Data retrieval and structure determination from synchrotron PD data

ALBA Synchrotron , 3th-5th Abril 2013

Cluster-based Direct Methods

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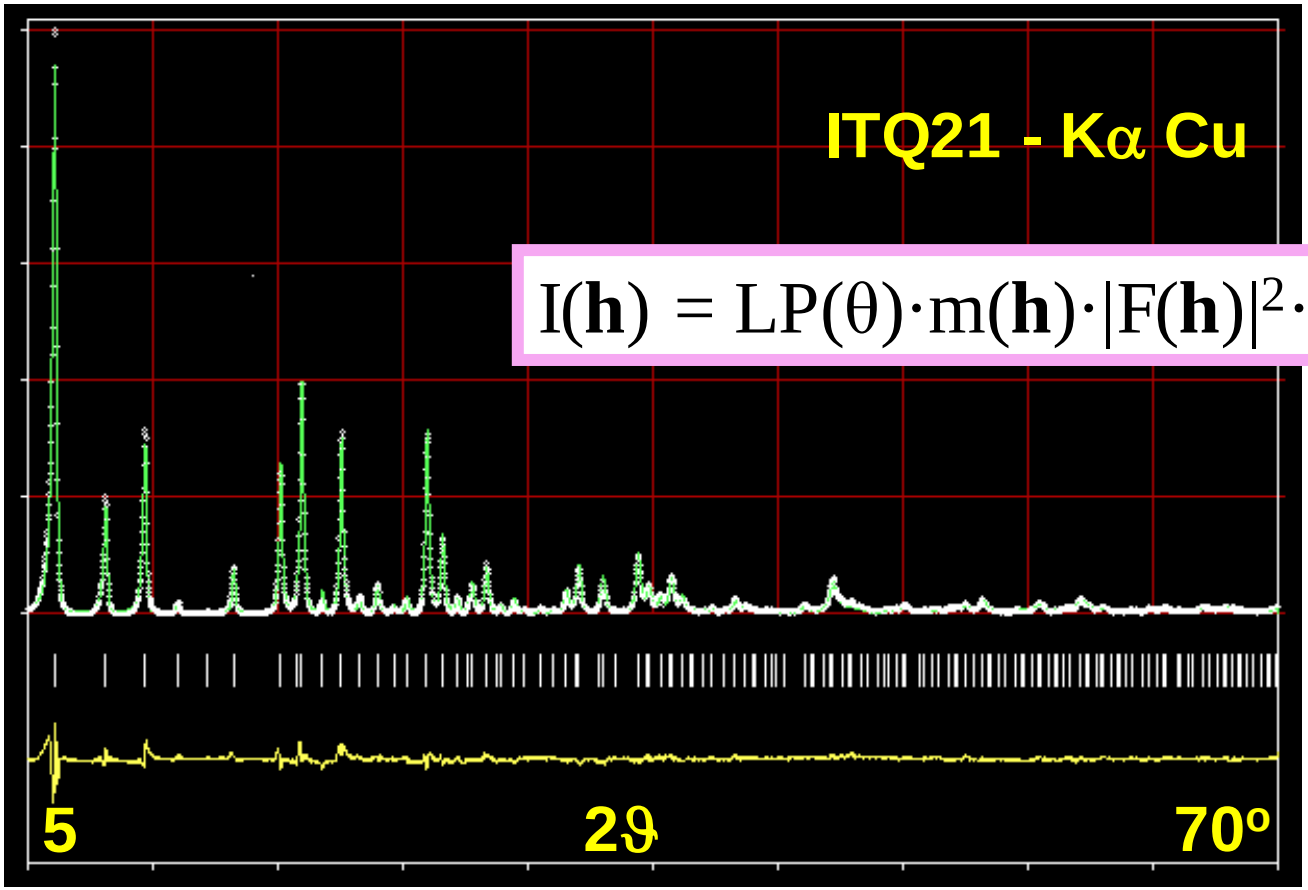
Summary

- **Hystorical remarks on the extraction of integrated intensities**
- **Progress in PD direct methods (DM)**
- **The concept of cluster-based DM**
- **Measuring geometries: strengths/limitations**
- **The S_P-FFT phasing method**
- **Application to inorganic/organic compounds**

Integrated intensities in powder diffraction



$$I(\mathbf{h}) = LP(\theta)\cdot m(\mathbf{h})\cdot |F(\mathbf{h})|^2 \cdot A(\theta)$$

Extracting intensity information from PD patterns

Individual fit: Clusters of intensities

Limited to simple structures (or to indexing)

Extracting intensity information from PD patterns

Individual fit: Clusters of intensities

Limited to simple structures (or to indexing)

Use of metric as constraint

Pawley/LeBail fit:
Integrated intensities for resolved reflections +
unknown intensities for unresolved reflections

PD patterns with systematic overlap

space groups affected by systematic overlap (%):

- **with cubic lattice: 33%**
- **with tetragonal lattice: 21%**
- **with hexagonal lattice: 65%**

Intensities improvement

Intensities are normally extracted either using the **Pawley (1981)** or the **LeBail (1988)** method. Other methods are (if applicable):

Squared Patterson function: Estermann, McCusker, Baerlocher (1992) J.Appl.Cryst. 25, 539-543

Anisotropic expansion: Shankland, David, Sivia (1997) J.Mater.Chem. 7, 569-572

Texture: Wessels, Baerlocher, McCusker (1999) Science 284, 477-479

Use of envelopes to aid phase determination: Brenner, McCusker, Baerlocher (1997) *J.Appl.Cryst.* 30 1167-1172

Isomorphous-replacement: Rius, Peral, Margiolaki,Torrelles (2005) *J.Appl.Cryst.* 38 906-911

How structure solution methods handle the overlapped regions

Direct methods
Atomicity + phases

Reflections in overlapped regions are ignored

Direct-space methods
Molecular geometry
+
conformational and positional parameters defined in direct space

Profile intensities are used (overlap difficulty disappears)

Progress in PD direct methods

(1995) Rius,Sañé,Miravitlles,Gies,Marler,Oberhagemann *Acta Cryst* A**51** 840-845
SF applied to intensity data at 2Å resolution
(1997) Grosse-Kunstleve,McCusker,Baerlocher *Acta Cryst* A**30** 985-995
FOCUS algorithm incorporating framework information
(1999) Rius,Miravitlles,Gies,Amigó *J.Appl.Cryst*. **32** 89-87
SF can be applied to data with systematic overlap
(2000) Rius,Torrelles,Miravitlles,Ochando,Reventós,Amigó *J.Appl.Cryst*. **33** 1208-1211
SF applied to data with accidental overlap (Overlapped E's are updated)
(2002) Altomare,Cuocci,Giacovazzo,Gugliardi,Moliterni,Rizzi *J.Appl.Cryst*. **35** 182-184
DM + Fourier recycling improvements

Progress in PD direct methods (cont.)

(2006) Wu,Leineweber,Spence,O'Keefe *Nature* **5** 647-652
CF and E's modification

(2007) Baerlocher,McCusker,Palatinus *Z.Kristallogr*. **222** 47-53
CF and histogram matching

(2007) Rius,Frontera *J.Appl.Cryst*. **40** 1035-1038
SF (in Fourier space) with E's treatment

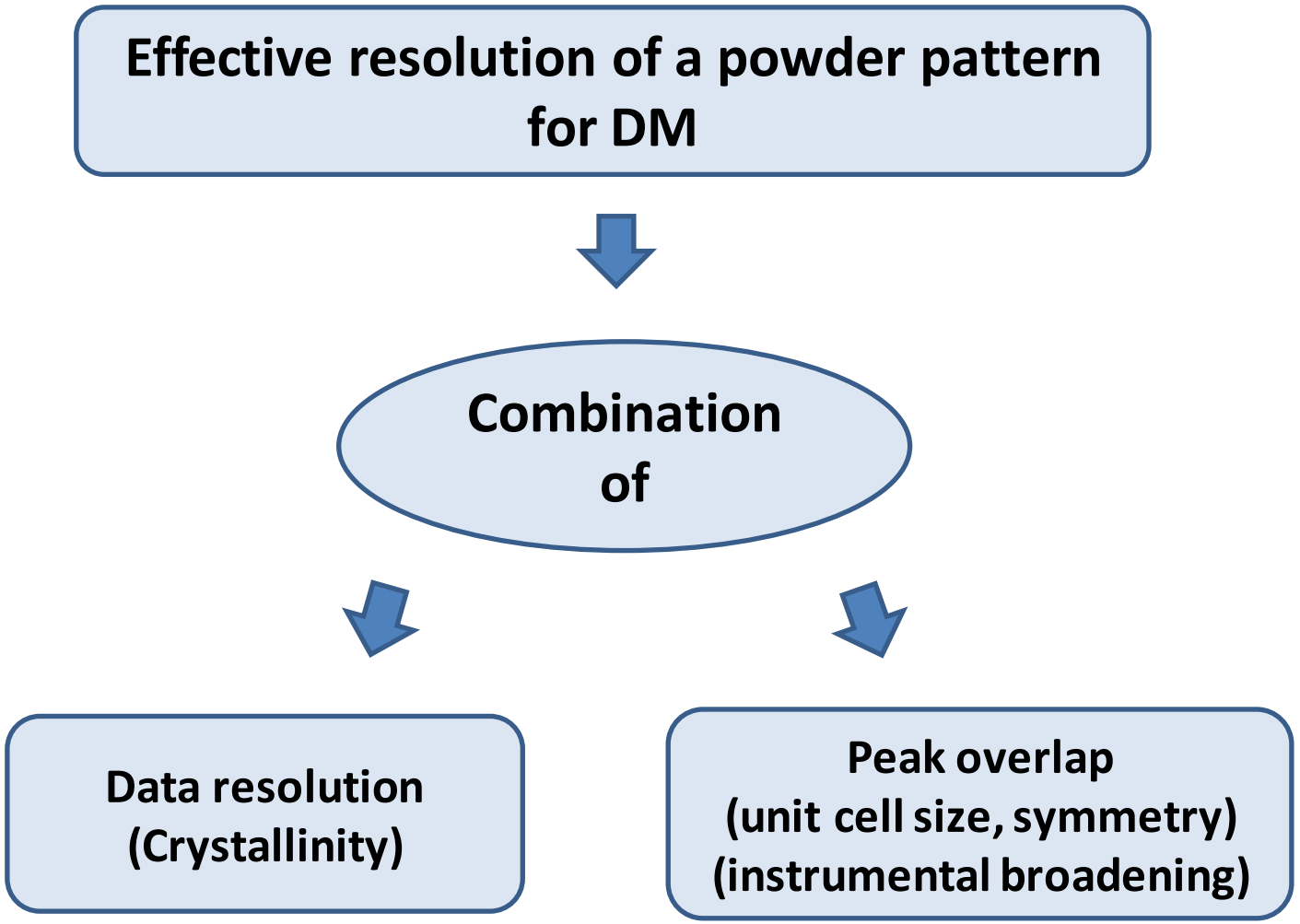
(2007) Baerlocher,Gramm,Massüger,McCusker,He,Hovmöller,Zou *Science* **315** 1113-16
CF and electron microscopy information

(2008) Altomare,Caliandro,Cuocci,Giacovazzo,Moliterni et al. *J.Appl.Cryst*. **41** 56-61
DM and simulated annealing

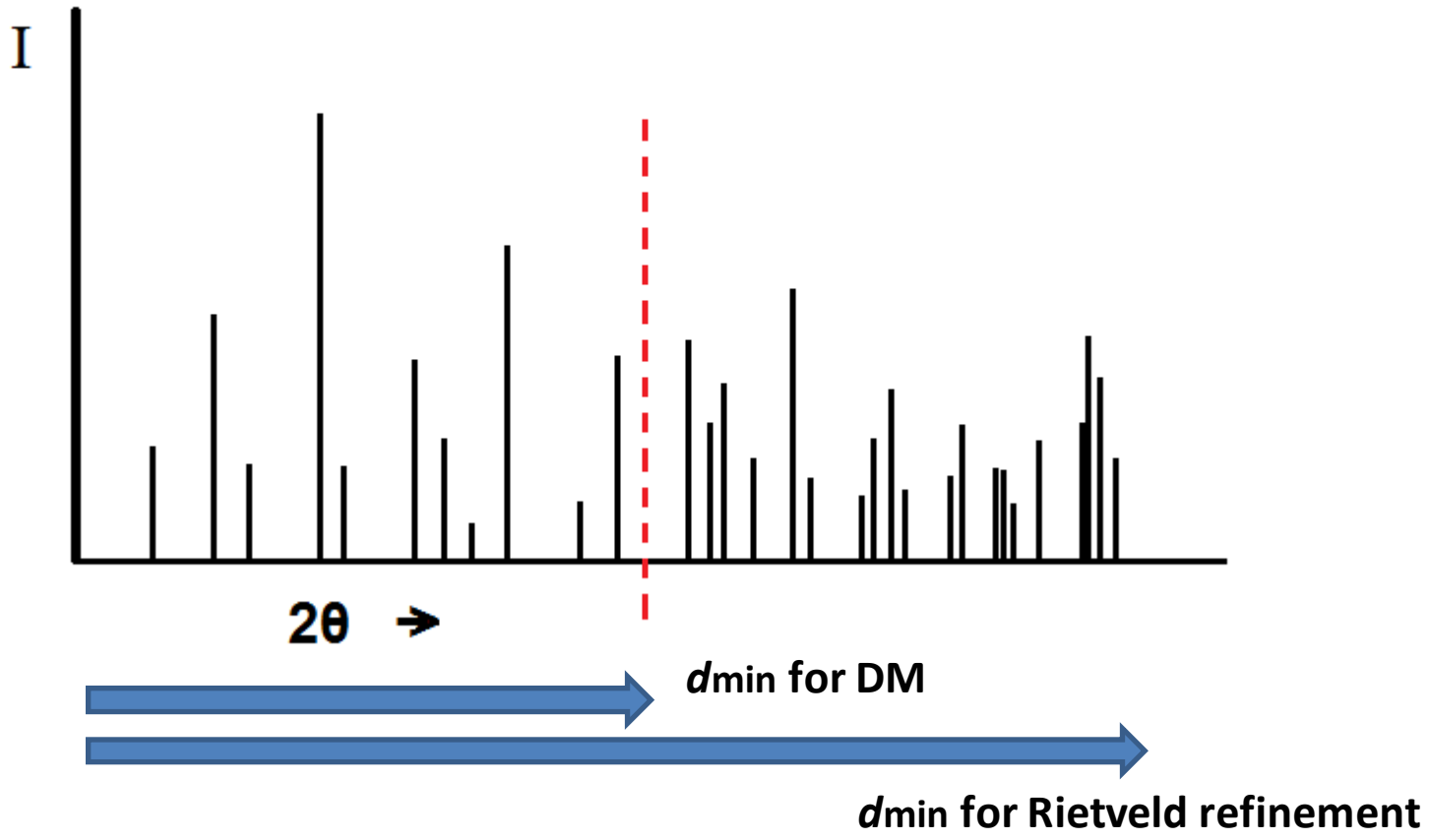
(2008) Altomare,Cuocci,Giacovazzo,Kamel,Moliterni,Rizzi *Acta Cryst*. A**64** 326-336
DM and minimally resolution biased electron-density maps

