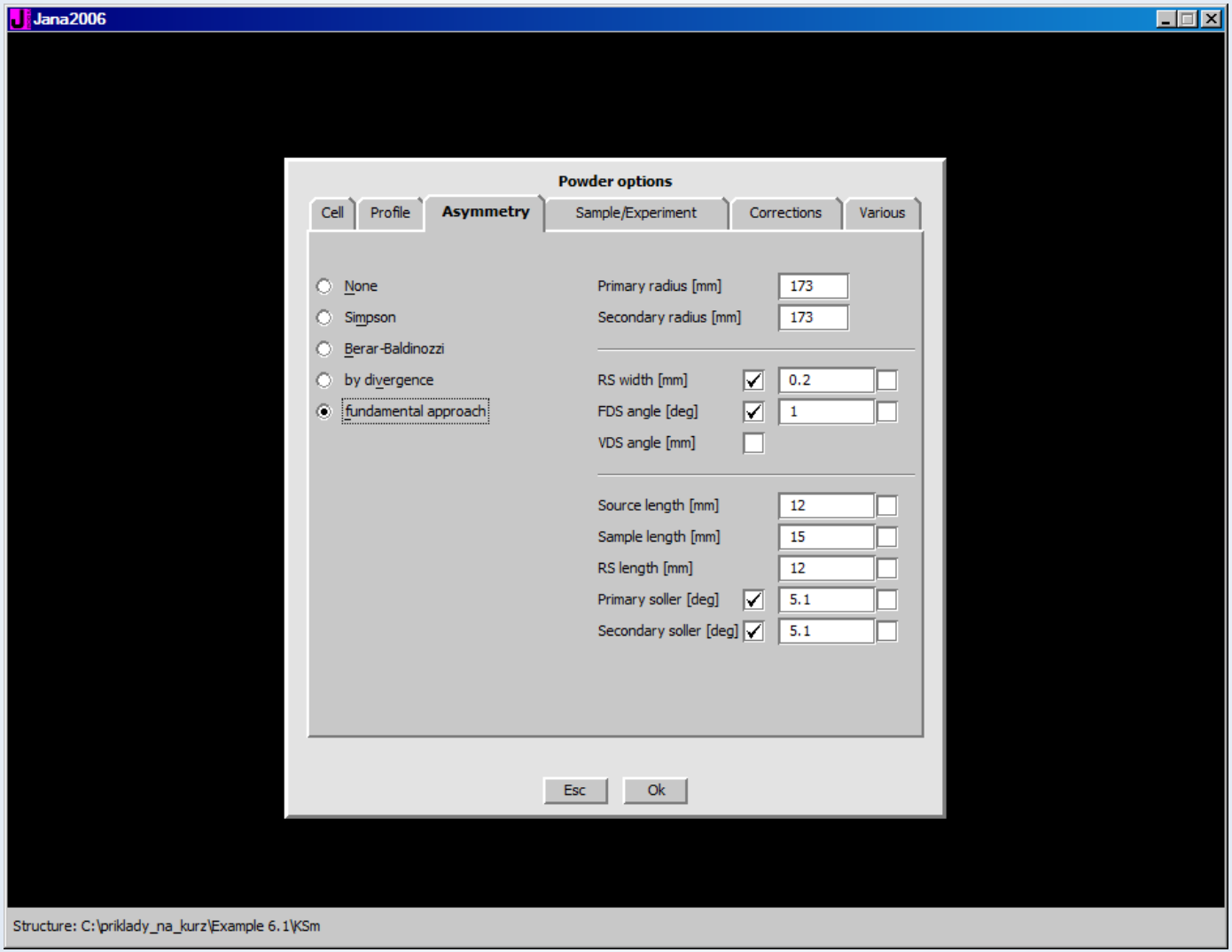
Peak asymmetry in Jana2006

Fundamental approach – it follows the method introduced by Cheary and Coelho, (1998), J.Appl.Cryst. **31**, 851-861.

This method can estimate the profile asymmetry just on the base of experimental parameters. For Bragg-Brentano geometry it works very nicely.

The method makes multiple convolution of several functions.

Peak asymmetry in Jana2006



Background correction

Legendre polynomials – set of orthonormal polynomials defined on the interval $\langle -1,+1 \rangle$. The measured interval have to be first linearly projected into this interval. Then their orthonormality considerably suppress their mutual correlations.

