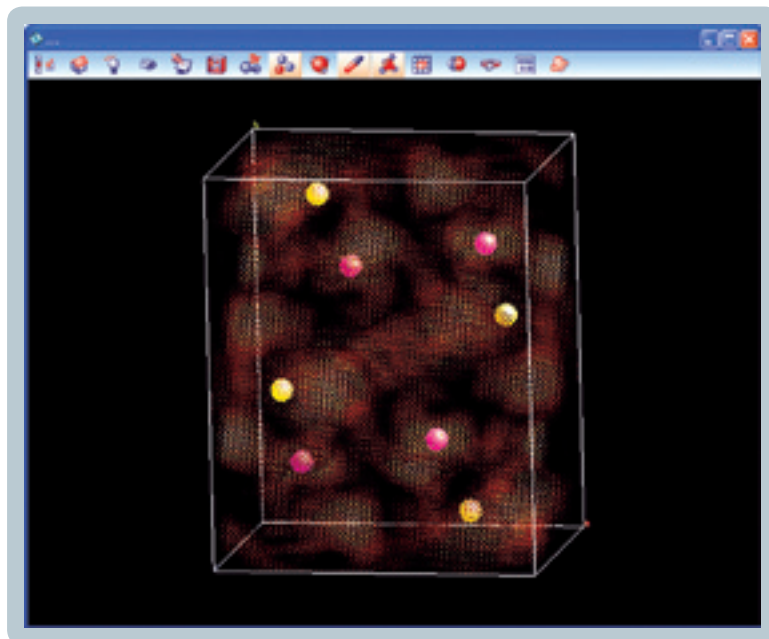


## 3D Fourier analysis - NEW

A highlight of the new TOPAS V4 is the 3D visualisation of (difference-) electron density distributions, including atom picking capabilities with recognition of special positions. This is the ideal tool for the completion of partial structure models from Simulated Annealing or Charge Flipping. The freely rotatable 3D display brings TOPAS's structure determination capabilities to perfection.

The most important features are:

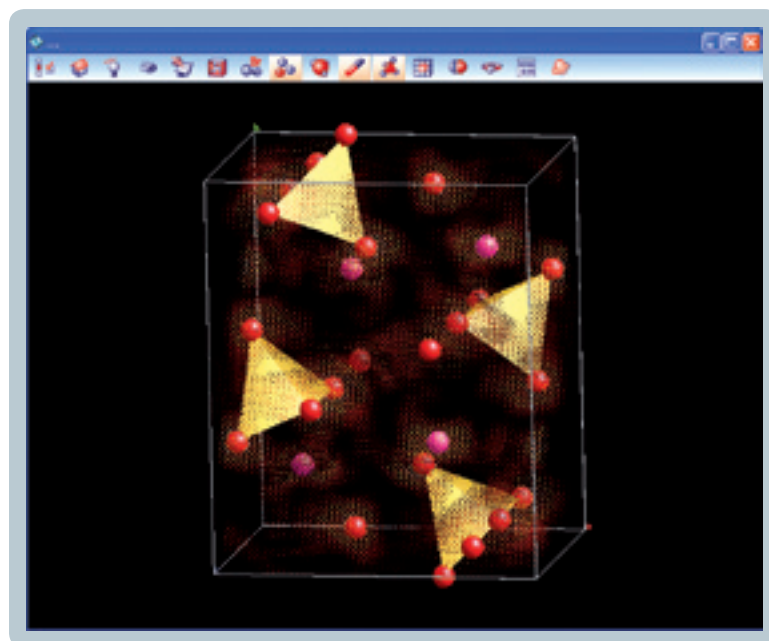
- 3D display of electron density with atom picking
- Observed, calculated, and difference electron density maps
- Allows for enlargement of the Ewald sphere with Fobs set to Fcalc
- Inserts missing reflections within the Ewald sphere with Fobs set to Fcalc
- Allows simultaneous display of electron densities, picked atoms, and crystal structures



3D difference electron density of PbSO<sub>4</sub> after Rietveld refinement with Pb and S positions only. The clouds represent the positions of the missing O atoms.

### References:

- Karakurt, T., Dinçer, M., Kahveci, B., Agar, E., Agar, A. & Sasmaz, S. (2003), *Acta Cryst.*, E59, 1616-1617.
- Oszlányi, G. & Sütő A. (2004): Ab initio structure solution by charge flipping, *Acta Cryst.*, A60, 134-141.
- Coelho, A. A. (2007): A Charge Flipping algorithm incorporating the tangent formula for solving difficult structures, *Acta Cryst.*, A36, 400-406.



Crystal structure of PbSO<sub>4</sub> after automatic atom picking from electron densities including correct assignment of special positions.

