STRUCTURE MODEL OPTIMIZATION

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THE CRYSTAL STRUCTURE SOLUTION PROCESS IN RECIPROCAL SPACE

- Cell Parameters Determination
  - Space Group Identification
    - Full Pattern Decomposition
      - Structure Solution in Reciprocal Space
        (default choice in EXPO)
        😊 Structure Model Optimization
        - Rietveld Refinement
PHASING PROCESS BY DIRECT METHODS

Peak search and chemical interpretation:
chemical species, bond distances and angles.

STRUCTURE MODEL OPTIMIZATION

Electron density map calculation: EMAP
Observed Fourier Synthesis
\[ \rho(r) = V^{-1} \sum_h |F_{oh}| \exp(i\phi_{calc}) \exp(-2\pi i \vec{h} \cdot r) \]

Difference Fourier Synthesis
\[ \Delta \rho(r) = V^{-1} \sum_h (|F_{oh}| - |F_{h_{calc}}|) \exp(i\phi_{calc}) \exp(-2\pi i \vec{h} \cdot r) \]

and also
\[ \Delta \rho(r) = V^{-1} \sum_h (2|F_{oh}| - |F_{h_{calc}}|) \exp(i\phi_{calc}) \exp(-2\pi i \vec{h} \cdot r) \]
Solve \rightarrow \rightarrow \rightarrow \text{ Fourier}
STRUCTURE MODEL OPTIMIZATION

The quality of the electron density map calculated after the phasing process by Direct Methods (DM) mainly depends on

- the experimental resolution
  atomic resolution is the best

- the reliability of the integrated intensities extracted from the experimental pattern (and used by DM for phasing)
  peak overlap should be low (percentage of independent observations larger than 50%)

- the structure complexity
  it depends on the number of non-H atoms in the asymmetric unit (30 is the current reasonable limit)
WHY DIRECT METHODS MODEL OPTIMIZATION

The structure model provided by DM is often approximate: some atoms are missed to the complete solution, additional false positions occur, some atoms are not perfectly located.

It is useful to optimize the model before the final Rietveld refinement, owing to the its modest convergence rate.

Structure and conformational analysis of a bidentate pro-ligand, C$_{37}$H$_{34}$N$_{2}$S$_{2}$, from powder synchrotron diffraction data and solid-state DFTB calculations

The molecular and crystalline structure of ethyl $\text{T}_{2}d_{2}d_{4}d_{5}d_{6}d_{7}d_{8}$-catalyzed hydrocarbons $1^{\circ}$-quαιn-
almel 4.1-cyclam (3)- $\text{H}$-carboxamide (4) was refined and refined from powder synchrotron X-ray diffraction data. The initial model for the structural solution in direct space using the simulated annealing algorithm implemented in DAFM (Diederik et al., 2009; J. Ant. Cryst. 2012-012) is available, performed a conformational study on the fluid dimerization of the naphthoquinoline system and the trans conformer of benzene rings of (4). The final model was chosen using experimental evidence from the NMR (Nuclear magnetic resonance) structure and X-ray diffraction data. The refined structure of the two space rings have the chain conformation, while both of the fused rings in the naphthoquinoline system have half-chair conformations determined with semi-empirical AM1 calculations to the refined structures the two space rings have the chain conformation, while both of the fused rings in the naphthoquinoline system have half-chair conformations determined with semi-empirical AM1 calculations. 25 non-H atoms in the asymmetric unit.
The EXPO team has developed innovative theories and computing procedures devoted to improve the structure model of organic/metal-organic and inorganic structures.

The model optimization requires that different methodological approaches must be adopted depending on the type of compound.

- Solve

- FOURIER RECYCLING (default for inorganic)
- RBM (default for organic/metal-organic)
- COVMAP
- RAMM
- SHIFT_AND_FIX

- Explore Trials
The model optimization default procedure for inorganic compound consists of eight blocks automatically executed; each block is constituted by weighted least squares (WLSQ) cycles combined with Fourier recycling (FR) of $(2|F_o|-|F_c|)$ map calculation ($|F_o|$ and $|F_c|$ are the observed and calculated structure factor moduli, respectively)

For each block, at the end of least squares, the $R_F$ residual value is evaluated

$$R_F = \frac{\sum_h |F_{oh}| - s \cdot |F_{ch}|}{\sum_h |F_{oh}|}$$

At the end of the eight blocks, the structure model corresponding to the smallest $R_F$ residual value is selected.