Chebyshev polynomials – they are also orthogonal but in difference sense

$$\int_{-1}^{1} w(x) p_i(x) p_j(x) = \delta_{ij}$$

Legendre polynomials: $w(x) = 1$

Chebyshev polynomials: $w(x) = 1/\sqrt{1-x^2}$

$w(x) = \sqrt{1-x^2}$

For data collected uniformly along diffraction interval we should preferably use the unique weight.

Background correction

Manual background – the background is expressed as a set of background intensities over the diffraction interval. The actual value is calculated by a linear interpolation. This method is can very effectively describe even very complicated background profiles. But it need some user assistance to select it properly. Moreover this first background estimation can be combined with some of previous continuous functions.

In the program Jana2006 there is a tool to select a first estimation of manual background but then you can interactively modify it.

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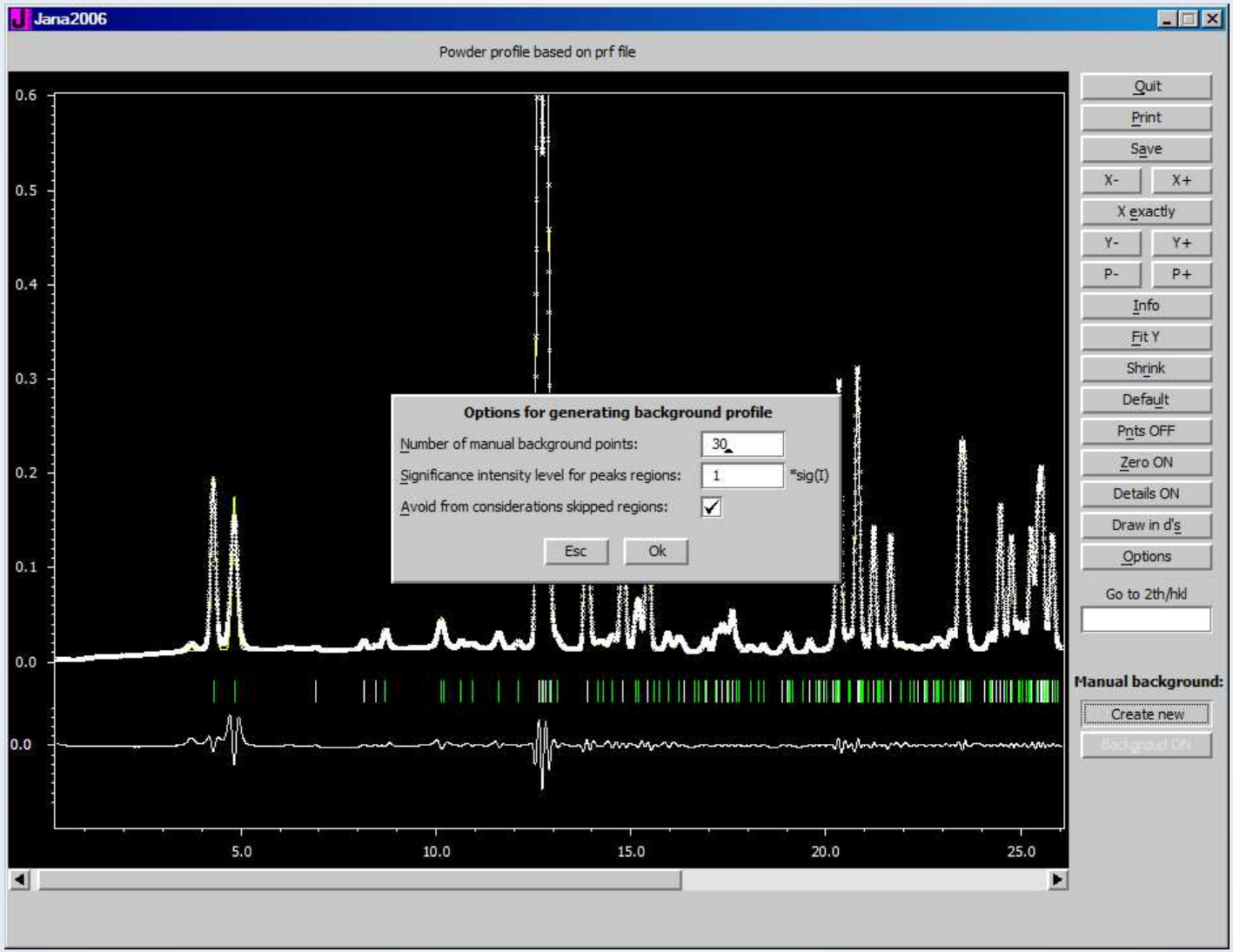
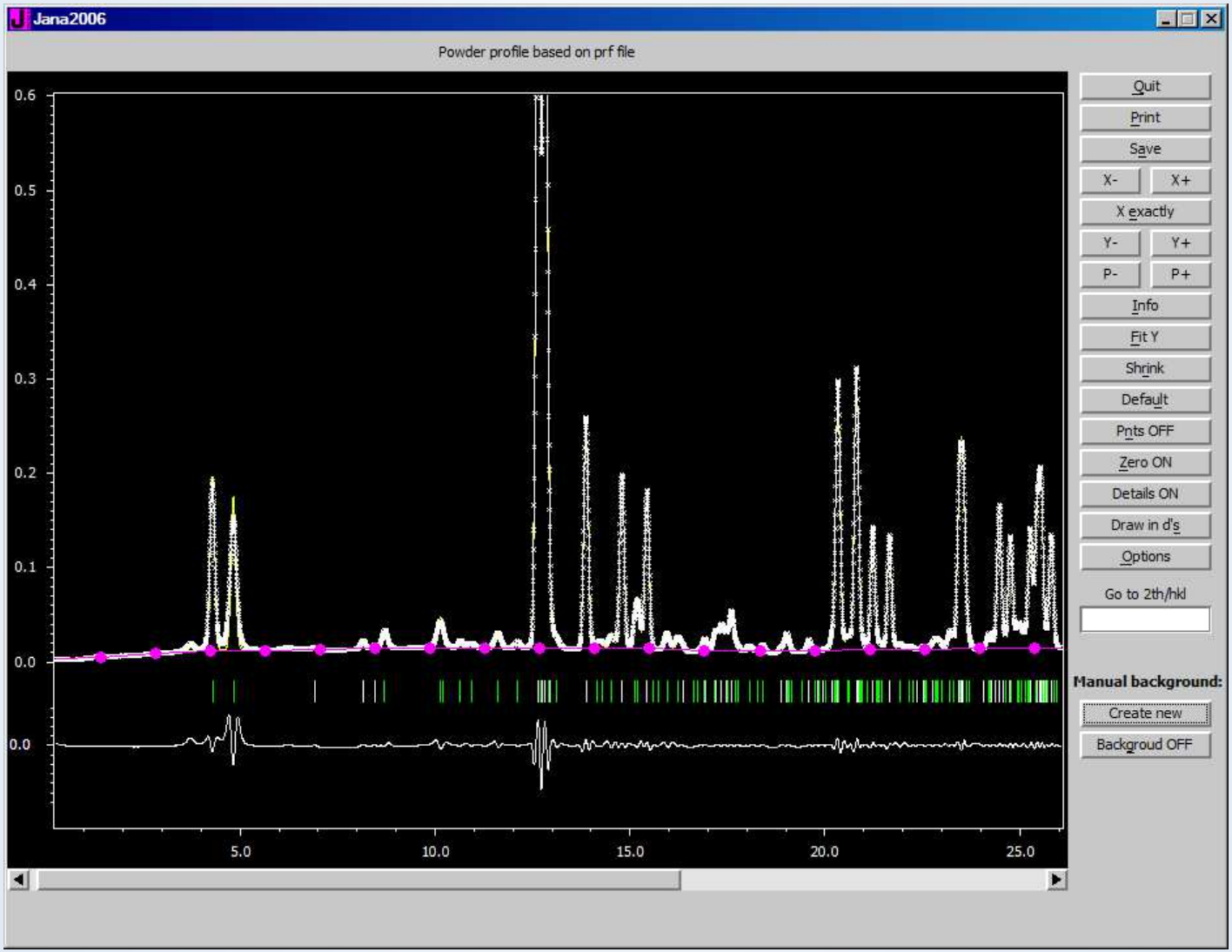
Background correction



Background correction



Zero shift

shift – it defines the zero shift (again in units of 0.01 deg). This value is to be added to the theoretical peak position to get a position in experimental profile

