QUESTIONNAIRE FOR the
STRUCTURE DETERMINATION BY POWDER DIFFRACTOMETRY ROUND ROBIN - 3

Please answer all questions as completely as possible. Provide one filled questionnaire for each data (samples 1 and 2).

Preferably, attach the results as one PDF file or as a MS Word document compressed by Winzip.

It is advised to complete the form as the structure determination progress.

O.0 Precise date of
- data download : Sat, 2 Feb 2008 19:56:00
- results submission : Tue, 29 Apr 2008 20:00:00

0.1 Is the first sample structure solvable with this quality of data ? Yes [x] No [ ]
0.2 Is the second sample structure solvable with this quality of data ? Yes [x] No [ ]
0.3 If not, what data would be required ?

Then, for each sample :

Ca-tartrate

1. Preliminary work

1.1 Did you obtained additional informations ?
(for instance from CSD or ICSD or ICDD databases)

Yes, from web and from the journals.

1.2 Did you obtained additional informations from the powder pattern ? If yes, how and what information ?
(for instance using the JCPDS-ICDD database)

Yes, the indexing and extinction conditions were checked.

1.3 Did you extract the structure factors ? Yes [ ] No [x]

1.3.1 If yes, which program(s) did you use ?
1.3.2 Give the angular range:
1.3.3 Give the number of extracted structure factors:
1.3.4 Give the Rp and Rwp (conventional Rietveld, background subtracted):
1.3.5 Give the Rp and Rwp (background not subtracted):

1.3.6 If not, did you use the whole pattern ?

Yes

1.3.7 Or a partial pattern (if yes, give the angular range):
1.3.8 If you use the whole or a partial pattern, did you keep fixed the profile parameters, and if yes, how did you obtained them ?

Yes, the profile parameters were refined by LeBail refinement with FullProf.

2- Structure solution

2.1 Did you use direct methods ? Yes [ ] No [x]

2.1.1 If yes, was it on the whole dataset ?
2.1.2 Or on a partial dataset ?
2.1.3 Give the number of reflections:
2.1.4 Which program(s) did you use ?
2.1.5 Did you modified intensities of closely neighbouring reflections ? If yes, explain how.

2.2 Did you use Patterson methods ? Yes [ ] No [x]

2.2.1 If yes, was it on the whole dataset ?
2.2.2 Or on a partial dataset ?
2.2.3 Give the number of reflections:
2.2.4 Which program(s) did you use ?
2.2.5 Did you modified intensities of closely neighbouring reflections ? If yes, explain how.

2.3 Did you use another method ? Yes [x] No [ ]

2.3.1 If yes, which method(s) (give details : molecule location by direct space - genetic algorithm, Monte Carlo, Simulated annealing, scratch, charge flipping, other) ?

Yes, direct space method with Simulated annealing in Parallel tempering mode.

2.3.2 Which program(s) did you use (name and reference) ?

Fox: Favre-Nicolin, V.; Cerny, R.: FOX, J. Appl. Crystallography 35 (2002) 734-743.
See also http://objcryst.sourceforge.net/Fox.

2.3.3 If you used direct space methods, how many independent molecules did you use (give details on these molecules)? How many degrees of freedom (total) ? How many torsion angles ?

Space group P-1.

1 semi-rigid molecule 2R,3R C4H4O6 with internal DoF and restraints as used in FOX
4 rigid molecules H2O
1 free atoms of Ca
In total 33 DoF + internal DoF of the C4H4O6 molecule.

2.4 Did you first locate the whole structure ? Yes [x] No [ ]

2.4.1 If not, how many atoms did you locate ?
27

2.4.2 Give their name and initial atomic coordinates

Atom	x	y	z	occ	Biso
Ca	0.7545	0.1228	0.0266	1.0000	1.5000
W1	0.6876	0.9884	0.4896	1.0000	2.5000
H11	0.8055	1.0304	0.5189	1.0000	2.5000
H12	0.6404	0.9383	0.3219	1.0000	2.5000
W2	0.2777	0.7500	0.6406	1.0000	2.5000
H21	0.3844	0.7520	0.6042	1.0000	2.5000
H22	0.2936	0.7068	0.7626	1.0000	2.5000
W3	0.3630	0.5799	0.0867	1.0000	2.5000
H31	0.3949	0.5265	0.1926	1.0000	2.5000
H32	0.4390	0.6677	0.1671	1.0000	2.5000
W4	0.6592	0.8274	0.8046	1.0000	2.5000
H41	0.6896	0.7587	0.6904	1.0000	2.5000

H42	0.7141	0.8157	0.9570	1.0000	2.5000
O1	0.9434	0.9229	0.6365	1.0000	2.5000
O2	0.7409	0.7705	0.3045	1.0000	2.5000
O3	0.9950	0.6675	0.6683	1.0000	2.5000
O4	1.1130	0.5337	0.2547	1.0000	2.5000
O5	1.0631	0.7589	0.0737	1.0000	2.5000
O6	0.7881	0.6420	-0.0845	1.0000	2.5000
C7	0.8905	0.8115	0.4493	1.0000	2.5000
C8	1.0133	0.7179	0.4894	1.0000	2.5000
C9	0.9788	0.6085	0.2588	1.0000	2.5000
C10	0.9419	0.6789	0.0719	1.0000	2.5000
H11	1.1413	0.7749	0.5423	1.0000	2.5000
H12	0.8687	0.5473	0.2294	1.0000	2.5000
H13	0.8766	0.6686	0.6585	1.0000	2.5000
H14	1.2130	0.6095	0.2784	1.0000	2.5000

2.4.3 Were the initial atomic coordinates taken from a known structure ? Yes [ ] No [x]
If yes, which one (give reference) ?

