Sample 1

Solving that structure from the poor powder data set distributed with this SDPD Round Robin 2 was not found to be possible before the $C_6N_4H_{20}$ molecular formula was established. But, once that molecule known, the structure was solved by using the ESPOIR 3.5 program, testing several hypotheses. The winning hypothesis was that the formula could be $Al_2F_{10}[C_6N_4H_{20}]$ with simple infinite chains of AlF_6 octahedra sharing trans-corners. ESPOIR was used in molecule location mode, describing the model with 3 objects : the $C_6N_4H_{20}$ molecule, an AlF_6 complete octahedron and an AlF_4 square plane, expecting that this square plane would go just between two AlF_6 octahedra in order to complete its own octahedron. This is exactly what happened. For avoiding the inversion center problems, the structure was searched in the Pc space group, using the first 100 hkl from the extracted structure factor amplitudes list.

ESPOIR data :

```
Al2F10 C6N4 ???
8.528655   7.394635  13.237077  90.0 128.765686  90.0
P C
1.54056 4 22 4 3 1 3
 0.01362   0.06704   0.00517   3
Al+3F-1 C   N
0
13. 13. 13. 13.
1.5 1.0 0.01
20000 600000 200000
300000 0.35 2 300
2 4                                !!! the AlF6 octahedron
1 6 0 0
1.5 0 0
10. 10. 10. 90. 90. 90.
0.0    0.0    0.00  1.00
0.18   0.0    0.00  1.00
-.18   0.00   0.00  1.00
0.00   0.18   0.00  1.00
0.00  -0.18   0.00  1.00
0.00   0.00   0.18  1.00
0.00   0.00  -0.18  1.00
2 4                                !!!  The AlF4 square plane
1 4 0 0
1.5 0 0
10. 10. 10. 90. 90. 90.
0.0    0.0    0.00  1.00
0.00   0.18   0.00  1.00
0.00  -0.18   0.00  1.00
0.00   0.00   0.18  1.00
0.00   0.00  -0.18  1.00
2 4                                !!!  The C6N4 molecule without H atoms
0 0 6 4
2.5 0 0
11.655 8.161 6.845 90.0000 101.54 90.0000
   0.50000   0.58274   0.00000    1.00
   0.50000   0.41726   0.00000    1.00
   0.60726   0.31804   0.08470    1.00
   0.60726   0.68196   0.08470    1.00
   0.39274   0.31804  -0.08470    1.00
   0.39274   0.68196  -0.08470    1.00
   0.60108   0.24623   0.28308    1.00
   0.60108   0.75377   0.28308    1.00
   0.39892   0.24623  -0.28308    1.00
   0.39892   0.75377  -0.28308    1.00
```

The best result is below :

```
Test number : 257
 18-Aug-2002     10 hour 17 min 46 Sec
```

```
  Object number  1 at test257.  Previous minimum R=0.276 at test257
     12 rot. acc.  150555 gen. and  150555 tested; Chi**2=0.367    , R=0.276
      3 trans. acc.   50184 tested
      5 events did not improve the fit, DAMP = 0.009998
  Object number  2 at test257.  Previous minimum R=0.276 at test257
     10 rot. acc.  149443 gen. and  149443 tested; Chi**2=0.367    , R=0.276
      3 trans. acc.   49813 tested
      1 events did not improve the fit, DAMP = 0.009998
  Object number  3 at test257.  Previous minimum R=0.276 at test257
      5 rot. acc.  150004 gen. and  150004 tested; Chi**2=0.367    , R=0.276
      9 trans. acc.   50000 tested
      5 events did not improve the fit, DAMP = 0.009998

 Final coordinates x,y,z and occupation numbers

Al1      0.00419    0.52987    0.49370      1.000
F1      -0.03287    0.54333    0.61324      1.000
F2       0.04125    0.51640    0.37416      1.000
F3       0.02923    0.28806    0.51310      1.000
F4      -0.02085    0.77167    0.47430      1.000
F5       0.27115    0.55442    0.61920      1.000
F6      -0.26277    0.50531    0.36821      1.000
Al2      0.00782    0.99247    0.01983      1.000
F7      -0.10492    0.93725   -0.14537      1.000
F8       0.12055    1.04770    0.18502      1.000
F9      -0.23815    1.02505   -0.02563      1.000
F10      0.25378    0.95990    0.06529      1.000
C1       0.44135    0.59381    0.45767      1.000
C2       0.47792    0.41424    0.46958      1.000
C3       0.34444    0.28373    0.35994      1.000
C4       0.26402    0.67864    0.33377      1.000
C5       0.65524    0.32941    0.59348      1.000
C6       0.57482    0.72432    0.56731      1.000
N1       0.43632    0.22320    0.29973      1.000
N2       0.32416    0.77397    0.26322      1.000
N3       0.59510    0.23408    0.66403      1.000
N4       0.48294    0.78485    0.62752      1.000
 18-Aug-2002     10 hour 20 min 10 Sec
```

