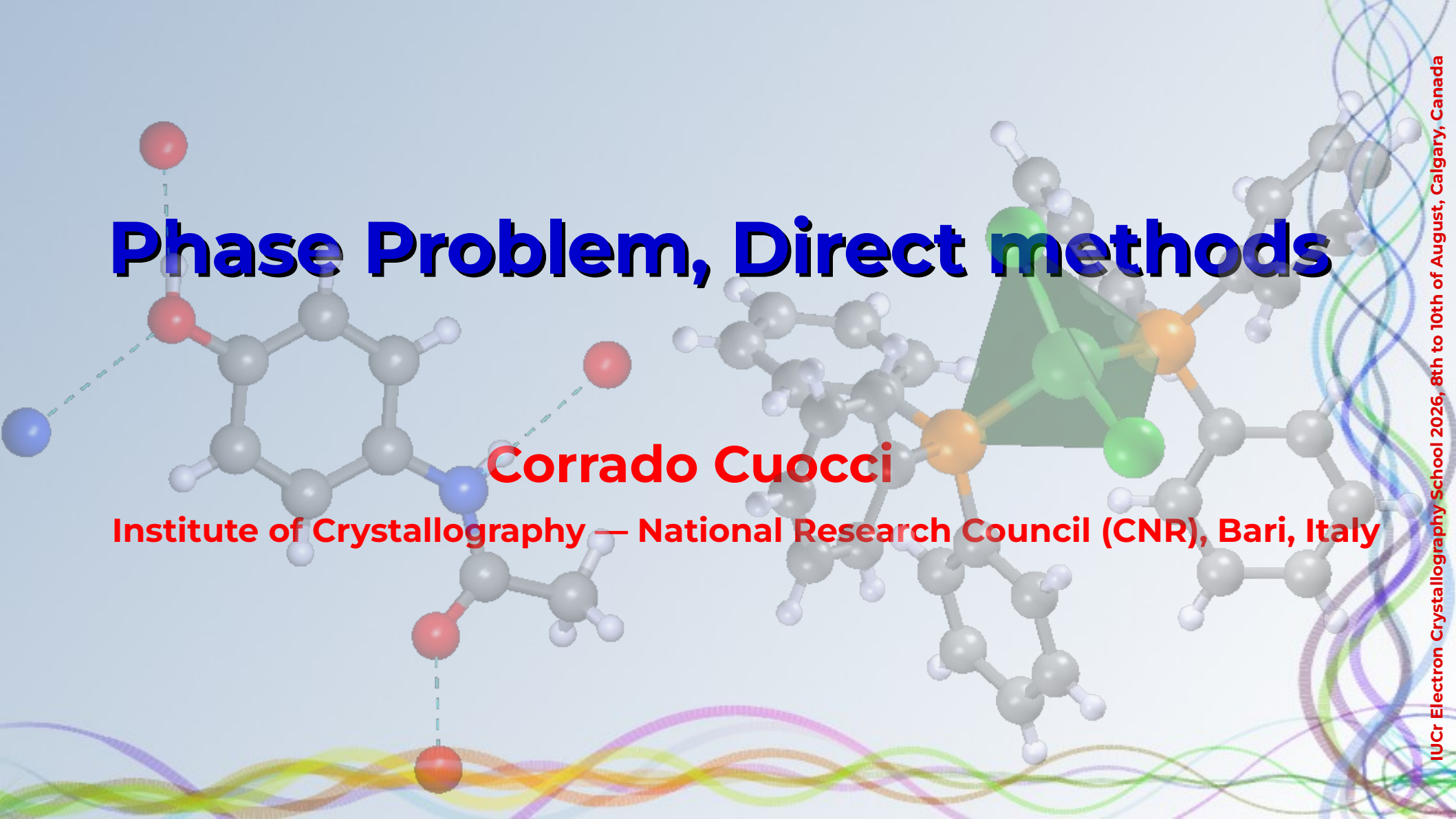


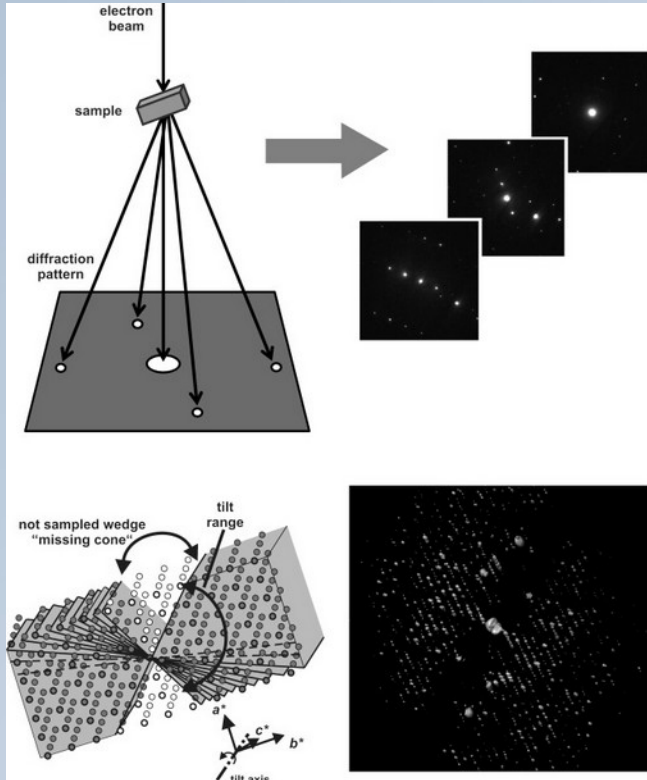
Phase Problem, Direct methods

The background features a complex molecular model with grey, white, red, and blue spheres representing atoms. A green polyhedral shape is superimposed on the model. To the right, a series of colorful, wavy lines in blue, green, yellow, and purple represent a diffraction pattern or electron density map.

Corrado Cuocci

Institute of Crystallography — National Research Council (CNR), Bari, Italy

The Phase Problem



Mugnaioli, E., Kolb, U. (2014). MICROPOROUS AND MESOPOROUS MATERIALS, 189, 107-114

Structure Factor

the total number of atoms in the unit cell

$$F_{\mathbf{h}} = \sum_{j=1}^N f_j \exp(2\pi i \mathbf{h} \cdot \mathbf{r}_j) \quad \leftarrow \quad F_{\mathbf{h}} = \int_V \rho(\mathbf{r}) \exp[2\pi i(\mathbf{h} \cdot \mathbf{r})] d\mathbf{r}$$

the atomic scattering factor

$$\mathbf{h} \cdot \mathbf{r}_j = \begin{pmatrix} h & k & l \end{pmatrix} \times \begin{pmatrix} x_j \\ y_j \\ z_j \end{pmatrix} = hx_j + ky_j + lz_j$$

$$F_{\mathbf{h}} = \sum_{j=1}^n f_j \exp[2\pi i(hx_j + ky_j + lz_j)]$$

Structure Factor

$$F_{\mathbf{h}} = \sum_{j=1}^n O_j t_j(s) f_j(s) \exp [2\pi i(hx_j + ky_j + lz_j)]$$

