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

Sample 2

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. The coordination of the cations (V, Sr) was estimated from the chemical formula and comparison with similar compounds from the ICSD.

The coordination of V was estimated as (a) tetrahedral V(O,F,OH)4 or (b) octahedral V(O,F,OH)6 with V - (O,F,OH) distances of about 1.7 Å.

The coordination of Sr was roughly modeled as a cube Sr(O,F,OH)8 with Sr - (O,F,OH) distances of about 2.7 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 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 ?
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 [x] No [ ]

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

a) 3 tetrahedrons VO4 and 5 cubes SrO8 :
48 position and rotation parameters of polyhedrons (considered as degrees of freedom) +
147 internal parameters of polyhedrons (distances, bond angles and dihedral angles, not considered as degrees of freedom) with limits +- 0.2 Å for distances and +- 11.45° for angles.
Three anti-bump distances were used, V-O = 1.6 Å, Sr-O = 2.5 Å and V-Sr = 3 Å.

Cost-function : 0.66 Rwp + 0.33 AntiBump

b) 3 octahedrons VO6 and 5 cubes SrO8 :
48 position and rotation parameters of polyhedrons (considered as degrees of freedom) +
165 internal parameters of polyhedrons (distances, bond angles and dihedral angles, not considered as degrees of freedom) with limits +- 0.2 Å for distances and +- 11.45° for angles.
Three anti-bump distances were used, V-O = 1.6 Å, Sr-O = 2.5 Å and V-Sr = 3 Å.

Cost-function : 0.66 Rwp + 0.33 AntiBump

Both modeling have yielded the same and correct positions for cations (V and Sr).

The positions of anions (O) were determined by third modeling using free atoms:

c) 3 V and 5 Sr atoms fixed in the positions found from (a) and (b) and 22 O atoms that were left to move :
66 position parameters for O atoms.
Two anti-bump distances were used, V-O = 1.6 Å and Sr-O = 2.5 Å.

Cost-function : 0.5 Rwp + 0.5 AntiBump

All O atoms were localized in correct positions by (c) modeling.

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) ?

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 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

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: 11:44:42.429

 => PCR file code: sample2
 => DAT file code: sample2              -> Relative contribution: 1.0000
 => Title: Dicvol 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:  0.79764 9.79764
 => Cos(Monochromator angle)=   0.6400
 => Absorption correction (muR-eff):   0.0000
 => Base of peaks: 2.0*HW*    7.00
 ==> Angular range, step and number of points:
     2Thmin:   1.5000  2Thmax:  80.0000  Step:   0.0050  No. of points:  15701
 =>-------> Pattern#  1
 => Crystal Structure Refinement for phase: 1
 => Scor: 2.1331

 ==> RESULTS OF REFINEMENT:


 => No. of fitted parameters:  100


------------------------------------------------------------------------------
 => Phase No.  1 Sr5V3(O/F/OH/H2O)22                    P 21/c
------------------------------------------------------------------------------

 =>  No. of reflections for pattern#:   1:  7926/2


 ==> ATOM PARAMETERS:

  Name    x     sx       y     sy       z      sz      B   sB  occ.  socc.  Mult
```

V1	0.23450(26)	0.92171(40)	0.47585(14)	1.348(30)	1.000(0)	4
V2	0.09986(26)	0.58025(42)	0.67301(14)	1.348(30)	1.000(0)	4
V3	0.57661(25)	0.58441(43)	0.65909(13)	1.348(30)	1.000(0)	4
Sr1	0.74054(13)	0.58909(22)	0.48234(7)	0.814(9)	1.000(0)	4
Sr2	0.49254(12)	0.91648(25)	0.35347(7)	0.814(9)	1.000(0)	4
Sr3	0.83506(16)	0.16601(18)	0.17103(9)	0.814(9)	1.000(0)	4
Sr4	0.15641(16)	0.15522(18)	0.32488(9)	0.814(9)	1.000(0)	4
Sr5	0.85021(13)	0.58486(23)	0.85298(7)	0.814(9)	1.000(0)	4
O1	0.92371(70)	0.42418(119)	0.10780(39)	0.091(30)	1.000(0)	4
O2	0.16630(68)	0.92841(115)	0.11053(39)	0.091(30)	1.000(0)	4
O3	0.77422(80)	0.65630(97)	0.04581(42)	0.091(30)	1.000(0)	4
O4	0.07560(84)	0.15026(100)	0.18695(46)	0.091(30)	1.000(0)	4
O5	0.60807(80)	0.14836(98)	0.18584(42)	0.091(30)	1.000(0)	4
O6	0.33905(77)	0.62438(90)	0.17003(42)	0.091(30)	1.000(0)	4
O7	0.76372(68)	0.59512(121)	0.72471(39)	0.091(30)	1.000(0)	4
O8	0.75823(65)	0.41605(128)	0.24464(39)	0.091(30)	1.000(0)	4
O9	0.47892(72)	0.37431(96)	0.26010(43)	0.091(30)	1.000(0)	4
O10	0.91541(86)	0.18145(102)	0.31015(47)	0.091(30)	1.000(0)	4
O11	0.61132(78)	0.81903(104)	0.64982(42)	0.091(30)	1.000(0)	4
O12	0.00973(70)	0.58733(108)	0.75423(40)	0.091(30)	1.000(0)	4
O13	0.07341(69)	0.41493(127)	0.38241(39)	0.091(30)	1.000(0)	4
O14	0.70226(69)	0.10295(111)	0.60640(37)	0.091(30)	1.000(0)	4
O15	0.86628(67)	0.06351(111)	0.46262(39)	0.091(30)	1.000(0)	4
O16	0.05388(79)	0.21615(100)	0.02731(44)	0.091(30)	1.000(0)	4
O17	0.69031(79)	0.77385(94)	0.38467(43)	0.091(30)	1.000(0)	4
O18	0.51796(80)	0.72060(99)	0.46773(44)	0.091(30)	1.000(0)	4
O19	0.62188(67)	0.06706(113)	0.46512(38)	0.091(30)	1.000(0)	4
O20	0.32677(72)	0.03142(99)	0.90850(42)	0.091(30)	1.000(0)	4
O21	0.57171(68)	0.42847(120)	0.40420(38)	0.091(30)	1.000(0)	4
O22	0.20150(74)	0.68332(97)	0.46742(44)	0.091(30)	1.000(0)	4

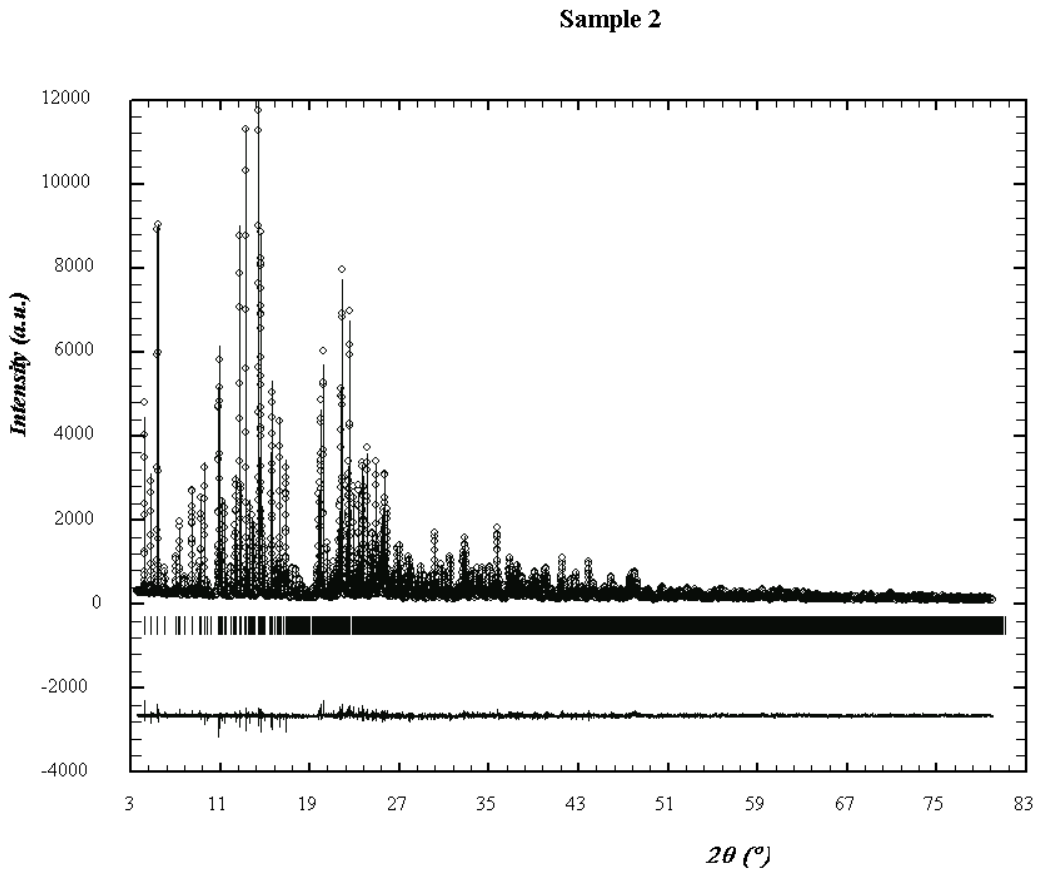
==> PROFILE PARAMETERS FOR PATTERN# 1

=> Cell parameters :	11.24286	0.00002
	8.19539	0.00002
	19.94848	0.00004
	90.00000	0.00000
	106.72866	0.00017
	90.00000	0.00000
=> overall scale factor :	0.000008395	0.000000015
=> Eta(p-v) or m(p-vii) :	0.80355	0.00000
=> Overall tem. factor :	0.00000	0.00000
=> Halfwidth parameters :	-0.00053	0.00000
	0.00205	0.00000
