$ZnAlF_5$·[TAZ]: an Al fluorinated MOF of MIL-53(Al) topology with cationic {Zn(1,2,4 triazole)}$^{2+}$ linkers.

Amandine Cadiau, Charlotte Martineau, Marc Leblanc, Vincent Maisonneuve, Annie Hémon-Ribaud, Francis Taulelle, Karim Adil

Electronic Supplementary Information

Experimental.

1. Synthesis. $ZnAlF_5$·[TAZ] has been synthesized from a stoechiometric mixture of ZnO (Merk), $Al(OH)_3$ (Merk), 1,2,4 triazole (Aldrich), hydrofluoric acid solution (40% HF, Prolabo) and water. The hydrothermal reaction has been performed at 160°C in a microwave oven (CEM Mars) for 1 h and the obtained solid powder has been washed with water and dried at room temperature.

2. X-ray diffraction. The powder X-ray diffraction pattern of $ZnAlF_5$·[TAZ] has been recorded at room temperature under air in a Bragg-Brentano geometry with a PANalytical MPD-PRO diffractometer using a CuK_α radiation in the $4 - 99° 2\theta$ range and a 0.017° interpolated step.

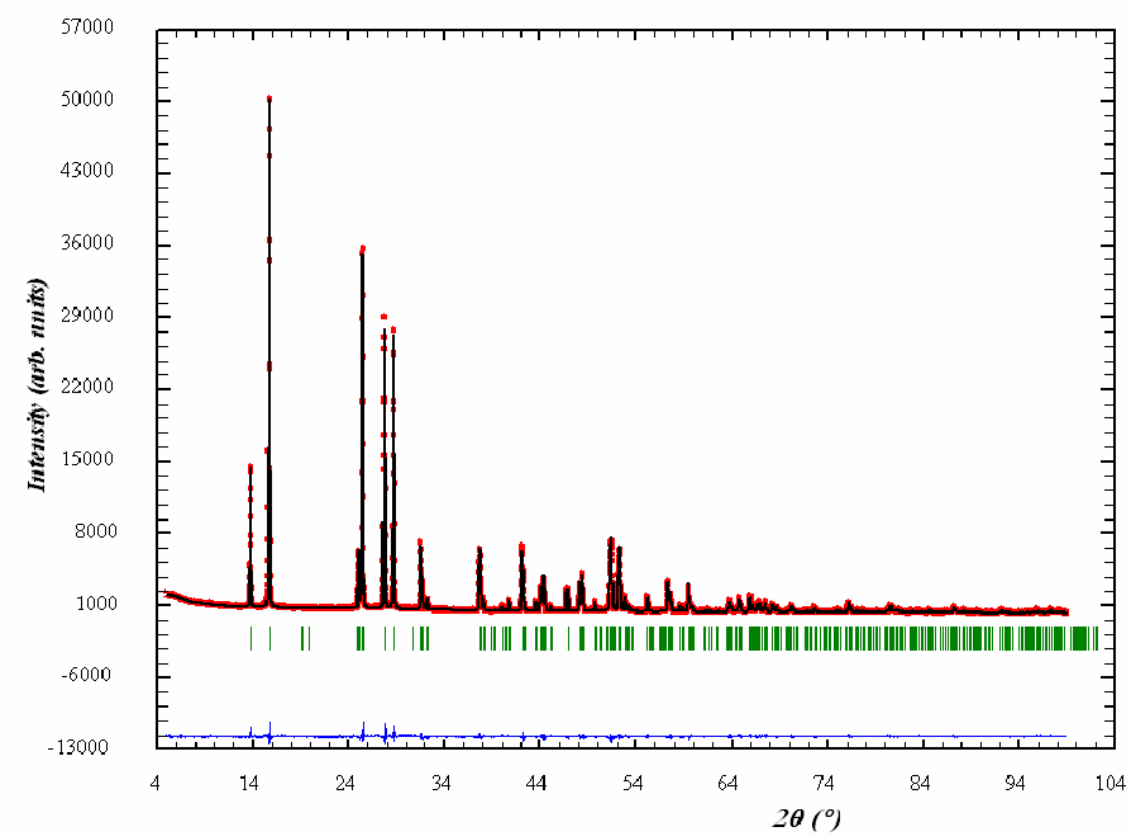
3. NMR experiments. The ^1H, ^{13}C ^{19}F and ^{27}Al (respective Larmor frequencies of 500.1, 125.76, 470.6 and 130.3 MHz) MAS NMR spectra of $ZnAlF_5$·[TAZ] were collected on an Avance 500 Bruker spectrometer (B_0 = 11.7 T) using 2.5 or 3.2 mm CP-MAS probes. The ^{19}F magic angle spinning (MAS) Hahn—echo spectrum of $ZnAlF_5$·[TAZ] was recorded at spinning frequency of 30 kHz, using 3.0 μs 90° pulses, an inter–pulse delay synchronized

with the rotor frequency and recycle delays of 20 s. The 2D ^{19}F double-quantum single-quantum (DQ—SQ) MAS correlation spectrum was recorded at a 30 kHz spinning frequency using the variant of the back—to—back (BABA)[i] pulse sequence spanning on multiple of two rotor periods to excite and reconvert the double—quantum coherences. 3.0 μs 90° pulses and excitation—reconversion delays of 66.67 μs (corresponding to two rotor periods) were used. 50 rotor—synchronized t_1 increments were accumulated with 16 transients each. Phase sensitive detection in the indirect dimension was obtained using the States method.[ii] The ^1H MAS (25 kHz) Hahn—echo spectrum of $ZnAlF_5$·[TAZ] was recorded using 2.5 μs 90° pulses, an inter–pulse delay synchronized with the rotor frequency and recycle delays of 5 s. The 2D ^1H DQ—SQ MAS (25 kHz) NMR correlation spectrum of $ZnAlF_5$·[TAZ] was recorded using the BABA recoupling scheme using 2.5 μs 90° pulses and excitation—reconversion delays of 80 μs. 32 rotor—synchronized t_1 increments were accumulated with 32 transients each. Phase sensitive detection in the indirect dimension was obtained using the States method. The ^{27}Al one pulse MAS (30 kHz) NMR spectrum was recorded using a 2.5 μs pulse length, 8192 transients and 1 s recycle delay. The ^{13}C CP-MAS (8 kHz) NMR spectrum of $ZnAlF_5$·[TAZ] was recorded using a 2 ms contact time, a ^1H 64-step small-phase incremental alternation (SPINAL-64)[iii] decoupling scheme (with an RF field corresponding to a 90 kHz nutation frequency) during the acquisition period and a 10 s recycle delay. The ^1H, ^{13}C ^{19}F and ^{27}Al chemical shifts were referenced to TMS, $CFCl_3$, and a 1 M solution of $Al(NO_3)_3$, respectively. All NMR spectra were reconstructed and drawn using the DMFIT software.[iv]