(2011) Rius *Acta Cryst* A67 63-67
Cluster-based SF (in Fourier space) with individual & overlapped I as data

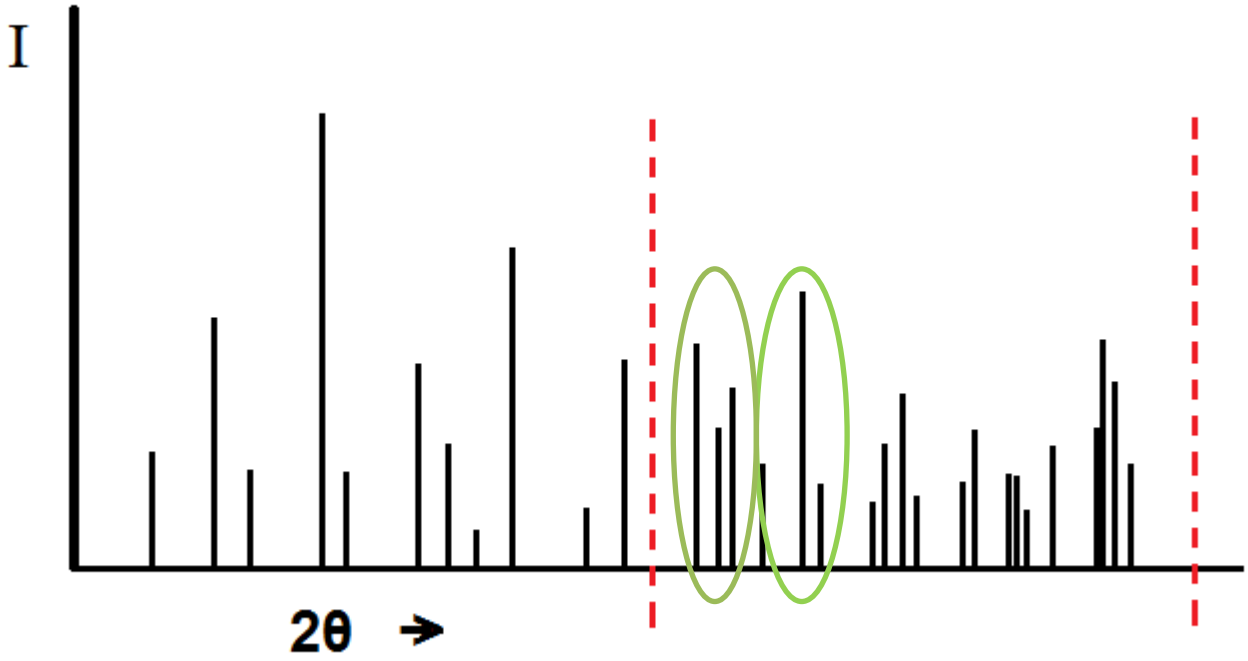
DM=conventional direct methods in reciprocal space
SF= direct methods sum function
CF= charge-flipping


Effective resolution of a powder pattern for DM
Combination of
Data resolution (Crystallinity)
Peak overlap (unit cell size, symmetry) (instrumental broadening)

Effective resolution for DM
Case 1: Only integrated intensities of resolved reflections



Effective resolution for DM
Case 2: Inclusion of integrated intensities of resolved clusters



*d*min for DM

*d*min for Rietveld refinement

Cluster-related quantities

Intensity of cluster *i*: $D_i = \sum_{k(i)} m_k \cdot |E_k|^2$

N. of contributors: $n_i = \sum_{k(i)} m_k$

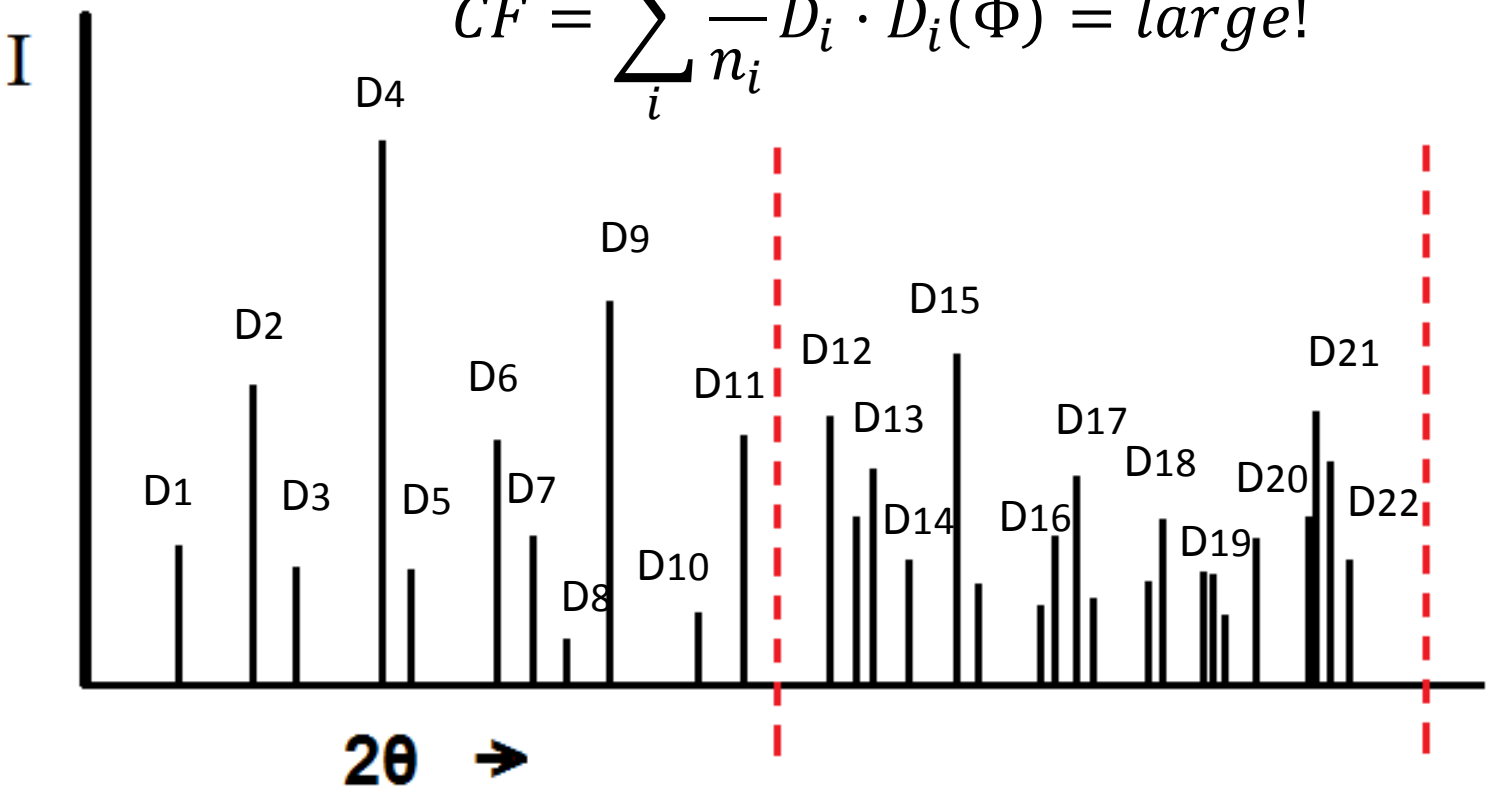
$$\mathrm{I_H} = \frac{D_i}{n_i}$$

Equidistributed intensities

(all reflections of one cluster have the same I)

1. The coincidence function

$$CF = \sum_i \frac{1}{n_i} D_i \cdot D_i(\Phi) = large!$$



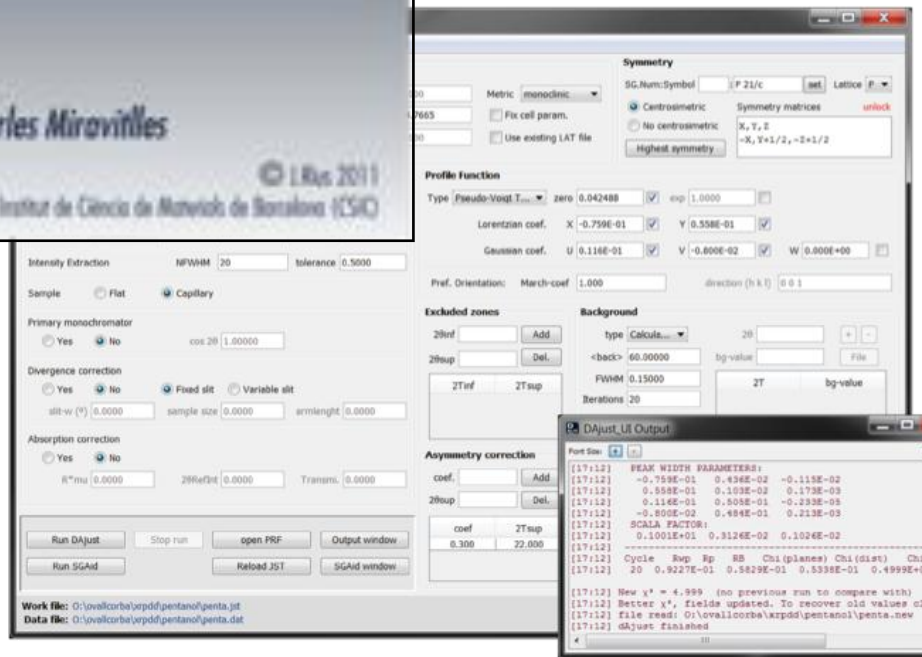
2. The cluster-based Residual

$$RV = 100 \cdot \frac{\sum_i \left| \sqrt{D_i} - \sqrt{D_i(\Phi)} \right|}{\sum_i \sqrt{D_i}}$$

D_i =observed intensity of cluster i

$D_i(\Phi)$= corresponding calculated intensity

Decomposition of a PD pattern into clusters



www.icmab.es/crystallography/software

J.Appl.Cryst. (2012) 45, 844-848

Space group estimation from refined metric and lattice type

SGAid Output

Click on a row to load symmetry matrices for the S.G into main window

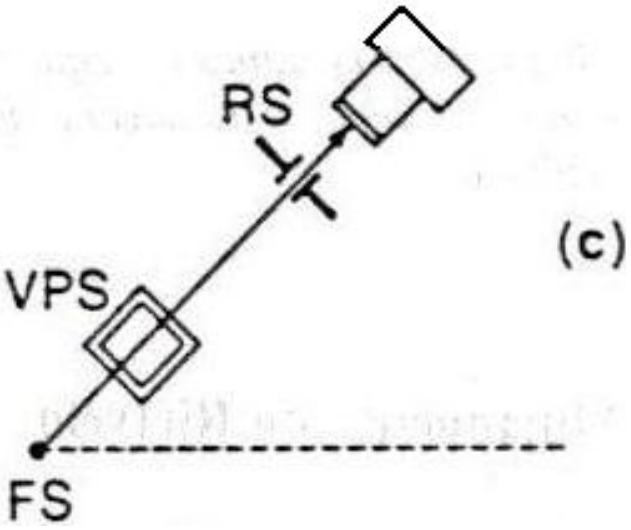
on top

SGNum	H-M symbol	RW (%)	NREF	Setting
11	P2(1)/m	**8,91**	608	abc
4	P2(1)	**8,91**	608	abc
3	P2	**8,91**	610	abc
10	P2/m	**8,91**	610	abc
6	Pm	**8,91**	610	abc
14	P2(1)/c	**9,19**	514	abc
13	P2/c	**9,19**	516	abc
7	Pc	**9,19**	516	abc
232	P2(1)/a	29,16	513	abc
235	Pa	29,16	515	abc
231	P2/a	29,16	515	abc
234	P2(1)/n	29,22	515	abc
236	Pn	29,22	517	abc
233	P2/n	29,22	517	abc

