

FROM STRUCTURE SOLUTIONS TO DATABASES

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OUTLINE

- ✻ Ab initio structure solution

 - ✻ Indexing

 - ✻ Structure solution

- ✻ Structure databases

 - ✻ Search match

 - ✻ Crystal structure databases

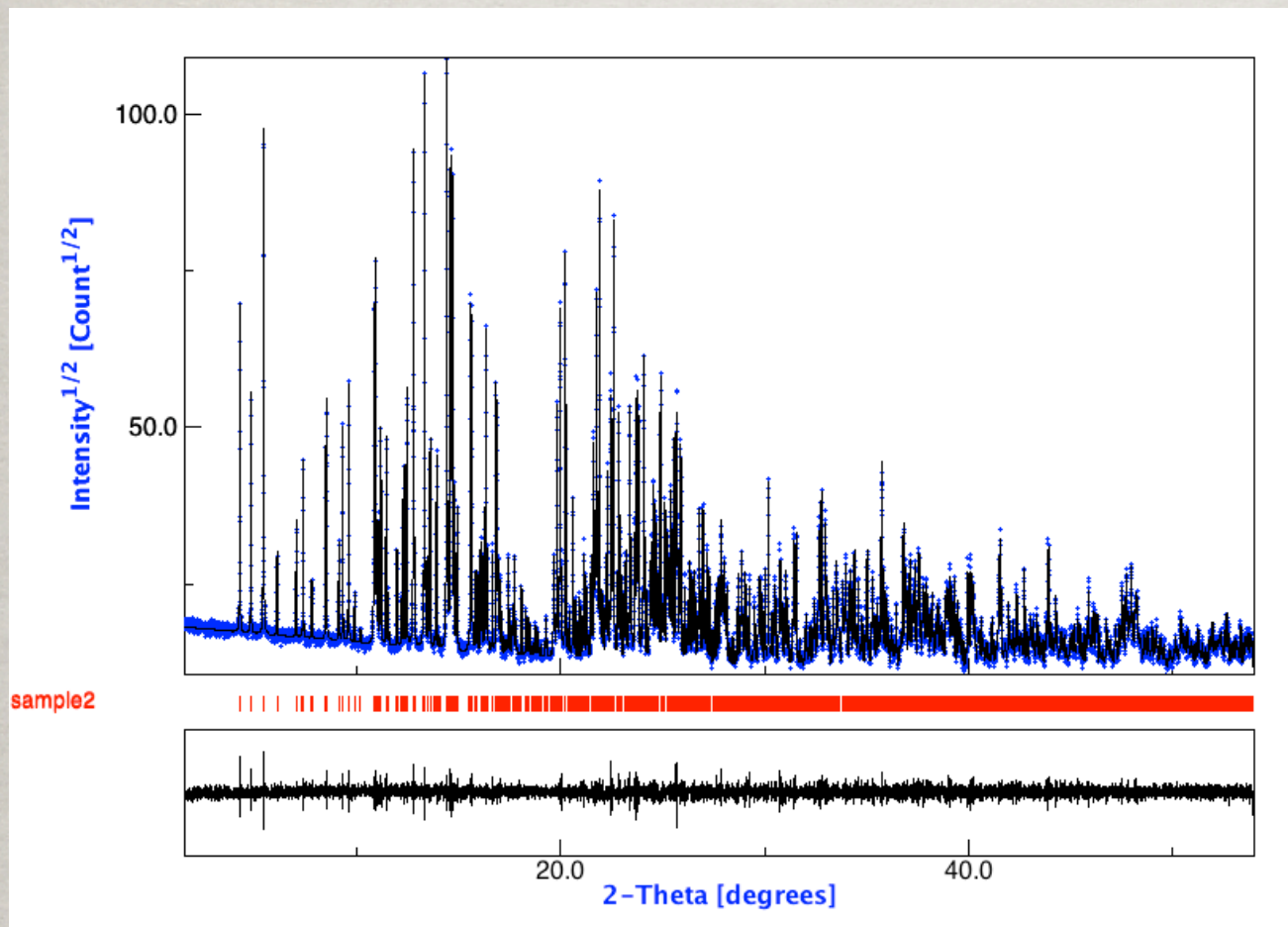
AB-INITIO STRUCTURE SOLUTION

- ✱ Collection of a very good experimental pattern
 - ✱ accurate peak positions
 - ✱ low noise, good intensities, sharp peaks
- ✱ Indexing (to determine symmetry, cell parameters and possible space groups)
- ✱ Atoms location
 - ✱ usual:
 - ✱ structure factors extraction
 - ✱ structure solving by specialized programs
 - ✱ direct:
 - ✱ atoms location from fitting
- ✱ Structure refinement (Rietveld)
- ✱ Structure validation (Platon?)

INDEXING AND SOLUTION IN MAUD

- ✱ Indexing:
 - ✱ smoothing, background subtraction and peak locations
 - ✱ peak list export for classical indexing programs (Dicvol, Treor etc.)
 - ✱ or genetic indexing or full pattern indexing (when no one else program succeed)
- ✱ Dicvol indexing results import and space group sorting (space group identification)
- ✱ Le Bail fitting for cell refinement and structure intensities extraction (with export for structure solution programs)
- ✱ Structure solutions:
 - ✱ Genetic algorithm
 - ✱ Electron density maps

LE BAIL FITTING



- ☼ monoclinic
- ☼ $R_w(\%) = 7.5$
- ☼ $a = 19.87820(5)$
- ☼ $b = 8.19454(2)$
- ☼ $c = 11.24154(3)$
- ☼ $\beta = 106.0656(2)$

SPACE GROUPS SORTING

Select the space group