4. *Ab initio* calculations. All calculations were conducted within the Kohn-Sham[v] density functional theory (DFT) using the NMR CASTEP[vi,vii] program in the Materials Studio 5.0 environment. For the structure optimization of $ZnAlF_5$·[TAZ], ultrasoft pseudopotentials were employed, with a plane-wave cut-off energy of 400 eV and a 2 x 2 x 2 Monkhorst-Pack[viii] *k*-point sampling grid. During the structure optimization, the cell parameters were set to that of

$ZnAlF_5$·[TAZ] as determined by XRPD and were kept constant. The Perdew, Burke and Ernzerhof (PBE)[ix] functionals were used in the generalized gradient approximation (GGA) for the exchange correlation energy. Magnetic properties were computed using the projector-augmented wave method (GIPAW)[x] method. For the calculation of the ^{27}Al electric field gradient (EFG) tensor parameters, a 2 x 2 x 2 Monkhorst-Pack *k*-point grid was used to sample the Brillouin zone, with a plane-wave basis set expanded to kinetics energy lower than 500 eV. For the calculation of the ^{19}F, ^{13}C and ^{1}H shielding tensor components, a 2 x 2 x 3 Monkhorst-Pack *k*-point grid was used to sample the Brillouin zone, with a plane-wave basis set expanded to kinetics energy lower than 500 eV.

Figure 1. Final profile refinement of $ZnAlF_5 \cdot [TAZ]$: observed (line), calculated (point), and difference (bottom) profiles of the XRPD pattern.



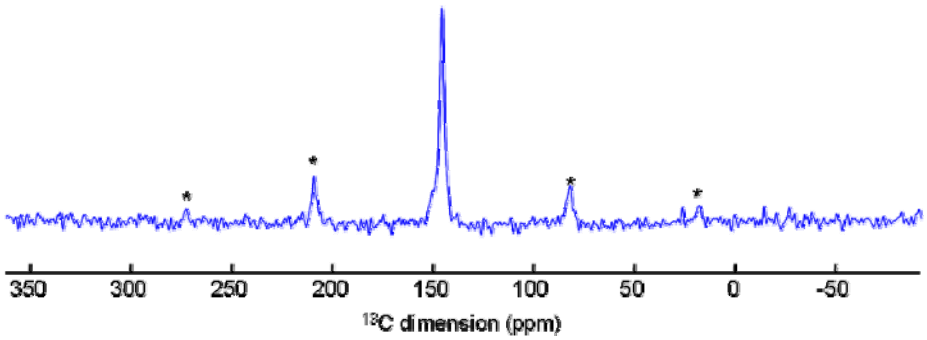


Figure 2. ^{13}C CP-MAS NMR Spectrum of $ZnAlF_5$·[TAZ]. The spinning sidebands are marked by *.

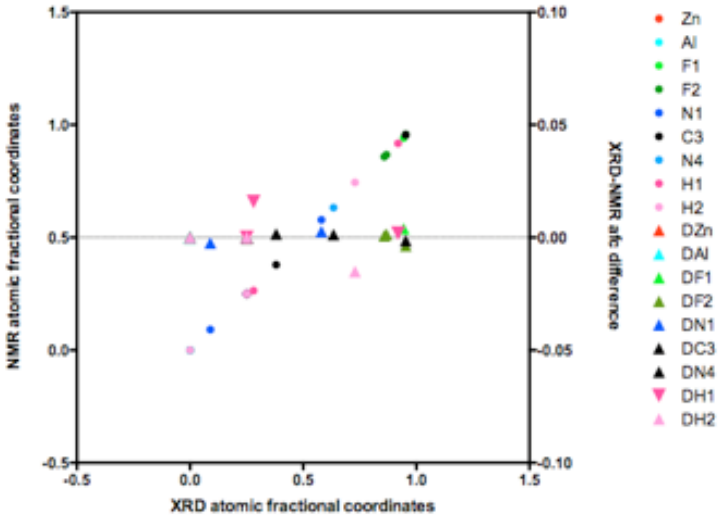


Figure 3. XPRD *vs* NMR atomic fractional coordinates (AFC) and XPDR AFC *vs* AFC difference in $ZnAlF_5$·[TAZ].

Table 1. Conditions of X-ray data collection and refinement parameters of $ZnAlF_5$·[TAZ].

	$ZnAlF_5$·[TAZ]
Formula	$ZnAlF_5C_2N_3H_3$
Formula weight	256.43
Crystal system	Orthorhombic
Space group	Imma
a (Å)	8.9288(1)
b (Å)	7.0989(1)
c (Å)	9.2593(1)
V ($Å^3$), Z	586.9(1), 4
Wavelength (Å)	1.54056
2θ range (°)	4-99
N. of independent reflections	379
N. intensity-dependent parameters	44
R_P, R_{wp}, R_B, R_F	0.077, 0.081, 0.031, 0.043

Table 2. Fractional atomic coordinates of $ZnAlF_5$·[TAZ] obtained from Rietveld refinement and from DFT optimization (in italic).

	x	y	z	Biso ($Å^2$)
Zn	¼	¼	¼	2.47(2)
	¼	*¼*	*¼*	
Al	0	0	0	2.22(9)
	0	*0*	*0*	
F(1)	0	¼	0.9447(3)	2.07(9)
	0	*¼*	*0.9408*	
F(2)	0.8587(2)	0.9539(2)	0.8685(2)	2.48(5)
	0.8579	*0.9575*	*0.8671*	
N(1)	0.5808(3)	¼	0.0894(4)	2.08(9)
	0.5781	*¼*	*0.0918*	
C(3)	0.3804(6)	¼	0.9535(5)	3.6(2)
	0.3788	*¼*	*0.9549*	
N(4)	0	¼	0.6338(6)	2.7(4)
	0	*¼*	*0.6325*	
H(1)	0.2803	¼	0.9193	15(1)
	0.2644	*¼*	*0.9174*	
H(2)	0	¼	0.7289	15(1)
	0	*¼*	*0.7440*	