Example of clustering: The file code.pow

609	-4	4	6	0.0000	4	3	1	13.378	1.28696	0.01716
610	-10	6	1	11.7608	4	3	0	13.385	1.28622	0.01717
611	-3	5	5	14.7751	4	3	0	13.392	1.28560	0.01717
612	19	3	2	21.6624	4	1	1	13.408	1.28408	0.01718
613	4	6	3	9.5681	4	6	1	13.423	1.28258	0.01719
614	-18	4	2	0.0000	4	6	0	13.430	1.28192	0.01719
615	-22	2	1	0.0000	4	6	0	13.434	1.28154	0.01720
616	1	5	5	0.0000	4	6	0	13.438	1.28122	0.01720
617	-12	4	5	0.0000	4	6	0	13.443	1.28076	0.01720
618	-14	2	6	12.0455	4	6	0	13.445	1.28051	0.01720
619	-6	6	3	8.5926	4	1	1	13.457	1.27940	0.01721
620	8	6	2	0.0000	4	3	1	13.474	1.27782	0.01722
621	18	4	1	3.7403	4	3	0	13.474	1.27775	0.01722
622	22	2	0	17.0356	4	3	0	13.479	1.27734	0.01723
623	2	4	6	6.7109	4	1	1	13.491	1.27619	0.01723
624	-8	2	7	8.5154	4	10	1	13.507	1.27466	0.01724
625	4	2	7	6.3116	4	10	0	13.516	1.27387	0.01725
626	-5	5	5	14.3962	4	10	0	13.522	1.27325	0.01725
627	-16	0	6	7.2488	2	10	0	13.527	1.27282	0.01726
628	15	5	1	3.4754	4	10	0	13.528	1.27268	0.01726
629	10	6	1	13.0145	4	10	0	13.533	1.27221	0.01726
630	-11	5	4	12.3077	4	10	0	13.541	1.27149	0.01727
631	-6	4	6	9.0573	4	10	0	13.544	1.27119	0.01727
632	-15	5	2	4.5380	4	10	0	13.551	1.27059	0.01727
633	-23	1	1	7.8477	4	10	0	13.555	1.27022	0.01727
634	23	1	0	12.7425	4	8	1	13.606	1.26546	0.01731
635	3	5	5	7.0988	4	8	0	13.613	1.26478	0.01731
636	9	5	4	11.0295	4	8	0	13.621	1.26402	0.01732
637	-22	2	2	4.9096	4	8	0	13.626	1.26358	0.01732
638	-11	1	7	0.0000	4	8	0	13.632	1.26305	0.01732
639	-21	3	1	0.0000	4	8	0	13.636	1.26264	0.01733
640	13	3	5	0.0000	4	8	0	13.643	1.26203	0.01733
641	7	1	7	15.3379	4	8	0	13.645	1.26189	0.01733

Debye – Scherrer geometry



Hart & Parrish (1989) *Mat. Res. Sym. Proc*., **143**, 185-19

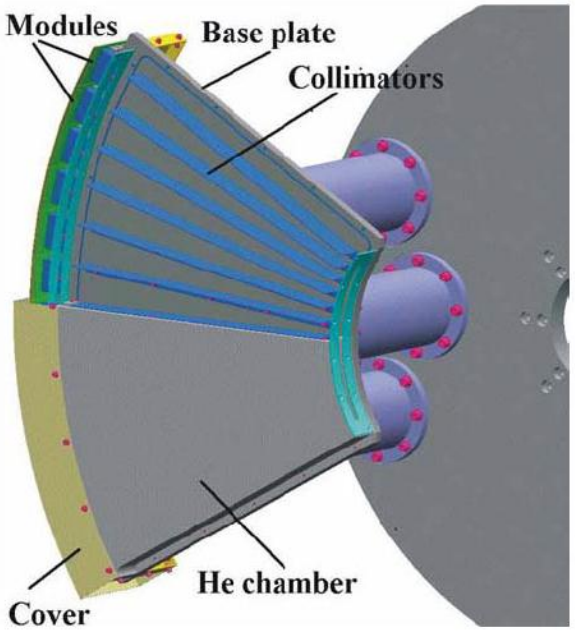
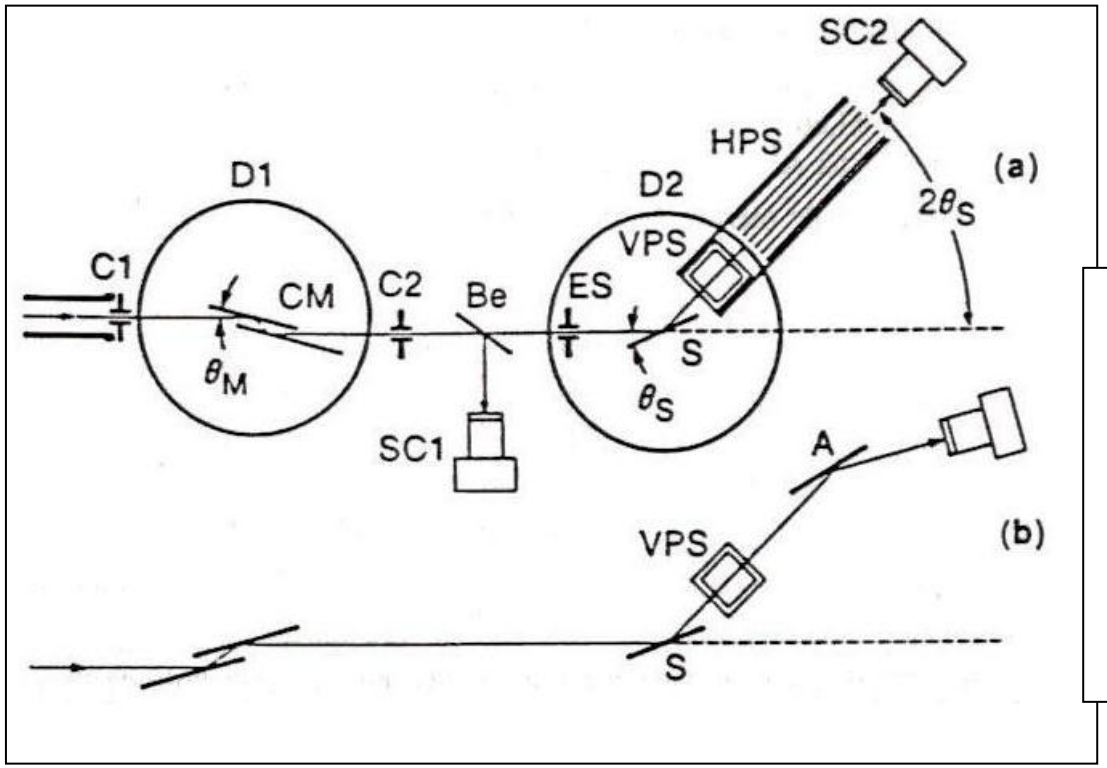


Figure 16: Mythen detector set-up of the SLS Powder Station, from [2]

Parallel beam geometries



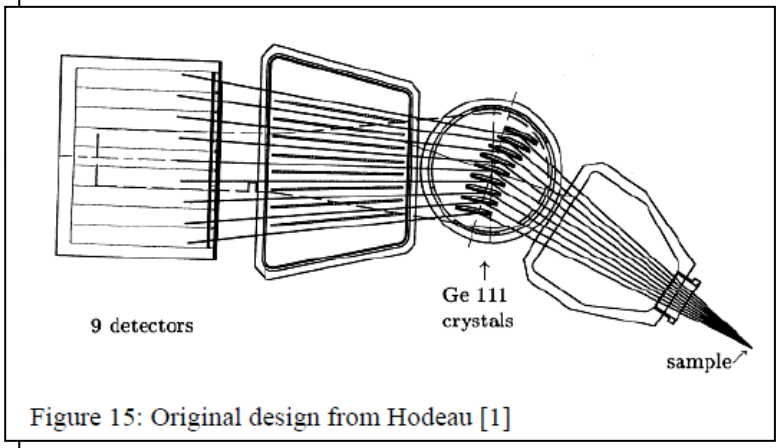


Figure 15: Original design from Hodeau [1]

MCA

Hart & Parrish (1989*) Mat. Res. Sym. Proc*., **143**, 185-19

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Debye-Scherrer geometry & a multistrip 1D detector like Mythen II: Their strengths

1) Enables the fast and simultaneous data collection of the whole 2-theta range (TIME-RESOLVED STUDIES e.g. reaction cinetics)

2) Useful for weak diffracting or unstable samples (STRUCTURAL & QUANTITATIVE STUDIES)

Debye-Scherrer geometry & a multistrip 1D detector like Mythen II: Their particularities

1) The Debye-Scherrer geometry is sensitive to the sample position at the focus. (MCA)

2) The instrumental function largely depends on the capillary diameter. The capillary must be embedded in the beam. (MCA)

3) Useful wavelength length is limited around $\lambda \approx 1$Å. Difficulties with heavy absorbing samples. (MCA)

4) Sensitive to sample fluorescence. (MCA)

Important for structure solution:

Ideal grain size: compromise between sample broadening and number of crystallites)

Use of rotating capillaries to reduce preferred orientation

Pure phase, no impurities (indexing)

All pattern portions should correspond to the same sample state

Minimize the amorphous contents (background modelling)

Coincidence function & Origin-free Patterson sum function

In terms of the clusters

$$CF = \sum_i \frac{1}{n_i} D_i \cdot D_i(\Phi)$$

In terms of the reflections (equidistributed intensities)

$$S_P = \sum_H (I_H - \langle I \rangle) \cdot {E_H}^2(\Phi)$$

P_o' P_c

$I_H \approx \langle I \rangle$ (H with no significant contribution)

$|(I_H - \langle I \rangle)| >> 0$ (H with largest contribution)

Symplifying S_P

$$S_P = \sum_{H} (I_H - \langle I \rangle) \cdot E_H^2(\Phi)$$

$M' \propto P'$

M = modulus function

$E_H(\Phi)$

$G_H(\Phi)$

$\rho = \rho^2$

- δP recycling phasing method
- S_P-*FFT* phasing method

The SP-FFT phasing method (1):

$$S_P = \sum_{\boldsymbol{H}} (I_{\boldsymbol{H}} - \langle I \rangle) \cdot \left\{ \frac{k}{V} \sum_{\boldsymbol{h}} E_{-\boldsymbol{h}} E_{\boldsymbol{h}-\boldsymbol{H}} e^{i(\psi_{\boldsymbol{H}} + \varphi_{-\boldsymbol{h}} + \varphi_{\boldsymbol{h}-\boldsymbol{H}})} \right\}$$

$G_{-\boldsymbol{H}}(\Phi)$

or also

$$S_P(\Phi) = k \cdot \sum_{\mathbf{h}} E_{-\mathbf{h}} e^{i\varphi_{-\mathbf{h}}} \left\{ \frac{1}{V} \sum_{\mathbf{H}} \left(I_{\mathbf{H}} - \langle I \rangle \right) e^{i\psi_{\mathbf{H}}} E_{\mathbf{h}-\mathbf{H}} e^{i\varphi_{\mathbf{h}-\mathbf{H}}} \right\}$$

For solving Φ, *Sp* has to be maximized

The SP-FFT phasing method (2):

$$S_P = k \cdot \sum_{\mathbf{h}} E_{-\mathbf{h}} e^{i\varphi_{-\mathbf{h}}} \left\{ \frac{1}{V} \sum_{\mathbf{H}} \left(I_{\mathbf{H}} - \langle I \rangle \right) e^{i\psi_{\mathbf{H}}} E_{\mathbf{h}-\mathbf{H}} e^{i\varphi_{\mathbf{h}-\mathbf{H}}} \right\}$$

$$\delta = FT^{-1}\left\{ \left(I_{\mathbf{H}} - \langle I \rangle \right) \cdot exp\, i\psi_{\mathbf{H}} \right\}$$

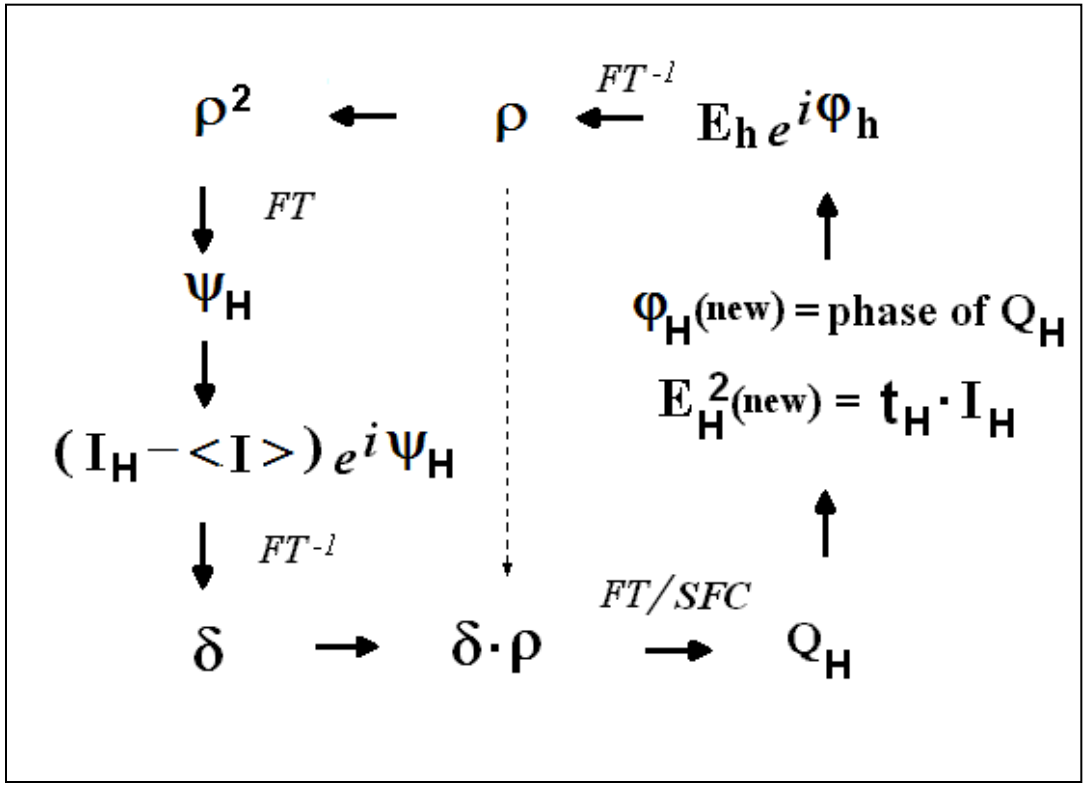
$$\rho = FT^{-1}\left\{ w_{\mathbf{H}} \cdot E_{\mathbf{H}} \cdot \exp i\varphi_{\mathbf{H}} \right\}$$

$$\varphi(\mathbf{h})_{new} = phase\ of\ \mathbf{Q_h}\ \ (= FT \text{ of } \{\rho \cdot \delta\})$$

J. Rius, Acta Cryst (2011) A**67**,63-67

Patterson-function direct methods for structure determination of organic compounds from powder diffraction data. XVI

Iterative cluster-based *SP-FFT* refinement



Initial phase values are combined with experimental and extrapolated amplitudes to give initial ρ values (upper right corner). The experimental cluster intensities are introduced via the (I_H - <*I*>) coefficients. New structure factor estimates are obtained either by Fourier inverting $\delta \cdot \rho$ (*FT*) or directly from the *N* top-ranked Fourier peaks (*SFC*) of $\delta \cdot \rho$. For overlapped reflections, E_H^2 values are updated every cycle while keeping the global intensity of each cluster constant.

