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 [x] No

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, 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

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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 perform 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 obtain from Fourier difference syntheses?
3.4 Give their name and atomic coordinates as they were obtained:

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Not applicable. Structure determined using step intensity data.

3.5 Did you make 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?

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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 synthesis 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.

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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
e  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

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<tr>
<td>A1</td>
<td>0.78452</td>
<td>0.48269</td>
<td>0.73825</td>
<td>2.1502</td>
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<tr>
<td>F1</td>
<td>0.94789</td>
<td>0.52828</td>
<td>0.91257</td>
<td>2.1502</td>
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<tr>
<td>F2</td>
<td>0.75303</td>
<td>0.53019</td>
<td>0.58449</td>
<td>2.1502</td>
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<tr>
<td>F3</td>
<td>0.80356</td>
<td>0.73792</td>
<td>0.78921</td>
<td>2.1502</td>
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<tr>
<td>F4</td>
<td>0.04570</td>
<td>0.47554</td>
<td>0.79229</td>
<td>2.1502</td>
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- Give details about constraints, restraints
Bond distances Al-F were restrained (using Lenard-Jones potentials) to be about 1.8 Å.