

QUESTIONNAIRE FOR the  
STRUCTURE DETERMINATION BY POWDER DIFFRACTOMETRY ROUND ROBIN - 2

Sample 1

0.2 Is the second sample structure solvable with this quality  
of data ? Yes [x] No [ ]

1. Preliminary work

1.1 Did you obtained additional informations from the  
chemical formula ? If yes, how and what information ?  
(for instance from CSD or ICSD or ICDD databases)

Yes. Most of necessary information was given on the SDPDRR2 page:

...  
- Cartesian coordinates (.xyz file) for the C<sub>6</sub>N<sub>4</sub>H<sub>20</sub> molecule, as found in  
C<sub>6</sub>N<sub>4</sub>H<sub>20</sub>.Cl<sub>4</sub>  
- The usual behaviour of Al in F environment is to form AlF<sub>6</sub> octahedra  
...

The Al - F distances were estimated from similar compounds from the ICSD as  
being about 1.8 Å.

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 [ ] 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 R<sub>p</sub> and R<sub>wp</sub> (conventional Rietveld, background subtracted):  
1.3.5 Give the R<sub>p</sub> and R<sub>wp</sub> (background not subtracted):  
  
1.3.6 If not, did you use the whole pattern ?  
1.3.7 Or a partial pattern (if yes, give the angular range):

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 ☒ No ☐

- 2.3.1 If yes, which method(s) (give details : molecule location by direct space - genetic algorithm, Monte Carlo, Simulated annealing, scratch, other) ?

Structural units location in direct space by Simulated annealing in the Parallel Tempering mode.

- 2.3.2 Which program(s) did you use ?

FOX.

- 2.3.3 If you used molecule location methods, how many independent molecules did you use (give details on these molecules)? How many degrees of freedom (total) ? How many torsion angles ?

2 octahedrons AlF6 and 1 molecule C6N4H20 :

18 position and rotation parameters of polyhedrons and molecule (considered as degrees of freedom)

+

30 internal parameters of 2 polyhedrons and molecule (distances, bond angles and dihedral angles, not considered as degrees of freedom) with limits  $\pm 0.2$  Å for distances and  $\pm 11.45^\circ$  for angles.

+

24 internal parameters of the molecule (9 bond distances, 8 bond angles and 7 dihedral angles, not considered as degrees of freedom) with limits  $\pm 0.03$  Å for distances and  $\pm 0.57^\circ$  for angles.

No anti-bump distances were used.

Cost-function : Rwp.

Impurity : Al(F,OH)3 x H2O pyrochlore structure.

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) ?

### 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 [ ]

See point 4.

- 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 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

Rietveld refinement with program Fullprof 2000:

```
*****
** PROGRAM FullProf.2k (Version 1.9c - May2001-LLB JRC) **
*****

      M U L T I  -- P A T T E R N
Rietveld, Profile Matching & Integrated Intensity
Refinement of X-ray and/or Neutron Data
```

Date: 14/11/2002 Time: 19:40:42.447

=> PCR file code: sample1\_final  
=> DAT file code: sample1 -> Relative contribution: 1.0000  
=> Title: Dicol solution: 1 (Automatic generated PCR file)

==> CONDITIONS OF THIS RUN FOR PATTERN No.: 1

=> Global Refinement of X-ray powder diffraction data  
=> Global Refinement of X-ray powder diffraction data  
Bragg-Brentano or Debye-Scherrer geometry  
=> The 5th default profile function was selected

=> Data supplied in free format for pattern: 1  
=> Wavelengths: 1.54056 1.54439  
=> Cos(Monochromator angle)= 1.0000  
=> Absorption correction (muR-eff): 0.0000  
=> Base of peaks: 2.0\*HW\* 7.00  
==> Angular range, step and number of points:  
2Thmin: 10.0000 2Thmax: 60.0000 Step: 0.0200 No. of points: 2501  
=>-----> Pattern# 1  
=> Crystal Structure Refinement for phase: 1  
=>-----> Pattern# 1  
=> Crystal Structure Refinement for phase: 2  
=> Scor: 3.2122

==> RESULTS OF REFINEMENT:

=> No. of fitted parameters: 57

-----  
=> Phase No. 1 Al2F10[C6N4H20] P c  
-----

=> No. of reflections for pattern#: 1: 440/2

==> ATOM PARAMETERS:

Name	x	sx	y	sy	z	sz	B	sB	occ.	socc.	Mult
Al1	0.57652( 277)		0.49493( 510)		0.06359( 221)		3.639(315)		1.000( 0)		2
Al2	0.50000( 0)		0.00000( 0)		0.00000( 0)		3.639(315)		1.000( 0)		2
F1	0.51905( 340)		0.73370( 234)		0.51814( 306)		8.155(314)		1.000( 0)		2
F2	-0.51905( 340)		-0.73370( 234)		-0.51814( 306)		8.155(314)		1.000( 0)		2
F3	0.60706( 255)		0.51991( 309)		0.43396( 183)		8.155(314)		1.000( 0)		2
F4	-0.60706( 255)		-0.51991( 309)		-0.43396( 183)		8.155(314)		1.000( 0)		2
F5	0.77741( 223)		0.51722( 442)		0.72546( 181)		8.155(314)		1.000( 0)		2
F6	-0.77741( 223)		-0.51722( 442)		-0.72546( 181)		8.155(314)		1.000( 0)		2
F7	0.22724( 200)		1.00880( 394)		0.86350( 171)		8.155(314)		1.000( 0)		2
F8	-0.22724( 200)		-1.00880( 394)		-0.86350( 171)		8.155(314)		1.000( 0)		2
F9	0.54388( 254)		0.02611( 346)		0.16292( 222)		8.155(314)		1.000( 0)		2
F10	-0.54388( 254)		-0.02611( 346)		-0.16292( 222)		8.155(314)		1.000( 0)		2
C1	0.07853( 387)		0.39203( 232)		0.04331( 376)		1.346(339)		1.000( 0)		2

C2	-0.07853( 387)	-0.39203( 232)	-0.04331( 376)	1.346(339)	1.000( 0)	2
C3	0.26419( 378)	0.71748( 452)	0.11184( 308)	1.346(339)	1.000( 0)	2
C4	-0.26419( 378)	-0.71748( 452)	-0.11184( 308)	1.346(339)	1.000( 0)	2
C5	0.08213( 445)	0.17806( 293)	-0.01817( 360)	1.346(339)	1.000( 0)	2
C6	-0.08213( 445)	-0.17806( 293)	0.01817( 360)	1.346(339)	1.000( 0)	2
N7	0.09981( 303)	0.72346( 360)	0.94279( 247)	1.346(339)	1.000( 0)	2
N8	-0.09981( 303)	-0.72346( 360)	-0.94279( 247)	1.346(339)	1.000( 0)	2
N9	0.31686( 614)	0.24502( 685)	0.18466( 550)	1.346(339)	1.000( 0)	2
N10	-0.02359( 569)	-0.26928( 604)	0.17050( 412)	1.346(339)	1.000( 0)	2

==> PROFILE PARAMETERS FOR PATTERN# 1

=> Cell parameters :

	10.32579	0.00086
	7.39600	0.00061
	13.24265	0.00107
	90.00000	0.00000
	139.90088	0.00229
	90.00000	0.00000

=> overall scale factor : 0.001620568 0.000028626

=> Eta(p-v) or m(p-vii) : 0.44592 0.03592

=> Overall tem. factor : 0.00000 0.00000

=> Halfwidth parameters : 0.06693 0.00000

-0.00790 0.00000

0.01358 0.00035

=> Preferred orientation: 0.00000 0.00000

0.00000 0.00000

=> Asymmetry parameters : 0.01914 0.00552

0.00864 0.00142

0.00000 0.00000

0.00000 0.00000

=> X and y parameters : 0.01252 0.00140

0.00000 0.00000

=> Strain parameters : 0.00000 0.00000

0.00000 0.00000

0.00000 0.00000

=> Size parameters (G,L): 0.00000 0.00000

0.00000 0.00000

-----  
=> Phase No. 2 Al(F,OH)3.xH2O - pyrochlore F d 3 m  
-----

=> No. of reflections for pattern#: 1: 32/2

==> ATOM PARAMETERS:

Name	x	sx	y	sy	z	sz	B	sB	occ.	socc.	Mult
F	0.31250( 0)		0.12500( 0)		0.12500( 0)		1.000( 0)		0.125( 0)		48
OH1	0.31250( 0)		0.12500( 0)		0.12500( 0)		1.000( 0)		0.125( 0)		48
Al	0.00000( 0)		0.00000( 0)		0.00000( 0)		1.000( 0)		0.083( 0)		16
OH2	0.37500( 0)		0.37500( 0)		0.37500( 0)		1.000( 0)		0.031( 0)		8