File 1. Instructions and results files from the cell determination using the McMaille software.

```
McMaille input parameters:
! Wavelength, zeropoint, Ngrid
 1.540598   0.0000 0
! Codes for symmetry
 1 1 1 1 0 0
! W, Nind
0.200  0
!Pmin, Pmax, Vmin, Vmax, Rmin, Rmax, Rmaxref
  2. 20. 8. 1000. 0.05 0.15 0.50
! Ntests, Nruns
 -100 20
!  2-theta   Intensity
   13.77965       14523.27
   15.73154       50162.91
   25.08183       6186.574
   25.44973       35930.96
   27.74953       28938.02
   28.72030       27508.07
   31.59583       7021.501
   37.60630       5260.167
   37.71967       6538.038
   40.83050       1066.698
   42.14981       6763.961
   44.13326       2758.817
   44.39032       3665.281
   46.82245       2581.186
   48.12358       3087.138
   48.43833       4143.285
   51.34167       7156.181
   51.45929       7268.446
   52.15933       5335.458
   52.25543       6552.193
   52.85062       1946.799
   55.19131       1955.880
   57.29116       3398.314
   59.45080       2872.215
   63.76271       1557.828
   65.94198       1963.232

================================================================
 McMaille version 4.00     by A. Le Bail - 2006 - alb@cristal.org
 ================================================================

 Using generic filename : camw28o2long-new

   Number of Processors :           1


 30-Mar-2010     10 hour 12 min 11 Sec

 McMaille input parameters:

 Wavelength :   1.54060 Zeropoint :   0.0000


 ============================================================================
     Monte Carlo cell generation
 ============================================================================

 Width of the columnar profile shape, W  =    0.2000

 Max non-indexed reflections, NIND  =    0


  Min/Max Rp, Rmaxref   5.0000001E-02  0.1500000      0.5000000

  N of runs           20
  N of MC events in cubic           100.000000000000
  N of MC events in tetra/hexa      2000.00000000000
  N of MC events in orthorhombic    40000.0000000000
  N of MC events in monoclinic      800000.000000000
  N of MC events in triclinic       16000000.0000000

     2-THETA     d(A)    Iobs       W
```

```
     13.780   6.4213 14523.270     0.200
     15.732   5.6287 50162.910     0.200
     25.082   3.5475  6186.574     0.200
     25.450   3.4971 35930.961     0.200
     27.750   3.2122 28938.020     0.200
     28.720   3.1058 27508.070     0.200
     31.596   2.8294  7021.501     0.200
     37.606   2.3899  5260.167     0.200
     37.720   2.3830  6538.038     0.200
     40.831   2.2083  1066.698     0.200
     42.150   2.1422  6763.961     0.200
     44.133   2.0504  2758.817     0.200
     44.390   2.0391  3665.281     0.200
     46.822   1.9387  2581.186     0.200
     48.124   1.8893  3087.138     0.200
     48.438   1.8777  4143.285     0.200
     51.342   1.7782  7156.181     0.200
     51.459   1.7744  7268.446     0.200
     52.159   1.7522  5335.458     0.200
     52.255   1.7492  6552.193     0.200
     52.851   1.7309  1946.799     0.200
     55.191   1.6629  1955.880     0.200
     57.291   1.6068  3398.314     0.200
     59.451   1.5535  2872.215     0.200
     63.763   1.4585  1557.828     0.200
     65.942   1.4154  1963.232     0.200


 dmax =    6.421270
 Be sure that your choice of max cell parameters
  ensures the exploration of all possibilities.


   Dmin =    1.395433

30-Mar-2010     10 hour 12 min 11 Sec


================================================================================
                RESULTS - RESULTS - RESULTS - RESULTS
================================================================================
                EXPLORED CELL PARAMETERS AND VOLUMES:
================================================================================


Cubic Monte Carlo search :
 Max a, V    20.00000       1000.000


Hexagonal/Trigonal/Rhomboedral Monte Carlo search :
 Max(a,c), V    20.00000       20.00000       1000.000


Tetragonal Monte Carlo search :
 Max(a,c), V    20.00000       20.00000       1000.000


Orthorhombic Monte Carlo search :
 Max(a,b,c), V    20.00000       20.00000       20.00000       1000.000


================================================================================
  FINAL LIST OF CELL PROPOSALS, sorted by McM20 :
================================================================================

      Global list as produced in the .ckm file
            (IN=number of indexed lines)
  The correct cell has some chances to be just below

IN  F.o.M.     Volume    V/V1       a         b         c        alpha    beta     gamma    Bravais
lattice

26   30.10     586.283  1.00      7.0958    9.2595    8.9231  90.000  90.000  90.000     P  Ortho
***

================================================================================
    FINAL LIST OF CELL PROPOSALS, sorted by F(20) :
```

```
 ================================================================================

      Global list (IN=number of indexed lines)
  The correct cell has some chances to be just below

IN  F(20)     Volume   V/V1      a         b         c        alpha   beta    gamma   Bravais
lattice

26   48.74    586.283  1.00      7.0958   9.2595   8.9231  90.000  90.000  90.000    P  Ortho
***

 ================================================================================
     FINAL LIST OF CELL PROPOSALS, sorted by M(20) :
 ================================================================================

      Global list   (IN=number of indexed lines)
  The correct cell has some chances to be just below

IN  M(20)     Volume   V/V1      a         b         c        alpha   beta    gamma   Bravais
lattice

26   40.40    586.283  1.00      7.0958   9.2595   8.9231  90.000  90.000  90.000    P  Ortho
***

 ================================================================================
  See for the highest F.o.M. above the cell(s) with highest symmetry, if any
  (Cubic, hexagonal, etc), they could correspond to the the right solution
 ================================================================================


  Cells sorted by symmetry

   Rp     Vol     Vol/V1 Ind Nsol    a        b         c      alpha  beta  gamma

   Orthorhombic cells

  0.073    586.283  1.00  26   1   7.0958   9.2595   8.9231  90.000  90.000  90.000

    "Best" cell with largest McM20 :
    --------------------------------

 PROGRAM *** CELREF ***  (J.LAUGIER & A.FILHOL 10/78)


 OBSERVABLE NUMBER    :    25
 ITERATION NUMBER :    10
 REFINEMENT CONSTRAINTS : NO
 INITIAL VALUES :
    ZERO    LAMBDA      A        B        C      ALPHA     BETA    GAMMA
      1.       0.       1.       1.       1.       0.       0.       0.
   0.000    1.5406   7.0958   9.2595   8.9231   90.000   90.000   90.000
 RECIPROCAL CELL :   0.14093  0.10800  0.11207   90.000   90.000   90.000
 VOLUME (A**3)  :      586.283

 NUMBER OF INDEPENDENT PARAMETERS :     4

 FINAL VALUES   : (STANDARD DEVIATIONS : 2nd LINE)

    ZERO    LAMBDA      A        B        C      ALPHA     BETA    GAMMA
  -0.016    1.5406   7.1003   9.2619   8.9283   90.000   90.000   90.000
   0.004    0.0000   0.0008   0.0009   0.0010    0.000    0.000    0.000
 RECIPROCAL CELL :   0.14084  0.10797  0.11200   90.000   90.000   90.000
 VOLUME (A**3)  :      587.146

    H     K     L  TH(OBS)    TH-ZERO    TH(CALC)     DIFF.

    0     1     1    13.780     13.764     13.765     -0.001
    1     1     0    15.732     15.716     15.714      0.002
    2     0     0    25.082     25.066     25.063      0.003
    1     1     2    25.450     25.434     25.434      0.000
    0     2     2    27.750     27.734     27.734      0.000
    2     1     1    28.720     28.705     28.703      0.002
    1     3     0    31.596     31.580     31.575      0.005
    1     3     2    37.606     37.591     37.589      0.002
    2     2     2    37.720     37.704     37.725     -0.021
    0     3     3    42.150     42.134     42.140     -0.006
    1     3     3    44.133     44.118     44.113      0.005
```

```
    3     1     2    44.390     44.375     44.376     -0.002
    2     4     0    46.822     46.807     46.804      0.003
    2     0     4    48.124     48.108     48.114     -0.006
    3     3     0    48.438     48.423     48.422      0.001
    2     4     2    51.342     51.326     51.321      0.005
    4     0     0    51.459     51.444     51.437      0.006
    1     3     4    52.159     52.144     52.138      0.006
    2     2     4    52.255     52.240     52.243     -0.003
    1     0     5    52.851     52.835     52.823      0.012
    1     5     2    55.191     55.176     55.176      0.000
    0     4     4    57.291     57.276     57.285     -0.009
    4     2     2    59.451     59.435     59.437     -0.002
    0     6     2    63.763     63.747     63.741      0.006
    2     6     0    65.942     65.926     65.932     -0.006

   M(20) =     40.40
   F(20) =     48.74 (  0.0045,  91)


  Final Rp on the .prf =   6.8966284E-02


 ================================================================================
          THE SELECTION OF THE "BEST" CELL
 based on McM20, Rp, F(20), M(20), V, high symmetry ?
           DEPENDS ON YOU, EXCLUSIVELY.

                    However...
   It is suggested that the correct cell could be :
 ================================================================================

IN  F.o.M.     Volume              a         b         c        alpha    beta     gamma    Bravais
lattice
26   30.10     586.283            7.0958    9.2595    8.9231   90.000   90.000   90.000     P  Ortho
***
    Found            3  time(s) head of the best lists

 ================================================================================
```