	0.00021	0.00000
=> 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 :	-0.02002	0.00000
	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:     0.0004    0.0000
 => Background Polynomial Parameters ==>
                               175.161    0.276645
                              -36.6960    0.254572
                               37.6840     0.00000
                              -62.0330     0.00000
                               38.7650     0.00000
                              -8.84290     0.00000

 => Cos( theta)-shift parameter :   0.0000  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: 15194
 =>  R-factors (not corrected for background) for Pattern:  1
 => Rp: 6.02     Rwp: 7.95     Rexp:    5.50 Chi2:  2.09      L.S. refinement
 => Conventional Rietveld R-factors for Pattern:  1
 => Rp: 12.3     Rwp: 13.5     Rexp:    9.33 Chi2:  2.09
 => Deviance: 0.325E+05     Dev*  :  2.140
 => DW-Stat.:    1.0098     DW-exp:     1.9631
 => N-sigma of the GoF:   94.859

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

 => N-P+C: 14374
 =>  R-factors (not corrected for background) for Pattern:  1
 => Rp: 6.06     Rwp: 8.01     Rexp:    5.47 Chi2:  2.14      L.S. refinement
 => Conventional Rietveld R-factors for Pattern:  1
 => Rp: 11.9     Rwp: 13.3     Rexp:    9.08 Chi2:  2.14
 => Deviance: 0.316E+05     Dev*  :  2.198
 => DW-Stat.:    1.0394     DW-exp:     1.9624
 => N-sigma of the GoF:       97.038

 => Global user-weigthed Chi2 (Bragg contrib.):2.21
 => Phase:  1
 => Bragg R-factor:  7.20       Vol: 1760.256( 0.006)  Fract(%):  100.00( 0.25)
 => Rf-factor= 10.2             ATZ: 3771.640          Brindley:  1.0000


              CPU Time:    75.320 seconds
                           1.255 minutes

 => Run finished at:     Date: 14/11/2002  Time: 11:45:57.728
```

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...).

Lachlan has large disk space.



Trials were made during the Rietveld refinement to decide which O atom is O, OH, H_2O or F by using the Bond Valences and interatomic distances. The Bond Valences sums and interatomic distances were calculated with Fullprof 2000 and the O atoms not summing to 2(+-0.2) were analyzed for a possibility to construct a hydrogen bonds network or to localize F atoms. No reliable solution was found.