Symmetry: monoclinic, Space group: P21/c:b2, Not present: 5, extra: 12, totalIndexed: 59

Symmetry: monoclinic, Space group: C2:b3, Not present: 11, extra: 6, totalIndexed: 39

Symmetry: monoclinic, Space group: Cm:b3, Not present: 11, extra: 6, totalIndexed: 39

Symmetry: monoclinic, Space group: C2/m:b3, Not present: 11, extra: 6, totalIndexed: 39

Symmetry: monoclinic, Space group: Pc:b2, Not present: 5, extra: 13, totalIndexed: 59

Symmetry: monoclinic, Space group: P2/c:b2, Not present: 5, extra: 13, totalIndexed: 59

Symmetry: monoclinic, Space group: C2:b1, Not present: 10, extra: 8, totalIndexed: 37

Symmetry: monoclinic, Space group: Cm:b1, Not present: 10, extra: 8, totalIndexed: 37

Symmetry: monoclinic, Space group: C2/m:b1, Not present: 10, extra: 8, totalIndexed: 37

Symmetry: monoclinic, Space group: P21/c:b3, Not present: 6, extra: 13, totalIndexed: 58

Symmetry: monoclinic, Space group: C2:c3, Not present: 14, extra: 5, totalIndexed: 36

Symmetry: monoclinic, Space group: Cm:c3, Not present: 14, extra: 5, totalIndexed: 36

Symmetry: monoclinic, Space group: C2/m:c3, Not present: 14, extra: 5, totalIndexed: 36

Symmetry: monoclinic, Space group: Pc:b3, Not present: 6, extra: 14, totalIndexed: 58

Symmetry: monoclinic, Space group: P2/c:b3, Not present: 6, extra: 14, totalIndexed: 58

Symmetry: monoclinic, Space group: Pc:c2, Not present: 8, extra: 13, totalIndexed: 56

Symmetry: monoclinic, Space group: P2/c:c2, Not present: 8, extra: 13, totalIndexed: 56

Symmetry: monoclinic, Space group: Cc:b1, Not present: 16, extra: 5, totalIndexed: 30