Files 2. Instructions and results files from the cell determination using the ESPOIR software.

```
! title
ZnAlF5triaz
!    a,     b,     c,    alpha,    beta,    gamma
   8.9288   7.0989   9.2592    90.000    90.000    90.000
! space group
I M M A
! lambda, radiation, N of atoms, types of atoms, N of objects, "|Fobs|" or patterns, iprint
1.54056   4  13   5   3   1 1
 0.040027  -0.016779   0.013392  5
! atom names, in 8A4
Zn  Al  F   C   N
! code for minimal distance contraints
 0
! maximum moves for each type of atom
   3.500   3.500   3.500   3.500   3.500
! annealing law, sigma, reject
  2.0000  1.0000  0.0010
! number of events for : print, maximum, save
     20000    200000    100000
! events for restart, rmax, ichi, number of runs
 40000  0.200   2  10
! object type and NPERM for object   1
   1   0
! number of atoms of each type in object   1
   1   0   0   0   0
! B overall, NOCC, NSPE for object   1
 1.0 0 0
! object type and NPERM for object   2
   2   4
! number of atoms of each type in object   2
   0   1   6   0   0
```

```
! B overall, NOCC, NSPE for object   2
 1.0 0 0
! cell parameters, and x, y, z, occup. for object   2
 10 10 10 90 90 90
0 0 0 1
0.18 0 0 1
-0.18 0 0 1
0 0.18 0 1
0 -0.18 0 1
0 0 0.18 1
0 0 -0.18 1
! object type and NPERM for object   3
   2   4
! number of atoms of each type in object   3
   0   0   0   2   3
! B overall, NOCC, NSPE for object   3
 3.0 0 0
! cell parameters, and x, y, z, occup. for object   3
9.2710 10.6220 3.7330 90.0000 94.9600 90.0000
 0.76300 0.23310 0.57670  1
 0.77070 0.11450 0.42790  1
 0.53910 0.18040 0.40940  1
 0.63460 0.08910 0.33080  1
 0.62450 0.27030 0.56490  1


 ============================================================
  ESPOIR VERSION 3.50     by A. Le Bail - 2000 -alb@cristal.org
 ============================================================

 Using generic filename : camw28o

 29-Mar-2010     18 hour  7 min 18 Sec

 Cell parameters :    8.9288   7.0989   9.2592    90.000    90.000    90.000

 Reciprocal cell parameters :    0.1120   0.1409   0.1080    90.000    90.000    90.000

 SPACE GROUP : I M M A

  BURZLAFF Crystal system :   2 Orthorhombic

 Wavelength :   1.54056

 X-ray data

 The fit will be on the pseudo powder pattern
    with U, V, W, STEPN  =    0.0400  -0.0168   0.0134   5.0000



 The model contains   3 objects

 Overall thermal parameter B =    1.00 for object  1


 Overall thermal parameter B =    1.00 for object  2


 Starting rigid model cell parameters :
                     10.0000  10.0000  10.0000    90.000    90.000    90.000

 Reciprocal cell parameters :    0.1000   0.1000   0.1000    90.000    90.000    90.000


 Starting coordinates x,y,z and occupation numbers

                       0.0000   0.0000   0.0000      1.00
                       0.1800   0.0000   0.0000      1.00
                      -0.1800   0.0000   0.0000      1.00
                       0.0000   0.1800   0.0000      1.00
                       0.0000  -0.1800   0.0000      1.00
                       0.0000   0.0000   0.1800      1.00
                       0.0000   0.0000  -0.1800      1.00

 Overall thermal parameter B =    3.00 for object  3
```

```
Starting rigid model cell parameters :
                     9.2710  10.6220   3.7330    90.000    94.960    90.000

Reciprocal cell parameters :    0.1083   0.0941   0.2689    90.000    85.040    90.000


Starting coordinates x,y,z and occupation numbers

                     0.7630   0.2331   0.5767      1.00
                     0.7707   0.1145   0.4279      1.00
                     0.5391   0.1804   0.4094      1.00
                     0.6346   0.0891   0.3308      1.00
                     0.6245   0.2703   0.5649      1.00


THE STRUCT. IS CENTRIC,
                              AND THE EQUIV. POS. ARE:
                        ROTATION MATRIX               TRANSLATION VECTOR
POS.  1    1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0  1.0  0.0000  0.0000  0.0000
POS.  2    1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0 -1.0  0.5000  0.5000  0.5000
POS.  3   -1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0 -1.0  0.5000  0.0000  0.5000
POS.  4   -1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0  1.0  0.0000  0.5000  0.0000
 Total number of symmetry operators =           16
   Full list of symmetry operators :
                        ROTATION MATRIX               TRANSLATION VECTOR
POS.  1    1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0  1.0  0.0000  0.0000  0.0000
POS.  2    1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0 -1.0  0.5000  0.5000  0.5000
POS.  3   -1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0 -1.0  0.5000  0.0000  0.5000
POS.  4   -1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0  1.0  0.0000  0.5000  0.0000
POS.  5    1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0  1.0  0.5000  0.5000  0.5000
POS.  6    1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0 -1.0  1.0000  1.0000  1.0000
POS.  7   -1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0 -1.0  1.0000  0.5000  1.0000
POS.  8   -1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0  1.0  0.5000  1.0000  0.5000
POS.  9   -1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0 -1.0  0.0000  0.0000  0.0000
POS. 10   -1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0  1.0  0.5000  0.5000  0.5000
POS. 11    1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0  1.0  0.5000  0.0000  0.5000