Summary of SP-FFT phasing:

A. Up to **NSET** trials starting from random phase values: $\Phi = \{ \varphi_{h1}, \varphi_{h2}, \ldots \}$.

B. Each trial consists of a maximum of **NCICLE** maximization cycles . It stops when RV is less than **RVMIN**.

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Patterson-function direct methods for structure determination of organic compounds from powder diffraction data. XVI

Summary of SP-FFT phasing:

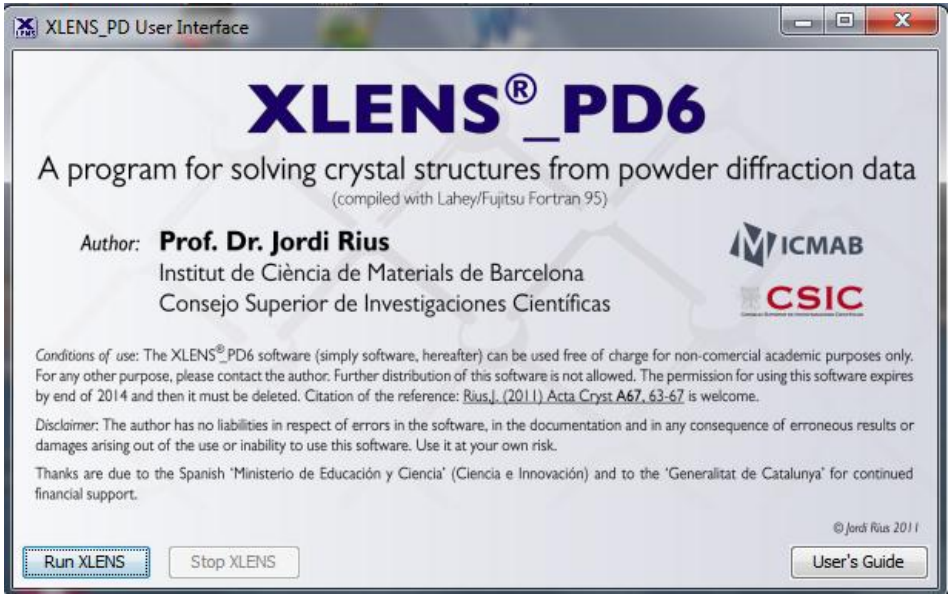
Constraints:

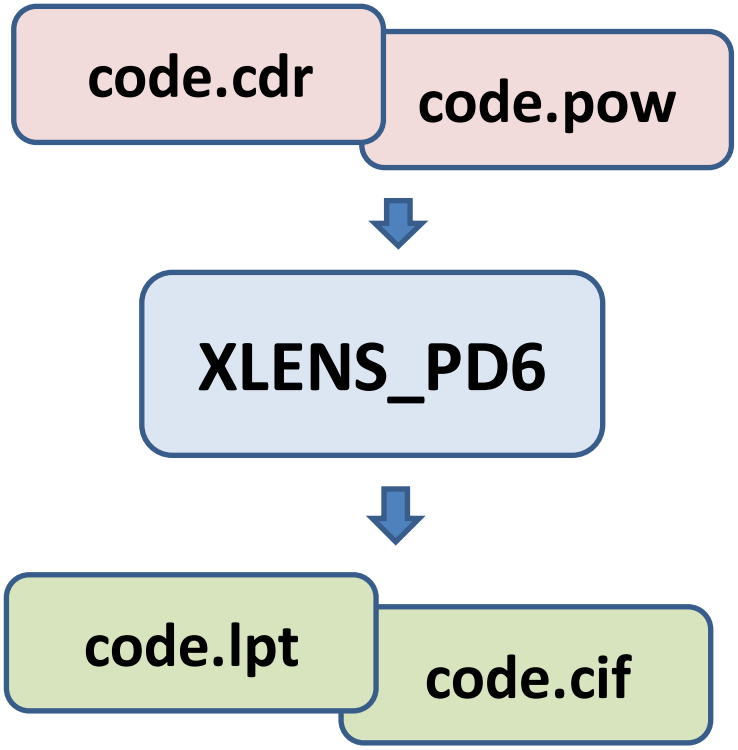
1) Known equidistributed intensities, $\{I_H\}$ (DSFOU)
2) Known E values
3) $\rho \propto \rho^2$, $\psi \approx \varphi$
4) Number of atoms in unit cell (NCELL)
5) Cluster intensity redistributed according to new E estimations

J. Rius, Acta Cryst (2011) A**67**,63-67

Patterson-function direct methods for structure determination of organic compounds from powder diffraction data. XVI

File organisation of XLENS_PD6





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INORGANIC V.S. ORGANIC COMPOUNDS IN POWDER DIFFRACTION

Principal differences:

1) IC tend to have smaller unit cells or higher metrics (less accidental peak overlap);

2) IC possess longer bond distances (less data resolution requirements);

3) IC contain stronger scatterers with higher contrast (smaller effective number of atoms);

4) IC are more stable (consistency of the measured data set over the whole 2-theta range).

Institute of Materials Science of Barcelona (CSIC)

Inorganic Chemistry

Article

pubs.acs.org/IC

High Proton Conductivity in a Flexible, Cross-Linked, Ultramicroporous Magnesium Tetraphosphonate Hybrid Framework

Rosario M. P. Colodrero,[†] Pascual Olivera-Pastor,[†] Enrique R. Losilla,[†] Daniel Hernández-Alonso,[†] Miguel A. G. Aranda,[†] Laura Leon-Reina,[‡] Jordi Rius,[§] Konstantinos D. Demadis,[∥] Bernard Moreau,[⊥] Didier Villemin,[⊥] Miguel Palomino,[#] Fernando Rey,[#] and Aurelio Cabeza*[,†]

Formula: $MgP_4O_{14.5}N_{2.5}C_{13.5}H_{37.5}$

S.G.: *C2/c*, *Z*=4

a= 29.654, *b*=8.622, *c*=9.704*Å*, *β*=95.49º

Volume: 2470 *Å*3

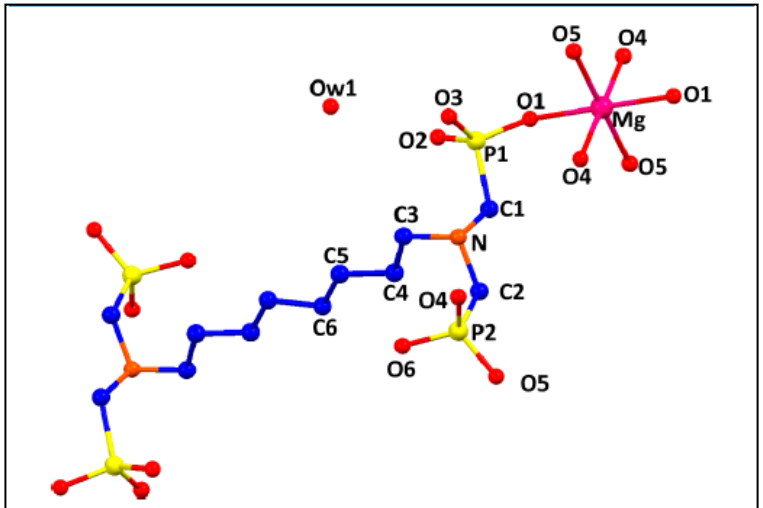
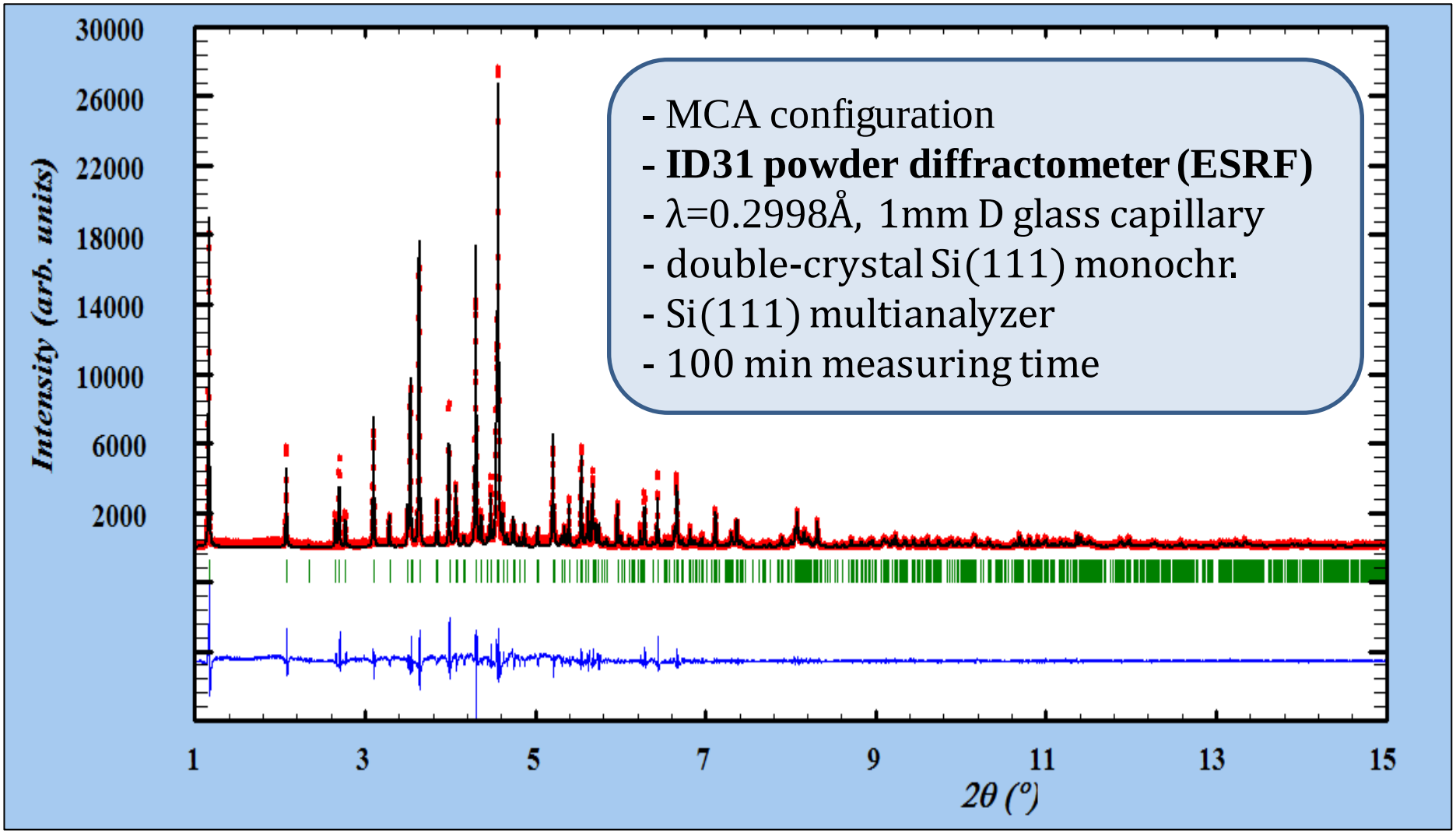


Figure 4. Selected ball–stick view for 1 with atoms labeled showing the octahedral environment of magnesium.