Symmetry: monoclinic, Space group: Cc:-b2, Not present: 16, extra: 5, totalIndexed: 30

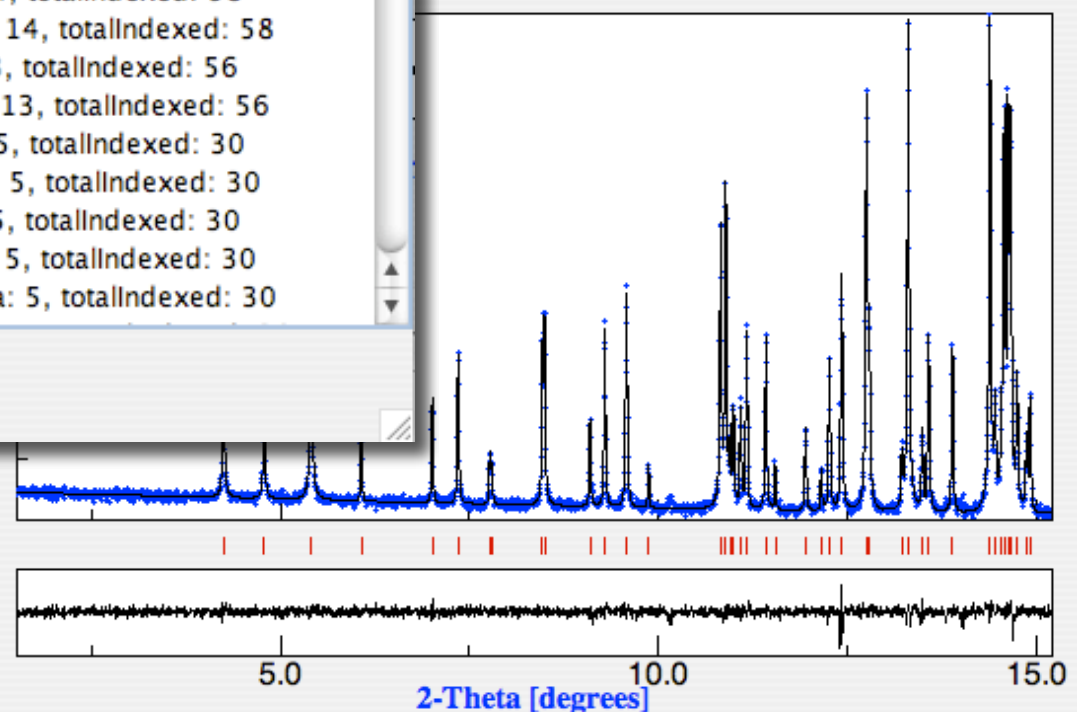
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Symmetry: monoclinic, Space group: Cc:-c3, Not present: 16, extra: 5, totalIndexed: 30

Symmetry: monoclinic, Space group: C2/c:b1, Not present: 16, extra: 5, totalIndexed: 30