POS. 12    1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0 -1.0  0.0000  0.5000  0.0000
POS. 13   -1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0 -1.0  0.5000  0.5000  0.5000
POS. 14   -1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0  1.0  1.0000  1.0000  1.0000
POS. 15    1.0  0.0  0.0  0.0 -1.0  0.0  0.0  0.0  1.0  1.0000  0.5000  1.0000
POS. 16    1.0  0.0  0.0  0.0  1.0  0.0  0.0  0.0 -1.0  0.5000  1.0000  0.5000

Title of run :

Overall standard deviation is  1.0000

Accept a move even if -  0.001000 > DELTA R > 0.

Maximum change in any coordinate is    3.500A for particles of type  1
Maximum change in any coordinate is    3.500A for particles of type  2
Maximum change in any coordinate is    3.500A for particles of type  3
Maximum change in any coordinate is    3.500A for particles of type  4
Maximum change in any coordinate is    3.500A for particles of type  5

Move amplitudes will be progressively reduced by MOVE=MOVE*(1.-NGEN/maxNGEN)**anneal
                  with anneal =  2.00

Writing summary every      20000 generated moves
Programme will run for   200000.0 moves saving every   100000.0 moves

Restart after  40000 events IF the R factor is still higher than  0.200

Number of different starting configurations to test :   10

Configuration contains    13 atoms of 5 types

REAL AND IM. X-RAY SCATTER. FACT.
zn   14.07430  3.26550  7.03180  0.23330  5.16520 10.31630  2.41000 58.70970
      1.30410 -1.61200  0.67800
al    6.42020  3.03870  1.90020  0.74260  1.59360 31.54720  1.96460 85.08860
      1.11510  0.20400  0.24600
f     3.53920 10.28250  2.64120  4.29440  1.51700  0.26150  1.02430 26.14760
      0.27760  0.06900  0.05300
c     2.31000 20.84390  1.02000 10.20750  1.58860  0.56870  0.86500 51.65120
      0.21560  0.01700  0.00900
n    12.21260  0.00570  3.13220  9.89330  2.01250 28.99750  1.16630  0.58260
```

```
     -11.52900  0.02900  0.01800

   Object    1 can be translated by     3.500

   Object    2 can be translated by     3.500

   Object    3 can be translated by     3.500



 Test number :   1
 29-Mar-2010     18 hour  7 min 18 Sec
  ISEED =    589470981

 Starting coordinates x,y,z and occupation numbers

Zn1     0.50668    0.10407    0.19500      1.000

 Starting coordinates x,y,z and occupation numbers

Al1     0.00000    0.00000    0.00000      1.000
F1      0.20159    0.00000    0.00000      1.000
F2     -0.20159    0.00000    0.00000      1.000
F3      0.00000    0.25356    0.00000      1.000
F4      0.00000   -0.25356    0.00000      1.000
F5      0.00000    0.00000    0.19440      1.000
F6      0.00000    0.00000   -0.19440      1.000


  Molecule connectivity, up to 1.8 Angstrom
           A1  A2   Distance


  Total number of non-H bonds in object   2 :    0

  Terminal bonds :
  Bond number  A1   A2

     No terminal bond found


 Ring analysis
        (the same ring may appear several times)

     No ring found


  Rotatable bonds : No   A1   A2

     No rotatable bond found


 The following   0 groups of atoms can rotate, if you wish
            (see the manual at the nobt parameter)


  End of molecule connectivity analysis


 Starting coordinates x,y,z and occupation numbers

C1      0.77140    0.34878    0.23164      1.000
C2      0.78477    0.17132    0.17187      1.000
N1      0.54496    0.26993    0.16444      1.000
N2      0.64696    0.13332    0.13287      1.000
N3      0.62801    0.40445    0.22690      1.000
```

```
 Molecule connectivity, up to 1.8 Angstrom
          A1  A2   Distance

         C1   C2     1.38113
         C1   N3     1.34054
         C2   C1     1.38113
         C2   N2     1.31041
         N1   N2     1.36213
         N1   N3     1.34023
         N2   C2     1.31041
         N2   N1     1.36213
         N3   C1     1.34054
         N3   N1     1.34023

 Total number of non-H bonds in object   3 :    5

 Terminal bonds :
 Bond number  A1   A2

    No terminal bond found



Ring analysis
        (the same ring may appear several times)
 5 atoms ring :   1  2  4  3  5  C1   C2   N2   N1   N3
 5 atoms ring :   1  5  3  4  2  C1   N3   N1   N2   C2
 5 atoms ring :   2  4  3  5  1  C2   N2   N1   N3   C1
 5 atoms ring :   3  4  2  1  5  N1   N2   C2   C1   N3
 5 atoms ring :   3  5  1  2  4  N1   N3   C1   C2   N2


 Rotatable bonds : No   A1   A2

    No rotatable bond found



The following   0 groups of atoms can rotate, if you wish
            (see the manual at the nobt parameter)


 End of molecule connectivity analysis



  Starting R value =     0.788


Test number :   9
29-Mar-2010     18 hour  9 min 14 Sec
 ISEED =    792486605

 Starting coordinates x,y,z and occupation numbers

Zn1     0.27802    0.78393    0.97998      1.000
Al1     0.00000    0.00000    0.00000      1.000
F1      0.20159    0.00000    0.00000      1.000
F2     -0.20159    0.00000    0.00000      1.000
F3      0.00000    0.25356    0.00000      1.000
F4      0.00000   -0.25356    0.00000      1.000
F5      0.00000    0.00000    0.19440      1.000
F6      0.00000    0.00000   -0.19440      1.000
C1      0.77140    0.34878    0.23164      1.000
C2      0.78477    0.17132    0.17187      1.000
N1      0.54496    0.26993    0.16444      1.000
N2      0.64696    0.13332    0.13287      1.000
N3      0.62801    0.40445    0.22690      1.000

  Object number  1 at test  9.  Previous minimum R=0.159 at test  1
      0 moves acc.       0 tested; Chi**2=0.812    , R=0.812
      0 perm. acc.       0 tested