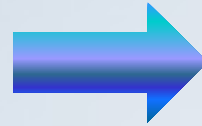
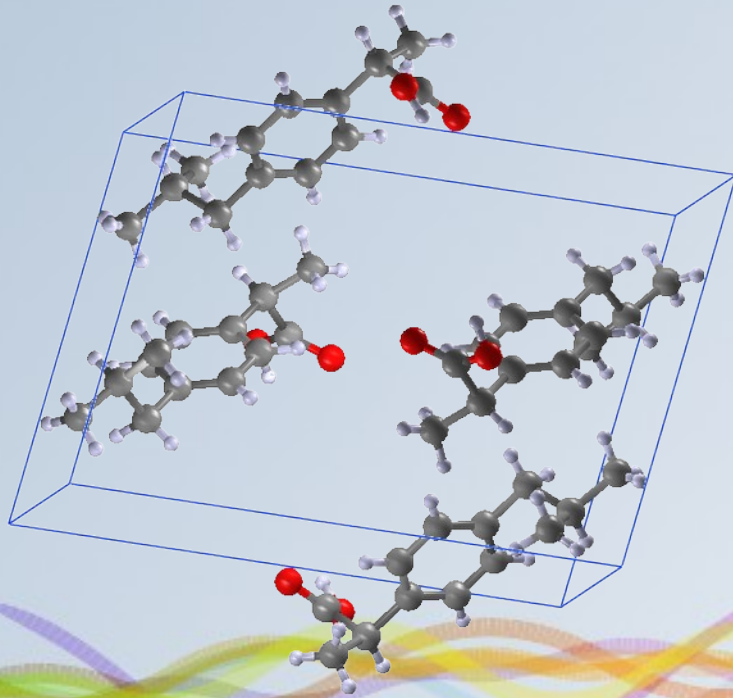
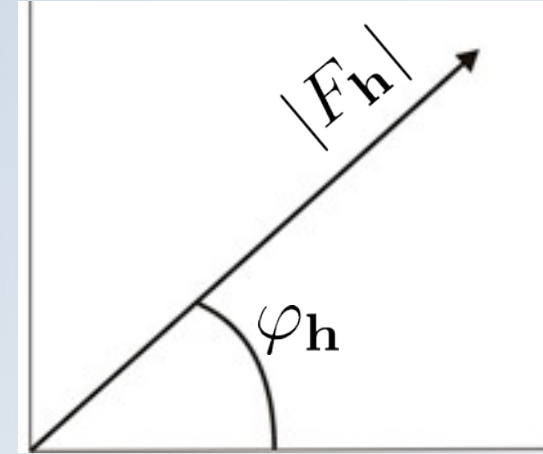
$s = \sin \theta_{\mathbf{h}} / \lambda$

the occupation factor

the temperature factor
(atomic displacement parameters)

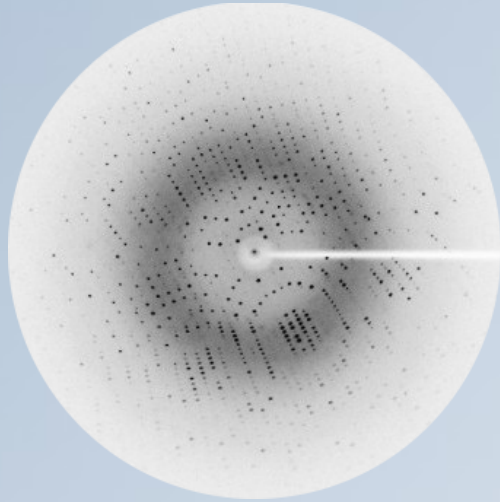
Phase Angle

$$F_{\mathbf{h}} = |F_{\mathbf{h}}| \exp(i\varphi_{\mathbf{h}})$$

 $F_{\mathbf{h}}$ 

Fourier Transform

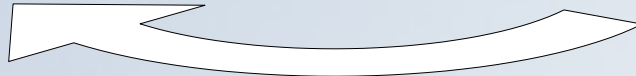
Reciprocal space



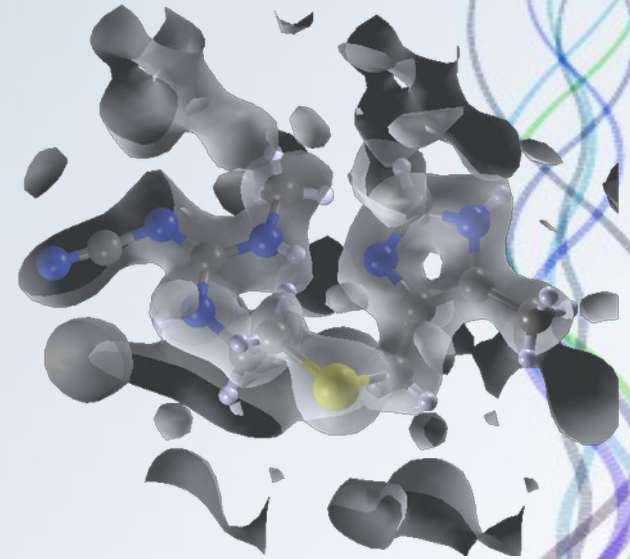
FT^{-1}



FT



Direct space



$$F(\mathbf{h}) = FT[\rho(\mathbf{r})]$$

$$F(\mathbf{h}) = \int_V \rho(\mathbf{r}) \exp[2\pi i(\mathbf{h} \cdot \mathbf{r})] d\mathbf{r}$$

$$\rho(\mathbf{r}) = FT^{-1}[F(\mathbf{h})]$$

$$\rho(\mathbf{r}) = \int_{V^*} F(\mathbf{h}) \exp[-2\pi i(\mathbf{h} \cdot \mathbf{r})] d\mathbf{h}$$

The electron-density equation

$$F_{\mathbf{h}} \xrightarrow{FT^{-1}} \rho(\mathbf{r})$$

$$\rho(\mathbf{r}) = FT^{-1} [F_{\mathbf{h}}] = \frac{1}{V_{cell}} \sum_{\mathbf{h}} F_{\mathbf{h}} \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

$$\rho(x, y, z) = \frac{1}{V_{cell}} \sum_h \sum_k \sum_l F_{hkl} \exp[-2\pi i(hx + ky + lz)] \quad \}$$

$$F_{hkl} = |F_{hkl}| \exp(i\varphi_{hkl}) = |F_{hkl}| (\cos \varphi_{hkl} + i \sin \varphi_{hkl}) \quad \}$$

$$\rho(x, y, z) = \frac{1}{V_{cell}} \sum_h \sum_k \sum_l |F_{hkl}| \cos[2\pi(hx + ky + lz) - \varphi_{hkl}]$$

Phase Problem

$$\rho(x, y, z) = \frac{1}{V_{cell}} \sum_h \sum_k \sum_l |F_{hkl}| \cos [2\pi(hx + ky + lz) - \varphi_{hkl}]$$

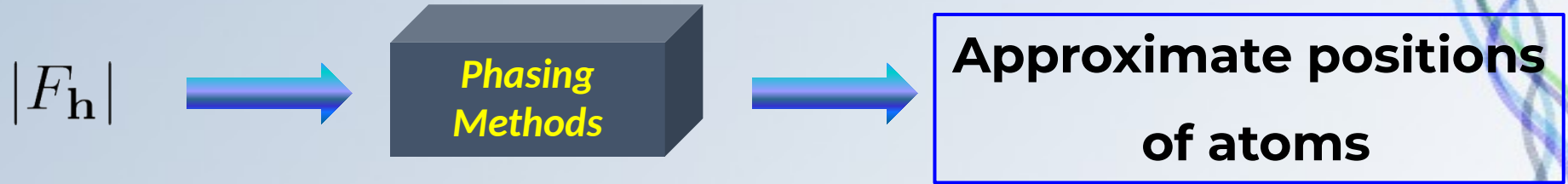
$$I_{hkl} = C |F_{hkl}|^2$$

Phase



Phase Problem

Accurate structure determination requires both the amplitudes and phases of the structure factors. This difficulty is known as the **phase problem**, as the phase information is missing from diffraction data.



Phases must be estimated and refined using appropriate **phasing methods**.

Phasing Methods



!!! The phase problem !!!

$$I = |F|^2$$
$$\varphi = ???$$

Traditional approaches:

- direct methods
- Patterson methods

Other methods:

- charge flipping
- molecular replacement

Direct space methods

Alternative expressions: real space, global optimization, global search

Direct Methods: the Theoretical Basis

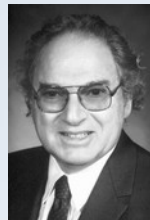
Direct Methods emerged in 1948, achieving full theoretical and practical maturity by the 1970s.

