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 : 2 Feb 2008
- results submission : 21 Apr 2008

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 :

sample 1

1. Preliminary work

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

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

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 ? No/Yes
1.3.7 Or a partial pattern (if yes, give the angular range):
For the structure solution in FOX: 7-70°
For the refinement in GSAS: whole pattern
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 ?
In FOX: fixed profile parameters obtained by the lebail module
In GSAS: refined profile parameters

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) ? Parallel tempering

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

Started as Z-matrix from Pb-tartrate example. Start with 1 rigid body tartrate molecule plus 5 free atoms: 1 x Ca and 4 x O
Later on: release 11 torsion angles one by one within window of 10° around starting model angle. Next Z-matrix converted to molecule description for finding final solution.

2.4 Did you first locate the whole structure ? Yes [X] 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
.....			
.....			
.....			

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

An initial Z-matrix tartrate molecule model was constructed with data from Pb-tartrate: Acta C 2002 58 vol.12 596-598 and inserted in the cell at an arbitrairy position.

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 ? FOX
3.3 If yes, how many additional atoms did you obtained from Fourier difference syntheses ? None
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 [ ] No [x], not for structure completion
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 ?

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: GSAS

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

Atom	x	y	z	B
.....see enclosed CIF file.....				
.....				
.....				

- Give details about constraints, restraints
  distance C4-O5 restrained to 1.31Å. Unrestrained O5 moves away from C4. This moving around hardly affects the R values.
  Tartrate H's included in the refinement at restrained position.

Based on the smooth Rietveld difference trace it was decided not to do a difference fourrier map search to look for additional atoms.

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 structure solution was going rather smooth and could have been completed within 2 days. By the step wise structure solution in FOX counting of the number of moves was to difficult.

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