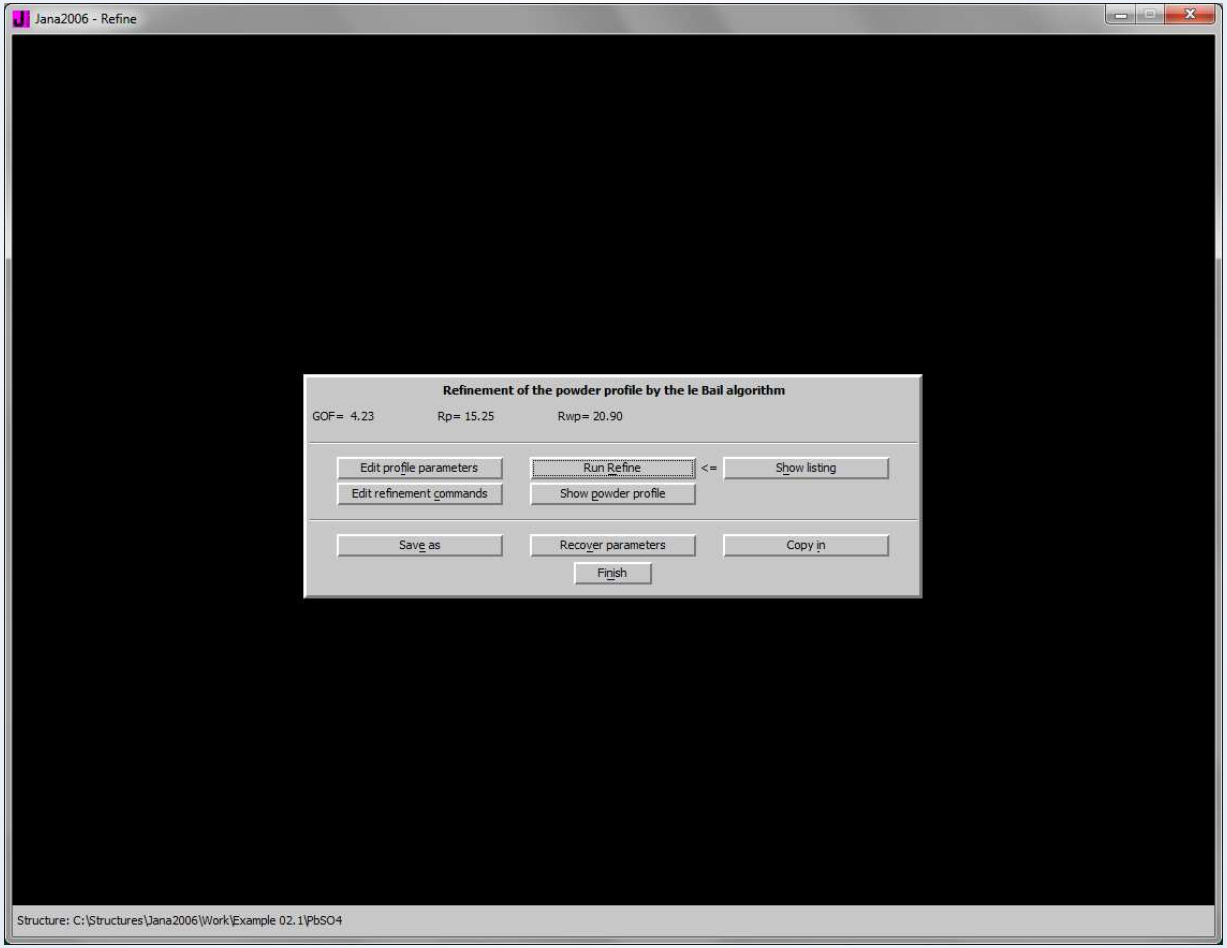
sycos – in analogy with the Fullprof: $\Delta 2\theta = \mathrm{c.cos}\theta$
is connected with a specimen displacement

sysin – in analogy with the Fullprof: $\Delta 2\theta = \mathrm{s.sin}2\theta$
is connected with a transparency correction

These three corrections are combined together to the actual peak position.