Choose Cancel

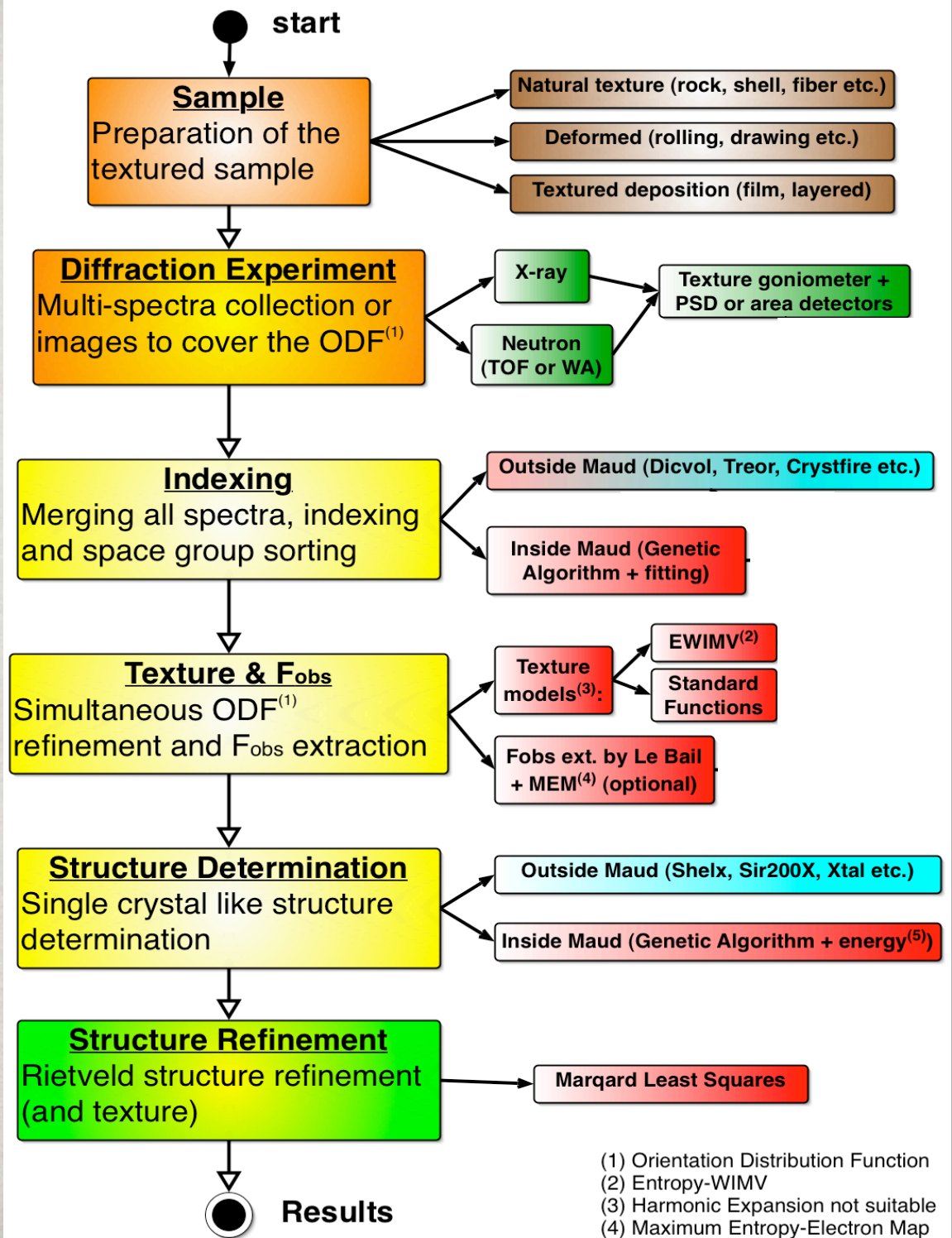
unknown



ATOM LOCATIONS

- ✱ Direct methods (Shelx, Sir2004...):
 - ✱ Some algorithms are used to solve the phase problem
 - ✱ They work best with single crystal structure factors
- ✱ Electron density maps, difference electron density maps and Patterson maps
 - ✱ using Fourier transform or maximum entropy methods
 - ✱ from electron density maps atom should then be located (not easy)
- ✱ Charge flipping algorithm: special technique to obtain electron density maps, as for the previous lot of reflections are required (ex. superflip)
- ✱ Direct atom locations:
 - ✱ genetic algorithms
 - ✱ simulated annealing
 - ✱ Montecarlo methods
- ✱ Special methods were developed for particular cases: envelopes, energy principles....