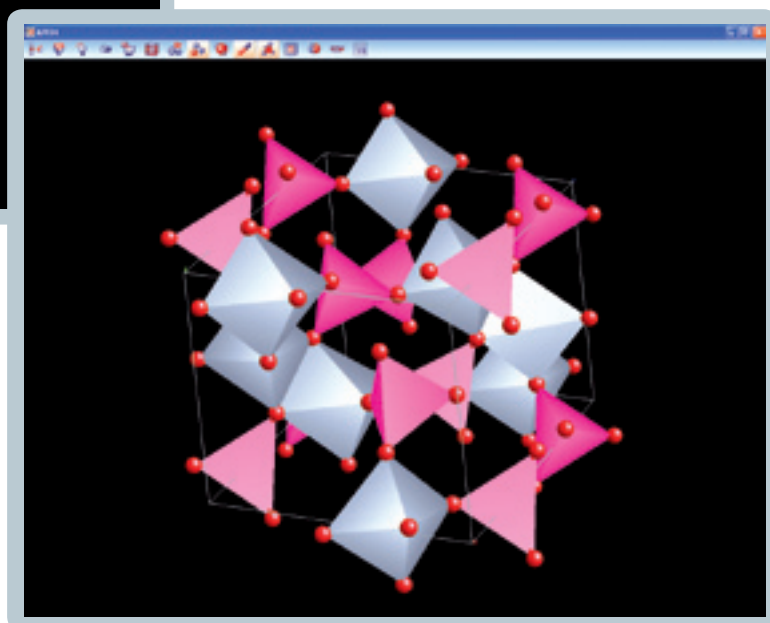
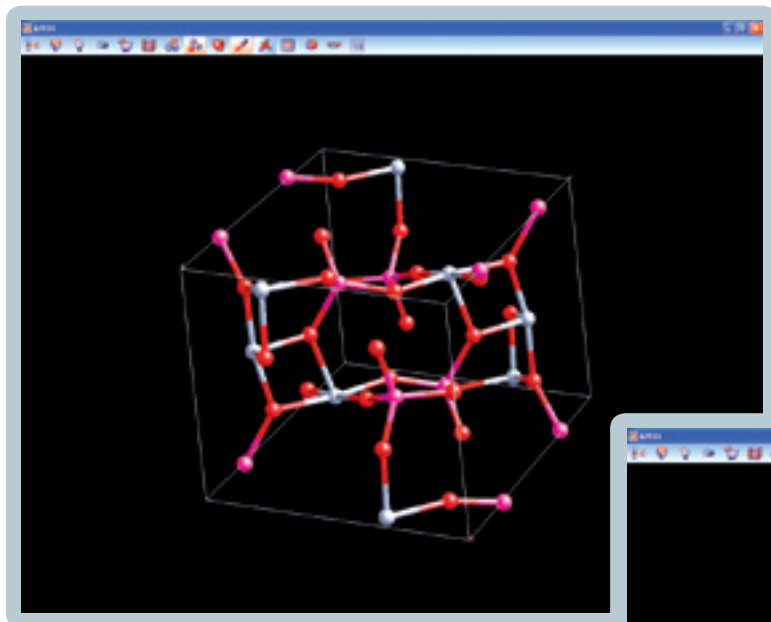
All configurations and specifications are subject to change without notice. Order No. DOC-H88-EXS022.  
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## STRUCTURE DETERMINATION WITH TOPAS: YOU CAN!

New!  
Version 4



TOPAS has been extended dramatically to bring routine structure determination into reach. The new version 4 now offers an outstanding suite of features making structure determination easy. These include:

- **Simulated Annealing,**
- **Charge Flipping, and**
- **3D Fourier analysis**

While each of these approaches is extremely successful on its own, the real beauty lies in their integration in TOPAS. By taking advantage of the particular strengths of each approach, many structures that previously were beyond reach can now be trivially solved.

General features are:

- Structure determination from single crystal and powder data
- Equally effective for organic, metallo-organic and inorganic structures
- Laboratory and synchrotron X-ray data, CW and TOF neutron data
- Structure size and complexity only limited by data quality
- Several of the largest and most-complex structures solved to date have been determined with TOPAS

# Simulated Annealing

TOPAS's unique Simulated Annealing approach was already introduced in 2000. It comprises two distinct techniques:

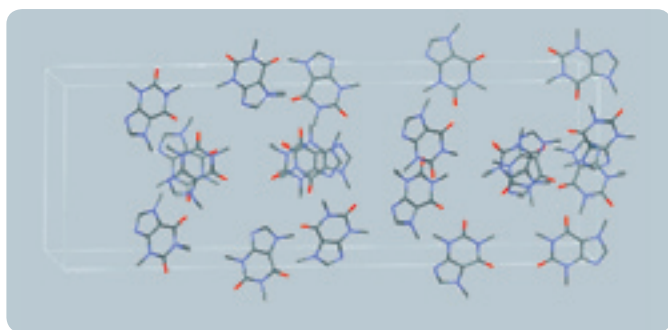
1. A classic two step approach using extracted intensities or single crystal data
2. A modified Rietveld method using step intensity data

In the classic two step approach intensities are first extracted from a powder pattern e.g. using Pawley and Le Bail refinements. In a second, independent step, these intensities are then used for structure analysis. Naturally, single crystal data can be used as well.

