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

Report on the structure solution of sample2 from X-ray diffraction data, data file La8W5O27-X-gsas.dat

0.0 Precise date of
- data download : *Fri, 22 Feb 2008 11:58 +0100*
- results submission : Sun, 24 Feb 2008 17:15 +0100

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 ?

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 ?
Jana2000

1.3.2 Give the angular range:
full, i.e. 2th=1.0-45.5

1.3.3 Give the number of extracted structure factors:
1162 - assumed 6/mmm Laue symmetry

1.3.4 Give the Rp and Rwp (conventional Rietveld, background subtracted): *18.04, 16,56*

1.3.5 Give the Rp and Rwp (background not subtracted): *5.43, 7.51*

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

Charge flipping

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

Superflip: http://superspace.epfl.ch/superflip; density analyzed by EDMA: http://superspace.epfl.ch/superflip; input file for Superflip and EDMA generated by Jana2000 http://www-xray.fzu.cz/jana/Jana2000/jana.html;

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

2.4.1 If not, how many atoms did you locate ?
10 - all La and W positions

2.4.2 Give their name and initial atomic coordinates

Atom	x	y	z
W1	0.666667	0.333333	0.000000
W2	0.000000	0.000000	0.083790
W3	0.000000	0.000000	0.250000
W4	0.333333	0.666666	0.167117
W5	0.666666	0.333333	0.135904
La1	0.760771	0.035954	0.167436
La2	0.038402	0.420728	0.083851
La3	0.740508	0.000000	0.000000
La4	0.064271	0.435729	0.250000
La5	0.666666	0.333333	0.218776

Note: La5 turned out later to be a half-occupied tungsten position.

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 ?

Jana2000

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 ?

Background subtracted Rp, Rwp: 8.50, 12.45
Background unsubtracted Rp, Rwp: 28.35, 27.54
RBobs, RBall: 15.30, 19.55
RFobs, RFall: 8.08, 8.57

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

Yes

3.5.4 Did you locate additional atoms at this stage ?

Yes, I located all oxygen atoms

3.5.5 And which one ?

Atom	x	y	z
O1	0.034943	0.184557	0.121949
O2	0.302263	0.490437	0.205942
O3	0.022529	0.174391	0.212548
O4	0.482395	0.298791	-0.043021
O5	0.176429	0.683720	0.129811
O6	-0.156397	-0.171336	0.044103
O7	0.704420	0.172327	0.111335
O8	0.517270	0.158539	0.169136
O9	0.521778	0.357818	0.035564
O10	0.830481	0.530460	0.250000

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:

All oxygen atoms could be located in one step. Indicators after refining all oxygen atoms, and changing La5-> W6:

Background subtracted Rp, Rwp: 20.90, 19.96
Background unsubtracted Rp, Rwp: 6.28, 8.58
RBobs, RBall: 7.45, 7.63
RFobs, RFall: 4.98, 4.83

4- Final refinement

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

Final reliability factors:

Background subtracted Rp, Rwp: 19.63, 17.76
Background unsubtracted Rp, Rwp: 5.87, 8.02
RBobs, RBwobs, RBall, RBwall: 4.48, 4.67, 6.00, 38.26
RFobs, RFwobs, RFall, RFwall: 3.19, 2.98, 3.70, 3.10

Atom	x	y	z	Uiso	Occupancy
W1	0.666667	0.333333	-0.00321(6)	0.0115(7)	
W2	0	0	0.08469(9)	0.0117(7)	
W3	0	0	0.25	0.0108(11)	
W4	0.333333	0.666667	0.16670(7)	0.0107(5)	
W5	0.666667	0.333333	0.13643(10)	0.0349(9)	
La1	0.7589(2)	0.0371(2)	0.16803(9)	0.0119(8)	
La2	0.0400(3)	0.4250(3)	0.08369(9)	0.0197(11)	
La3	0.7436(4)	0	0	0.0151(12)	
La4	0.0593(4)	0.4285(5)	0.25	0.0147(15)	
W6	0.666667	0.333333	0.21998(12)	0.0129(12)	0.5
O1	0.028(2)	0.174(2)	0.1202(6)	-0.015(4)	
O2	0.317(3)	0.497(3)	0.2070(7)	0.008(6)	
O3	0.017(3)	0.170(3)	0.2127(6)	-0.002(5)	
O4	0.477(3)	0.288(3)	-0.0442(7)	0.021(7)	
O6	-0.152(3)	-0.190(3)	0.0453(8)	0.017(6)	
O7	0.709(3)	0.181(3)	0.1079(6)	0.008(5)	
O8	0.523(2)	0.161(3)	0.1764(7)	0.020(6)	
O9	0.595(3)	0.458(3)	0.0280(7)	0.028(7)	
O10	0.823(3)	0.534(3)	0.2571(11)	-0.012(7)	0.5

For details see the attached CIF-file

- Give details about constraints, restraints

No constraints or restraints applied. All heavy atoms refined anisotropically, oxygen atoms left isotropic.

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 think the most difficult part of the structure solution could be the determination of the symmetry. I performed the profile fitting assuming 6/mmm point-group symmetry, and I then used Superflip's option "derivesymmetry" to derive the symmetry from the resulting density. The symmetry -62c came out clearly, and from that point on the structure solution was quite easy. All 10 heavy-atom positions were located in the density obtained by charge flipping (using program EDMA), and the oxygen atoms were located in difference Fourier synthesis. The whole process took about two hours, out of which most of the time took the profile fitting.

Since the challenge has been "structure solution", I didn't spend too much time on getting the best possible profile fit (the asymmetry could be probably described better than I did, for example), nor did I perform detailed analysis of the structure, like trying to go to a subgroup in order to remove the disorder on the W6 position, or taking too much care of absorption correction and negative displacement parameters of a few oxygens. In short, the result I submit is a result of a "routine structure solution", which was, as I understand, the purpose of the challenge.