The model is carefully analyzed: poorly connected low peak intensity atoms characterized by large refined thermal factors are discarded.
Model optimization

**WEIGHTED LEAST SQUARES**
(atomic coordinates and isotropical thermal factors refinement)

(2|F_o| - |F_c|) CALCULATION
(new Fourier map)

A very effective weighting scheme has been identified

(2|F_o| - |F_c|) map \leftrightarrow |F_o| + (|F_o| - |F_c|) map

It aims at recovering missing atoms

In the model analysis step unreliable peaks are discarded.

**R_F CALCULATION**
(agreement between observed |F_o| and calculated |F_c| structure factor moduli)

**THE SMALLEST R_F MODEL**

**MODEL ANALYSIS**

**FINAL RIETVELD REFINEMENT**
Least squares in structure refinement by single crystal data are usually carried out for minimizing

\[ R = \sum_h w_h (|F_{oh}| - |F_{ch}|)^2 \]

The summation is over the total number of measured reflections

- \( w_h \) is a suitable weight associated to each reflection; it usually represents an estimate of the precision of the measured quantity
- \( |F_{oh}| \) is the measured (experimental) structure factor modulus
- \( |F_{ch}| \) is the calculated structure factor modulus
LEAST SQUARES FOR SINGLE CRYSTAL DATA

If $|F_{ch}|$ is expanded by Taylor series

$$R = \sum_h w_h \left( \Delta |F_h| - \sum_k \frac{\delta |F_{ch}|}{\delta x_k} \Delta x_k \right)^2,$$

($x$ are the parameters to be refined)

The normal equations are obtained by setting to zero the derivatives of $R$ with respect to the parameters to be refined.

The shift $\Delta x_k$ on parameters can be derived

$$x_k = x_k + \Delta x_k \quad \text{refined parameters}$$
LEAST SQUARES FOR SINGLE CRYSTAL DATA

\[ F_h = \sum_{j} f_j \exp[2\pi i (hx_j + ky_j + lz_j)] \exp[-B_j \sin^2 \theta/\lambda^2] \]

\( f_j \) is the scattering factor of the \( j \)-th atom.

(the summation is over the number of atoms in the unit cell).

Atomic coordinates and thermal factors are refined.

In case of single crystal, the observed structure factor moduli estimates are reliable and least squares are able to improve atom positions.

The success of least squares strongly depends on the ratio ‘\( \text{number of observations (reflections)} \)/\( \text{number of parameters to be refined} \)’.
In case of powder data, the observed structure factor moduli estimates are affected by errors (that is particularly true for overlapping reflections). In addition, the number of observations is usually small (particularly for organic structures because of their rapid scattering factor decay).

Single crystal type least squares may be unsuccessful.

Special strategies can be adopted in terms of:

- quantity to be minimized
- weighting scheme
In case of powder data, for the overlapping reflections, the intensity of a cluster is more reliable than the single intensity. We developed an approach based on the use of the cluster intensities as observations in the least squares.

That choice may be not profitable because of the small number of clusters (observations).

A more effective approach, available in the current version, was developed and introduced in EXPO.
The main features of the EXPO approach are

- least squares minimization on reflection integrated intensities

- a robust weighting scheme capable of compensating the low accuracy of the overlapping reflection intensities

- a delayed partitioning of the observed intensity of each cluster of overlapped reflections proportionally to the calculated intensity of each reflection
In EXPO, least squares for optimizing the structure model aim at minimizing the following residual

\[ R = \sum_{h} w_h (I_{oh} - I_{ch})^2 \]

The summation is over the total number of reflections in the experimental pattern

- \( w_h \) is a suitable weight
- \( I_{oh} \) is the observed integrated intensity for the reflection \( h \), extracted from the experimental pattern \( (I_{oh} \propto |F_{oh}|^2) \)
- \( I_{ch} \) is the corresponding intensity calculated from the model \( (I_{ch} \propto |F_{ch}|^2) \)

