Sample 1

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)

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 [ ] 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 ? Yes
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) ?

Molecule location in direct space, Simulated annealing, structure determination using step intensity data

2.3.2 Which program(s) did you use ? TOPAS

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 ?

- Start with 3 „rigid" bodies (including 2 for AlF6 octahedra). For the molecule 3 rot and 3 trans degrees of freedom and 4 torsion angles were used.

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

Not applicable. Structure determined using step intensity data.

3.5 Did you made first Rietveld refinements without preliminary Fourier difference syntheses ? Yes [] No []
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
.....			
.....			
.....			

Not applicable. Structure determined using step intensity data. Final refinement to bring profile shapes in.

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

.....

.....

.....

Not applicable. Structure determined using step intensity data. Final refinement to bring profile shapes in.

4- Final refinement

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

SG: Pc
a 8.53440
b 7.39785
c 13.24539
be 128.77996
R_exp 2.347
R_exp(background subtracted) 3.739
R_wp 5.787
R_wp(background subtracted) 9.221
R_p 4.267
R_p(background subtracted) 7.439
Weighted_Durbin_Watson 0.383
gof 2.466
Rbragg 9.58

Atom	x	y	z	B
Al1	0.78452	0.48269	0.73825	2.1502
F1	0.94789	0.52828	0.91257	2.1502
F2	0.75303	0.53019	0.58449	2.1502
F3	0.80356	0.73792	0.78921	2.1502
F4	0.04570	0.47554	0.79229	2.1502

F5	0.52717	0.44834	0.68172	2.1502
Al2	0.82934	0.98781	0.76584	2.1502
F6	0.83379	0.95480	0.63160	2.1502
F7	0.76047	0.95832	0.87402	2.1502
F8	0.10806	0.01494	0.86721	2.1502
F9	0.60276	0.12389	0.64083	2.1502
F10	0.87791	0.24048	0.79642	2.1502
C1	0.29625	0.44648	0.74786	2.1502
C2	0.30655	0.62873	0.74921	2.1502
C3	0.45607	0.73457	0.87097	2.1502
C4	0.43343	0.33378	0.86802	2.1502
C5	0.16937	0.74143	0.62905	2.1502
C6	0.14673	0.34064	0.62610	2.1502
N7	0.63682	0.78572	0.88341	2.1502
N8	0.59393	0.24363	0.87208	2.1502
N9	0.24148	0.75215	0.55151	2.1502
N10	0.24731	0.20898	0.59768	2.1502
H11	0.70673	0.68681	0.89647	2.1502
H12	0.67812	0.32671	0.88211	2.1502
H13	0.71342	0.86074	0.95008	2.1502
H14	0.66114	0.16647	0.93816	2.1502
H15	0.59798	0.84011	0.81105	2.1502
H16	0.53814	0.18398	0.79837	2.1502
H17	0.03552	0.68950	0.57608	2.1502
H18	0.06604	0.42362	0.55389	2.1502
H19	0.49793	0.66269	0.94539	2.1502
H20	0.49539	0.41028	0.94370	2.1502
H21	0.35516	0.81544	0.59507	2.1502
H22	0.30535	0.12229	0.65744	2.1502
H23	0.14871	0.80624	0.47625	2.1502
H24	0.15633	0.16023	0.51992	2.1502
H25	0.26355	0.64113	0.53702	2.1502
H26	0.34018	0.26601	0.59877	2.1502
H27	0.39215	0.84325	0.87061	2.1502
H28	0.35486	0.24246	0.87100	2.1502
H29	0.16101	0.86245	0.65354	2.1502
H30	0.05738	0.27580	0.63518	2.1502

- Give details about constraints, restraints

Bond distances Al-F were restrained (using Lenard-Jones potentials) to be about 1.8A