Key points in DM development include

- ♦ Sayre's equation (1952)
- ♦ Structure invariants by Hauptman and Karle (1953)
- ♦ Cochran's three-phase probability distribution (1955)
- ♦ Hauptman and Karle's **tangent formula** (1956)

Automated direct methods software

- ♦ MULTAN (Main, 1980)
- ♦ SIMPEL (Schenk, 1988)
- ♦ SHELX-76 (Sheldrick, 1976)
- ♦ SIR (Giacovazzo, 1982)



Herbert A. Hauptman



Jerome Karle

DM practically solved the phase problem for small molecules, earning H. Hauptman and J. Karle the 1985 **Nobel Prize in Chemistry**.

Constraints on the electron density

- Discrete atoms (atomicity)

$$F_{\mathbf{h}} \xrightarrow{FT^{-1}} \rho(\mathbf{r})$$

$$E_{\mathbf{h}} \xrightarrow{FT^{-1}} \text{point-atom structure}$$

$$|E_{\mathbf{h}}|^2 = \frac{|F_{\mathbf{h}}|^2}{\langle |F_{\mathbf{h}}|^2 \rangle} \quad \langle |F_{\mathbf{h}}|^2 \rangle = \epsilon_{\mathbf{h}} \sum_{i=1}^N f_i^2$$

- Non-negative electron density (positivity)

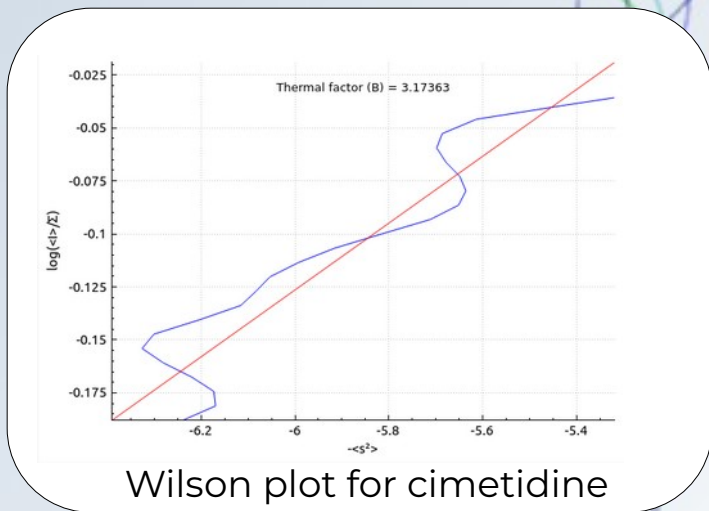
$$\rho(\mathbf{r}) > 0$$

A Typical Direct Methods Procedure

Scaling and normalization of the structure factors $F_h \rightarrow E_h$

$$|F_h|_{obs}^2 = K |F_h|^2 = K |F_h^0|^2 \exp(-2Bs^2)$$

$$\ln \left(\frac{\langle |F_h|_{obs}^2 \rangle_s}{\sum_s^0} \right) = \ln K - 2B \langle s^2 \rangle$$



$$s = \sin(\theta) / \lambda \quad \sum_s^0 = \sum_{j=1}^N (f_j^0)^2$$

$$|E_h|^2 = \frac{|F_h|_{obs}^2}{\langle |F_h|_{obs}^2 \rangle} = \frac{|F_h|_{obs}^2}{K \exp(-2Bs^2) \epsilon_h \sum_{j=1}^N (f_j^0)^2}$$

Normalized Structure Factors

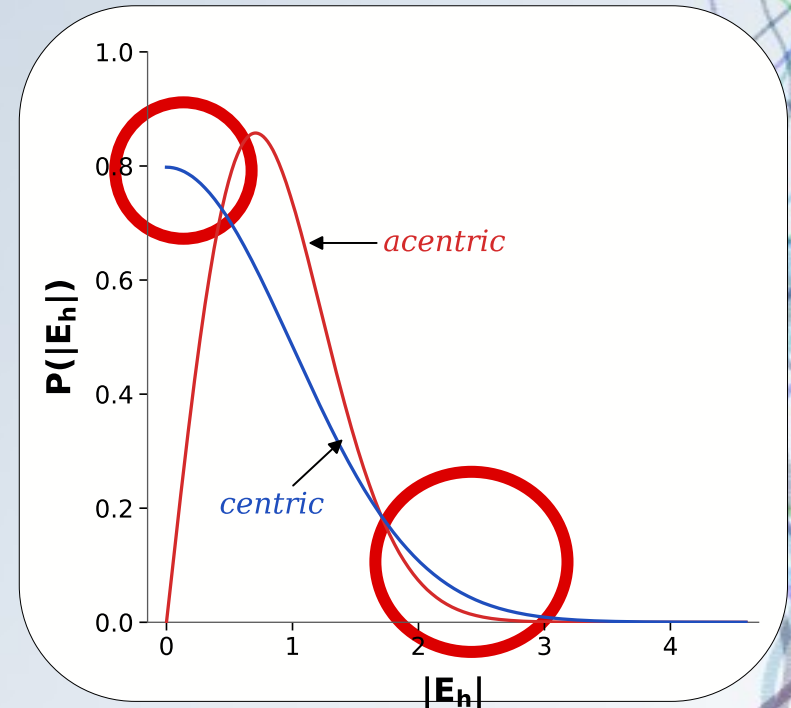
Centric structures

$$P_1(|E|) = \sqrt{\frac{2}{\pi}} \exp(-|E|^2/2)$$

Acentric structures

$$P_1(|E|) = 2|E| \exp(-|E|^2)$$

Normalization helps to reveal underlying symmetry (i.e. inversion centre) in the data.



Comparison of theoretical and experimental distribution functions

	exp. data	acentric	centric	hypercentric	a. c. h.
mod(E)	0.865	0.886	0.798	0.718	*
E**2	1.000	1.000	1.000	1.000	
E**3	1.419	1.329	1.596	1.916	*
E**4	2.364	2.000	3.000	4.500	*
E**5	4.497	3.323	6.383	12.260	*
E**6	9.593	6.000	15.000	37.500	*
mod(E**2-1)	0.805	0.736	0.968	1.145	*
(E**2-1)**2	1.364	1.000	2.000	3.500	*
(E**2-1)**3	4.501	2.000	8.000	26.000	*
(mod(E**2-1))**3	4.974	2.415	8.691	26.903	*

Structure Invariant Definition

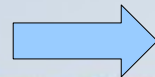
$$F_{\mathbf{h}} = |F_{\mathbf{h}}| \exp(i\varphi_{\mathbf{h}}) = \sum_{j=1}^n f_j \exp(2\pi i \mathbf{h} \cdot \mathbf{r}_j)$$

The origin is moved by a general vector $\mathbf{r}_0$

$$F_{\mathbf{h}} \longrightarrow F'_{\mathbf{h}}$$

$$\begin{aligned} F'_{\mathbf{h}} &= |F'_{\mathbf{h}}| \exp(i\varphi'_{\mathbf{h}}) = \sum_{j=1}^n f_j \exp[2\pi i \mathbf{h} \cdot (\mathbf{r}_j - \mathbf{r}_0)] \\ &= \exp(-2\pi i \mathbf{h} \cdot \mathbf{r}_0) \sum_{j=1}^n f_j \exp(2\pi i \mathbf{h} \cdot \mathbf{r}_j) \\ &= \exp(-2\pi i \mathbf{h} \cdot \mathbf{r}_0) F_{\mathbf{h}} \end{aligned}$$

Shifting the origin by a vector $\mathbf{r}_0$



$$\begin{aligned} |F'_{\mathbf{h}}| &= |F_{\mathbf{h}}| \\ \varphi'_{\mathbf{h}} &= \varphi_{\mathbf{h}} - 2\pi i \mathbf{h} \cdot \mathbf{r}_0 \end{aligned}$$

$|F_{\mathbf{h}}|$ is a structure-invariant quantity, i.e. independent of the choice of origin, whereas $\varphi_{\mathbf{h}}$ is not.