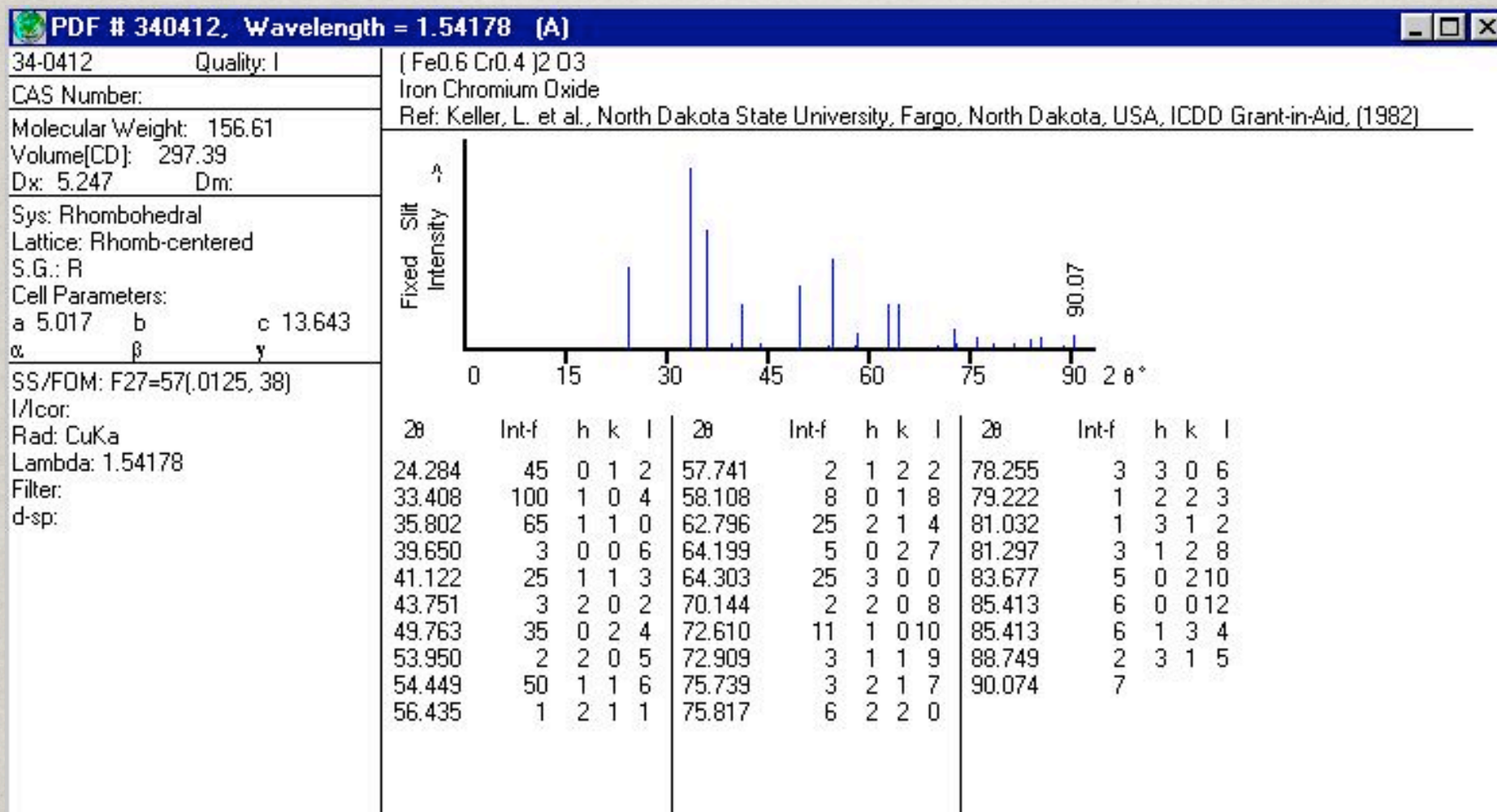
Texture can be used for structure solution (better extraction for overlapped peaks)



STRUCTURE SOLUTION: WHEN?