3- Structure completion

3.1 Did you performed Fourier difference syntheses before refining the structure by the Rietveld method ? Yes [ ] No [x]
3.2 If yes, with what program ?
3.3 If yes, how many additional atoms did you obtained from Fourier difference syntheses ?
3.4 Give their name and atomic coordinates as they were obtained

Atom	x	y	z
.....			
.....			
.....			

3.5 Did you made first Rietveld refinements without preliminary Fourier difference syntheses ? Yes [x] No [ ]
3.5.1 If yes, with what program ?

Topas.

3.5.2 What were the Rp and Rwp (background subtracted AND not subtracted) and RB and RF that you obtained at the first Rietveld application ?

??

3.5.3 Did you get the structure factors from this result and performed a Fourier difference synthesis ?

No

3.5.4 Did you locate additional atoms at this stage ?

No

3.5.5 And which one ?

Atom	x	y	z
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....

3.5.6 If you repeated Rietveld refinements and Fourier synthese several times before to complete the model, give the number of times and which atoms you locate and the Rp, Rwp RB, RF at each times.

Atom	x	y	z
.....	.....	.....	.....
.....	.....	.....	.....
.....	.....	.....	.....

4- Final refinement

- Give the final atomic coordinates, thermal parameters, standard deviations, Reliability factors.....

Atom	x	y	z	Occupancy		Biso
Ca	0.738(1)	0.126(1)	0.006(2) occ	Ca	1	beq bCa 0.4790`_0.3128
W1	0.65307	1.02214	0.60613 occ	O	1	beq bW 0.5637`_0.4169
H11	0.76730	1.00876	0.60088 occ	H	1	beq = bW;
H12	0.67222	1.08846	0.75860 occ	H	1	beq = bW;
W2	0.34830	0.77825	0.66048 occ	O	1	beq = bW;