==> PROFILE PARAMETERS FOR PATTERN# 1

```

=> Cell parameters      :
      9.82571    0.00121
      9.82571    0.00121
      9.82571    0.00121
      90.00000    0.00000
      90.00000    0.00000
      90.00000    0.00000

=> overall scale factor :    0.000109303    0.000001668
=> Eta(p-v) or m(p-vii) :    1.19177    0.11524
=> Overall tem. factor  :    4.91889    0.49050
=> Halfwidth parameters :    0.50181    0.04821
      -0.00790    0.00000
      0.00985    0.00129
=> Preferred orientation:    0.00000    0.00000
      0.00000    0.00000
=> Asymmetry parameters :    0.00000    0.00000
      0.00000    0.00000
      0.00000    0.00000
      0.00000    0.00000
=> X and y parameters   :   -0.03092    0.00633
      0.00000    0.00000
=> Strain parameters     :    0.00000    0.00000
      0.00000    0.00000
      0.00000    0.00000
=> Size parameters (G,L):    0.00000    0.00000
      0.00000    0.00000

==> GLOBAL PARAMETERS FOR PATTERN#  1

=> Zero-point:          0.0130    0.0021
=> Cos( theta)-shift parameter :   -0.0147    0.0000
=> Sin(2theta)-shift parameter :    0.0000    0.0000

==> RELIABILITY FACTORS WITH ALL NON-EXCLUDED POINTS FOR PATTERN:  1

=> Cycle:  1 => MaxCycle:  1
=> N-P+C:  2444
=> R-factors (not corrected for background) for Pattern:  1
=> Rp: 3.65    Rwp: 4.78    Rexp:  2.35 Chi2:  4.13    L.S. refinement
=> Conventional Rietveld R-factors for Pattern:  1
=> Rp: 20.2    Rwp: 16.4    Rexp:  8.09 Chi2:  4.13
=> Deviance: 0.101E+05    Dev*   :  4.121
=> DW-Stat.:  0.5549    DW-exp:  1.9223
=> N-sigma of the GoF:  109.417

==> RELIABILITY FACTORS FOR POINTS WITH BRAGG CONTRIBUTIONS FOR PATTERN:  1

=> N-P+C:  2314
=> R-factors (not corrected for background) for Pattern:  1
=> Rp: 3.71    Rwp: 4.84    Rexp:  2.34 Chi2:  4.26    L.S. refinement
=> Conventional Rietveld R-factors for Pattern:  1
=> Rp: 19.5    Rwp: 16.3    Rexp:  7.87 Chi2:  4.26
=> Deviance: 0.985E+04    Dev*   :  4.258
=> DW-Stat.:  0.5677    DW-exp:  1.9215
=> N-sigma of the GoF:  111.015

```

```
=> Global user-weighted Chi2 (Bragg contrib.):4.36
=> Phase: 1
=> Bragg R-factor: 12.4          Vol: 651.413( 0.093)  Fract(%): 92.26( 2.26)
=> Rf-factor= 10.5             ATZ: 800.110          Brindley: 1.0000
=> Phase: 2
=> Bragg R-factor: 7.04          Vol: 948.619( 0.203)  Fract(%): 7.74( 0.49)
=> Rf-factor= 5.22             ATZ: 2735.260          Brindley: 1.0000
```

```
CPU Time:      2.886 seconds
              0.048 minutes
```

```
=> Run finished at:      Date: 14/11/2002  Time: 19:40:45.341
```

5-

The setting of the monoclinic cell giving the greater monoclinic angle was used for keeping the standardized setting (cell choice no. 1) of the space groups according to ITC.

The structure was solved in the centrosymmetric space group P2/c. When it was recognized that the chiral molecule is placed on the inversion center, the model was transformed to the non-centrosymmetric space group Pc and refined.

For a lack of time and also not to spend too much time on refinement of the structure already refined (this is the structure solution RR and not structure refinement RR) the constrained was not set-up very properly. Correctly one should refine the octahedrons AlF<sub>6</sub> and the C<sub>6</sub>N<sub>4</sub>H<sub>20</sub> molecule as semi-rigid bodies, because of low quality of the data. We have just constrained all the structure with the exception of the atoms N9 and N10 as having the symmetry of the inversion center. The inversion-center related pairs:

```
F1 - F2
F3 - F4
F5 - F6
F7 - F8
F9 - F10
C1 - C2
C3 - C4
C5 - C6
N7 - N8
```

Therefore the precision of our interatomic distances is rather low.

Sample 1