- ✻ First use the search-match (next)
- ✻ Check for every similar compound
- ✻ Check literature and structural databases
- ✻ Repeat the experiment in case
- ✻ If nothing similar is found then go for the ab initio structure solution
- ✻ After indexing better to do the search again

SEARCH MATCH



OPEN DATA AND CRYSTALLOGRAPHY DATABASES

- ✿ Open access on the Web :

- ✿ PDB (proteins)

- ✿ NDB (nucleic acids)

- ✿ AMCSD (minerals)

- ✿ COD (small/medium crystal structures, based on donations)

- ✿ Toll databases :

- ✿ CSD (organic, organometallic)

- ✿ ICSD (inorganic, minerals)

- ✿ CRYSTMET (metals, intermetallics)

- ✿ ICDD (powder patterns)

OTHER STRUCTURE DATABASES

- ✻ For all metallic/intermetallic phases: The Pearson books (in paper format)
- ✻ For some simple structures: Wyckoff, Crystal Structures (book)
- ✻ Literature: all IUCr journals like Acta Cryst., J. Appl. Cryst. etc.

SEARCHING THE COD

<http://www.crystallography.net/>

Netscape

File Edit View Go Bookmarks Tools Window Help

http://www.crystallography.net/ Search

Search

(Output limited to 300 entries maximum, see the [hints and tips](#))

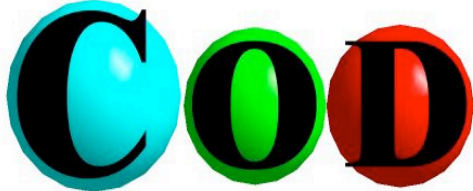
text (1 or 2 words)		
1 to 8 elements	P	O
NOT these elements		
volume min and max		
strict number of elements		
Reset	Send	

a (min - max)		
b		
c		
alpha	90	90
beta	90	90
gamma	90	90
Reset	Send	

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Document: Done (0.11 secs)

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http://www.crystallography.net/ Search

Result : There are 300 entries in the selection

Searching elements including P O

CIF file	Formula : - C 5.00 H 17.00 O 8.00 N 2.00 P 2.00 Al 1.00 - Comments : S. PHAN THANH J. MARROT J. RENAUDIN V. MAISONNEUVE [H~3~N(CH~2~)~5~NH~3~].AlP~2~O~8~H, a One-Dimensional Aluminophosphate Space group : P2~1~n Cell volume: 1319.90 Cell parameters: 7.8783 ; 10.4689 ; 16.0680 ; 90.000 ; 95.147 ; 90.000
CIF file	Formula : - O 24.00 P 6.00 Cu 3.00 Fe 4.00 - Comments : unknown Space group : P -1 Cell volume: 406.34 Cell parameters: 7.9296 ; 9.3275 ; 6.2555 ; 107.160 ; 101.011 ; 105.830
CIF file	Formula : - O 8.00 P 2.00 Cu 3.00 - Comments : unknown Space group : P -1 Cell volume: 140.70 Cell parameters: 4.8564 ; 5.2907 ; 6.1872 ; 72.363 ; 86.996 ; 68.547
CIF file	Formula : - O 7.00 P 2.00 Cu 2.00 - Comments : unknown Space group : C 2/c Cell volume: 480.48 Cell parameters: 6.8513 ; 8.1178 ; 9.1535 ; 90.000 ; 109.300 ; 90.000
CIF file	Formula : - H 5.00 O 7.00 P 1.00 V 1.00 - Comments : Le Bail, A Ferey, G Amoros, P Beltran=Porter, D Villeneuve, G Crystal Structure of β -V O (H P O~4~) (H~2~O)2 Solved from X-Ray Powder Diffraction Journal of Solid State Chemistry 79 (1989) 169 176

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DATABASES IN MAUD

- ✻ Maud has a small structure database that can be expanded by the user
- ✻ Phases, Instruments can be stored in the databases in CIF format
- ✻ The CIF format is the Crystallographic Information Format developed by the IUCr
- ✻ Maud can import from any file containing phases or instruments in CIF format
- ✻ A special function can submit a solved or refined structure directly to the COD database online