Structure Invariant Definition

A structure invariant is a product of structure factors whose phase is independent of the choice of origin.

The most general structure invariant is represented by the product:

$$F_{\mathbf{h}_1} F_{\mathbf{h}_2} \dots F_{\mathbf{h}_m} = |F_{\mathbf{h}_1} F_{\mathbf{h}_2} \dots F_{\mathbf{h}_m}| \exp[i(\varphi_{\mathbf{h}_1} + \varphi_{\mathbf{h}_2} + \dots + \varphi_{\mathbf{h}_m})]$$

when

$$\mathbf{h}_1 + \mathbf{h}_1 + \dots + \mathbf{h}_m = 0$$

Every structure invariant must satisfy the condition that the sum of the Miller indices equals **ZERO**

Triplet invariants:

$$F_{\mathbf{h}} F_{\mathbf{k}} F_{-\mathbf{h}-\mathbf{k}} = |F_{\mathbf{h}} F_{\mathbf{k}} F_{-\mathbf{h}-\mathbf{k}}| \exp[i(\varphi_{\mathbf{h}} + \varphi_{\mathbf{k}} + \varphi_{-\mathbf{h}-\mathbf{k}})]$$

Quartet invariants:

$$F_{\mathbf{h}} F_{\mathbf{k}} F_{\mathbf{l}} F_{-\mathbf{h}-\mathbf{k}-\mathbf{l}} = |F_{\mathbf{h}} F_{\mathbf{k}} F_{\mathbf{l}} F_{-\mathbf{h}-\mathbf{k}-\mathbf{l}}| \exp[i(\varphi_{\mathbf{h}} + \varphi_{\mathbf{k}} + \varphi_{\mathbf{l}} + \varphi_{-\mathbf{h}-\mathbf{k}-\mathbf{l}})]$$

Probability methods

$$\Phi_{\mathbf{hk}} = \varphi_{\mathbf{h}} + \varphi_{-\mathbf{k}} + \varphi_{\mathbf{k}-\mathbf{h}}$$

$$|F_{\mathbf{h}}| = |F_{-\mathbf{h}}| \longrightarrow I_{\mathbf{h}} = I_{-\mathbf{h}} \quad \text{Friedel's law}$$

$$\varphi_{\mathbf{h}} = -\varphi_{-\mathbf{h}}$$

$$\Phi_{\mathbf{hk}} = \varphi_{\mathbf{h}} + \varphi_{-\mathbf{k}} + \varphi_{\mathbf{k}-\mathbf{h}} = \varphi_{\mathbf{h}} - \varphi_{\mathbf{k}} - \varphi_{\mathbf{h}-\mathbf{k}}$$

The probability formulae for triplet invariants derived by Cochran:

$$P(\Phi_{\mathbf{hk}}) = \frac{1}{2\pi I_0 G_{\mathbf{hk}}} \exp(G_{\mathbf{hk}} \cos(\Phi_{\mathbf{hk}}))$$

Equal atoms:

$$G_{\mathbf{hk}} = \frac{2}{\sqrt{N}} |E_{\mathbf{h}} E_{\mathbf{k}} E_{\mathbf{h}-\mathbf{k}}|$$

$$\sigma_n = \sum_{j=1}^N Z_j^n$$

Non-equal atoms:

$$G_{\mathbf{hk}} = 2\sigma_3 \sigma_2^{-3/2} |E_{\mathbf{h}} E_{\mathbf{k}} E_{\mathbf{h}-\mathbf{k}}|$$

I_0 is the modified Bessel function of zero order

Z is the atomic number

Probability methods

$P(\Phi_{hk})$ is a so-called **von Mises distribution**

$$G_{hk} = \frac{2}{\sqrt{N}} |E_h E_k E_{h-k}|$$

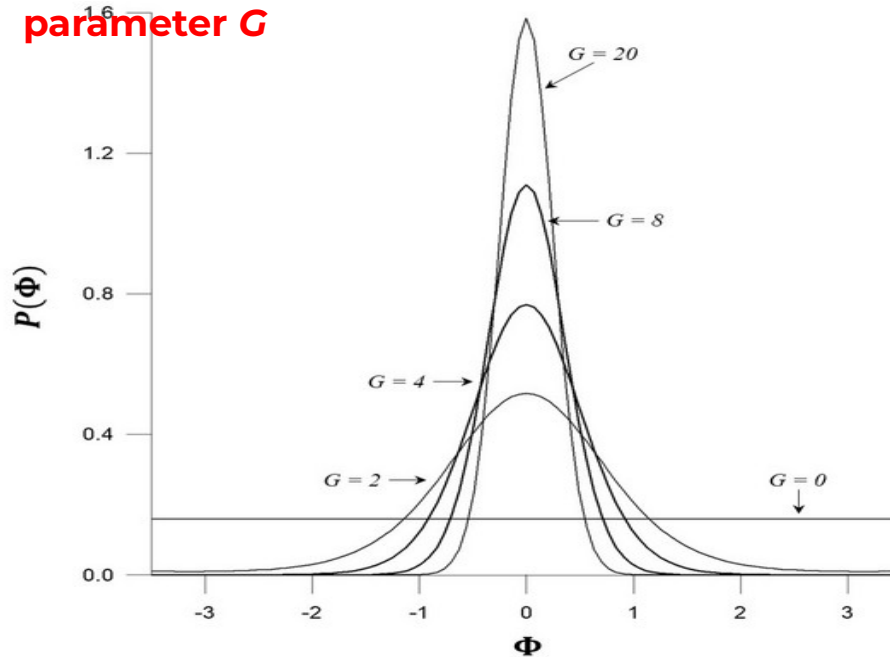
$$\Phi_{hk} = \varphi_h - \varphi_k - \varphi_{h-k} \approx 0$$

$\approx$ indicates 'is distributed about'

$$\varphi_h \approx \varphi_k + \varphi_{h-k}$$

Which shows that if the phases of two structure factors are known then the phase of the third one may be estimated.

Probability distributions based on Cochran's formula for different values of the parameter G



Structure Factor Phase Estimate (the Tangent Formula)

If more than one pair of phases

$$\varphi_{\mathbf{h}} \approx \varphi_{\mathbf{k}_j} + \varphi_{\mathbf{h}-\mathbf{k}_j} \quad j=1,2,\dots,r$$

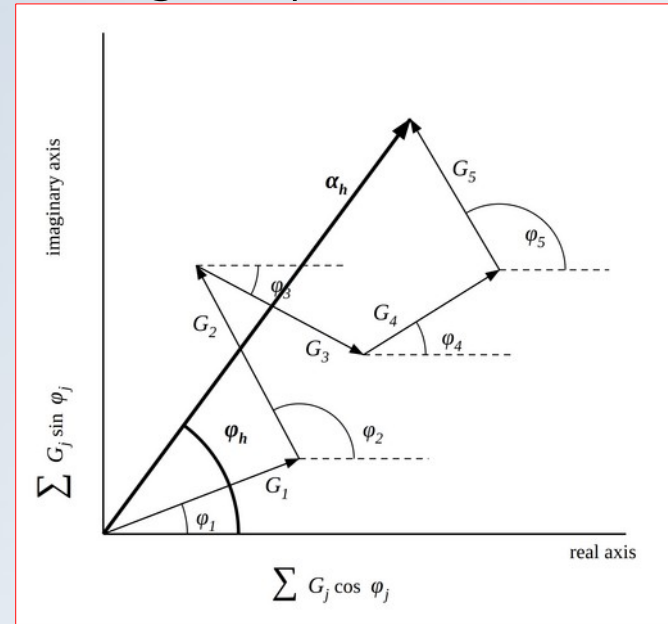
are known, all defining the same phase $\varphi_{\mathbf{h}}$ through triplet relations

$$\tan \varphi_{\mathbf{h}} \approx \frac{\sum_j^r G_j \sin(\varphi_j)}{\sum_j G_j \cos(\varphi_j)} = \frac{A_{\mathbf{h}}}{B_{\mathbf{h}}}$$

$$G_j = G_{\mathbf{h}\mathbf{k}_j}$$

gives the most probable value of $\varphi_{\mathbf{h}}$

$$\alpha_{\mathbf{h}} = \sqrt{A_{\mathbf{h}}^2 + B_{\mathbf{h}}^2} \quad \text{reliability parameter}$$



Graphical representation of the tangent formula: is visualized in the Argand plane as the resultant of the sum of five complex vectors $G_j \exp(i\varphi_j)$.