The modified Rietveld method is characterized by a seamless integration of simulated annealing techniques, allowing the determination of crystal structures from scratch. As no preceding intensity extraction is required, all problems associated with peak overlap (intensity partitioning) are avoided.

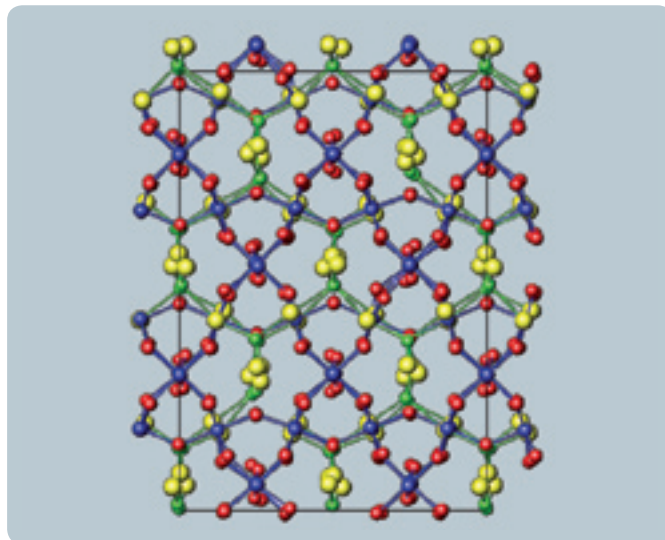
TOPAS's Simulated Annealing approach

- is particularly strong for low resolution data ( $d \gg 1\text{\AA}$ ), especially if
  - heavy atom positions are approximately known, possibly obtained from Charge Flipping
  - bond length, angle and torsion restraints can be formulated, e.g. via rigid bodies
- can be successful even in the presence of one (or more) additional phase(s). Even two unknown crystal structures can be solved simultaneously
- solved several of the largest and most-complex structures to date, see the examples



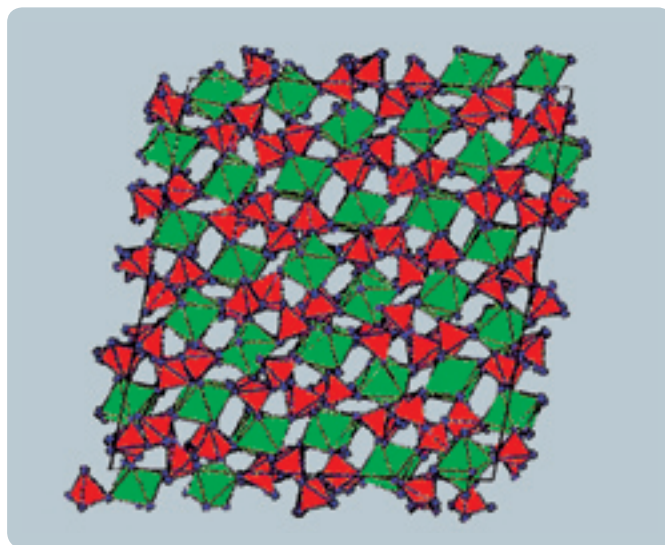
Structure determination and refinement of Anhydrous Caffeine from laboratory powder data (Bruker AXS D8 ADVANCE).

Lehmann & Stowasser (2006), Chem. Eur. J., 13(10), 2908 - 2911.



Structure determination and refinement of  $\alpha$ - $\text{Bi}_2\text{Sn}_2\text{O}_7$  from combined synchrotron and neutron powder data.

Evans et al. (2003), J. Mater. Chem., 13(9), 2098-2103.



Structure determination and refinement of  $\text{Mo}_2\text{P}_4\text{O}_{15}$  from single crystal data (Bruker AXS SMART 6000).