Experimental powder pattern



Portion of file code.pow

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631	-6	4	6	9.0573	4	10	0	13.544	1.27119	0.01727
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635	3	5	5	7.0988	4	8	0	13.613	1.26478	0.01731
636	9	5	4	11.0295	4	8	0	13.621	1.26402	0.01732
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638	-11	1	7	0.0000	4	8	0	13.632	1.26305	0.01732
639	-21	3	1	0.0000	4	8	0	13.636	1.26264	0.01733
640	13	3	5	0.0000	4	8	0	13.643	1.26203	0.01733
641	7	1	7	15.3379	4	8	0	13.645	1.26189	0.01733

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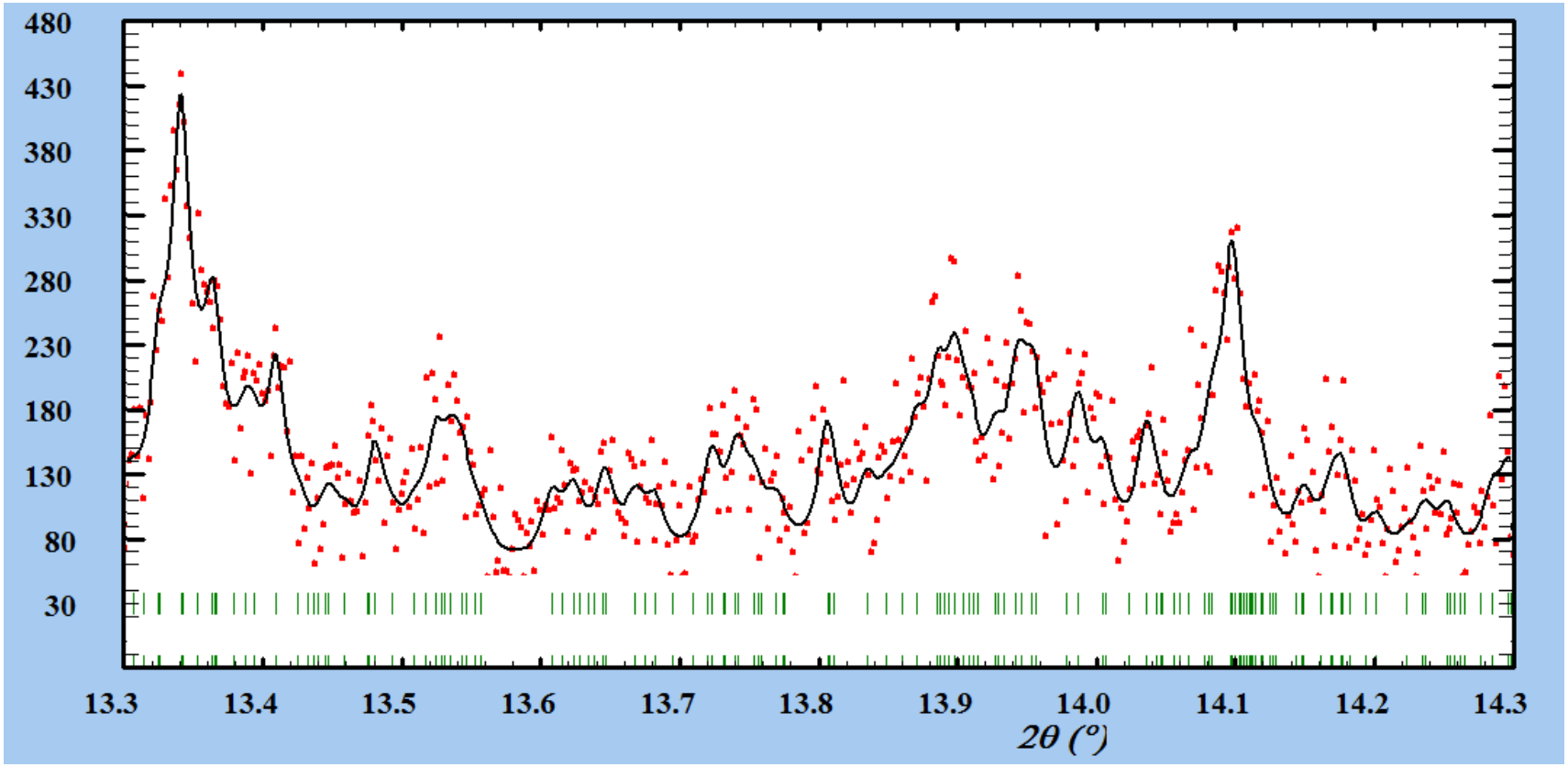
Powder pattern region between 1.3-1.2Å



File code.cdr

MgODTMP, Z=4, C2/c, L=0.2998A
CELL
29.55472 8.61463 9.70192 90.0000 95.4581 90.0000
LATTICE
C-
SYMMETRY
X,Y,Z
X,-Y,1/2+Z
CONTENTS
CL MG O N C H
16 4 52 8 48 128
&CONTROL NSET=3, **RVMIN**=42., **DSFOU**=1.15/

NSET (50)

NCELL

NCICLE

DSFOU

RVMIN

PHASE REFINEMENT DETAILS:

TOTAL NUMBER OF E-VALUES = 861 D-SPACING CUT-OFF = 1.15
NUMBER OF STRONG/WEAK REFLECTIONS = 210/651
NUMBER OF SETS AND CYCLES/SET = 3 40

PHASE REFINEMENT WITH POWDER DATA:

SET	CC	R_ALL	R_HGH	NPKS	NCYCLE	SEED	CENDEV
1	0.892	53.07	41.08	18	41	0.2500000	18.609
2	0.891	48.39	39.35	17	41	0.5000000	31.444
3	**0.895**	**41.76**	**32.66**	**17**	**41**	**0.7500000**	**18.364**

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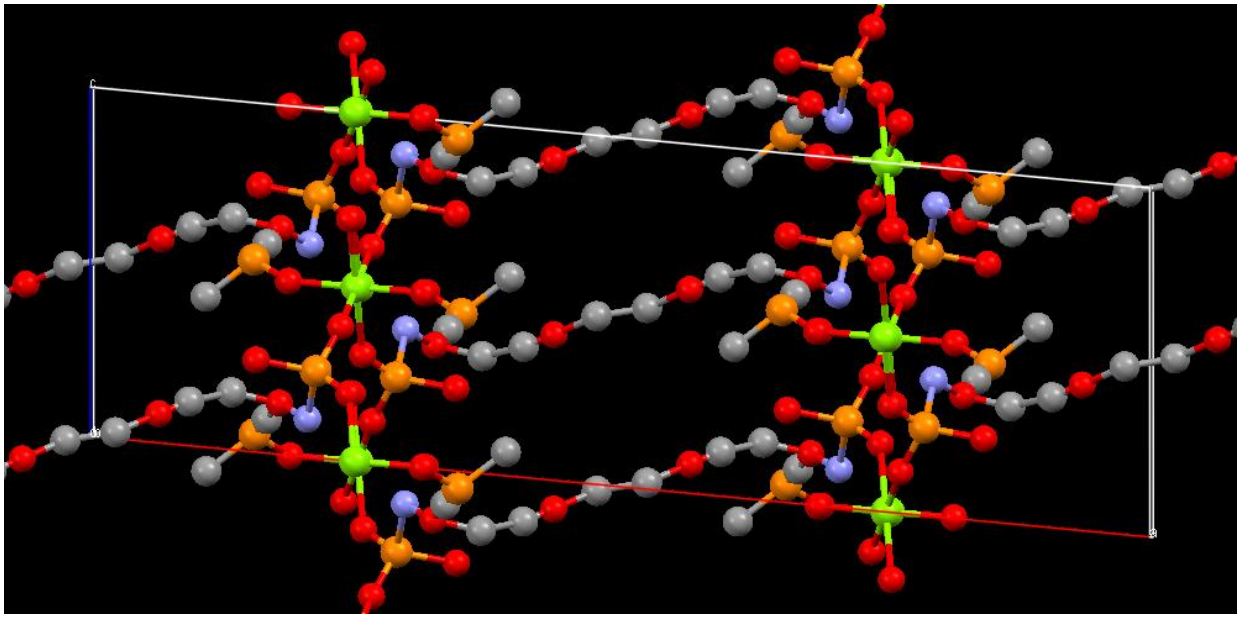
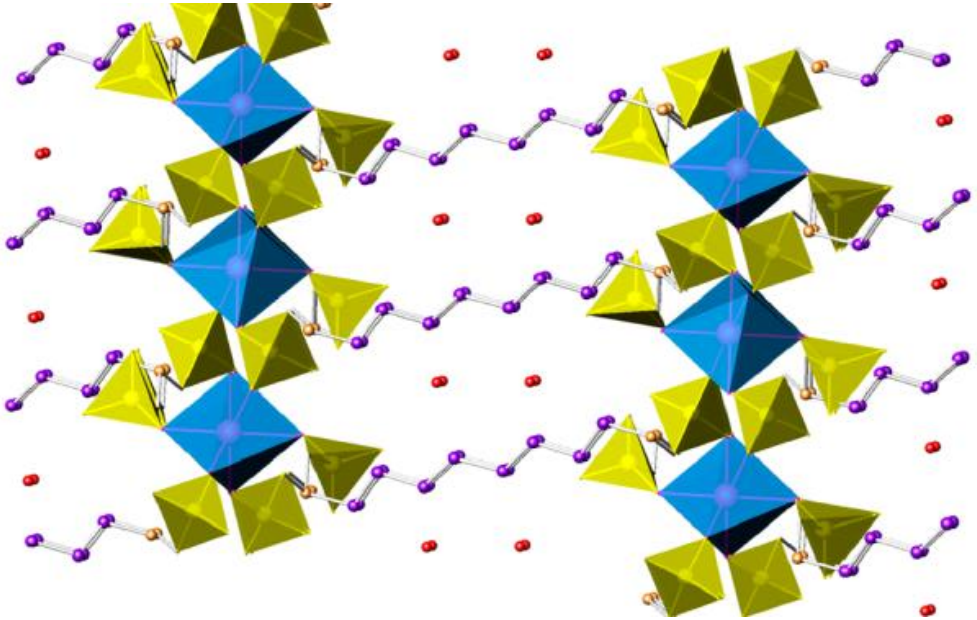
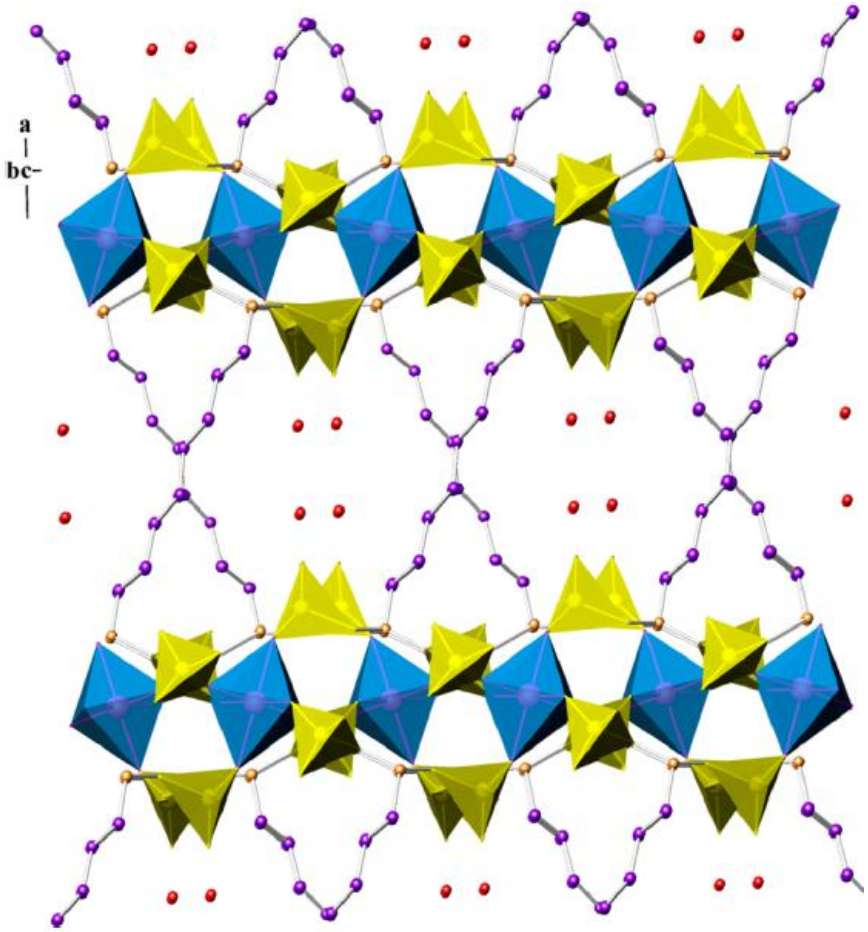
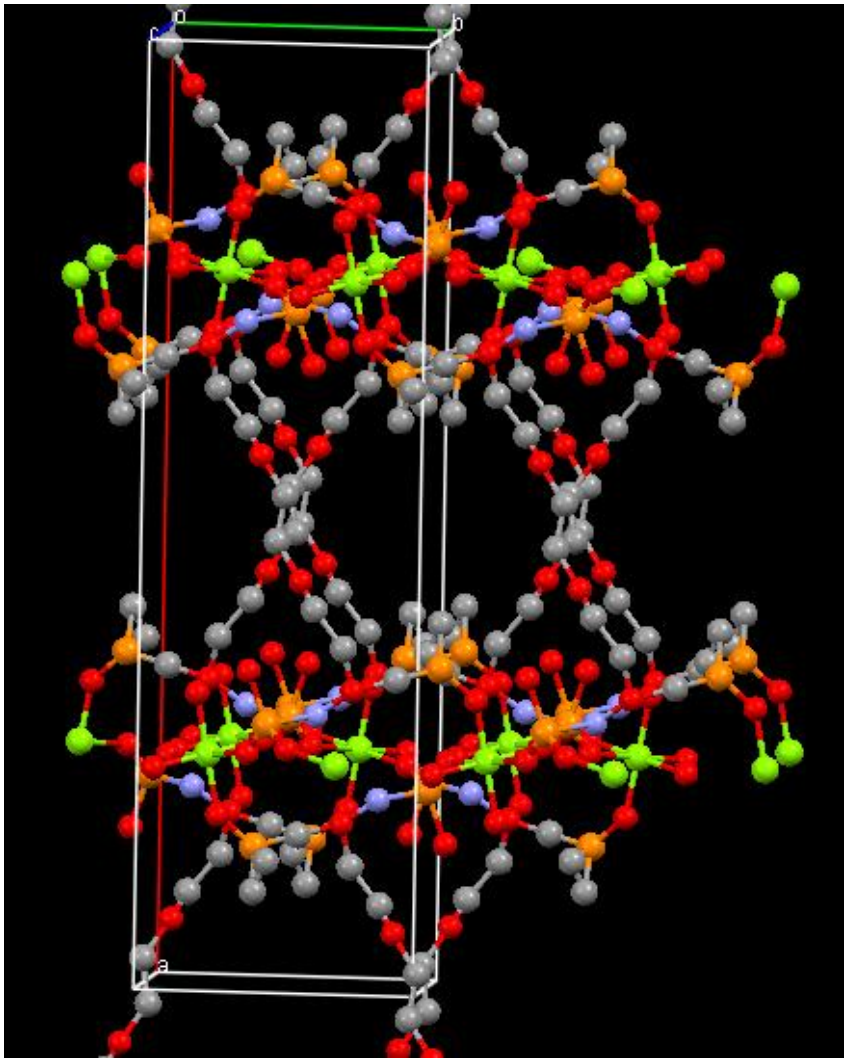





