From structure solutions to databases

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Outline

- Ab initio structure solution
  - Indexing
  - Structure solution
- Structure databases
  - Search match
  - Crystal structure databases

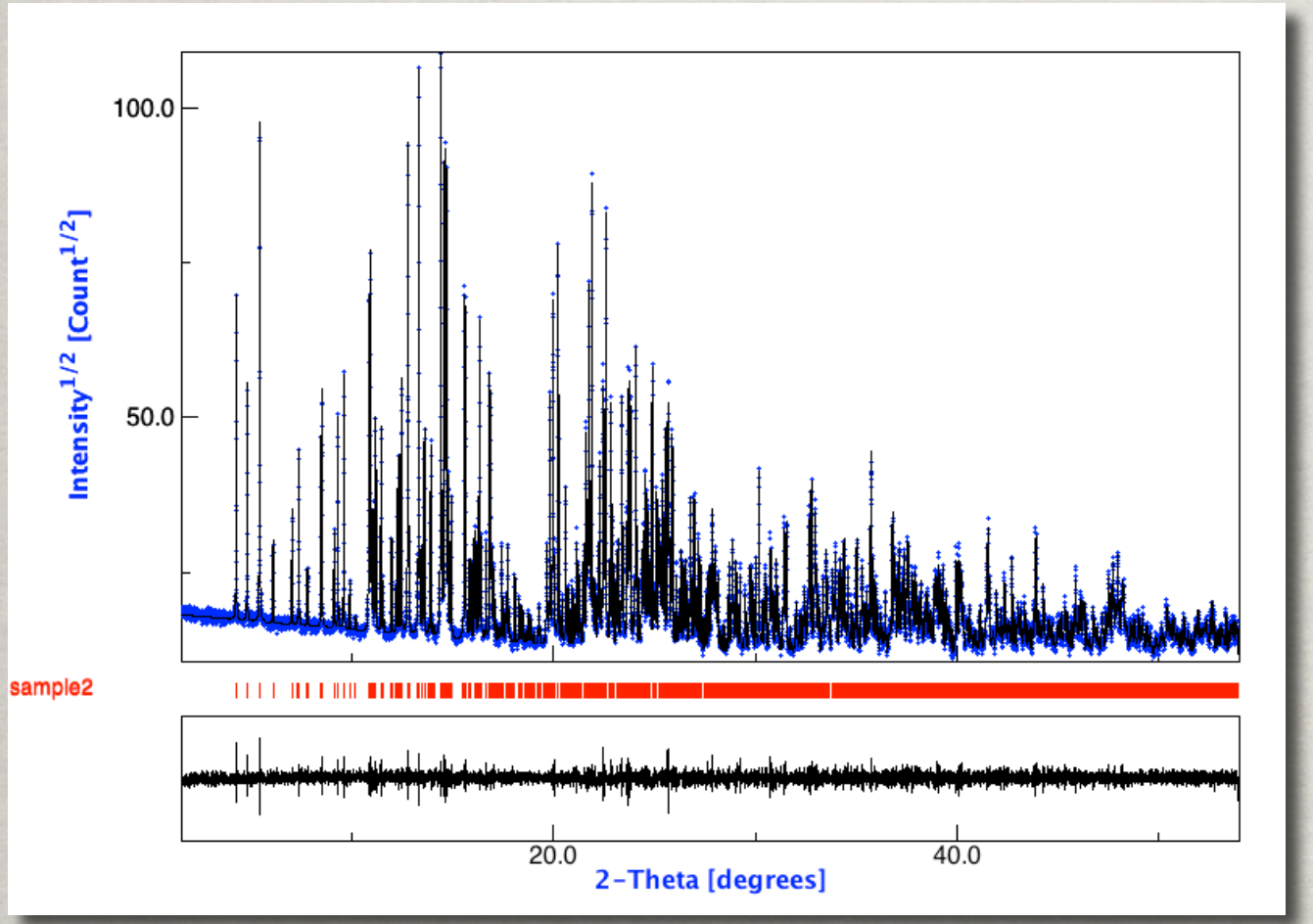
AB-INITIO STRUCTURE SOLUTION

- 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

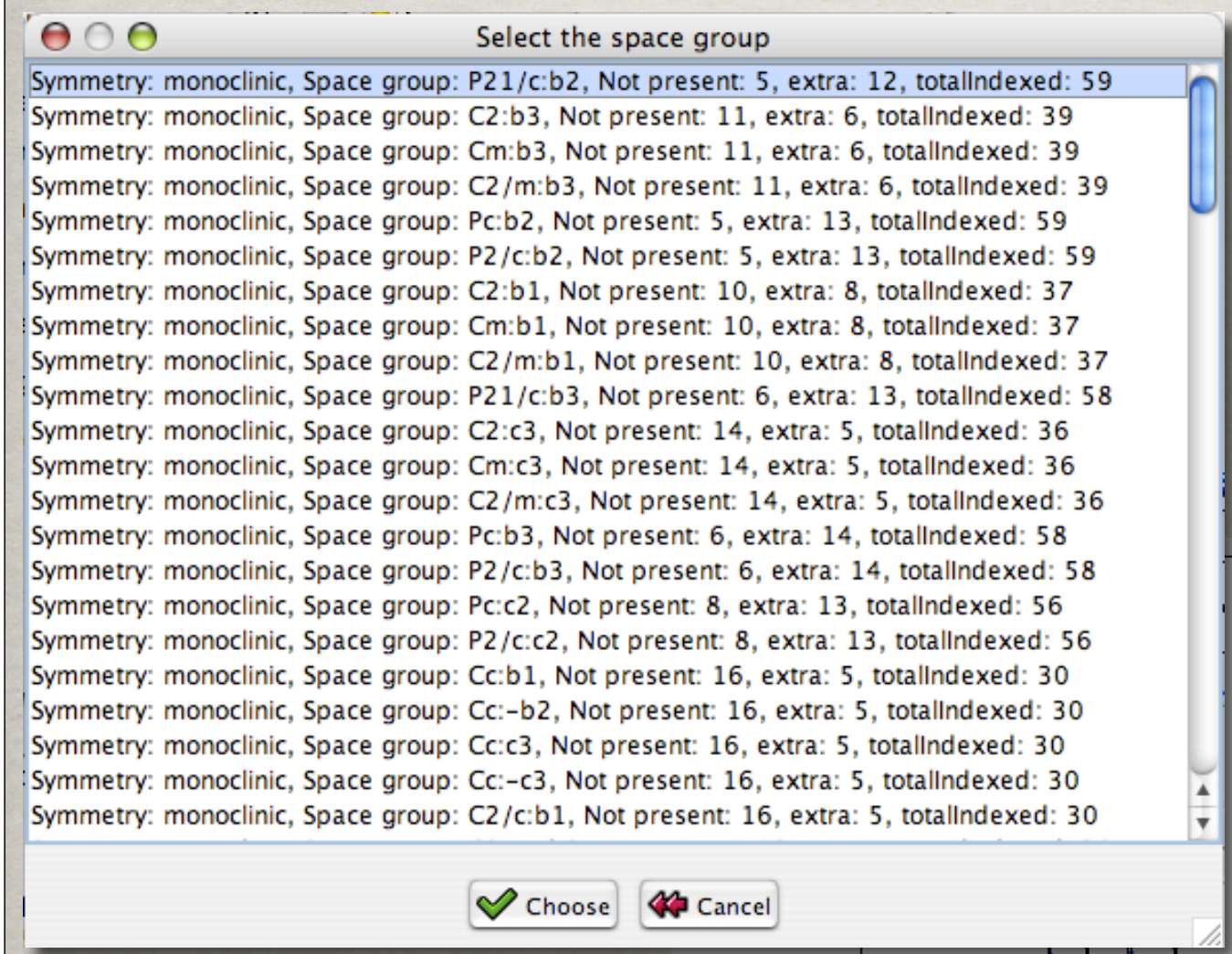
- Indexing:
  - smoothing, background subtraction and peak locations
  - peak list export for classical indexing programs (Dicvol, Treor etc.)
  - or genetic indexing or full pattern indexing (when no one else program succeed)
- Dicvol indexing results import and space group sorting (space group identification)
- 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
- Rw(%)=7.5
- a=19.87820(5)
- b=8.19454(2)
- c=11.24154(3)
- β=106.0656(2)