      0 events did not improve the fit, DAMP = 1.000000
  Object number  2 at test  9.  Previous minimum R=0.159 at test  1
      0 rot. acc.       0 gen. and       0 tested; Chi**2=0.812    , R=0.812
      0 trans. acc.       0 tested
```

```
    0 events did not improve the fit, DAMP = 1.000000
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
    0 rot. acc.       0 gen. and       0 tested; Chi**2=0.812    , R=0.812
    0 trans. acc.       0 tested
    0 events did not improve the fit, DAMP = 1.000000

Object number  1 at test  9.  Previous minimum R=0.159 at test  1
    7 moves acc.   20362 tested; Chi**2=0.210    , R=0.210
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.482427
Object number  2 at test  9.  Previous minimum R=0.159 at test  1
   12 rot. acc.   15185 gen. and   15185 tested; Chi**2=0.210    , R=0.210
    0 trans. acc.    5061 tested
    0 events did not improve the fit, DAMP = 0.484848
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   15236 gen. and   15236 tested; Chi**2=0.210    , R=0.210
    3 trans. acc.    5078 tested
    0 events did not improve the fit, DAMP = 0.483428

Object number  1 at test  9.  Previous minimum R=0.159 at test  1
    7 moves acc.   32081 tested; Chi**2=0.200    , R=0.200
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.269138
Object number  2 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   23973 gen. and   23973 tested; Chi**2=0.200    , R=0.200
    0 trans. acc.    7990 tested
    0 events did not improve the fit, DAMP = 0.270977
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   24163 gen. and   24163 tested; Chi**2=0.200    , R=0.200
    3 trans. acc.    8054 tested
    0 events did not improve the fit, DAMP = 0.267041

Object number  1 at test  9.  Previous minimum R=0.159 at test  1
    7 moves acc.   40841 tested; Chi**2=0.196    , R=0.196
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.150067
Object number  2 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   30718 gen. and   30718 tested; Chi**2=0.196    , R=0.196
    0 trans. acc.   10239 tested
    0 events did not improve the fit, DAMP = 0.148734
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
   14 rot. acc.   30911 gen. and   30911 tested; Chi**2=0.196    , R=0.196
    4 trans. acc.   10303 tested
    0 events did not improve the fit, DAMP = 0.145764

Object number  1 at test  9.  Previous minimum R=0.159 at test  1
    7 moves acc.   40873 tested; Chi**2=0.195    , R=0.195
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.149695
Object number  2 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   30748 gen. and   30748 tested; Chi**2=0.195    , R=0.195
    1 trans. acc.   10249 tested
    0 events did not improve the fit, DAMP = 0.148271
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
   14 rot. acc.   30945 gen. and   30945 tested; Chi**2=0.195    , R=0.195
    4 trans. acc.   10314 tested
    0 events did not improve the fit, DAMP = 0.145249

Object number  1 at test  9.  Previous minimum R=0.159 at test  1
    8 moves acc.   54958 tested; Chi**2=0.175    , R=0.175
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.030846
Object number  2 at test  9.  Previous minimum R=0.159 at test  1
   13 rot. acc.   41298 gen. and   41298 tested; Chi**2=0.175    , R=0.175
    2 trans. acc.   13765 tested
    0 events did not improve the fit, DAMP = 0.030295
Object number  3 at test  9.  Previous minimum R=0.159 at test  1
   15 rot. acc.   41593 gen. and   41593 tested; Chi**2=0.175    , R=0.175
    6 trans. acc.   13864 tested
    0 events did not improve the fit, DAMP = 0.028278

Object number  1 at test  9.  Previous minimum R=0.158 at test  9
   13 moves acc.   62648 tested; Chi**2=0.158    , R=0.158
    0 perm. acc.       0 tested
    1 events did not improve the fit, DAMP = 0.009997
Object number  2 at test  9.  Previous minimum R=0.158 at test  9
   15 rot. acc.   47230 gen. and   47230 tested; Chi**2=0.158    , R=0.158
```

```
     9 trans. acc.   15743 tested
     0 events did not improve the fit, DAMP = 0.009994
  Object number  3 at test  9.  Previous minimum R=0.158 at test  9
    16 rot. acc.   47359 gen. and   47359 tested; Chi**2=0.158    , R=0.158
    11 trans. acc.   15786 tested
     1 events did not improve the fit, DAMP = 0.009994

  Object number  1 at test  9.  Previous minimum R=0.158 at test  9
    13 moves acc.   64299 tested; Chi**2=0.158    , R=0.158
     0 perm. acc.       0 tested
     1 events did not improve the fit, DAMP = 0.009997
  Object number  2 at test  9.  Previous minimum R=0.158 at test  9
    15 rot. acc.   48529 gen. and   48529 tested; Chi**2=0.158    , R=0.158
     9 trans. acc.   16176 tested
     0 events did not improve the fit, DAMP = 0.009994
  Object number  3 at test  9.  Previous minimum R=0.158 at test  9
    16 rot. acc.   48577 gen. and   48577 tested; Chi**2=0.158    , R=0.158
    12 trans. acc.   16192 tested
     1 events did not improve the fit, DAMP = 0.009994

  Object number  1 at test  9.  Previous minimum R=0.158 at test  9
    13 moves acc.   66395 tested; Chi**2=0.195    , R=0.158
     0 perm. acc.       0 tested
     1 events did not improve the fit, DAMP = 0.009997
  Object number  2 at test  9.  Previous minimum R=0.158 at test  9
    15 rot. acc.   50079 gen. and   50079 tested; Chi**2=0.195    , R=0.158
     9 trans. acc.   16692 tested
     0 events did not improve the fit, DAMP = 0.009994
  Object number  3 at test  9.  Previous minimum R=0.158 at test  9