a
bc

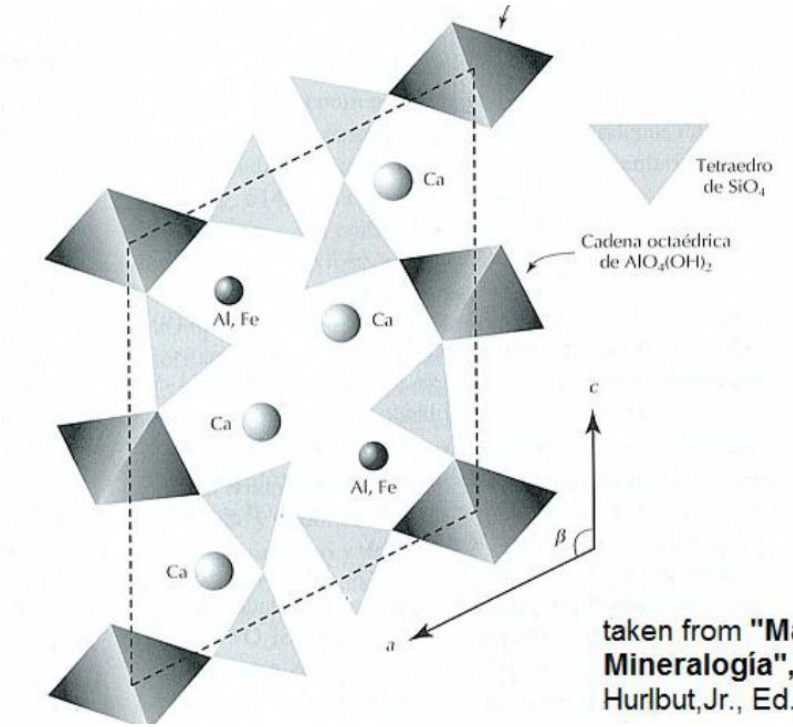


Epidote

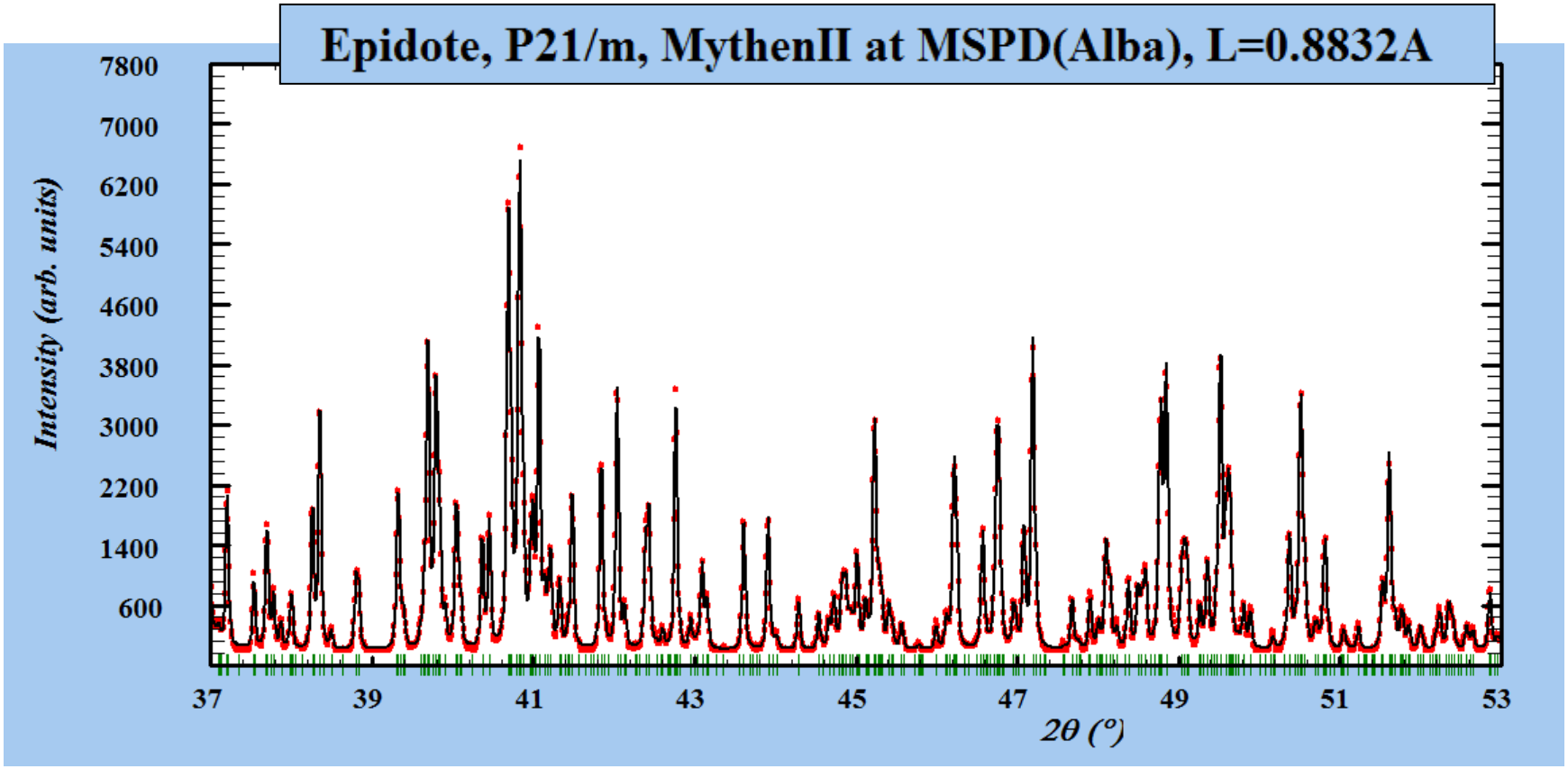
$Ca_2 (Al,Fe)Al_2O(SiO_4)(Si_2O_7)(OH)$

Ito et al. (1954) *Acta Cryst.* **7**, 53

Epidote, P 21/m, Z=4, H2 Ca4 Fe2 Al4 Si6 O26, ALBA
CELL
8.89884 5.63482 10.23989 90.0000 116.3046 90.0000
LATTICE
P-
SYMMETRY
X,Y,Z
X,-Y+1/2,Z
CONTENTS
Ca Si Al O H
4 6 6 26 2
&CONTROL
DSFOU=1.15,nset=50, RVMIN=37.00/



taken from **"Manual de Mineralogía"**, C. Klein & C. Hurlbut,Jr., Ed. Reverté, p.515



d	FWHM
5	0.032
2.5	0.036
1.5	0.038
1.1	0.042
1.0	0.049

Instrumental function: X=0.031, $W^{0.5}$=0.021
Particle size: L(Y=0.001)

Detail of Epidote's POW file

-3	2	8	254.2808	4	2	1	44.816	1.15844	0.04609
-2	4	5	129.9447	4	2	0	44.834	1.15801	0.04610
-7	2	3	252.7049	4	1	1	44.858	1.15743	0.04611
-6	3	2	54.4837	4	3	1	44.909	1.15618	0.04613
-7	2	4	79.7563	4	3	0	44.910	1.15615	0.04613
-5	3	6	148.3612	4	3	0	44.913	1.15609	0.04613
4	2	4	153.5328	4	1	1	44.942	1.15536	0.04615
-6	3	4	231.8338	4	3	1	44.998	1.15401	0.04617
4	4	0	240.7965	4	3	0	45.004	1.15386	0.04617
3	3	4	0.0000	4	3	0	45.025	1.15336	0.04618
2	2	6	191.0977	4	3	1	45.103	1.15146	0.04622
-4	4	4	122.5692	4	3	0	45.123	1.15098	0.04622
1	4	4	94.2585	4	3	0	45.138	1.15062	0.04623
-1	4	5	177.6180	4	4	1	45.204	1.14901	0.04626
2	3	5	46.8021	4	4	0	45.219	1.14867	0.04627
-6	0	8	64.4076	2	4	0	45.219	1.14865	0.04627
7	0	0	496.4752	2	4	0	45.220	1.14863	0.04627
0	0	8	165.6602	2	5	1	45.255	1.14780	0.04628
-3	3	7	131.0370	4	5	0	45.269	1.14745	0.04629
3	4	2	153.8234	4	5	0	45.285	1.14707	0.04629
-4	2	8	158.8148	4	5	0	45.299	1.14674	0.04630
-2	2	8	163.1143	4	5	0	45.311	1.14646	0.04630

EPIDOTE

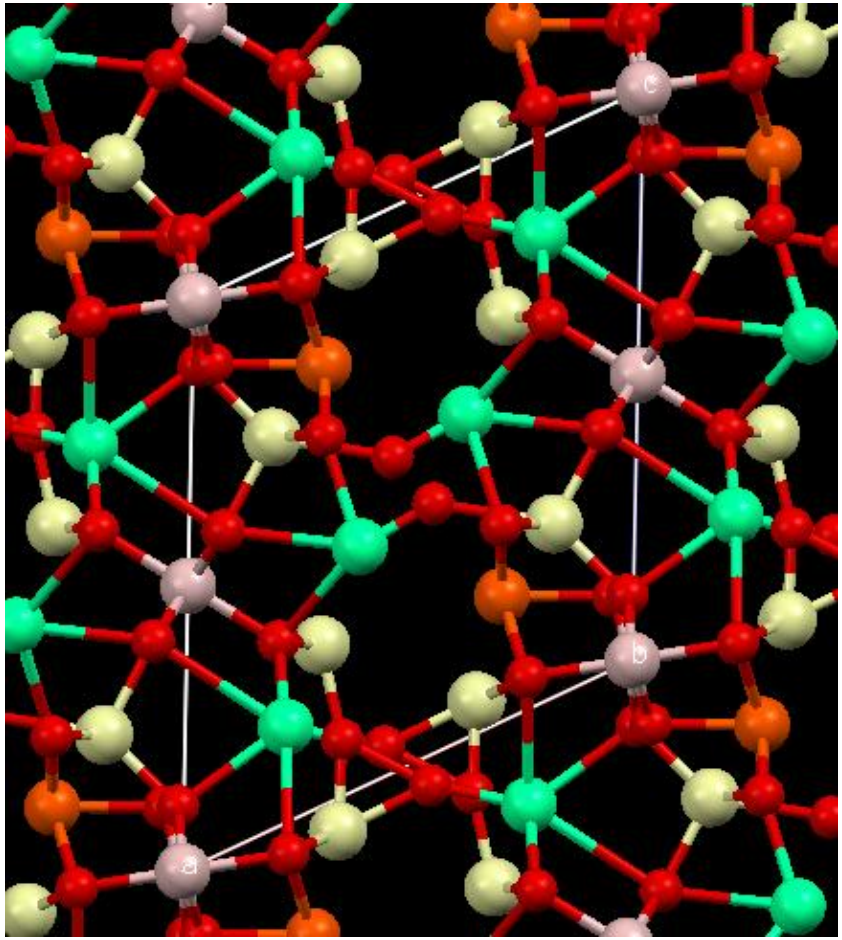
PHASE REFINEMENT DETAILS WITH XLENS_PD6:

TOTAL NUMBER OF E-VALUES = 360 D-SPACING CUT-OFF = 1.15
NUMBER OF STRONG/WEAK REFLECTIONS = 81/279
NUMBER OF SETS AND CYCLES/SET = 50 26
B GLOBAL = 0.4

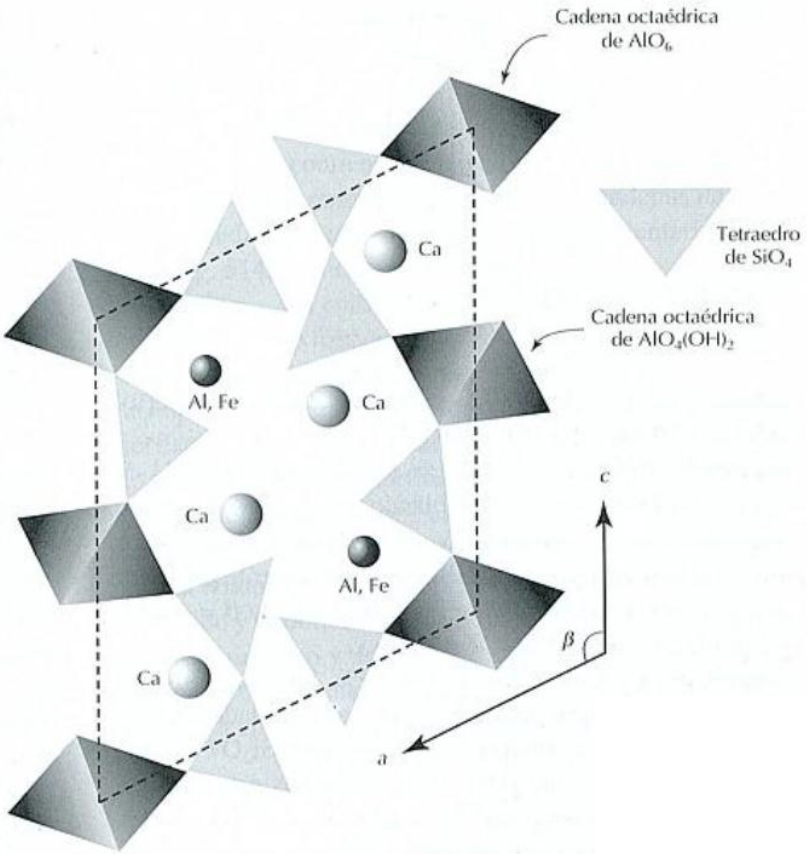
PHASE REFINEMENT WITH POWDER DATA:

SET	CC	R_ALL	R_HGH	NPKS	NCYCLE	CCD	SEED	NEG.-RO
1	0.873	37.20	42.27	18	27	0.882	0.0196078	0.262
2	0.868	37.68	42.80	18	27	0.878	0.0392157	0.257
3	0.917	37.37	42.75	18	22	0.883	0.0588235	0.266
.....								
26	0.869	37.13	41.77	18	27	0.884	0.5098039	0.257
27	0.873	38.36	42.63	18	27	0.881	0.5294118	0.261
28	**0.871**	**36.98**	**41.75**	**18**	**27**	**0.884**	**0.5490196**	**0.263**

CIF view from XLENS_PD6



taken from **"Manual de Mineralogía"**, C. Klein & C. Hurlbut,Jr., Ed. Reverté, p.515



Application of cluster-based DM to organic compounds

DM can cope with molecular compounds, only if intensity data reach **1.1-1.2 Å** resolution. This is the high angle portion of PD patterns where

1. peak overlap is most severe,

2. intensity statistics poorer and

3. any inconsistency in the data set (e.g. the variation in unit cell dimensions or radiation damage during data acquisition) is most critical.