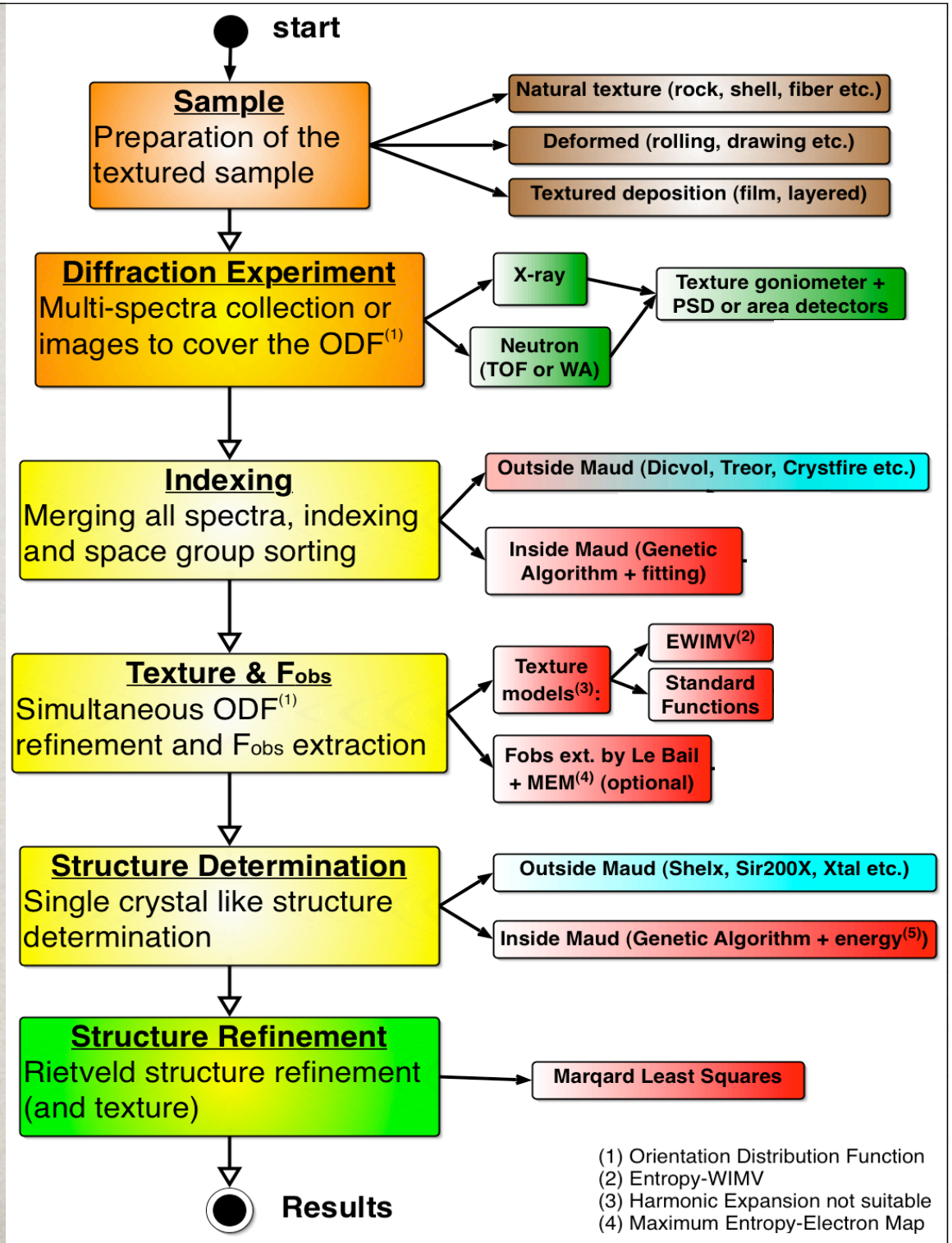
Space groups sorting



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

PDF # 340412, Wavelength = 1.54178 (A)

34-0412 Quality: I

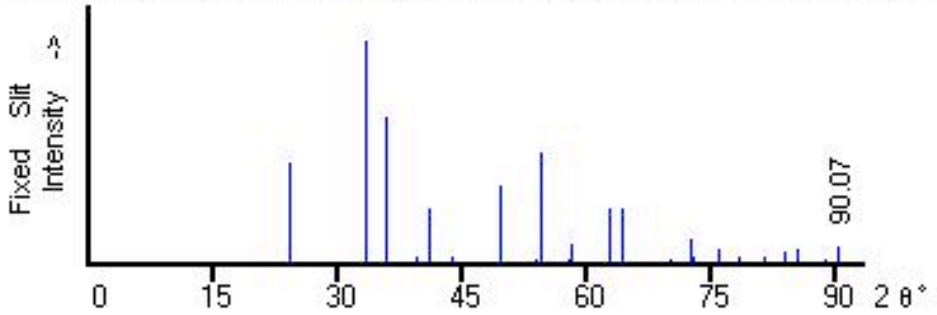
CAS Number:

Molecular Weight: 156.61
Volume[CD]: 297.39
Dx: 5.247 Dm:

Sys: Rhombohedral
Lattice: Rhomb-centered
S.G.: R
Cell Parameters:
a 5.017 b c 13.643
α β γ

SS/FOM: F27=57(.0125, 38)
I/Icor:
Rad: CuKa
Lambda: 1.54178
Filter:
d-sp:

(Fe0.6 Cr0.4)2 O3
Iron Chromium Oxide
Ref: Keller, L. et al., North Dakota State University, Fargo, North Dakota, USA, ICDD Grant-in-Aid, (1982)



2θ	Int-f	h	k	l	2θ	Int-f	h	k	l	2θ	Int-f	h	k	l
24.284	45	0	1	2	57.741	2	1	2	2	78.255	3	3	0	6
33.408	100	1	0	4	58.108	8	0	1	8	79.222	1	2	2	3
35.802	65	1	1	0	62.796	25	2	1	4	81.032	1	3	1	2
39.650	3	0	0	6	64.199	5	0	2	7	81.297	3	1	2	8
41.122	25	1	1	3	64.303	25	3	0	0	83.677	5	0	2	10
43.751	3	2	0	2	70.144	2	2	0	8	85.413	6	0	0	12
49.763	35	0	2	4	72.610	11	1	0	10	85.413	6	1	3	4
53.950	2	2	0	5	72.909	3	1	1	9	88.749	2	3	1	5
54.449	50	1	1	6	75.739	3	2	1	7	90.074	7			
56.435	1	2	1	1	75.817	6	2	2	0					

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)

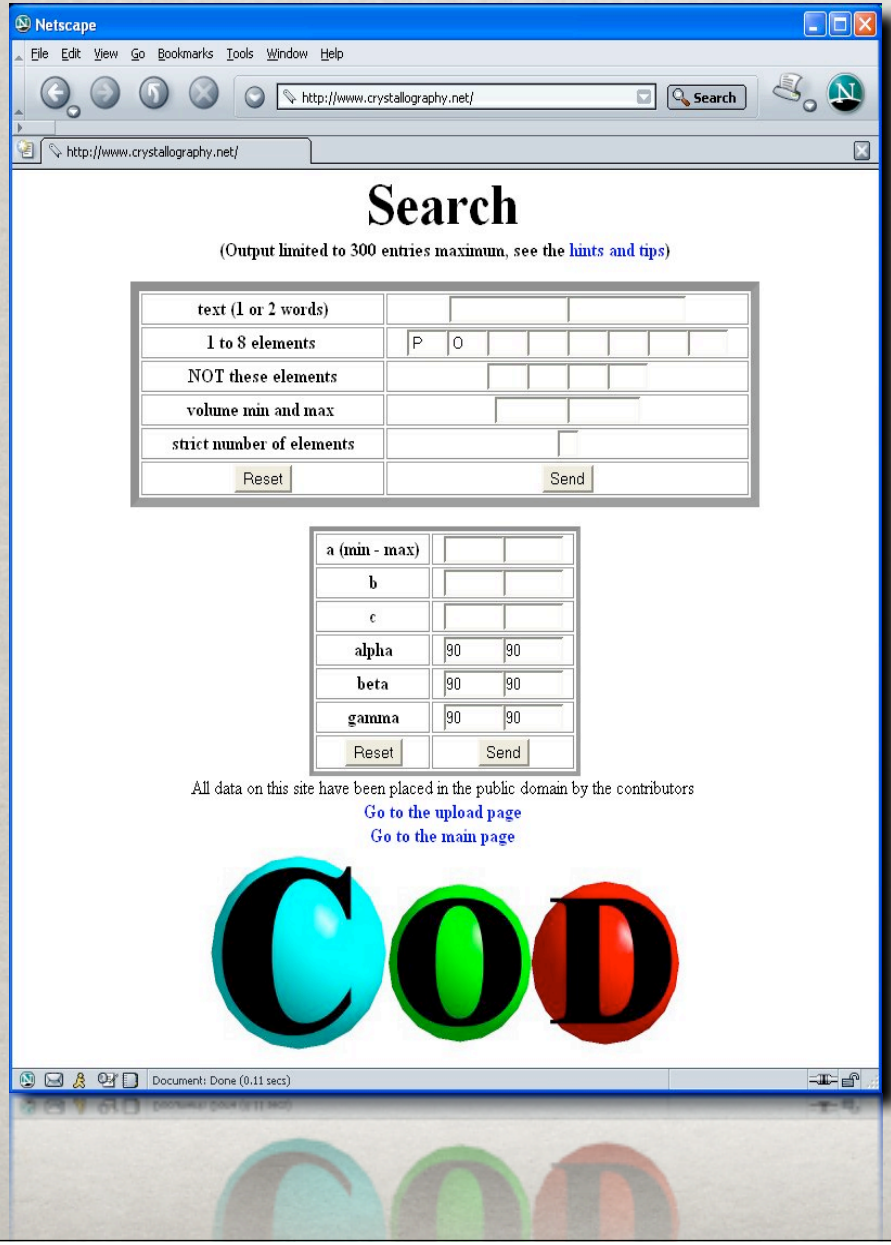
- Toll databases :
  - CSD (organic, organometallic)
  - ICSD (inorganic, minerals)
  - CRYSTMET (metals, intermetallics)
  - ICDD (powder patterns)