A Typical Direct Methods Procedure

Scaling and normalization of the structure factors $F_h \rightarrow E_h$

Triplet and negative quartet invariants are found among the reflections with largest E_h

$$\varphi_j \approx \varphi_{\mathbf{k}_j} + \varphi_{\mathbf{h}-\mathbf{k}_j}$$
$$\tan \varphi_{\mathbf{h}} \approx \frac{\sum_r G_j \sin(\varphi_j)}{\sum_j G_j \cos(\varphi_j)}$$

Random phases assignment

Phase Extension

Phase Refinement

DM phase set (trial) and FOM

$N_{\max}$ trial?

No

A Typical Direct Methods Procedure

Scaling and normalization of the structure factors $F_h \rightarrow E_h$

Triplet and negative quartet invariants are found among the reflections with largest E_h

Tangent formula procedure

Random phases assignment

Phase Extension

Phase Refinement

DM phase set (trial) and FOM

$N_{\max}$ trial?

No

A Typical Direct Methods Procedure

Scaling and normalization of the structure factors $F_h \rightarrow E_h$

Triplet and negative quartet invariants are found among the reflections with largest E_h

Select the most reliable trials

Tangent formula procedure

E-map calculation

$$\rho(\mathbf{r}) = FT^{-1}[w_h E_h] = \frac{1}{V_{cell}} \sum_h w_h |E_h| \exp(i\varphi_h) \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

A Typical Direct Methods Procedure

Scaling and normalization of the structure factors $F_h \rightarrow E_h$

Triplet and negative quartet invariants are found among the reflections with largest E_h

Tangent formula procedure

Select the most reliable trials

E-map calculation

Structure refinement and completion

Select a new trial

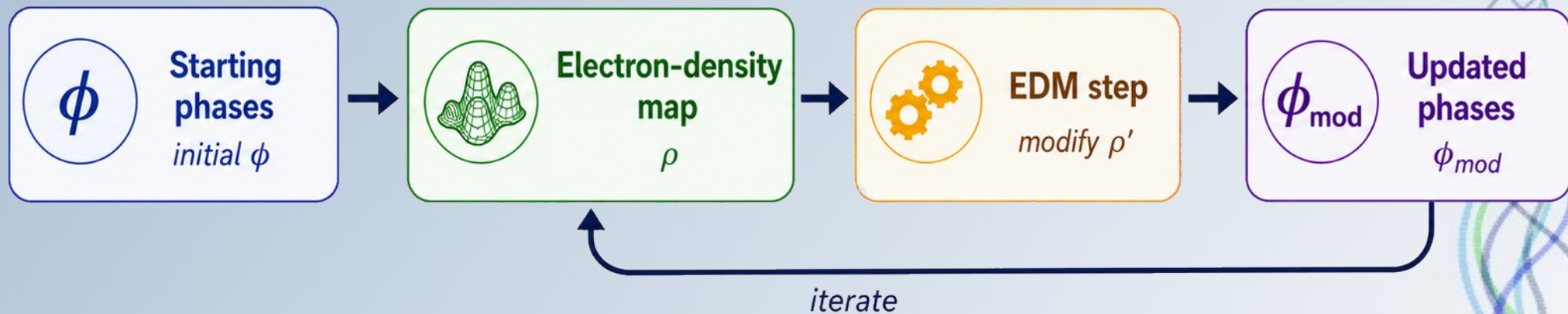
Suitable model?

Structure model

No

Yes

DSR (Direct Space Refinement)



Typical EDM operators



Output: cleaner map and improved phases

Completing and refining the structure

Errors in the model: missing atoms, position errors, errors in the thermal parameters

A Fourier series having as coefficients $F_{\mathbf{h}}^c$

$$\rho_c(\mathbf{r}) = \frac{1}{V} \sum_{\mathbf{h}} F_{\mathbf{h}}^c \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

Positions of the atoms
of the given model

A Fourier series having as coefficients $F_{\mathbf{h}}^o = |F_{\mathbf{h}}^o| \exp(i\varphi_{\text{true}})$

$$\rho_o(\mathbf{r}) = \frac{1}{V} \sum_{\mathbf{h}} F_{\mathbf{h}}^o \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

The true structure

$$\Delta\rho(\mathbf{r}) = \rho_o(\mathbf{r}) - \rho_c(\mathbf{r}) = \frac{1}{V} \sum_{\mathbf{h}} (F_{\mathbf{h}}^o - F_{\mathbf{h}}^c) \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

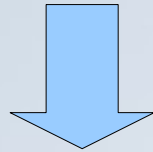
How much the initial model
deviates from the real structure

Completing and refining the structure

Difference Fourier synthesis method

φ_{true} are unknown

$$\Delta\rho(\mathbf{r}) = \rho_o(\mathbf{r}) - \rho_c(\mathbf{r}) = \frac{1}{V} \sum_{\mathbf{h}} (F_{\mathbf{h}}^o - F_{\mathbf{h}}^c) \exp(-2\pi i \mathbf{h} \cdot \mathbf{r})$$

