

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 : Sunday, April 20, 2008. 7:00 AM
- results submission : Thursday, May 1, 9:50 PM

0.1 Is the first sample structure solvable with this quality of data ? Yes No

0.2 Is the second sample structure solvable with this quality of data ? I played with it a bit, but made no serious attempt. Probably solvable with tools at hand, but not time or motivation.

0.3 If not, what data would be required ?

Then, for each sample :

Following answers are only for sample 1.

1. Preliminary work

1.1 Did you obtained additional informations ?

(for instance from CSD or ICSD or ICDD databases)

I looked at tartrate.cif that you furnished. I skimmed through the CSD to get a sense of the degree of flexibility in tartrate salts. Concluded that the configuration of planar C-C-C-C backbone is pretty much universal. I was a bit concerned that the sample might be DL or a meso-tartrate, but guessed that you probably wouldn't have done that.

1.2 Did you obtained additional informations from the powder pattern ? If yes, how and what information ?

(for instance using the JCPDS-ICDD database)

None

1.3 Did you extract the structure factors ? Yes No

1.3.1 If yes, which program(s) did you use ? Topas

1.3.2 Give the angular range: 7 to 53.5

1.3.3 Give the number of extracted structure factors: 196

1.3.4 Give the Rp and Rwp (conventional Rietveld, background subtracted): Rp = 3.705%, Rwp=5.042%

1.3.5 Give the Rp and Rwp (background not subtracted): N/A

- 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 ?

Refined profile and lattice parameters in a Pawley fit over angular range (196 reflections to 53.5 degrees). I didn't have the geometric parameters of the diffractometer, so I just winged it with the FP crystallite size and strain parameters in topas, and adjustable axial divergence. It wasn't in the information provided, but it was evident from the fit that there was K-alpha-2 present, which I included in the lineshape fit.

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) ?

I used direct space, parallel tempering of extracted intensities, with correlation coefficients from the Pawley fit.

2.3.2 Which program(s) did you use (name and reference) ?
Not released or named - Parallel tempering adaptation of PSSP.

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 ?

Two independent tartrates, each with Ca tethered in a likely location to two oxygen atoms. 8 independent oxygen atoms. The Ca and carboxyl torsions were varied over a limited range relative to planar. So each Ca tartrate was described by 3 translations, three rotations, and three torsions. 8 independent oxygen were 3 translations each, for a grand total of 42 parameters.

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
.....			
.....			
.....			

2.4.3 Were the initial atomic coordinates taken from a known structure ? Yes No

If yes, which one (give reference) ?

I assume you mean the baseline structure for real space. I took it from the reference you provided, Hawthorne et al., Acta B 38, 2461 (1982).

3- Structure completion

3.1 Did you performed Fourier difference syntheses before refining the structure by the Rietveld method ? Yes No

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 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 ? Rwp=16.65%, Rp=11.3%, background not subtracted.

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 ?

3.5.5 And which one ?

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

.....

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 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	B
Ca1	-0.08085262	0.1709038	-0.2162526	0.8800544
Ca2	0.2667451	0.7040273	0.4502548	0.8800544
C11	0.2219382	0.08508906	0.1019197	-1.201111
C12	0.1569518	0.1655314	0.2873039	-1.201111
C13	0.3058912	0.2271451	0.5148265	-1.201111
C14	0.2599072	0.3233946	0.7052927	-1.201111
O11	0.1357225	0.07001087	-0.115363	-1.201111
O12	0.3597962	0.03743745	0.1764286	-1.201111
O13	0.04954378	0.2679547	0.2270628	-1.201111
O14	0.4580055	0.2943965	0.4753301	-1.201111
O15	0.1059214	0.3039086	0.7148046	-1.201111
O16	0.3839281	0.4213604	0.8488662	-1.201111
C21	-0.01275034	0.535238	0.5439603	-1.201111
C22	-0.056267	0.664757	0.6707219	-1.201111
C23	-0.05292774	0.6623105	0.9106952	-1.201111
C24	-0.09455366	0.7876489	1.054011	-1.201111
O21	0.03320791	0.5330483	0.3637949	-1.201111
O22	-0.02514466	0.4362111	0.6268099	-1.201111
O23	0.0760628	0.7785787	0.6872838	-1.201111
O24	0.1122364	0.6254826	1.040564	-1.201111
O25	0.02535453	0.8704503	1.236707	-1.201111
O26	-0.253543	0.8020576	0.9784097	-1.201111
O1	0.2746908	0.7869181	0.9012306	0
O2	0.3561118	0.8100445	0.1641496	0
O3	0.3804389	0.9535596	0.6913836	0
O4	0.7143557	0.2206624	0.3843348	0
O5	0.3686795	0.5001773	0.2097239	0
O6	0.6988174	0.358161	1.028696	0
O7	0.7649951	0.9150282	0.511257	0
O8	0.7451038	0.06718621	1.017575	0

Give details about constraints, restraints

Chemically similar bond lengths within tartrate constrained to be equal. carboxyl groups constrained to be planar. I am not quoting esd's because the numbers put out by the least squares software are

not relevant to the source of error here, which is probably systematic rather than statistical. Also, refinements with rigid bodies are difficult to calculate even statistical esd's of fractional coordinates.

- 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...).

I am very suspicious of some of the short O-O distances, but I'm not going to spend any more time worrying about it. Without information to the contrary, I assume that there was constant counting time per point, which limits ability to get information from the high angle part of the pattern. Also, I would trust capillary data more than flat plate, so I feel that I have an excuse for deficiencies of the fit and atomic model. No apologies for negative thermal parameters from flat plate data and a lab source. I spent approx. 10 hours on the solution and refinement, spread over several days.