With R = 27.6%, this is not a fantastically convincing model, but the fact is that the AlF_4 square plane was now placed in the neighbouring of the AlF_6 octahedra, forming an infinite chain of corner sharing octahedra running along the b axis. Very interesting…

Therefore, the model was introduced into the Rietveld method (FULPROF) for refinement. This was done by considering that the P2/c was highly probable since in the Pc description above, the two Al atoms and the center of the C_6N_4 part of the molecule were fully compatible with being on inversion centers.

Below is the result of the Rietveld refinement (.sum Fullprof file) :

```
        *******************************************************
        ** 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: 17/07/2002  Time: 20:21:21.150

 => PCR file code: saupoudre
 => DAT file code: saupoudre            -> Relative contribution: 1.0000
 => Title: Saupoudre...

 ==> 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)=   0.7998
 => 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: 3.7248

 ==> RESULTS OF REFINEMENT:


 => No. of fitted parameters:   40


-------------------------------------------------------------------------------
 => Phase No.  1 phase tetra insaturée                    P 2/C
-------------------------------------------------------------------------------

 =>  No. of reflections for pattern#:   1:   450/2


 ==> ATOM PARAMETERS:

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

 AL1   0.00000(   0)  0.50000(   0)  0.50000(   0)  0.800(  0)  0.500(  0)     2
 F1    0.21210( 200)  0.46261( 266)  0.52098( 138)  0.800(  0)  1.000(  0)     4
 F2   -0.02668( 263)  0.75739( 181)  0.51516( 185)  0.800(  0)  1.000(  0)     4
 F3    0.09673( 217)  0.46819( 244)  0.65852( 128)  0.800(  0)  1.000(  0)     4
 AL2   0.00000(   0)  0.00000(   0)  0.50000(   0)  0.800(  0)  0.500(  0)     2
 F4    0.03290( 206)  0.02603( 280)  0.37203( 113)  0.800(  0)  1.000(  0)     4
 F5    0.26070( 163)  0.00455( 313)  0.61538( 128)  0.800(  0)  1.000(  0)     4
 C1    0.54111( 566)  0.07997( 275)  0.02192( 355)  0.800(  0)  1.000(  0)     4
 C2    0.75978( 348) -0.24710( 421)  0.13014( 236)  0.800(  0)  1.000(  0)     4
 C3    0.61739( 531)  0.19097( 394)  0.11913( 341)  0.800(  0)  1.000(  0)     4
 N1    0.62993( 383) -0.22678( 429)  0.14584( 283)  0.800(  0)  1.000(  0)     4
 N2    0.57043( 339)  0.28904( 278)  0.19224( 218)  0.800(  0)  1.000(  0)     4

 ==> PROFILE PARAMETERS FOR PATTERN#  1

 => Cell parameters      :
                            8.52842   0.00040
                            7.39318   0.00043
                           13.23733   0.00068
                           90.00000   0.00000
                          128.77281   0.00371
                           90.00000   0.00000

 => overall scale factor :    0.001272333   0.000009859
 => Eta(p-v) or m(p-vii) :    0.81889   0.02111
 => Overall tem. factor  :    0.00000   0.00000
 => Halfwidth parameters :    0.01362   0.00000
                              0.06704   0.00000
                              0.00333   0.00040
 => Preferred orientation:    0.00000   0.00000
                              0.00000   0.00000
 => Asymmetry parameters :    0.00553   0.00000
                              0.00332   0.00000
                              0.00000   0.00000
                              0.00000   0.00000
 => X and y parameters   :    0.00000   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

-------------------------------------------------------------------------------
 => Phase No.  2 Pyrochlore                               F D 3 M
-------------------------------------------------------------------------------
```

=> No. of reflections for pattern#:   1:   30/2

==> ATOM PARAMETERS:

Name	x	sx	y	sy	z	sz	B	sB	occ.	socc.	Mult
Al	0.00000	0	0.00000	0	0.00000	0	0.800	0	0.160	0	16
F	0.31660	0	0.12500	0	0.12500	0	1.800	0	0.480	0	48

==> PROFILE PARAMETERS FOR PATTERN# 1

=> Cell parameters :	9.82038	0.00044
	9.82038	0.00044
	9.82038	0.00044
	90.00000	0.00000
	90.00000	0.00000
	90.00000	0.00000
=> overall scale factor :	0.000021890	0.000000269
=> Eta(p-v) or m(p-vii) :	0.90489	0.02956
=> Overall tem. factor :	0.00000	0.00000
=> Halfwidth parameters :	0.01362	0.00000
	0.06704	0.00000
	0.00333	0.00040
=> Preferred orientation:	0.00000	0.00000
	0.00000	0.00000
=> Asymmetry parameters :	0.00553	0.00000
	0.00332	0.00000
	0.00000	0.00000
	0.00000	0.00000
=> X and y parameters :	0.00000	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: -0.0068 0.0000
=> 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: 10 => MaxCycle: 10
=> N-P+C: 2461
=> R-factors (not corrected for background) for Pattern: 1
=> Rp: 4.25   Rwp: 5.89   Rexp: 2.36 Chi2: 6.22   M.L. refinement
=> Conventional Rietveld R-factors for Pattern: 1
=> Rp: 22.0   Rwp: 19.6   Rexp: 7.86 Chi2: 6.22
=> Deviance: 0.152E+05   Dev* : 6.164
=> DW-Stat.:   0.3710   DW-exp: 1.9082
=> N-sigma of the GoF: 183.263

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

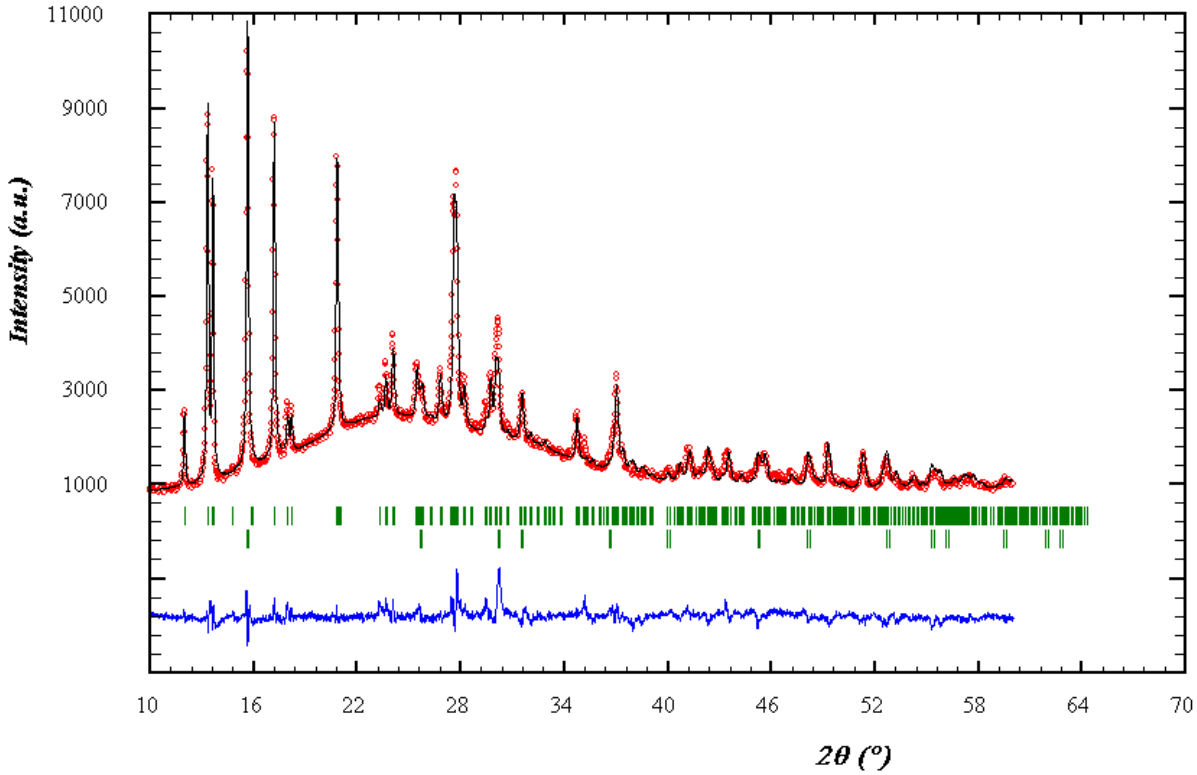
=> N-P+C: 2336
=> R-factors (not corrected for background) for Pattern: 1
=> Rp: 4.34   Rwp: 5.99   Rexp: 2.35 Chi2: 6.48   M.L. refinement
=> Conventional Rietveld R-factors for Pattern: 1
=> Rp: 21.4   Rwp: 19.5   Rexp: 7.66 Chi2: 6.48
=> Deviance: 0.150E+05   Dev* : 6.422
=> DW-Stat.:   0.3751   DW-exp: 1.9067
=> N-sigma of the GoF: 187.444