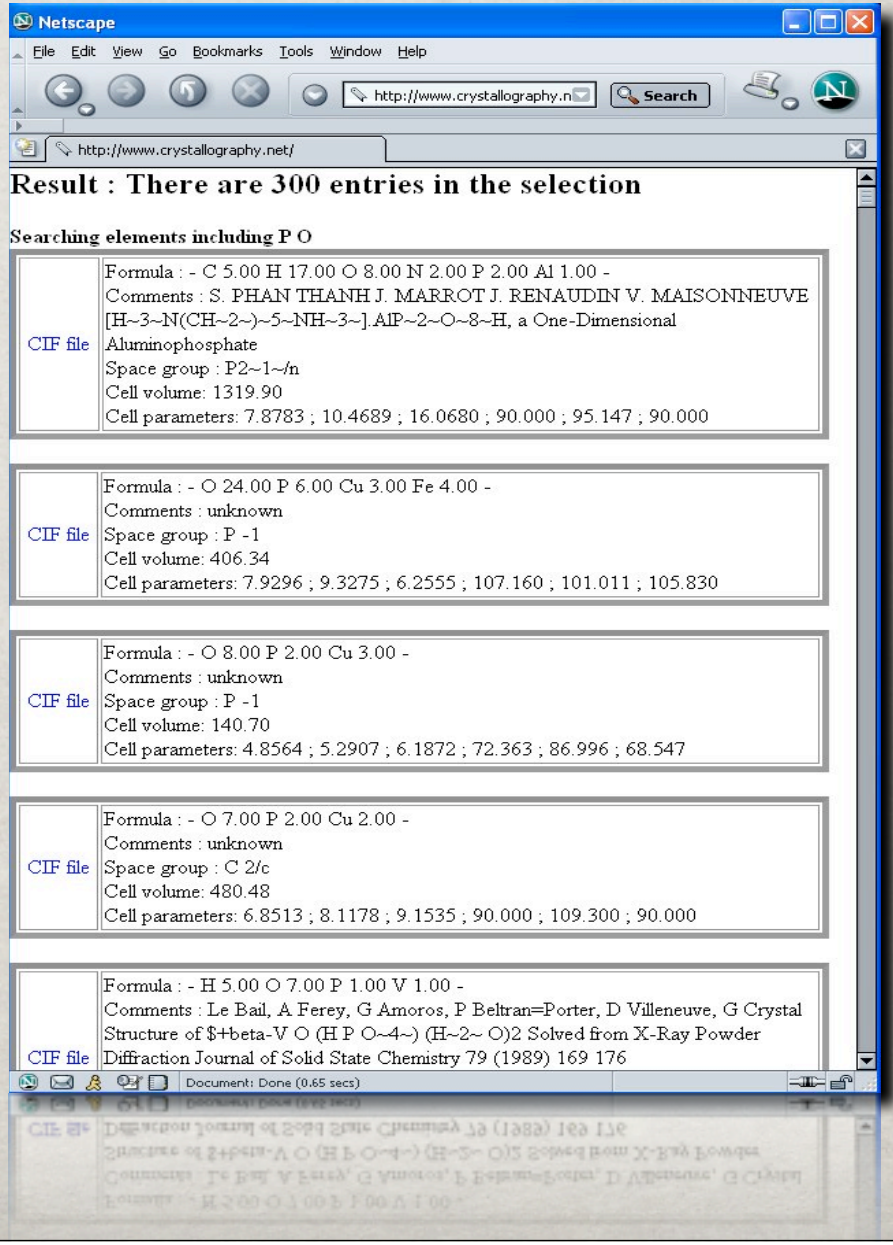
Other structure Databases

- 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/





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