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 : **Thu, 7 Feb 2008 16:30**
- results submission : **Tue, 30 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 ?
For the second sample, even if the quality of experimental data is good, probably additional chemical information is needed

Then, for each sample :

SAMPLE 1

1. Preliminary work

1.1 Did you obtained additional informations ?
(for instance from CSD or ICSD or ICDD databases)
NO

1.2 Did you obtained additional informations 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 ?
EXPO200x, an alpha version of EXPO
1.3.2 Give the angular range: **7.0 – 89.705**
1.3.3 Give the number of extracted structure factors: **780**
1.3.4 Give the Rp and Rwp (conventional Rietveld, background subtracted): **Rp (back. subtracted)=9.933%**
1.3.5 Give the Rp and Rwp (background not subtracted): **Rp =3.35%, Rwp = 5.989%**
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 ?
NO, the starting values are automatically provided by EXPO200x and then refined.

2- Structure solution

2.1 Did you use direct methods ? Yes [**X**] No [ ]

2.1.1 If yes, was it on the whole dataset ? **YES**
2.1.2 Or on a partial dataset ? **NO**
2.1.3 Give the number of reflections: **780**
2.1.4 Which program(s) did you use ? **EXPO200x**

2.1.5 Did you modified intensities of closely neighbouring reflections ? If yes, explain how.
YES, as performed by EXPO200x.

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, Simulated Annealing.

2.3.2 Which program(s) did you use (name and reference) ?
EXPO200x

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 ?

The number of independent molecules is 6:
- 1 tartrate molecule
- 4 water molecules
- 1 Ca atom
The total number of degrees of freedom is 38: 33 external DOFs and 5 torsion angles were refined.
The file tartrate1.mol (Molfile format) included in the zip file tartrate.zip contains the structure model used by EXPO200x as starting point for the Simulated Annealing. Hydrogen atoms are included.

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

In case of Direct Methods application an automatic run of EXPO200x gave the following atomic coordinates (see also tartrate_DM1.cif file and the EXPO200x output file tartrate_DM.out):

	Atom	x	y	z	B(iso)
1)	Ca1	0.186	0.272	0.336	6.105
2)	C1	0.015	0.163	0.913	0.031
3)	O1	0.288	0.637	1.084	2.331
4)	O2	-0.309	0.037	0.284	2.161
5)	O3	0.025	0.347	0.630	2.630
6)	O4	-0.359	-0.102	-0.166	4.100
7)	O5	-0.023	0.082	0.305	4.867

8)	O6	0.315	0.514	0.620	6.310
9)	O7	0.364	0.204	0.681	6.238
10)	O8	0.350	0.376	1.166	7.034
11)	O9	0.147	0.666	1.016	6.879
12)	O10	-0.027	0.397	0.190	9.607
13)	C2	0.884	0.141	0.462	4.887
14)	C3	0.864	0.188	0.815	6.730
15)	C4	0.863	0.250	0.572	9.621
16)	C6	0.685	0.629	0.982	11.656
17)	C7	0.056	0.153	0.877	3.898

In case of Simulated Annealing application EXPO200x gave the following structure model (see also tartrate_SA.cif file and the corresponding Simulated Annealing results in tartrate_SA.out file):

Atom		x	y	z	B(iso)
1)	Ca1	0.186	0.770	0.840	3.587
2)	O1	-0.027	0.892	0.685	3.587
3)	O2	-0.288	0.876	0.423	3.587
4)	O3	0.037	0.673	0.424	3.587
5)	O4	0.021	0.837	0.127	3.587
6)	O5	-0.015	0.592	-0.176	3.587
7)	O6	-0.290	0.535	-0.203	3.587
8)	O7	0.378	0.727	1.207	3.587
9)	O8	0.333	0.879	0.635	3.587
10)	O9	0.309	1.006	1.106	3.587
11)	O10	0.348	0.600	0.683	3.587
12)	C1	-0.149	0.837	0.490	3.587
13)	C2	-0.123	0.711	0.318	3.587
14)	C3	-0.128	0.740	0.089	3.587
15)	C4	-0.143	0.614	-0.116	3.587
16)	H1	0.451	0.677	1.143	6.000
17)	H2	0.251	0.664	1.158	6.000
18)	H3	0.295	0.920	0.727	6.000
19)	H4	0.264	0.875	0.484	6.000
20)	H5	0.289	1.053	1.239	6.000
21)	H6	0.259	1.034	1.004	6.000
22)	H7	0.380	0.551	0.583	6.000
23)	H8	0.293	0.544	0.736	6.000
24)	H9	-0.217	0.639	0.261	6.000
25)	H10	-0.248	0.779	0.030	6.000
26)	H11	0.027	0.605	0.379	6.000
27)	H12	0.013	0.909	0.213	6.000

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

If yes, which one (give reference) ?