=> Global user-weigthed Chi2 (Bragg contrib.):6.56
=> Phase: 1
=> Bragg R-factor: 14.1   Vol: 650.717(0.059) Fract(%):   85.71(0.88)
=> Rf-factor= 13.0   ATZ: 744.080   Brindley: 1.0000
=> Phase: 2
=> Bragg R-factor: 12.2   Vol: 947.076(0.074) Fract(%):   14.29(0.11)
=> Rf-factor= 8.26   ATZ: 1343.630   Brindley: 1.0000

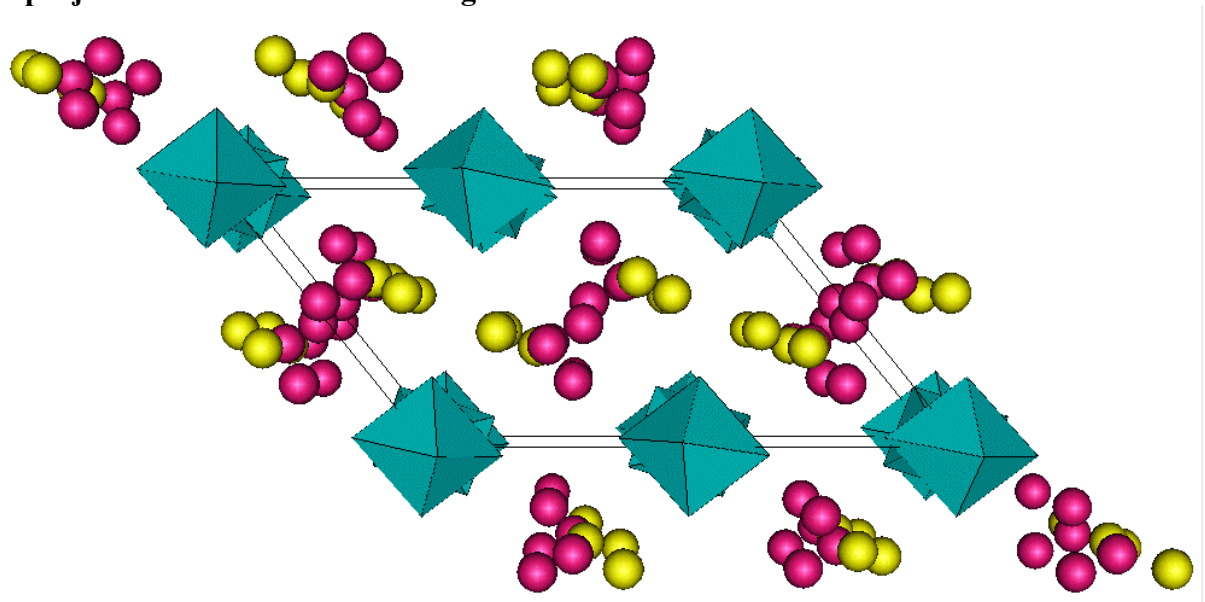
```
                CPU Time:     42.842 seconds
                               0.714 minutes

 => Run finished at:      Date: 17/07/2002  Time: 20:22:04.050
```

The final plot :



A projection of the structure anong the b axis :



CONCLUSION

More work is needed for adding the numerous H atoms, using distance constraints. The current interatomic distances are quite bad. Also, it has to be verified that the centrosymmetric P2/c is the good one instead of Pc. This is certainly possible for the $C_6N_4H_{20}$ molecule which was found to have an inversion center at its gravity center in the $C_6N_4H_{20}.Cl_4$ compound crystal structure. Having a better powder pattern would help… Anyway, this was certainly a good choice for testing all these direct space structure solution by molecule location programs, don't you think so ?