Part of these limitations can be experimentally overcome with a fast read-out solid-state microstrip detector.

Structure of (S)-(+)-Ibuprofen from PD

Relevant information:

Chemical name: 2-[4-(2-methylpropyl)phenyl] propanoic acid

Formula of cyclic hydrogen-bonded dimer: C_{26} H_{16} O_4, Z=2

Space group: $P2_1$

Volume: 1246 Å^3

Data measured with MYTHEN-II by F. Gozzo (SLS)

A.FREER*,J.BUNYAN, N.SHANKLAND AND D. SHEEN. *Acta Cryst.* (1993). C49, 1378-1380

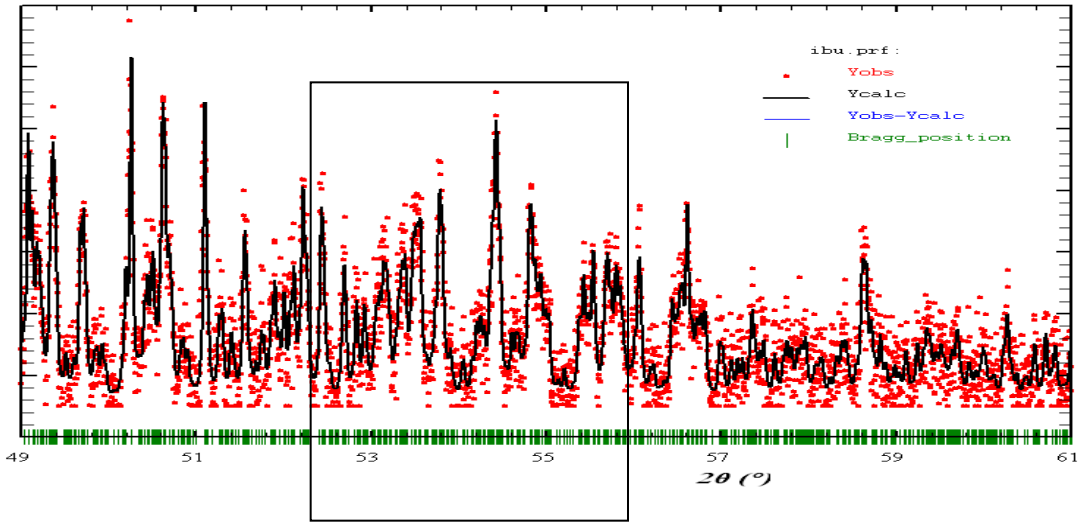
d	FWHM
>10Å	0.014°
2.5	0.023
1.5	0.031
1.1	0.040

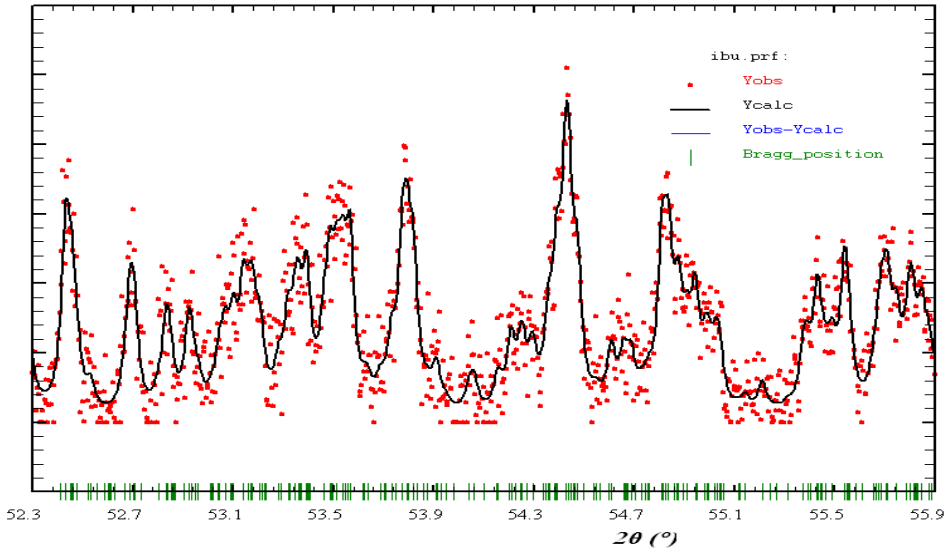
0.5 x FWHM

25 x FWHM

Cauchy prof.

1.00097 Å





Institut de Ciència de Materials de Barcelona (J.Rius)

Summary of DM with XLENS:

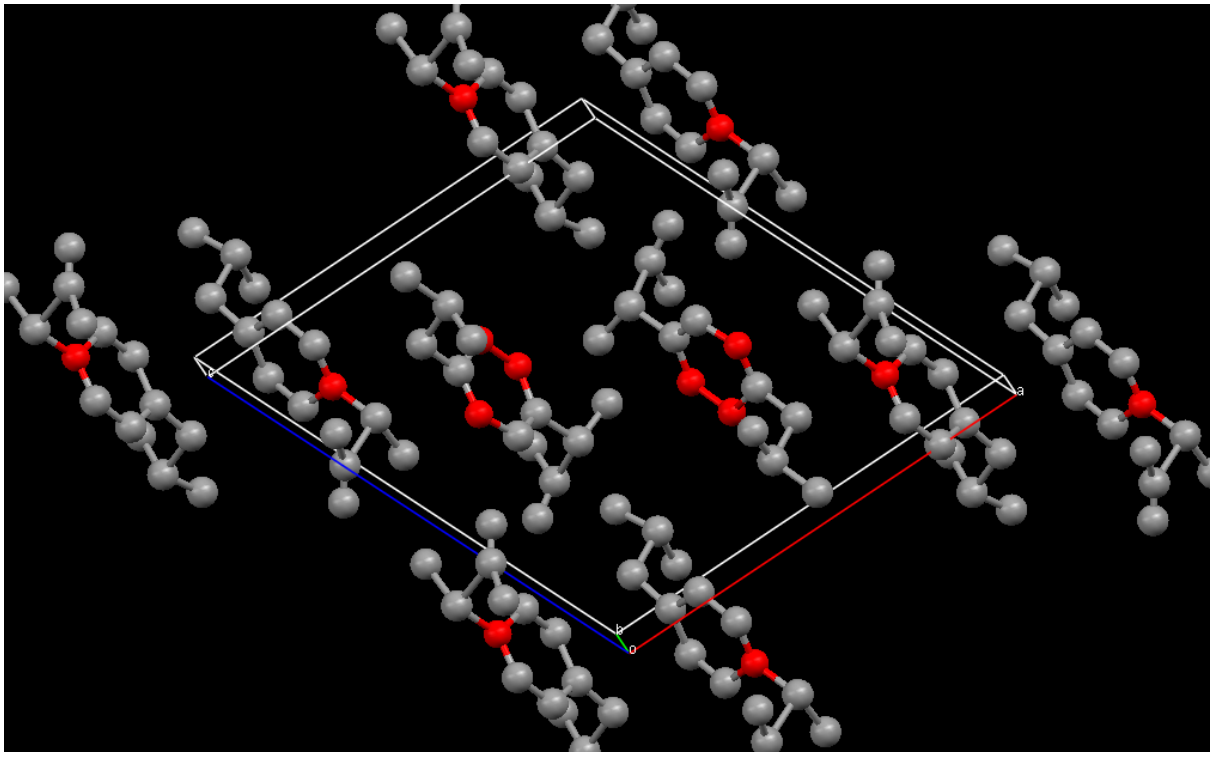
d_{min}= 1.10Å

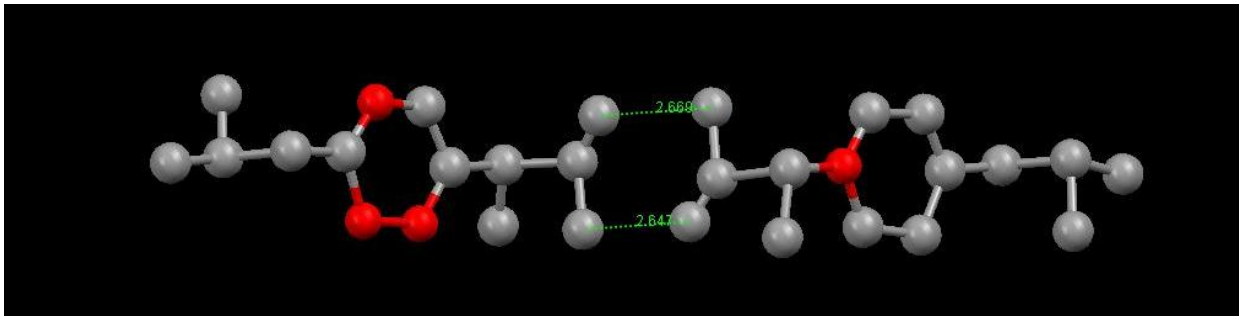
B_{over}= 5.2 Å^2

Success rate= 7 / 25
CC_{true}: 0.92-0.89
CC_{wrong}: <0.85

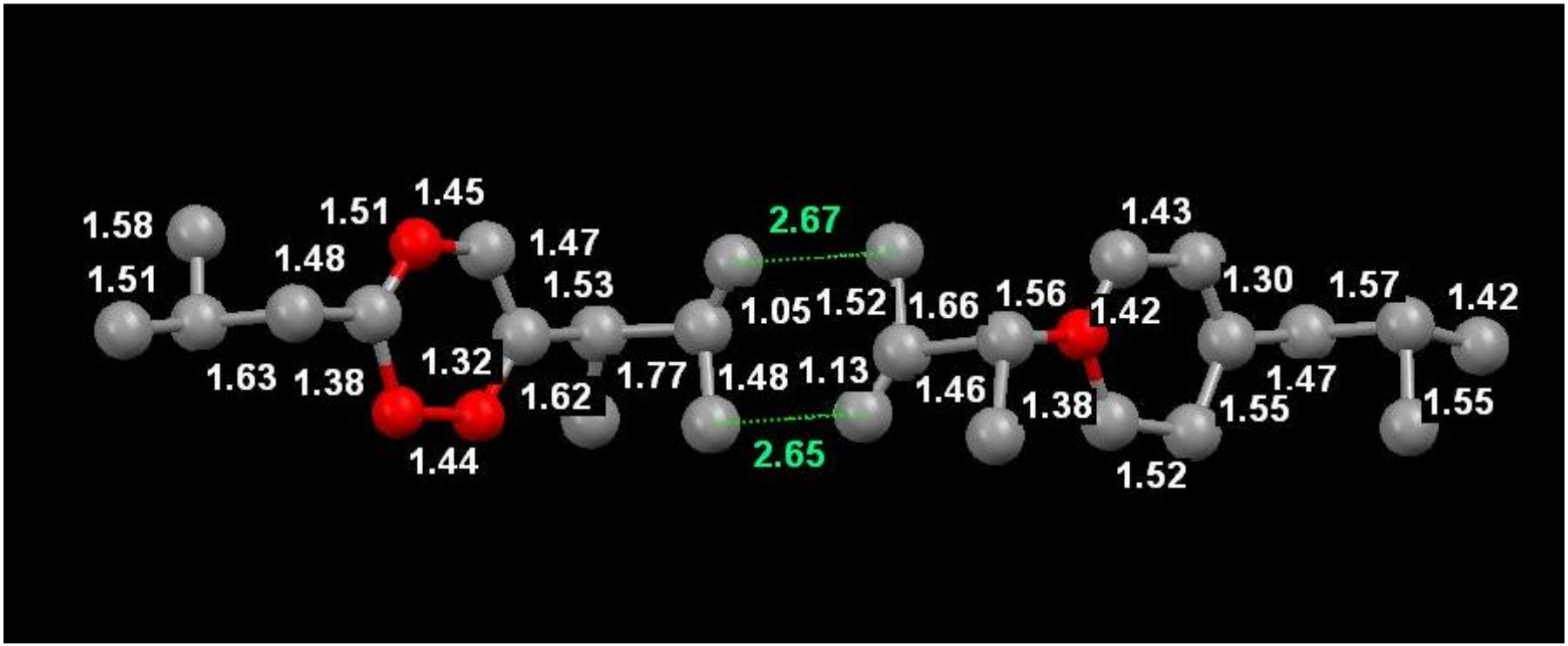
N. strong refl.: 284

N. refl. with E's > 1.15= 795





Intramolecular distances directly from DM from powder data



Rius,J. (2011) *Acta Cryst* A**67**, 63-67

Cluster-based DM applied to organic compounds:

(-) Riboflavin (as-received from Aldrich)

$C_{17}\ H_{20}\ O_6\ N_4$, Z= 4

$P\ 2_1\ 2_1\ 2_1$

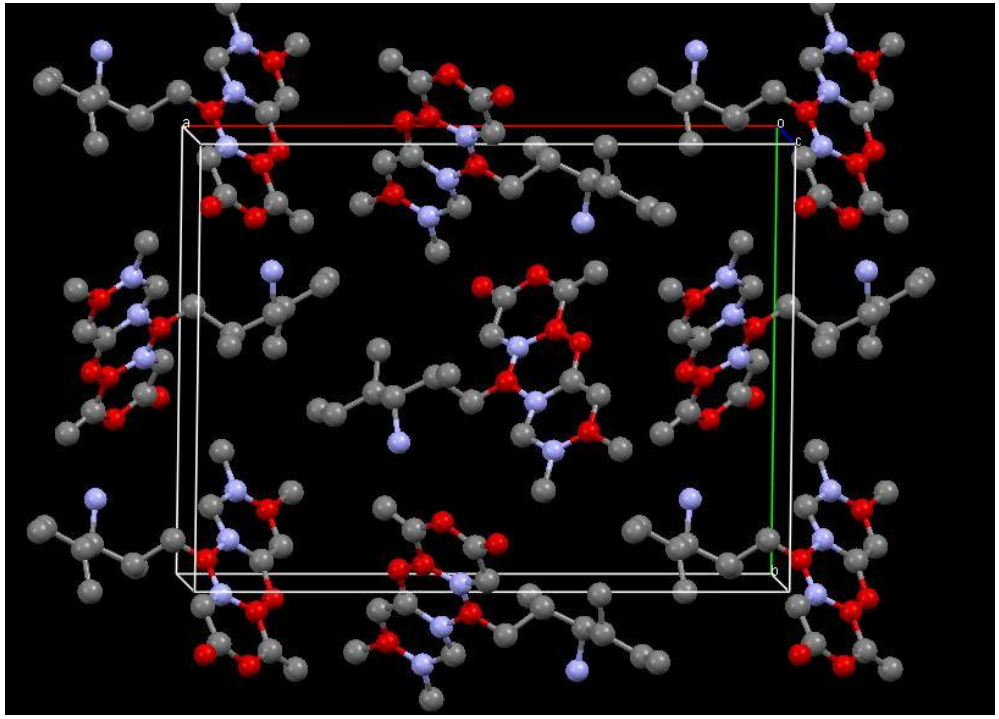
V = 1644Å^3

Data measured with Mythen-II by I. Peral (Alba)

1) Capillar diameter: 0.5mm

2) Partially focused beam

3) Data merged with DMERGE (Vallcorba *et al*., 2013)

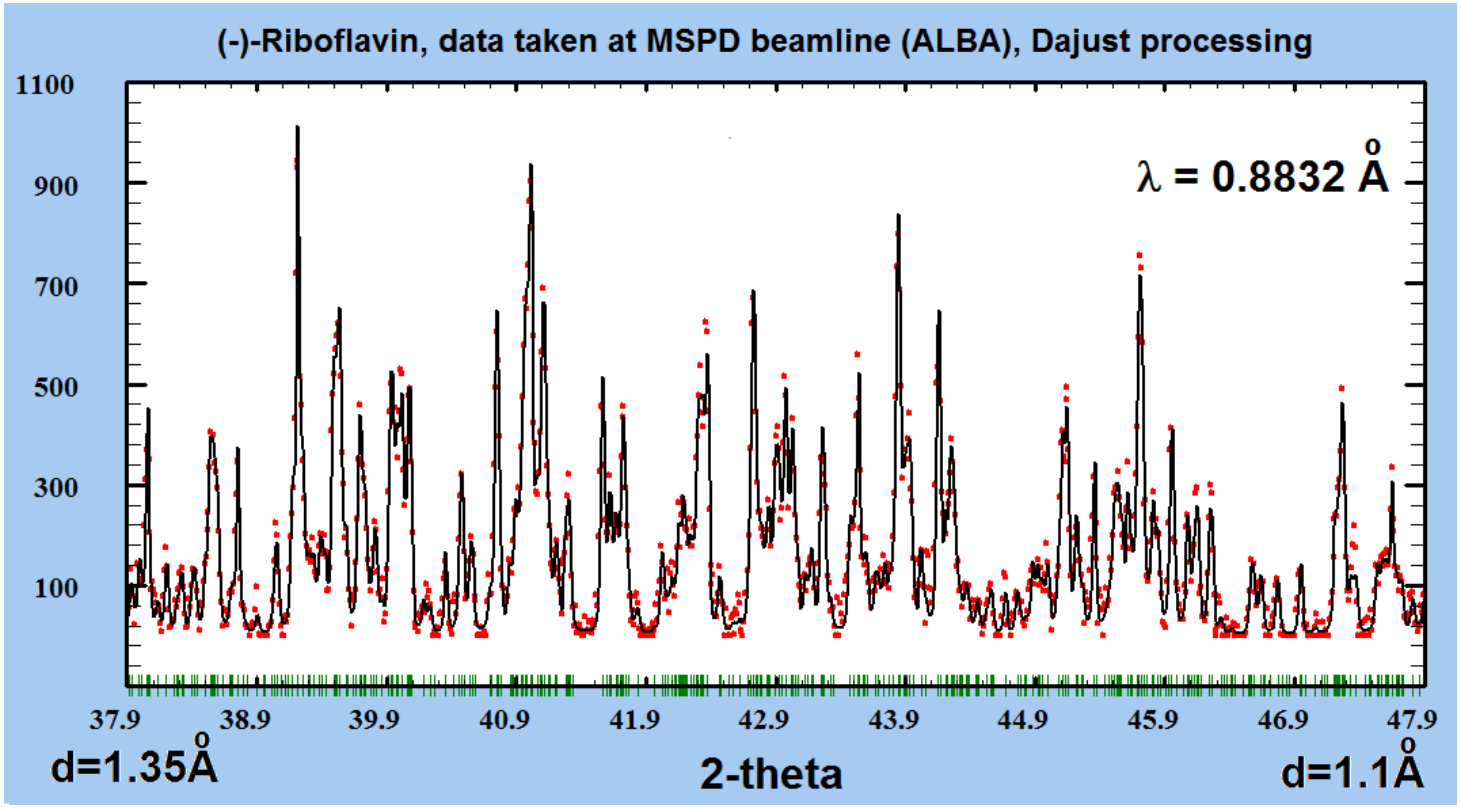


Riboflavin (cont.)

d	FWHM
10	0.030
2.5	0.032
1.5	0.034
1.1	0.037
1.0	0.038

t =0.5

25 x FWHM



Instrumental function: X=0.027, $W^{0.5}$=0.028

Particle size: L(Y=0.002)

Riboflavin (cont.)

10	1	4	0.0000	8	1	1	46.787	1.11222	0.03641
5	10	3	0.0000	8	1	1	46.815	1.11160	0.03641
4	7	4	0.0000	8	1	1	46.845	1.11092	0.03642
14	2	3	89.2807	8	2	1	46.925	1.10914	0.03643
18	2	0	40.9234	4	2	0	46.933	1.10895	0.03644
17	4	1	0.0000	8	1	1	46.964	1.10827	0.03644
8	12	1	27.0505	8	1	1	47.042	1.10652	0.03646
17	5	0	0.0000	4	1	1	47.090	1.10547	0.03646
10	10	2	0.0000	8	1	1	47.112	1.10499	0.03647
16	6	1	0.0000	8	1	1	47.138	1.10441	0.03647
10	2	4	32.9969	8	6	1	47.185	1.10337	0.03648
6	13	0	51.0640	4	6	0	47.188	1.10331	0.03648
8	11	2	86.1605	8	6	0	47.193	1.10319	0.03648
11	11	0	50.5460	4	6	0	47.205	1.10292	0.03649
12	10	1	0.0000	8	6	0	47.214	1.10272	0.03649
8	9	3	102.1831	8	6	0	47.224	1.10252	0.03649
9	12	0	135.4458	4	4	1	47.251	1.10191	0.03650
11	7	3	83.0958	8	4	0	47.260	1.10172	0.03650
12	6	3	67.8728	8	4	0	47.263	1.10165	0.03650
8	5	4	0.0000	8	4	0	47.266	1.10158	0.03650
5	12	2	73.8244	8	1	1	47.314	1.10053	0.03651
9	4	4	84.9742	8	1	1	47.349	1.09977	0.03651
5	13	1	0.0000	8	1	1	47.428	1.09805	0.03653
15	8	0	5.0178	4	2	1	47.454	1.09746	0.03653
18	0	1	16.0265	4	2	0	47.468	1.09716	0.03654
13	8	2	38.0784	8	3	1	47.517	1.09610	0.03654
5	7	4	82.2240	8	3	0	47.519	1.09606	0.03655
14	9	0	0.0000	4	3	0	47.531	1.09581	0.03655

Riboflavin (cont.)

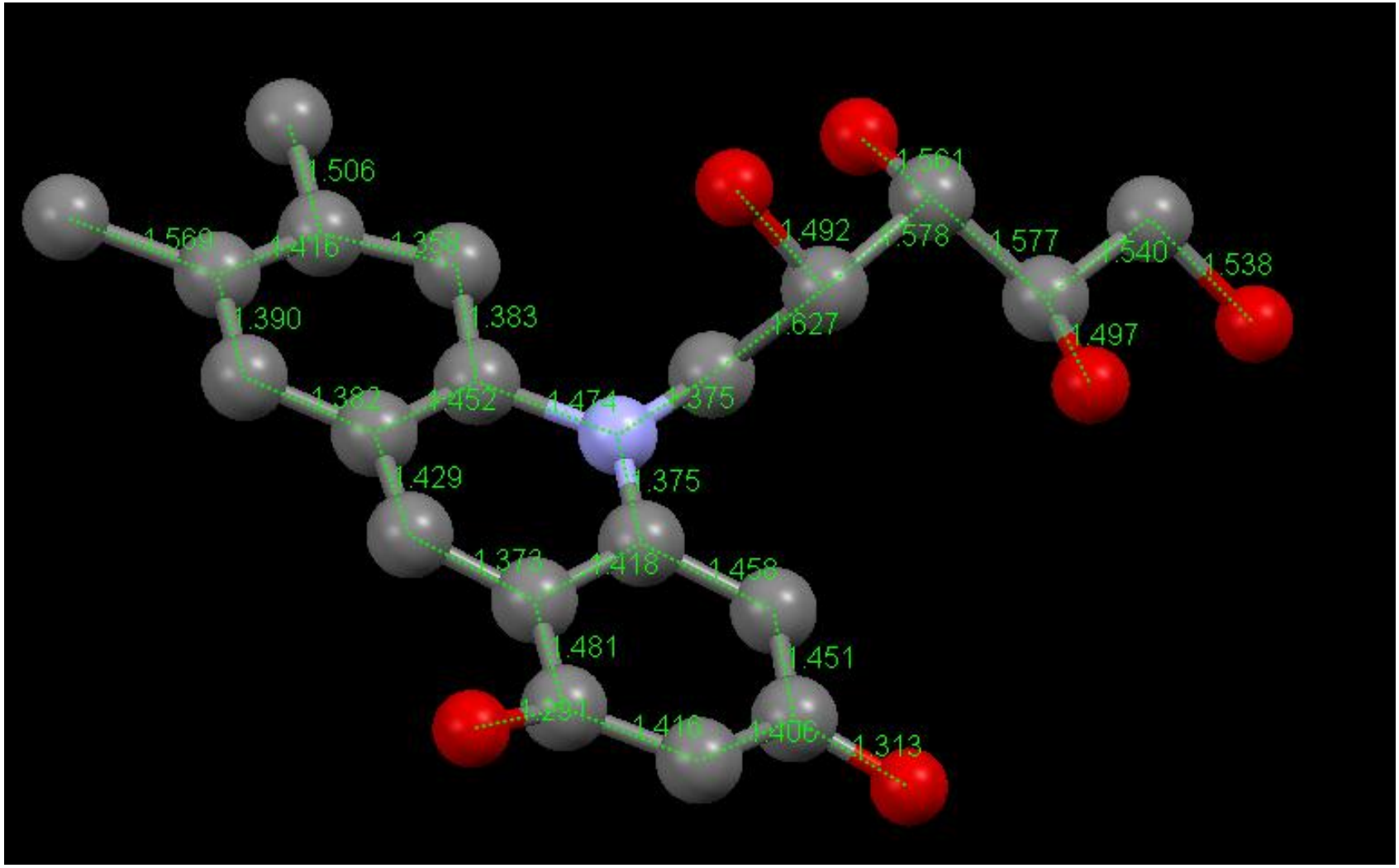
PHASE REFINEMENT DETAILS WITH XLENS_PD6:

TOTAL NUMBER OF E-VALUES = 906 D-SPACING CUT-OFF = 1.05
NUMBER OF STRONG/WEAK REFLECTIONS = 215/691
NUMBER OF SETS AND CYCLES/SET = 20 43
B GLOBAL = 1.94

PHASE REFINEMENT WITH POWDER DATA:

SET	CC	R_ALL	R_HGH	NPKS	NCYCLE	CCD	SEED	NEG.-RO
1	0.865	43.47	38.37	27	44	0.876	0.0476191	0.295
2	0.836	45.47	37.28	28	44	0.866	0.0952381	0.291
3	0.829	45.18	37.17	28	44	0.868	0.1428571	0.295
.....								
12	0.868	40.67	36.56	28	44	0.887	0.5714286	0.298
13	0.881	45.03	39.05	29	38	0.867	0.6190476	0.299
14	**0.919**	**28.62**	**33.27**	**27**	**44**	**0.939**	**0.6666667**	**0.300**

Riboflavin (cont.)



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