\( w_h \) is able to take into account the unavoidable errors on the integrated intensity estimates of overlapping reflections
LEAST SQUARES WEIGHTING SCHEME

\[ w_h = w_{1h} \cdot w_{2h} \]

\[ w_{1h} = w_{ah} \cdot w_{bh} \quad w_{2h} = 1/[1+4 \cdot (m_h \cdot LP_h \cdot |F_{oh}|)^2] \]

\[ w_{ah} = \exp[-0.5 \cdot (\sin\theta_h / \lambda)^2] \quad w_{bh} = \sqrt{I_{oh} / I_{oh}'} \]

\( m_h \) is the reflection multiplicity; \( LP_h \) is the Lorentz-polarization factor; \( \theta_h \) is the reflection angular position (Bragg angle); \( \lambda \) is the radiation wavelength;

\[ I_{oh} = m_h \cdot LP_h \cdot |F_{oh}|^2 \]

\[ I_{oh}' = I_{oh} + \sum_{k \neq h} (1 - q_{kh}^2) \cdot I_{ok}, \quad q_{kh}^2 = \frac{|2\theta_k - 2\theta_h|}{\text{FWHM}_h} \]

The summation is over the reflections in overlapping with \( h \)

\( \text{FWHM}_h \) is the full width at half maximum of the \( h \) reflection peak
LEAST SQUARES WEIGHTING SCHEME

\[ w_{1h} = w_{ah} \cdot w_{bh} \]
\[ w_{ah} = \exp[-0.5 \cdot (\sin \theta_h / \lambda)^2] \]
\[ w_{bh} = \frac{I_{oh}'}{I_{oh}} \]

\( w_{1h} \) decreases with

- increasing \( \theta_h \) (it is more difficult to carefully model overlapping and background at high \( 2\theta_h \) angle region of the experimental pattern)
- increasing the number of reflections in overlapping (in particular, when the overlapping neighbouring intensities are large with respect to \( I_{oh} \))

\[ w_{2h} = \frac{1}{1 + 4 \cdot (m_h \cdot L P_h \cdot |F_{oh}|)^2} \]

\( w_{2h} \) is the typical weight used to avoid that least squares are dominated by the largest intensities
We try to improve the integrated intensity $I_{oh}$ estimate ($I_{oh} \propto |F_{oh}|^2$) associated to each $h$ overlapping reflection, before the $(2|F_o|-|F_c|)$ Fourier calculation and after least squares.

The observed intensity of each cluster of overlapping reflections is ‘partially’ partitioned proportionally to the calculated intensity of each reflection in the cluster.

$$I_{oh}^p = w_{1h} \cdot I_{oh} + K \cdot (1 - w_{1h}) \cdot I_{ch}$$

K is a scale factor, given by

$$K = \frac{\sum_k (1 - w_{1k}) \cdot I_{ok}}{\sum_k (1 - w_{1k}) \cdot I_{ck}}$$

The summation goes over the reflections belonging to the cluster.

K fixes the condition that the overall intensity of the cluster must not be changed after the partition.

If the reflection intensity $I_{oh}$ is well estimated ($w_{1h}$ is $\sim 1$), $I_{oh}^p$ slightly differs from the observed $I_{oh}$; on the contrary, if $w_{1h} \sim 0$, $I_{oh}^p$ is proportional to the calculated intensity.
MODEL OPTIMIZATION FOR ORGANIC COMPOUNDS

For organic compounds the experimental resolution $RES (d_h$, the smallest interplanar distance value) is usually far away from the atomic one because of their rapid scattering factor decay.

$$(d_h)_{\text{min}} = \left( \frac{\lambda}{2\sin \theta_{\text{max}}} \right)$$

resolution 1.5 Å
It is well known that limited experimental data resolution modifies the Fourier synthesis (even at atomic resolution)

\[ \rho(r) = \frac{2}{V} \sum_{h=0}^{\infty} \sum_{k=-\infty}^{\infty} \sum_{l=-\infty}^{\infty} |F_H| \cos[(\varphi_H - 2\pi \overline{H} \cdot r)] \]