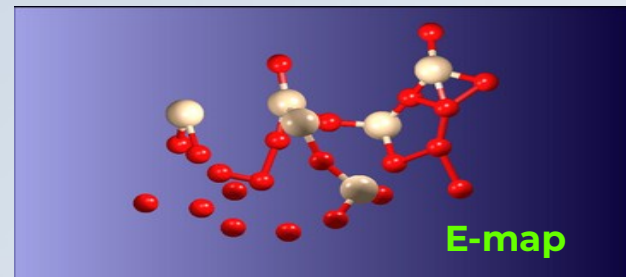
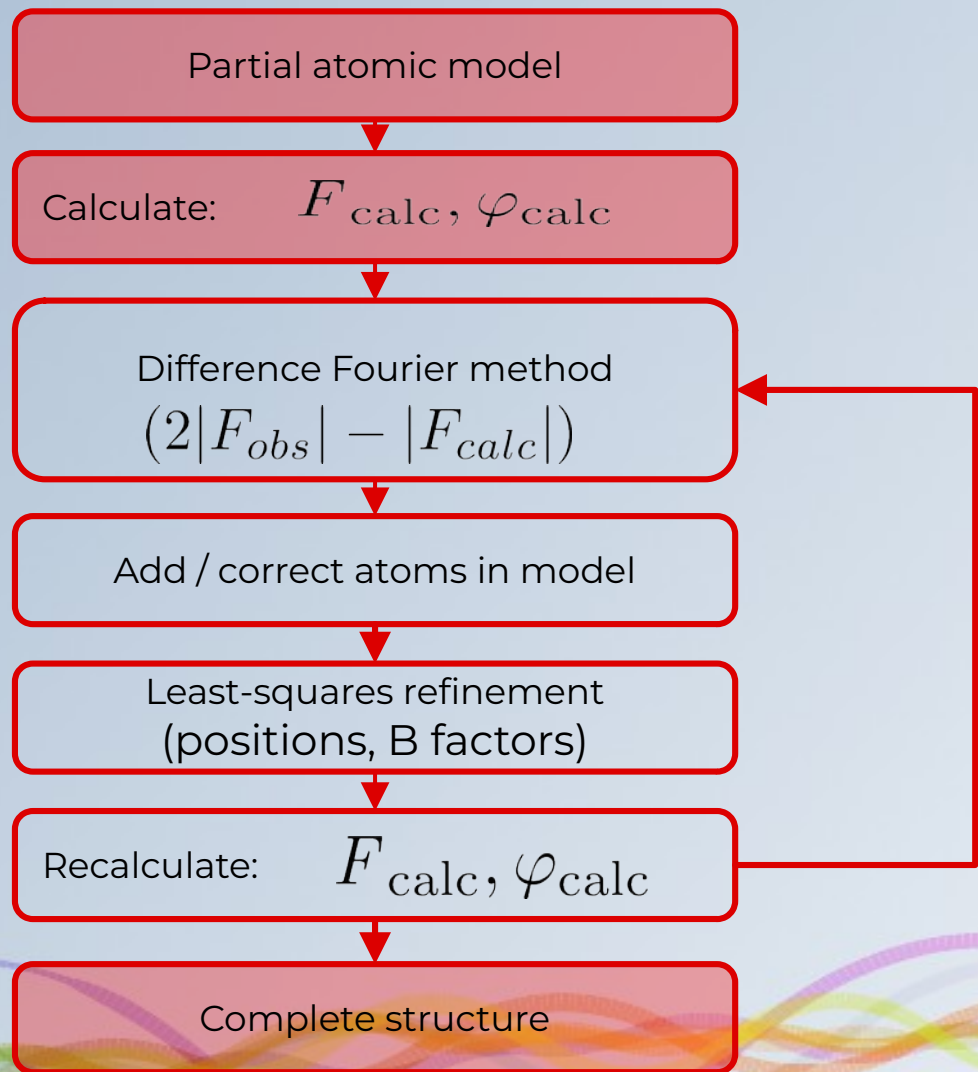


$$\varphi_{\text{true}} \approx \varphi_{\mathbf{h}}^c$$

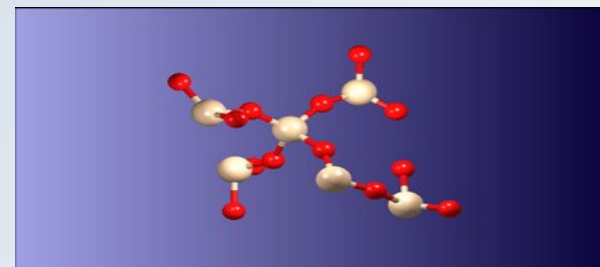
$$\Delta\rho(\mathbf{r}) = \frac{1}{V} \sum_{\mathbf{h}} (|F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c|) \exp(-2\pi i \mathbf{h} \cdot \mathbf{r} + i\varphi_{\mathbf{h}}^c)$$

$$\rho_{\text{best}}(\mathbf{r}) = \frac{1}{V} \sum_{|\mathbf{h}|} (2|F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c|) \exp(-2\pi i \mathbf{h} \cdot \mathbf{r} + i\varphi_{\mathbf{h}}^c)$$

Completion of the Crystal Structure and Preliminary Refinement



**Kinematical
Model
Refinement**



Least-squares method

Functions to minimize:

$$M = \sum_{\mathbf{h}} w_{\mathbf{h}} (|F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c|)^2 \quad M' = \sum_{\mathbf{h}} w'_{\mathbf{h}} (|F_{\mathbf{h}}^o|^2 - |F_{\mathbf{h}}^c|^2)^2$$

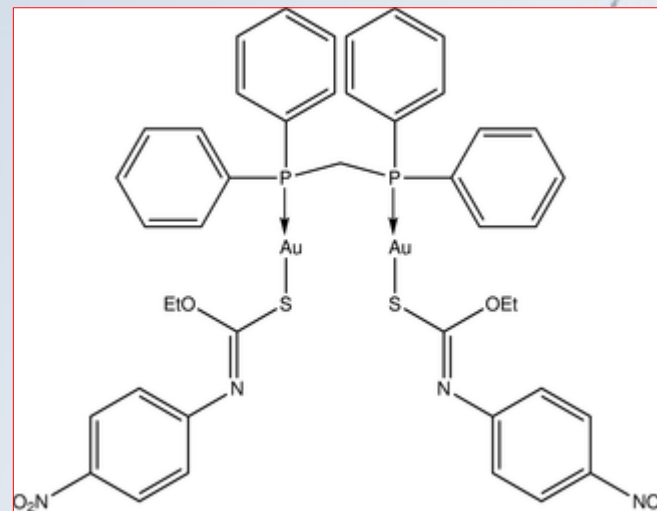
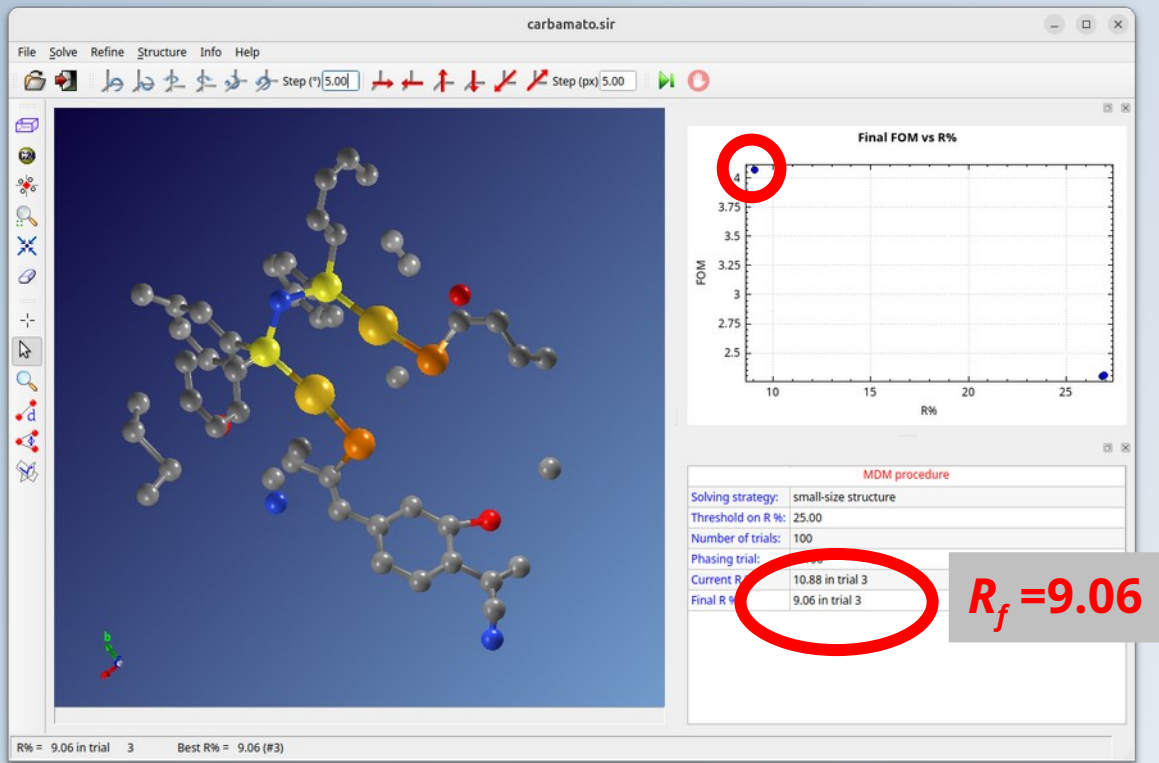
Residual Factors (R-factors):

$$wR_f = \left[\frac{\sum_{\mathbf{h}} w_{\mathbf{h}} (|F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c|)^2}{\sum_{\mathbf{h}} w_{\mathbf{h}} |F_{\mathbf{h}}^o|^2} \right]^{1/2} \quad R_f = \frac{\sum_{\mathbf{h}} ||F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c||}{\sum_{\mathbf{h}} |F_{\mathbf{h}}^o|}$$

$$S = \left[\frac{\sum_{\mathbf{h}} w_{\mathbf{h}} (|F_{\mathbf{h}}^o| - |F_{\mathbf{h}}^c|)^2}{N_R - N_P} \right]^{1/2}$$

Correct Solution Identification

For X-ray data, the correct solution is marked by an outstanding value of the FOM ($R_f \ll 25\%$).