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H21 0.22370     0.77280    0.61817 occ    H    1  beq = bW;
H22 0.38003     0.72759    0.77089 occ    H    1  beq = bW;
W3  0.35945     0.57185    0.27287 occ    O    1  beq = bW;
H31 0.26718     0.49292    0.20564 occ    H    1  beq = bW;
H32 0.34045     0.62800    0.41190 occ    H    1  beq = bW;
W4  0.68301     0.75984    0.95278 occ    O    1  beq = bW;
H41 0.71373     0.68190    1.00085 occ    H    1  beq = bW;
H42 0.55725     0.74295    0.88819 occ    H    1  beq = bW;
O1  0.91474     0.60977    0.03264 occ    O    1  beq !bM 4.
O2  1.03116     0.56581    0.39403 occ    O    1  beq = bM;
O3  0.88341     0.88845    0.33882 occ    O    1  beq = bM;
O4  0.81139     0.78712    0.69823 occ    O    1  beq = bM;
O5  0.78934     0.58810    0.64882 occ    O    1  beq = bM;
O6  1.07881     0.66603    0.87830 occ    O    1  beq = bM;
C7  0.96161     0.63865    0.26268 occ    C    1  beq = bM;
C8  0.92437     0.77442    0.36800 occ    C    1  beq = bM;
C9  0.94070     0.78536    0.61652 occ    C    1  beq = bM;
C10 0.93616     0.67306    0.72005 occ    C    1  beq = bM;
H1  0.82266     0.78900    0.24690 occ    H    1  beq = bM;
H2  0.88018     0.73540    0.44460 occ    H    1  beq = bM;
H3  0.99715     0.94840    0.37912 occ    H    1  beq = bM;
H4  0.74055     0.69522    0.60054 occ    H    1  beq = bM;
```

Space group P-1.
r_exp 1.951 r_wp 11.726 r_p_dash 13.538 gof 6.012 r_bragg 3.90

- Give details about constraints, restraints

The C4H4O6 molecule was refined on Z-matrix description with 5 free bond angles and 4 free torsion angles.
The water molecules were refined as rigid body.
The e.s.d. of the internal angles and molecule rotation angles were better than 2 deg for the C4H4O6 molecule.
The e.s.d. of the rotation angles of the water molecules were as bad as 90 deg!
The e.s.d. of the positional parameters of the C4H4O6 and water molecules were better than 0.006.
Ca was refined as free atom.
The Biso were constrained as given in the table. The Biso of the C4H4O6 molecule was fixed.
Following antibum restraints were used:

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Anti_Bump(2, W1, O4,    2.9, 0.00000001)
Anti_Bump(2, W2, O2,    2.9, 0.00000001)
Anti_Bump(2, H2, O4,    1.8, 0.00000001)
Anti_Bump(2, H2, C10,    1.8, 0.00000001)
Anti_Bump(2, C9, O3,    1.9, 0.00000001)
```
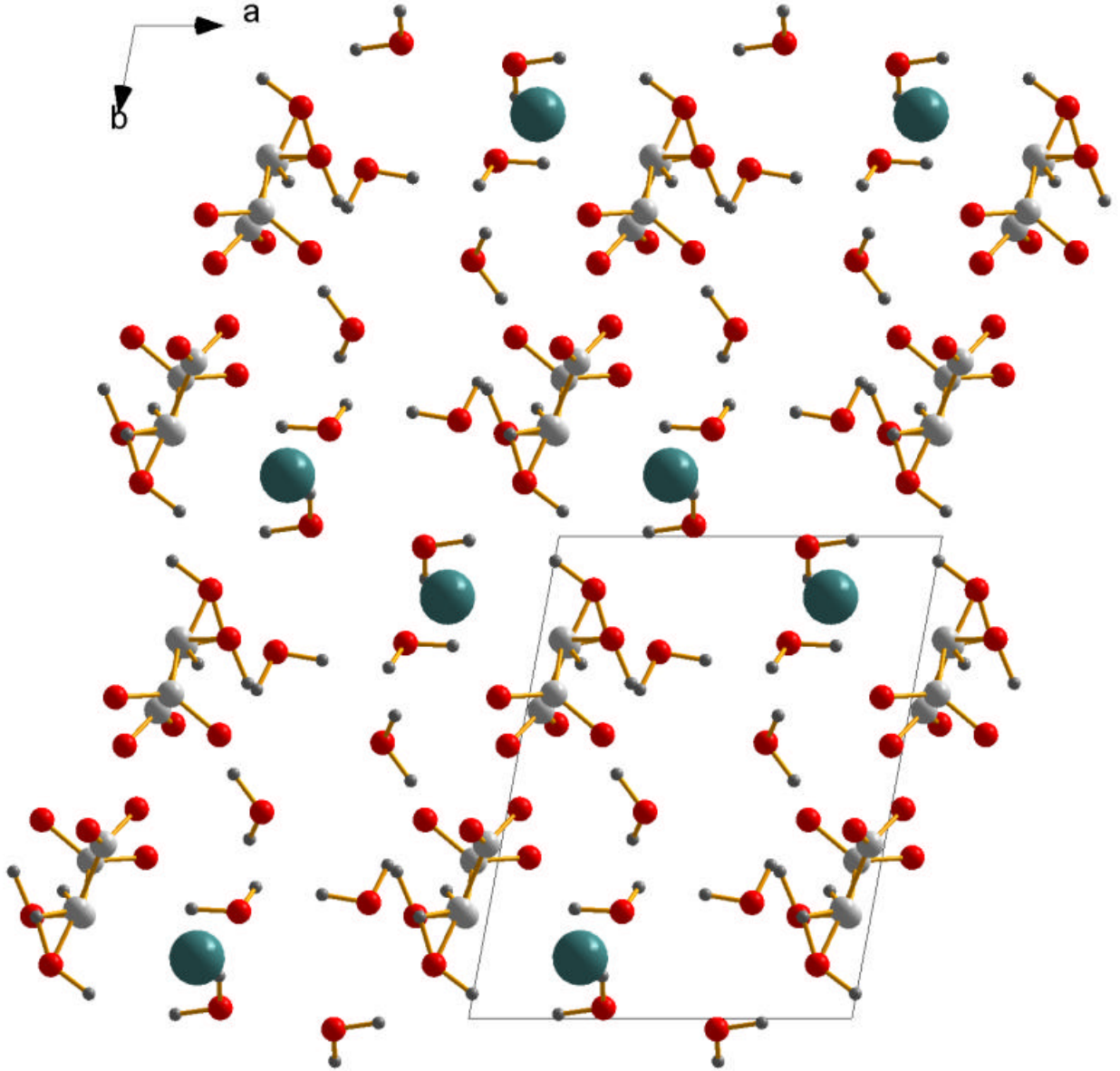

The preferred orientation of the planes (110) was refined by spherical harmonics of the 8^{th} order.

5- Feel free to add any intermediate results (list of extracted structure factors, software decisive input and output data...) or comments you might consider as essential (details on hardware, time for solving the structure, number of moves by Monte Carlo or molecule position trial, any picture...).

The key problem of the structure solution was the preferred orientation of the planes (110). It was modeled in FOX by March-Dollase correction and in Topas by spherical harmonics of the 8^{th} order. The planes (110) can be identified in the final structure as planes containing C4H4O6 molecules and Ca atoms. The preferred orientation including the hkl indices of the preferred orientation planes was solved ab-initio during the structure solution in FOX.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