The map is an imperfect representation of the true electron density: the worse the experimental resolution, the less accurate the Fourier representation of the electron density

When \( RES > 1 \) Å the resolution effects may be critical
MODEL OPTIMIZATION FOR ORGANIC COMPOUNDS

One point-atom structure at 1.54 Å
• Regions of the electron density map in the unit cell become negative

• Peaks are broadened and surrounded by ripples (positive and negative with decreasing amplitude)

• Peaks are misplaced from their correct positions

• Peaks have distorted intensities
RESOLUTION BIAS MINIMIZATION PROCEDURE

RBM

RBM is a very powerful procedure of EXPO providing an electron density map more informative with respect to the case in which the resolution effects have not been corrected.

RBM is able to face the perennial problem of limited experimental data resolution. In case of powder, the resolution bias is mixed with errors on both structure factor moduli and phases.

RBM corrects the electron density map by typical resolution bias errors: peak broadening, ripples, intensity distortion, peak shifts.
RBM PROCEDURE

The method requires the generalization of the traditional concept of gaussian-like peak: each $j$-th atomic peak, in the electron density map, is replaced by a two-component function, constituted by the \textit{main peak} and by the corresponding \textit{ripples}

\[
\rho_j(\mathbf{r} - \mathbf{r}_j) = \rho_{\text{[main]}}^j (\mathbf{r} - \mathbf{r}_j) + \rho_{\text{[ripples]}}^j (\mathbf{r} - \mathbf{r}_j)
\]

Each component of the generalized peak is mathematically modelled in its corresponding existence domain: \textit{e.g.}, the $j$-th main peak has its own existence domain while the ripples extend over all the rest of the unit cell
RBM PROCEDURE

RBM consists of the two following main steps

- subtracting ripples corresponding to each experimental electron density peak

\[ \rho(r) = \rho(r) - \sum_j \rho_{ripples} (r - r_j) \]

- best fitting each \( j \)-th main peak in its own domain by gaussian function

\[ \rho(r) \approx \sum_{j=1}^{N} c_j G(r; \sigma_j, r_j) \]
The EXPO default procedure for optimizing structure model of organic and metal-organic compounds is based on successive cycles of

$$2|F_o| - |F_c|$$ map calculation suitably corrected by RBM

and

weighted least squares (restraints on distances and angles are applied)

EXPO automatically recognizes a compound as organic (or metallorganic) or inorganic and applies the designed model optimization strategy
MODEL OPTIMIZATION IN EXPO

DM model

UTM-1 zeolite

WLSQ-FR model

Clomipramine hydrochloride
C\textsubscript{19}H\textsubscript{24}ClN\textsubscript{2}·Cl

RBM model

Ethyl 1',2',3',4',4a',5',6',7' - octahydrodispiro[cyclohexane -1,2' - quinazoline-4',1'' - cyclohexane]-8' -carbodithioate

RBM model
The resolution bias correction in reciprocal space

The correction by RBM in the direct space can be translated into the reciprocal space. The structure factors can be modified (resolution dependent correction) in order to provide electron density map showing truncation effects smaller than the canonical case.

\[ F_h \xrightarrow{\text{mod}} F_{h \text{ mod}} \]

\[ \Sigma \]

\[ \rho(r) \]

\[ \rho'(r) \]

\[ \Sigma \]

The use of the modified structure factors locates more reliable peaks.

Also this non-default strategy (alternative to standard RBM in direct space), mainly based on atomic scattering factor correction, is available in EXPO for reducing the typical resolution bias errors of the electron density map.
Refine --> RBM --> RBM (Direct and Reciprocal Space)
COVMAP is a non-default strategy in EXPO for optimizing a structure model obtained at the end of an *ab initio* solution process, in particular for recovering missing positions.

COVMAP starts from the available non-interpretable structural model containing very few well positioned and a large set of misplaced peaks.
THE COVMAP METHOD

A quite common case

Only a few atoms (the yellow ones) are close to their correct positions, the others are incorrectly located or are so far from the correct positions that chemical bonds cannot be established.