    16 rot. acc.   50125 gen. and   50125 tested; Chi**2=0.195    , R=0.158
    12 trans. acc.   16708 tested
     1 events did not improve the fit, DAMP = 0.009994

 Final coordinates x,y,z and occupation numbers

Zn1     0.24855    0.27173    0.75015      1.000
Al1     0.00245    0.02667    0.98425      1.000
F1      0.04112    0.26667    1.03468      1.000
F2     -0.03623   -0.21333    0.93382      1.000
F3      0.15353   -0.05243    1.09777      1.000
F4     -0.14864    0.10577    0.87073      1.000
F5      0.13019    0.04756    0.83471      1.000
F6     -0.12529    0.00578    1.13379      1.000
C1      0.40110    0.27150    0.07666      1.000
C2      0.40308    0.26583   -0.07242      1.000
N1      0.63573    0.22757    0.01732      1.000
N2      0.54511    0.23762   -0.10077      1.000
N3      0.54043    0.24951    0.12788      1.000
 29-Mar-2010     18 hour  9 min 36 Sec

 End of this test




 Test number :  10
 29-Mar-2010     18 hour  9 min 36 Sec
  ISEED =    871129947

 Starting coordinates x,y,z and occupation numbers

Zn1     0.97147    0.38258    0.57480      1.000
Al1     0.00000    0.00000    0.00000      1.000
F1      0.20159    0.00000    0.00000      1.000
F2     -0.20159    0.00000    0.00000      1.000
F3      0.00000    0.25356    0.00000      1.000
F4      0.00000   -0.25356    0.00000      1.000
F5      0.00000    0.00000    0.19440      1.000
F6      0.00000    0.00000   -0.19440      1.000
C1      0.77140    0.34878    0.23164      1.000
C2      0.78477    0.17132    0.17187      1.000
N1      0.54496    0.26993    0.16444      1.000
N2      0.64696    0.13332    0.13287      1.000
```

```
N3      0.62801    0.40445    0.22690      1.000

  Object number  1 at test 10.  Previous minimum R=0.158 at test  9
      0 moves acc.       0 tested; Chi**2=0.796    , R=0.796
      0 perm. acc.       0 tested
      0 events did not improve the fit, DAMP = 1.000000
  Object number  2 at test 10.  Previous minimum R=0.158 at test  9
      0 rot. acc.       0 gen. and       0 tested; Chi**2=0.796    , R=0.796
      0 trans. acc.       0 tested
      0 events did not improve the fit, DAMP = 1.000000
  Object number  3 at test 10.  Previous minimum R=0.158 at test  9
      0 rot. acc.       0 gen. and       0 tested; Chi**2=0.796    , R=0.796
      0 trans. acc.       0 tested
      0 events did not improve the fit, DAMP = 1.000000

  Object number  1 at test 10.  Previous minimum R=0.158 at test  9
      4 moves acc.   35445 tested; Chi**2=0.245    , R=0.245
      0 perm. acc.       0 tested
      0 events did not improve the fit, DAMP = 0.219328
  Object number  2 at test 10.  Previous minimum R=0.158 at test  9
      9 rot. acc.   26806 gen. and   26806 tested; Chi**2=0.245    , R=0.245
      0 trans. acc.    8935 tested
      0 events did not improve the fit, DAMP = 0.215203
  Object number  3 at test 10.  Previous minimum R=0.158 at test  9
      4 rot. acc.   26911 gen. and   26911 tested; Chi**2=0.245    , R=0.245
      1 trans. acc.    8969 tested
      0 events did not improve the fit, DAMP = 0.213259

 Final coordinates x,y,z and occupation numbers

Zn1     0.24353    0.77392    0.75674      1.000
Al1     0.00000    0.00000    0.00000      1.000
F1     -0.04884    0.22568   -0.07506      1.000
F2      0.04884   -0.22568    0.07506      1.000
F3      0.12572    0.11353    0.12457      1.000
F4     -0.12572   -0.11353   -0.12457      1.000
F5      0.14983   -0.02170   -0.12899      1.000
F6     -0.14983    0.02170    0.12899      1.000
C1      0.53070    0.60816    0.85278      1.000
C2      0.38247    0.55272    0.85605      1.000
N1      0.41705    0.81353    0.99078      1.000
N2      0.32081    0.68121    0.93845      1.000
N3      0.54910    0.76271    0.93405      1.000
 29-Mar-2010     18 hour  9 min 49 Sec

   h   k   l   Fcalc    Fobs     d(A)   2-theta   FWHM

   1   0   1  76.236  47.959   6.427  13.766   0.109
   0   1   1  98.309 103.747   5.634  15.717   0.109
   0   0   2  30.992  13.251   4.630  19.155   0.108
   2   0   0  23.091   4.642   4.464  19.871   0.108
   1   1   2   6.358  10.147   3.557  25.014   0.108
   0   2   0 109.194  83.499   3.549  25.067   0.108
   2   1   1 109.940 110.114   3.499  25.435   0.108
   2   0   2 164.497 153.918   3.214  27.737   0.108
   1   2   1  76.547 112.776   3.107  28.707   0.108
   1   0   3  16.324  22.933   2.917  30.622   0.109
   3   0   1  14.308  19.626   2.833  31.549   0.109
   0   1   3 102.432  84.601   2.830  31.583   0.109
   0   2   2  32.838  53.741   2.817  31.740   0.109
   2   2   0  25.997  40.714   2.778  32.192   0.109
   2   1   3  46.030  61.833   2.390  37.595   0.111
   2   2   2  82.329  63.550   2.382  37.730   0.111
   3   1   2  44.192  32.510   2.361  38.082   0.111
   0   0   4  25.652   0.000   2.315  38.873   0.112
   0   3   1  14.679   8.242   2.293  39.265   0.112
   1   2   3  27.828  21.216   2.254  39.973   0.112
   4   0   0  36.594  26.315   2.232  40.373   0.112
   3   2   1  36.003  35.907   2.214  40.711   0.113
   3   0   3 116.874 117.741   2.142  42.144   0.113
   1   1   4  40.449  26.173   2.137  42.260   0.114
   4   1   1  49.901  34.962   2.075  43.576   0.114
   2   0   4  44.492  39.829   2.055  44.028   0.115
   1   3   2  48.471  51.161   2.051  44.125   0.115
   2   3   1  70.173  67.525   2.039  44.382   0.115
   4   0   2  34.870  40.549   2.011  45.051   0.115
   0   2   4  82.091  79.764   1.939  46.816   0.117
```