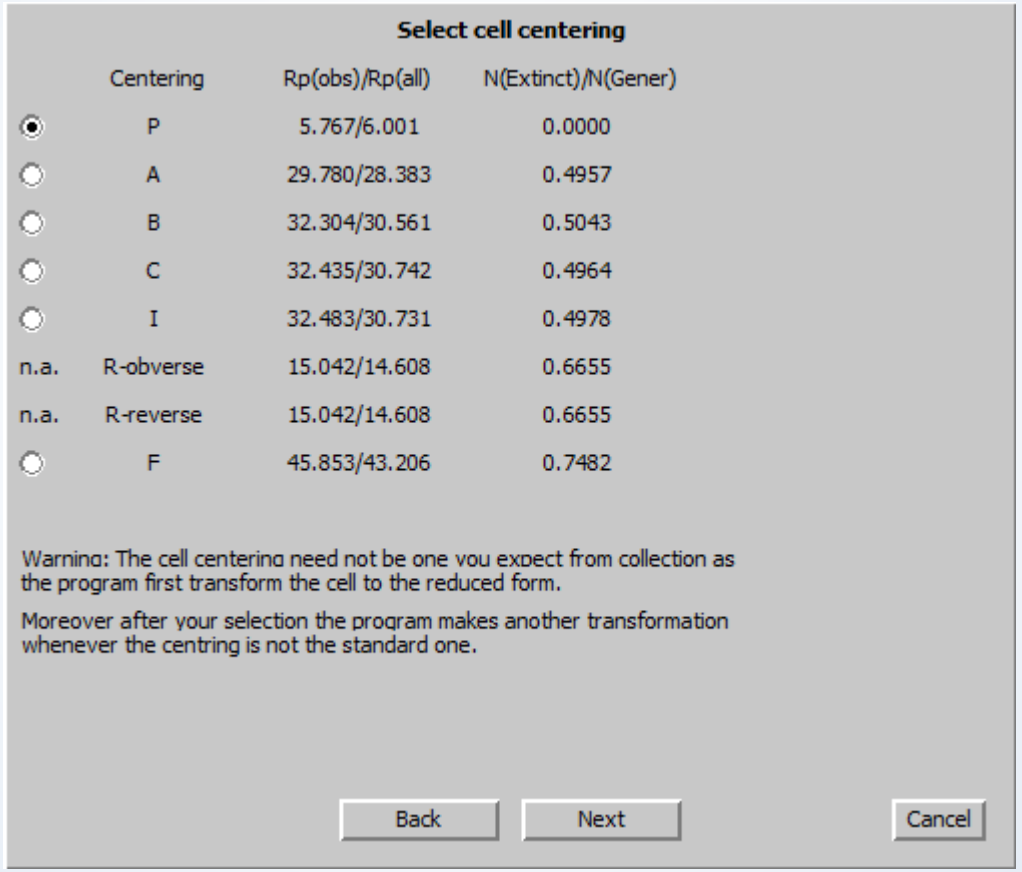
le Bail technique

Based on a peak decomposition and subsequent refinement of profile parameters.