COVMAP is able to improve such a model

**CAMPHOR O₂C₁₀**

*unpubl. data by courtesy of Dr. M. Brunelli*
THE COVMAP METHOD

COVMAP has been inspired by a mathematical technique enabling to calculate the covariance and/or correlation between two points of an electron density map, no matter the quality of the structure model.

The technique operates in the reciprocal space, by exploiting suitable Fourier series involving structure factors as components of the Fourier coefficients.

COVMAP translates the covariance concept in direct space.

COVARIANCE CONCEPT

*the density in one point depends on the density in another point of the map if their covariance is not vanishing*
Around each positioned atom (the pivot atom), at bond distance, we expect that one or more atoms are present in the correct structure (the condition is violated in case of isolated atom).

COVMAP introduces the covariance concept: the largest intensity peaks in the electron density map are considered pivots, and for each of them, the reasonable expectation that some other peaks would be present at bond distance is transformed in electron density modifications.
THE COVMAP METHOD

Three kinds of information are exploited by COVMAP

1. the chemical interpretation of the structure model peaks (according to their intensities in the electron density map)

2. some basic crystal-chemical rules, essentially the bond distances expected for the pivot atoms

3. the crystallographic residual $R_F$ corresponding to the model modified by COVMAP. It is expected to be smaller than $R_F$ corresponding to the original improvable model
THE COVMAP METHOD

All the **pivot pairs** \((i, j)\) of non-bonded atom positions, with distances compatible (within the electron density errors) with the presence of one atom bridging \(i\) and \(j\) atoms, are considered

\[
\rho_c(\mathbf{r}) \text{ is the electron density map corresponding to the current model}
\]

\[
\rho_d(\mathbf{r}) \text{ is the difference electron density map}
\]

A **new working map** \(\rho_n(\mathbf{r})\) (first set to 0) is created:

\[
\text{If } \rho_c(\mathbf{r}) > 0 \text{ and } \rho_d(\mathbf{r}) > 0
\]

\[
\rho_n(\mathbf{r}) = \rho_c(\mathbf{r}) \cdot w(\mathbf{r})
\]

\(w(\mathbf{r})\) (integer positive factor) is the number of times in which the positional vector \(\mathbf{r}\) falls within the range of the expected bond distance from each position of each pivot pair

\(w(\mathbf{r})\) vanishes in the points where atoms are not expected to be connected with the pivot pairs.
A peak search on $\rho_n(\mathbf{r})$ is carried out and new peak positions ($nd$) are added to the starting number ($ns$) of the improvable model.

The model constituted by $ns+nd$ peaks is finally retained and chemically interpreted if

$$R_{F_{ns+nd}} < 1.2 \cdot R_{F_{ns}}$$

otherwise the work by COVMAP is cancelled.

COVMAP has been suitably combined with WLSQ (Weighted Least Squares) and RBM (Resolution Bias Minimization) procedures, which relocate, refine or reject the new peaks.

This strategy highlights the benefits obtained by COVMAP.
THE COVMAP BASED METHOD

RBM
Correction of the electron density map by resolution bias and chemical interpretation of the corresponding structure model.

COVMAP
Modification of the structure model by introducing the covariance concept.

WLSQ
Structure model refinement by least squares with restraints on bond distances and angles.

FINAL MODEL
THE COVMAP BASED METHOD

- **RBM** provides the structure model to COVMAP

- **COVMAP** locates new atomic positions

- **WLSQ** validate the new peaks

- **RBM** relocates in a better way and/or rejects the survived positions and so on cyclically

COVMAP is able to introduce supplementary structural information; RBM and WLSQ can discard false and improve good COVMAP indications
THE COVMAP BASED METHOD

S-BUPIVACAINE HYDROCHLORIDE
C_{18}H_{28}N_{2}O·HCl
22 non-H positions in the asymmetric unit
RES=1.46 Å

DM

ONLY 13 POSITIONS CLOSE TO THE TRUE ONES

RBM-COVMAP-WLSQ

SOLUTION OBTAINED IN ~2 minutes
THE COVMAP BASED METHOD

CAMPHOR
$\text{O}_2\text{C}_{10}$
12 non-H positions in the asymmetric unit

DM

RBM-COVMAP-WLSQ

ONLY 3 POSITIONS CLOSE TO THE TRUE ONES
Refine →→→ RBM →→→ RBM - COVMAP
RBM-COVMAP-WLSQ is a very powerful tool for recovering the full structure from approximate structure models

If we start from a random model (not from DM model)?