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  4   2   0  97.601  98.595   1.890  48.114   0.118
  0   3   3 118.509 110.122   1.878  48.433   0.118
  3   2   3  24.636  42.499   1.834  49.664   0.119
  1   0   5  25.889  24.523   1.813  50.277   0.120
  0   1   5  26.964  27.328   1.792  50.919   0.120
  2   2   4 103.648 112.064   1.778  51.332   0.121
  0   4   0 160.686 174.812   1.775  51.447   0.121
  3   1   4  28.623  28.903   1.770  51.609   0.121
  5   0   1  40.953  62.200   1.753  52.118   0.121
  4   1   3  85.856  88.711   1.753  52.141   0.122
  4   2   2  94.297  94.890   1.749  52.245   0.122
  2   3   3  46.067  58.181   1.731  52.846   0.122
  3   3   2  20.377  35.405   1.720  53.220   0.123
  1   4   1  24.111  23.091   1.711  53.522   0.123
  2   1   5  77.465  60.318   1.663  55.189   0.125
  0   4   2   1.639   3.390   1.657  55.398   0.125
  2   4   0   9.898  10.987   1.649  55.688   0.125
  1   3   4  29.282  15.539   1.627  56.515   0.126
  5   1   2   7.698   7.927   1.622  56.704   0.126
  1   2   5   4.677   7.049   1.615  56.983   0.127
  4   0   4 126.443 127.813   1.607  57.290   0.127
  4   3   1  74.722  72.219   1.599  57.583   0.128
  3   0   5  59.182  48.424   1.572  58.668   0.129
  5   2   1   7.551  23.728   1.572  58.678   0.129
  2   4   2  90.950  90.616   1.554  59.447   0.130
  5   0   3  55.571  59.875   1.546  59.781   0.130
  0   0   6  48.231  39.979   1.543  59.887   0.130
  1   4   3  27.895  21.388   1.516  61.067   0.132
  3   4   1  32.392  25.266   1.504  61.612   0.133
  6   0   0   9.680  17.489   1.488  62.345   0.134
  1   1   6  27.386  16.491   1.487  62.401   0.134
  4   2   4   2.503  19.454   1.464  63.500   0.135
  2   0   6  64.600  65.785   1.459  63.758   0.136
  0   3   5  45.795  62.260   1.458  63.766   0.136
  3   3   4  27.152  29.158   1.446  64.364   0.137
  6   1   1  49.722  67.075   1.439  64.738   0.137
  3   2   5  23.380  45.004   1.438  64.798   0.137
  4   3   3  48.409  33.140   1.437  64.828   0.137
  5   2   3  25.242  26.645   1.417  65.851   0.139
  6   0   2  80.428  60.625   1.417  65.872   0.139
  0   2   6  95.456 105.997   1.415  65.951   0.139
  0   4   4  55.736  38.892   1.408  66.311   0.140
  0   5   1  54.393  77.634   1.403  66.580   0.140
  4   1   5  57.122  63.258   1.397  66.905   0.141
  4   4   0  59.142  51.919   1.389  67.351   0.141
  5   1   4   6.433  33.012   1.387  67.488   0.142
  2   3   5  72.027  49.706   1.386  67.511   0.142
  6   2   0  92.202  93.098   1.372  68.287   0.143
  3   4   3  39.857  48.161   1.367  68.611   0.143
  5   3   2   2.777  16.371   1.362  68.864   0.144
  2   2   6  17.960  16.394   1.349  69.636   0.145
  3   1   6   8.360  12.502   1.345  69.867   0.146
  2   4   4  35.908  30.050   1.343  69.987   0.146
  1   5   2   7.854  16.214   1.342  70.058   0.146
  2   5   1  47.056  56.613   1.339  70.249   0.146
  4   4   2  24.429  25.633   1.331  70.748   0.147
  6   1   3  55.542  27.020   1.317  71.577   0.149
  6   2   2  26.161  21.523   1.316  71.664   0.149
  1   0   7  24.115  39.087   1.308  72.128   0.150
  0   1   7  73.667  69.002   1.300  72.649   0.151
 Final RF factor    =   0.2007589
 Final Rp(F) factor =   0.1581771

End of this test
```

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