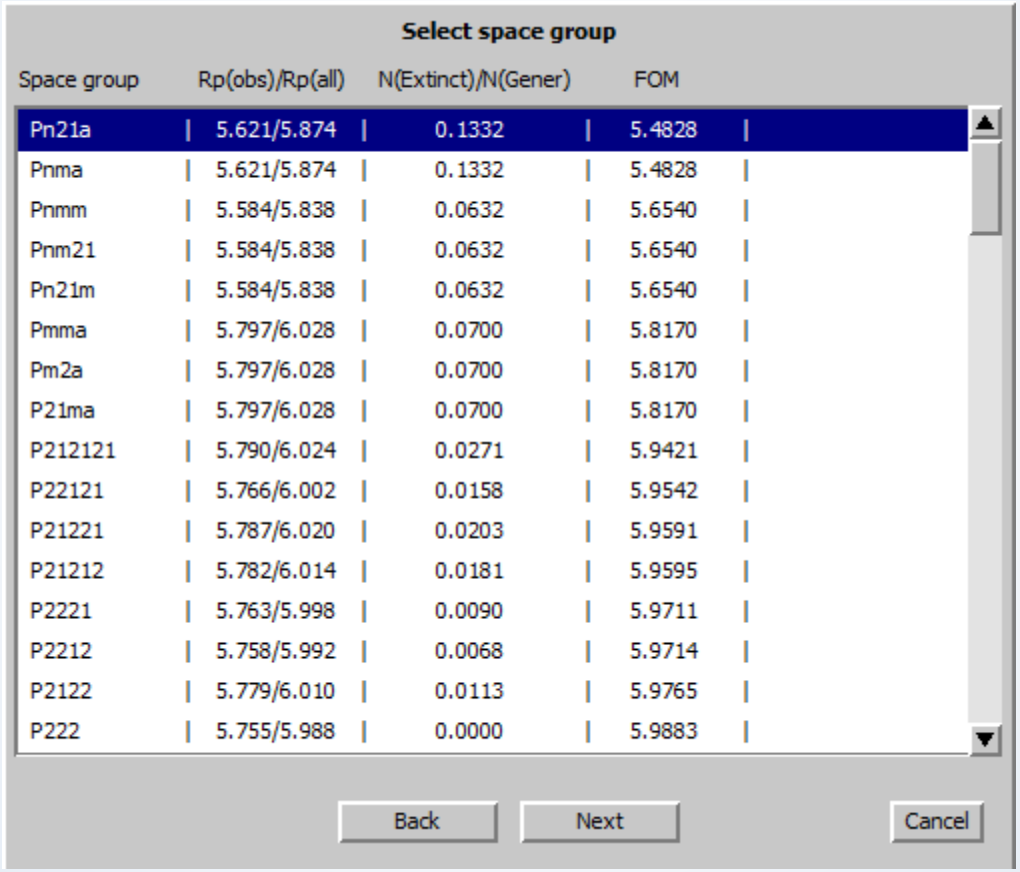
le Bail technique

In Jana2006 is also used to predict symmetry by comparing of profile fits for different space groups:

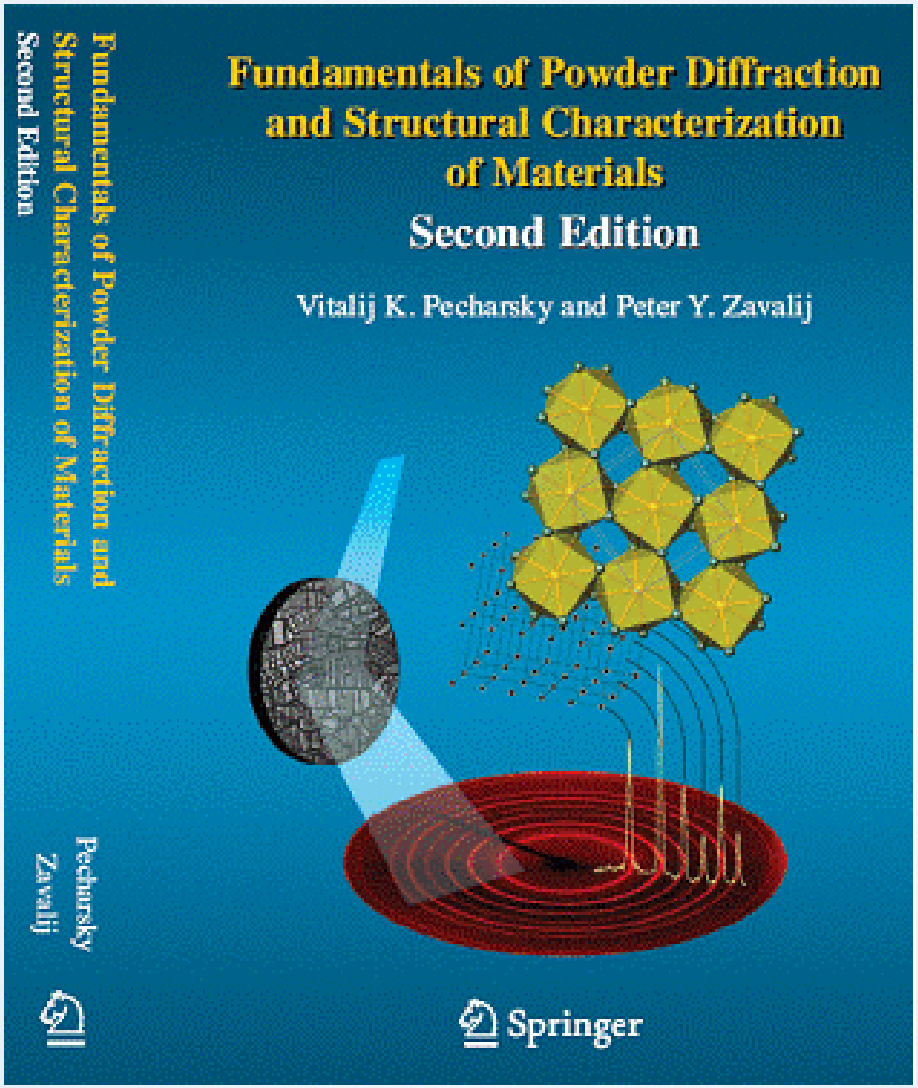


le Bail technique

In Jana2006 is also used to predict symmetry by comparing of profile fits for different space groups:



Recommendation



Questions???

Thank you for your attention

Examples

Example 2.1 – PbSO4 … simple regular structure, solution, refinement
Example 2.2 – Y2O3 … different asymmetry corrections
Example 6.1 – KSm … simple modulated structure