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.

0.0 Precise date of
- data download : 28-Feb-08
- results submission : 20-Mar-08

0.1 Is the first sample structure solvable with this quality of data? Yes [ ] No [ ] N/A (have not taken a look at it yet)
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:

1. Preliminary work

1.1 Did you obtain additional information?
(for instance from CSD or ICSD or ICDD databases)
ICSD

1.2 Did you obtained additional information from the powder pattern? If yes, how and what information?
(for instance using the JCPDS-ICDD database)
No

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

1.3.1 If yes, which program(s) did you use? GSAS
1.3.2 Give the angular range: 7-70deg
1.3.3 Give the number of extracted structure factors: ~800
1.3.4 Give the Rp and Rwp (conventional Rietveld, background subtracted): 4.75/7.40%
1.3.5 Give the Rp and Rwp (background not subtracted): 3.87/5.74%

1.3.6 If not, did you use the whole pattern?
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?

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 [x] No [ ]

2.2.1 If yes, was it on the whole dataset? No
2.2.2 Or on a partial dataset? Yes
2.2.3 Give the number of reflections: ~150
2.2.4 Which program(s) did you use? SHELX
2.2.5 Did you modify intensities of closely neighbouring reflections? If yes, explain how.

Overlapping peaks were filtered out

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)?

Other, see Section 5

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

Forcite (Accelrys Materials Studio)

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?

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

2.4.1 If not, how many atoms did you locate?
2.4.2 Give their name and initial atomic coordinates

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

2.4.3 Were the initial atomic coordinates taken from a known structure? Yes [ ] No [ ]
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 [x] No [ ]
3.2 If yes, with what program? SHELXL
3.3 If yes, how many additional atoms did you obtained from Fourier difference syntheses? 3
3.4 Give their name and atomic coordinates as they were obtained

One of the tartrate COO groups was located in a series of Fourier syntheses using different subsets of the originally extracted Fobs's. I did not keep incomplete models but the coordinates were pretty close to the final ones.

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

3.5.4 Did you locate additional atoms at this stage?

3.5.5 And which one?

Water oxygen atoms around Ca were located at this stage (GSAS)

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

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4- Final refinement

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

See the enclosed "tartrate.cif" file

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- Give details about constraints, restraints

Water molecules modelled as rigid bodies, C-C/C-O distances were restrained

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...).

Positions of hydrogen atoms were obtained from geometry optimization with molecular mechanics module Forcite (COMPASS forcefield) of the Accelrys Materials Studio.