This is not always true for Electron Diffraction data.
The lowest value of R_f does not necessarily indicate the best solution.

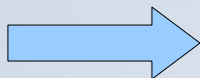
Main Parameters in Phasing Process

Data Resolution

When data resolution is around 1 Å or better, phase determination can normally be obtained *ab initio* by direct methods

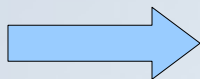
Data Completeness

>70–80% completeness



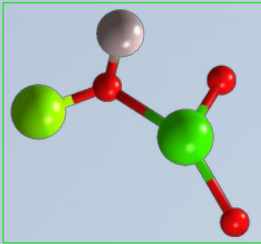
Structure solution is often feasible

<60% completeness

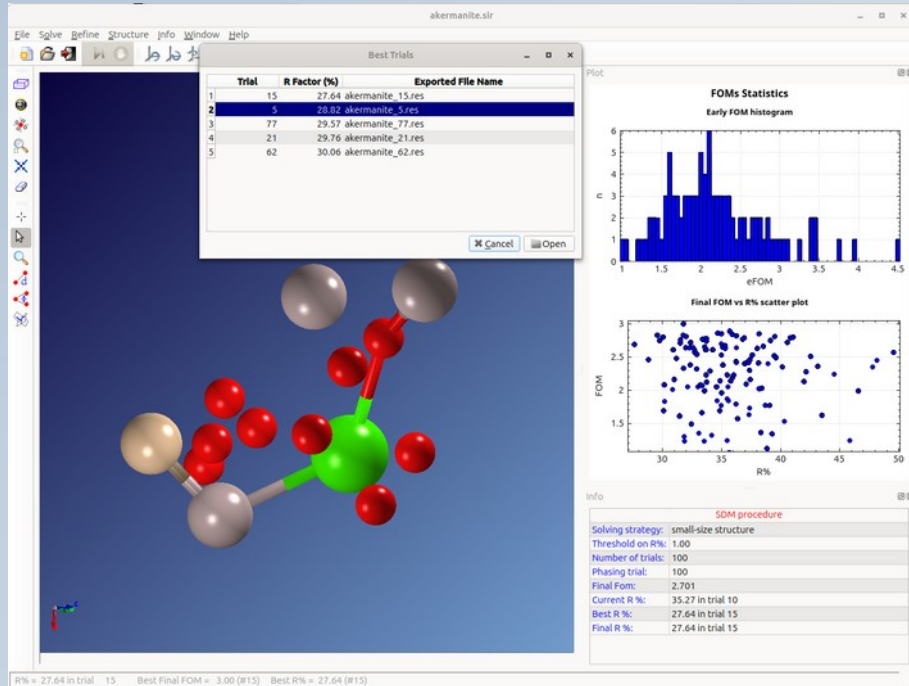


Solution becomes difficult or unstable, depending also on resolution and data quality

Main Parameters in Phasing Process: Data Completeness



Akermanite* PED Data



Data up to 0.4 Å
Completeness ≈ 23%

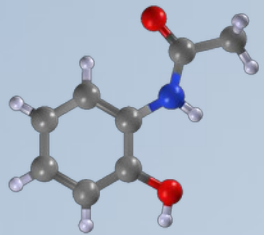
Distribution of reflections up to 0.40 Ang.

Angstrom resolution	No. of expected	No. of observed	No. of missing	% of completeness
up to 10.0	0	0	0	0
10.0 - 8.0	0	0	0	0
8.0 - 6.0	0	0	0	0
6.0 - 5.0	2	2	0	100
5.0 - 4.0	1	1	0	100
4.0 - 3.0	4	3	1	75
3.0 - 2.5	3	3	0	100
2.5 - 2.0	9	6	3	67
2.0 - 1.8	7	4	3	57
1.8 - 1.6	9	4	5	44
1.6 - 1.4	14	9	5	64
1.4 - 1.3	9	4	5	44
1.3 - 1.2	16	8	8	50
1.2 - 1.1	16	8	8	50
1.1 - 1.0	26	14	12	54
1.0 - 0.9	41	17	24	41
0.9 - 0.8	58	22	36	38
0.8 - 0.7	94	35	59	37
0.7 - 0.6	166	53	113	32
0.6 - 0.5	309	83	226	27
0.5 - 0.4	661	63	598	10
All	1445	339	1106	23

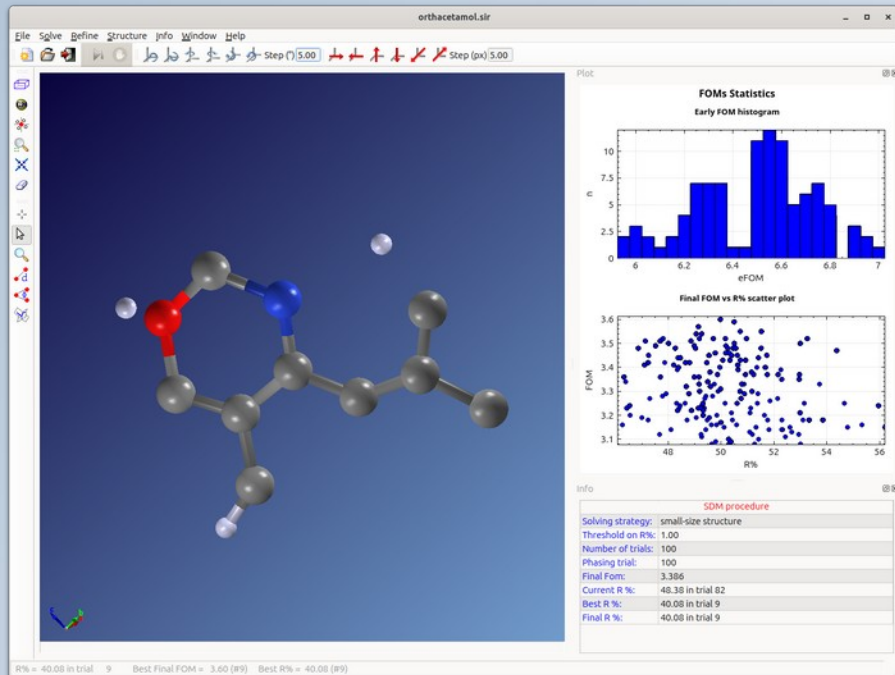
% of completeness up to 0.40 Ang. resolution = 23.46

*Gemmi et al., Amer. Miner. 92, 408 (2007)

Main Parameters in Phasing Process: Data Completeness



Orthocetamol* STEM-3DED Data



Distribution of reflections up to 0.78 Ang.