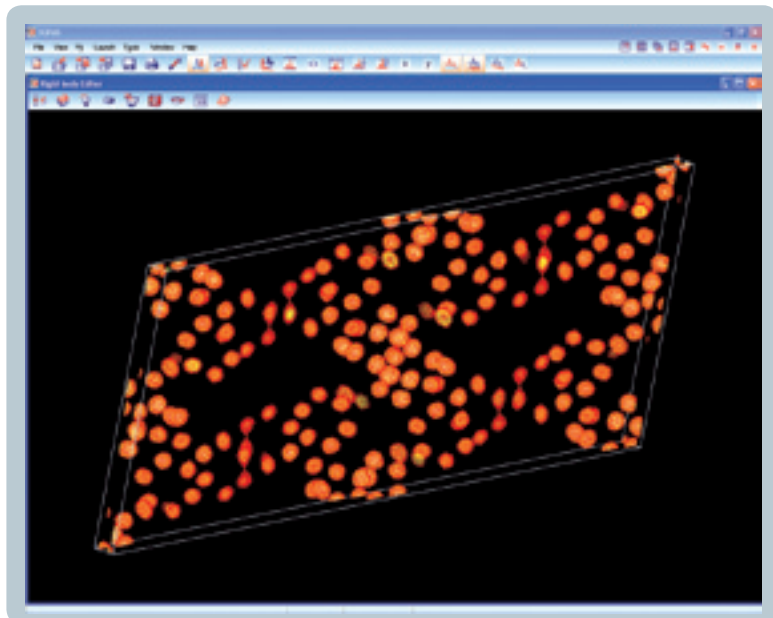
Lister et al. (2004), Chem. Commun., 2540-2541.

## Charge Flipping - NEW

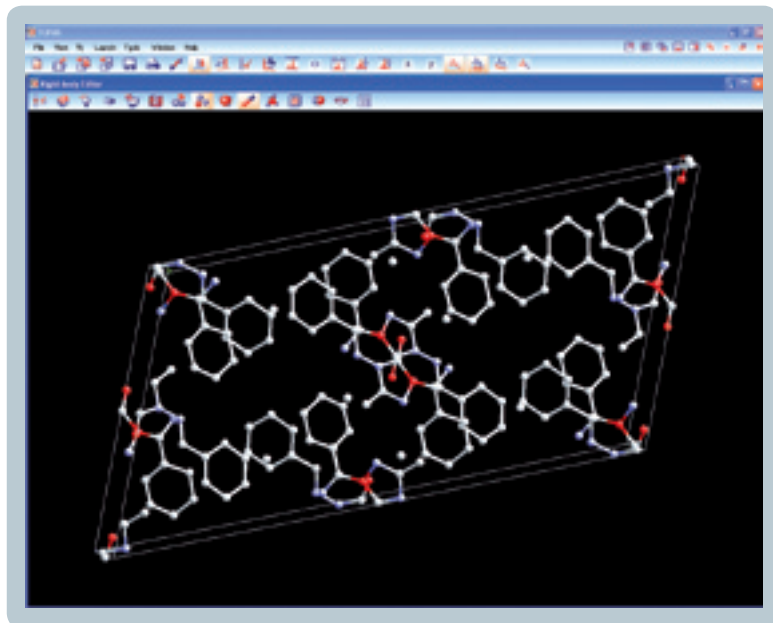
The Charge Flipping method for ab initio structure determination has been implemented in TOPAS V4 with a number of enhancements (Oszlányi & Sütö, 2004; Coelho, 2007). Charge Flipping is an iterative procedure to reconstruct approximate electron densities from diffraction data. The method

- requires only lattice parameters and reflection intensities,
- requires no preliminary chemical information such as unit cell contents, bond distances, bond angles, or connectivity,
- requires no symmetry apart from the input intensities, and thus
- is highly robust towards symmetry ambiguity.

With atomic resolution data ( $d \leq 1 \text{ \AA}$ ) difficult structures can often be trivially solved, especially in cases of disorder or pseudo-symmetry. Partial solutions such as heavy atoms positions or molecular fragments are the ideal input for final structure completion using either the Simulated Annealing or the 3D Fourier analysis method.



Electron density distribution of  $C_{17}H_{14}N_4O_2$  as found by charge flipping. Diffraction data taken from Karakurt et al. (2003).



Crystal structure of  $C_{17}H_{14}N_4O_2$  after automatic atom picking from electron densities.

Charge Flipping features include:

- Tangent formula can be used in each Charge Flipping iteration
- Can operate in any space group and not just P1
- Space group symmetry restraints can be applied to the electron density for each Charge Flipping iteration
- For powder data the A-matrix from a Pawley refinement can be used to attribute intensities to E-values for each Charge Flipping iteration
- Real time 3D display of electron density with optional atom picking
- A new electron density atom picking routine that is both fast and independent of atom size
- Histogram matching, low density elimination, and random omitting and insertion of atoms
- Extremely fast, requires only seconds to minutes per run.