RAMM: a new RAndom Model based Method for solving ab initio crystal structure by EXPO package

RAMM skips Direct Methods and applies RBM-COVMAP-WLSQ to fully random starting models
THE RAMM METHOD

Random starting model is generated.

- Weighed least-squares refine atomic fractional coordinates and isotropic displacement parameters.
- The discrepancy factor $R_F$ corresponding to the refined random model is calculated.

- The starting model (SM) corresponding to the smallest $R_F$ value ($R_{FSM}$) is selected among the $n_{random}$ iterations.

RBM-COVMAP-WLSQ optimize SM providing the final model (FM).

- The discrepancy factor $R_{FFM}$ corresponding to FM is calculated.

 selection of the best refined random starting model (SM)

SM optimization (FM is the optimized model)

The model corresponding to the smallest $R_{FFM}$ is selected among the $n_{trial}$ iterations.

NO $R_{FFM} < R_{FSM}$? NO no more iterations

YES

The correct solution is FM.

Selection of the best FM

n_{random} iterations

n_{trial} iterations
Solve --> RAMM
THE RAMM METHOD

RAMM is a useful approach alternative to Direct Methods in case of their failure.

RAMM is usually able not only to find the correct solution, but also to recognize it.

It gives results comparable to and in some cases better than those obtained by Direct Methods.

RAMM requires long execution time (it increases with the number of atoms in the asymmetric unit).
THE RAMM METHOD

EXPO DEFAULT

18 non-H positions in the asymmetric unit

RAMM
• Least squares are carried out by using experimental structure factor moduli as observations, suitably weighted.

• Fourier map: difference map contribution depends on atomic content.

• For the final optimization, weighted least squares are used in case of inorganic compounds (wLSQ); for organic structures, the resolution bias modification (RBM) method is applied.
## THE SHIFT_AND_FIX METHOD

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<th>Unit cell content</th>
<th>Number of non-H atoms in the asymmetric unit</th>
<th>Resolution (Å)</th>
<th>Space group</th>
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Conventional X-ray diffraction data
THE SHIFT_ANDFIX METHOD

Silicoaluminophosphate molecular sieve SAPO-40

THE SHIFT_AND_FIX METHOD

![Graph showing comparison between Default and Shift_and_Fix methods. The graph displays the number of solutions for different values. Default has an average of 4.1 solutions, while Shift_and_Fix has an average of 6.7 solutions.]
Suggestions for structure solution in the reciprocal space

Successfull solution by default run

Interpretable but uncomplete model: Save the model (Export structure)

Cycling RBM (organic/metal-organic) or Fourier Recycling (inorganic)

Cycling COVMAP

Cycling Shift_and_Fix

Improving the extraction of the structure factor moduli from the experimental pattern (if possible)

Solve by Simulated Annealing (if possible)

Uninterpretable and uncomplete model

NO

YES

YES

YES

YES

YES
Scientific Program

Tuesday, 1st October 2019

**EXPO Software**

Angela Altomare, Nicola Corriero, Corrado Cuocci,
Aurelia Falcicchio, Anna Moliterni, Rosanna Rizzi

14:30-16:00 EXPO Computer session: Solution by Direct Methods and structure model optimization

16:00-16:30 Coffee break
We invite contribute of papers that, while discussing the followed computational, methodological, and/or experimental strategies, point out the essential and advanced contribution of powder diffraction in identifying the unknown crystal structure of a compound.

Keywords:
Structure solution methods
Qualitative analysis
Quantitative analysis
Structure refinement
Structure determination

**Special Issue**
Crystal Structure Characterization by Powder Diffraction

**Guest Editor:**
Dr. Angela Altomare
Dr. Rosanna Rizzi
Institute of Crystallography, National Research Council-CNR, Bari, Italy
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**Submission Deadline:** 31 December 2019
200 CHF discounts for the paper submit by the deadline
Thank you for your kind attention