NO in case of Direct Methods application, YES in case of Simulated Annealing. The initial atomic coordinates (file tartrate1.mol) used by Simulated Annealing correspond to the first structure model in the file tartrate.cif included in the sample1.zip supplied by the Round Robin–3 Organizers.

3- Structure completion

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

EXPO200x automatically provided the whole structure. The automatic EXPO200x run performs Fourier difference based procedures.

3.2 If yes, with what program ?

EXPO200x

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 [**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 ?

After the Rietveld application performed on the structure model supplied by Simulated Annealing (file tartrate_SA.cif) EXPO200x supplied the following values:

Rp = 4.545% Rwp = 6.159% (Background NOT subtracted)
Rp = 12.661% Rwp = 13.164% (Background subtracted)
RB = 9.667% RF = 6.279%

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.....

The final atomic coordinates supplied by EXPO200x are (see also the file tartrate_SA_riet.cif):

Atom	x	y	z	B
1) Ca1	0.18224	0.76863	0.82916	4.14958
2) O1	-0.03838	0.89353	0.69069	5.56299
3) O2	-0.28529	0.86921	0.41888	4.20983
4) O3	0.02624	0.67528	0.39187	5.71544
5) O4	0.02041	0.83112	0.11050	0.23167

6)	O5	-0.01631	0.58477	-0.18035	0.97688
7)	O6	-0.30461	0.53380	-0.21745	3.44425
8)	O7	0.37564	0.72787	1.18863	3.53062
9)	O8	0.32875	0.87661	0.62296	3.96483
10)	O9	0.32278	1.00384	1.11158	0.85331
11)	O10	0.34022	0.59963	0.67450	4.32078
12)	C1	-0.13537	0.84783	0.49213	2.55738
13)	C2	-0.10878	0.70660	0.33303	13.78161
14)	C3	-0.15209	0.74563	0.08183	1.28124
15)	C4	-0.11652	0.62496	-0.09645	1.23609
16)	H1	0.45100	0.67700	1.14300	6.00072
17)	H2	0.25100	0.66400	1.15800	6.00072
18)	H3	0.29500	0.92000	0.72700	6.00072
19)	H4	0.26400	0.87500	0.48400	6.00072
20)	H5	0.28900	1.05300	1.23900	6.00072
21)	H6	0.25900	1.03400	1.00400	6.00072
22)	H7	0.38000	0.55100	0.58300	6.00072
23)	H8	0.29300	0.54400	0.73600	6.00072
24)	H9	-0.21700	0.63900	0.26100	6.00072
25)	H10	-0.24800	0.77900	0.03000	6.00072
26)	H11	0.02700	0.60500	0.37900	6.00072
27)	H12	0.01300	0.90900	0.21300	6.00072

Final reliability factors:

Rp = 4.545% Rwp = 6.159% 'expected Rwp' = 1.998% GoF = 3.082 (Background NOT subtracted)

Rp= 12.661% Rwp = 13.164% 'expected Rwp' = 4.271% (Background subtracted)

- Give details about constraints, restraints

Hydrogen atoms were not refined during Rietveld application. The only restraint applied is the positivity of thermal factor.

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 was solved by EXPO200x, an alpha version of EXPO. You can require this version by sending an e-mail to caterina.chiarella@ic.cnr.it.

EXPO200x has been used both for Direct Methods and for Simulated Annealing application. We compared the structure models supplied by both structure solution methods and they are very similar.

The solution by Direct Methods was carried out by using EXPO200x program (input file tartrate_DM.exp). After the full pattern decomposition step, the solution was not obtained at the first phasing trial. It was recognized at the third trial (exploring the phasing trials was easily activated by graphic interface), see the corresponding output file tartrate_DM.out. The file tartrate_DM1.cif corresponds to the structure model obtained at the end of the EXPO200x run. Two of the 17 atoms provided by the program were graphically deleted because they appeared to be chemically unreliable, two wrongly labelled atoms have been correctly relabelled and reconnected using the graphic options of EXPO200x. The final model have been saved on the file tartrate_DM2.cif. That model should be refined further on by Rietveld method but we don't give these results because we provide only one list of refined positions, obtained applying the Rietveld method at the end of Simulated Annealing approach.

The solution by Simulated Annealing was carried out by using EXPO200x program (input file tartrate_SA.exp). The starting fragment tartrate1.mol will be automatically read by EXPO200x. 10 runs are executed in about 16h on a Linux HP xw6400 Workstation (CPU Intel Xeon 3 GHz) and two plausible and similar solutions have been obtained. The Simulated Annealing results are summarized in the file tartrate_SA.out and the file

tartrate_SA.cif contains the structure model corresponding to the best figure merit run.

The Rietveld refinement of structure model and profile parameters was carried out automatically by EXPO200x *via* **graphic interface. We didn't try to modify the automatic strategy in the Rietveld refinement in order to improve the results. Hydrogen atoms were not refined.**

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