Angstrom resolution	No. of expected	No. of observed	No. of missing	% of completeness
up to 10.0	0	0	0	0
10.0 - 8.0	0	0	0	0
8.0 - 6.0	4	4	0	100
6.0 - 5.0	4	2	2	50
5.0 - 4.0	4	3	1	75
4.0 - 3.0	17	17	0	100
3.0 - 2.5	20	14	6	70
2.5 - 2.0	47	42	5	89
2.0 - 1.8	33	26	7	79
1.8 - 1.6	66	56	10	85
1.6 - 1.4	88	70	18	80
1.4 - 1.3	68	58	10	85
1.3 - 1.2	95	78	17	82
1.2 - 1.1	140	115	25	82
1.1 - 1.0	186	161	25	87
1.0 - 0.9	291	243	48	84
0.9 - 0.8	443	364	79	82
0.8 - 0.7	101	31	70	31
All	1607	1284	323	80

% of completeness up to 0.78 Ang. resolution = 79.90

Data up to 0.78 Å
Completeness ≈ 80%

*Andrusenko et al., Angew. Chem. Int. Ed. 2019, 58, 10919–10922

Structure Solution of the complex silicate $\text{Ca}_2\text{FeAl}_2\text{Si}_3\text{O}_{13}$ by Direct Methods

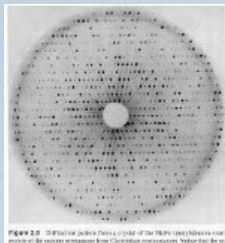
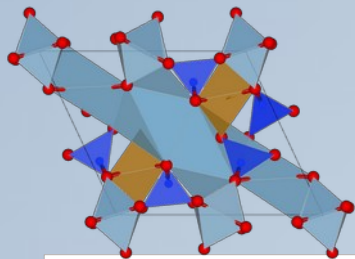
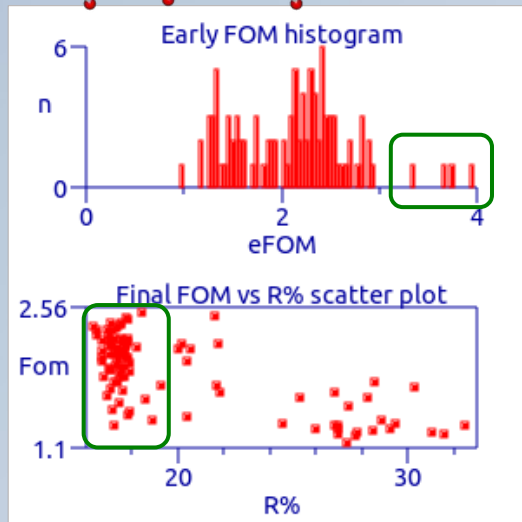


Figure 2.5. Diffraction pattern (image of the film) (left) and the corresponding intensity map (right) of the single-crystal diffraction pattern of $\text{Ca}_2\text{FeAl}_2\text{Si}_3\text{O}_{13}$.

Cell: 8.86 5.62 10.11 90 115.3 90

Space Group: $P2_1/m$



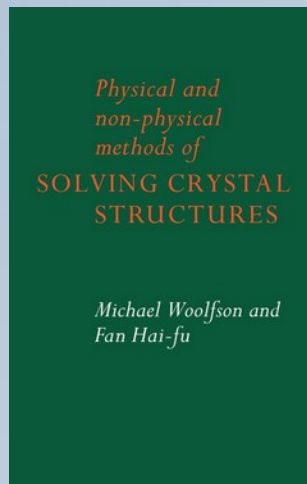
Res: 0.77 $R_{\text{int}} = 14.27\%$

Angstrom	Number	Rint
up to 10.0	0	----
10.0 - 8.0	1	3.00%
8.0 - 6.0	0	----
6.0 - 5.0	1	16.79%
5.0 - 4.0	5	5.59%
4.0 - 3.0	10	7.61%
3.0 - 2.5	13	7.17%
2.5 - 2.0	31	10.02%
2.0 - 1.8	22	12.12%
1.8 - 1.6	37	11.39%
1.6 - 1.4	51	11.64%
1.4 - 1.3	37	13.76%
1.3 - 1.2	69	14.29%
1.2 - 1.1	74	15.48%
1.1 - 1.0	114	14.44%
1.0 - 0.9	165	16.38%
0.9 - 0.8	257	16.25%
0.8 - 0.7	116	16.60%
All	1003	14.27%

Distribution of reflections up to 0.77 Ang.				
Angstrom resolution	No. of expected	No. of observed	No. of missing	% of completeness
up to 10.0	0	0	0	0
10.0 - 8.0	2	1	1	50
8.0 - 6.0	1	0	1	0
6.0 - 5.0	1	1	0	100
5.0 - 4.0	7	5	2	71
4.0 - 3.0	12	10	2	83
3.0 - 2.5	17	13	4	76
2.5 - 2.0	36	31	5	86
2.0 - 1.8	24	22	2	92
1.8 - 1.6	45	39	6	87
1.6 - 1.4	62	51	11	82
1.4 - 1.3	41	38	3	93
1.3 - 1.2	79	69	10	87
1.2 - 1.1	83	76	7	92
1.1 - 1.0	130	118	12	91
1.0 - 0.9	188	167	21	89
0.9 - 0.8	295	265	30	90
0.8 - 0.7	133	118	15	89
All	1156	1024	132	89
% of completeness up to 0.77 Ang. resolution = 88.58				

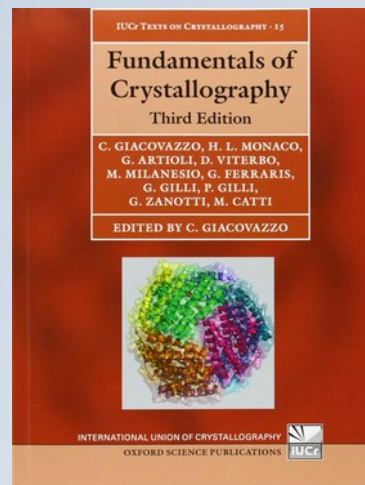
The merging residual value: $R_{\text{int}} = \frac{\sum |F_o^2 - \langle F_o^2 \rangle|}{\sum F_o^2}$

Recommended Reading on Direct Methods



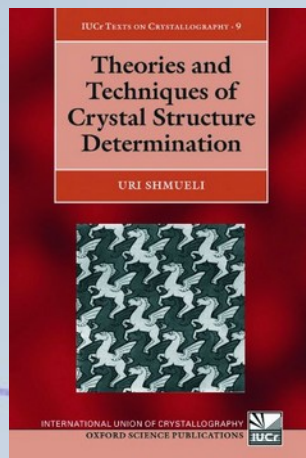
Physical and Non-Physical Methods of Solving Crystal Structures 1st Edition

by Michael M. Woolfson, Fan Hai-Fu



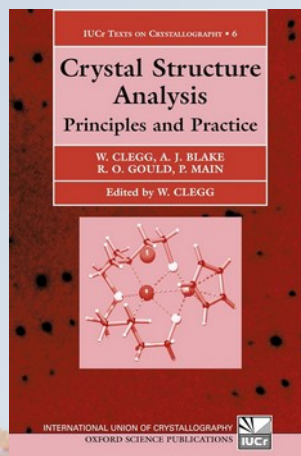
Fundamentals of Crystallography (International Union of Crystallography Texts on Crystallography) 3rd Edition

by Carmelo Giacovazzo, Hugo Luis Monaco, Gilberto Artioli, Davide Viterbo, & 6 more



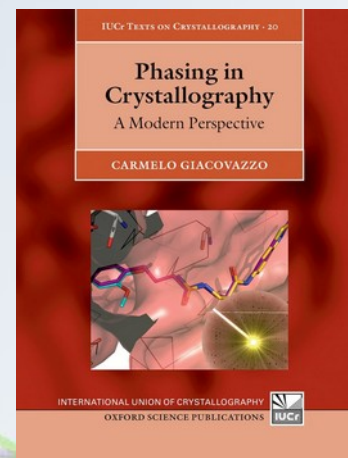
Theories and Techniques of Crystal Structure Determination

by Uri Shmueli



Crystal Structure Analysis: Principles and Practice

by Peter Main, Clegg William, Alexander J. Blake, Robert O. Gould



Phasing in Crystallography: A Modern Perspective

by Carmelo Giacovazzo