

FullProf
***for magnetic structure
determination and refinement***
example: $\text{KTb}_3\text{F}_{12}$

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**Steps for magnetic structure
determination using powder
diffraction**

Step

Input

Propagation vector(s)
SuperCell

*Peak positions of
 \Leftarrow magnetic reflections
Cell parameters*

Symmetry Analysis
BasIreps

*Propagation vector
 \Leftarrow Space Group
Atom positions*

**Magnetic structure
solution (Sim. Ann.)**
FullProf

*Integrated intensities
 \Leftarrow Atomic components
of basis functions*



Problems of magnetic structure refinement using powder diffraction

Input

**Magnetic structure
Refinement**
FullProf

*Complete structural
⇐ model should be
provided*

Different runs of SAnn jobs may give you an idea of the degeneracy of solutions for your particular problem.

In many cases the number of free parameters is too much high to be refined by LSQ: try to reduce the number of parameters or make soft constraints.

Use spherical components of Fourier coefficients in order to have better control of the amplitude of the magnetic moment

NEUTRONS
FOR SCIENCE

Tutorial Magnetic Structure of $\text{KTb}_3\text{F}_{12}$

- ☞ A simple case where symmetry analysis gives a very important information to interpret the results.
- ☞ Space Group: $I4/m$, prop. vector $\mathbf{k}=(1,0,0)$ ($a=7.63\text{\AA}$, $c=7.52\text{\AA}$), 2 magnetic atoms $\{\text{Tb}_a, \text{Tb}_b\}$ in special positions (3 magnetic atoms per primitive unit cell)
Eight 1D-Irreps some of them are complex.
- ☞ The final magnetic structure is reduced to the refinement of only a single parameter.

NEUTRONS
FOR SCIENCE

Steps for using the Simulated Annealing option within *FullProf* for Magnetic Structure Determination

- 1- Refine the crystal structure in the paramagnetic state
1'- If there is a structural phase transition at T_N/T_C , then refine the crystal structure in the ordered state using high-Q reflections without magnetic contribution
- 2- Indexing: determine the propagation vector
- 3- In the ordered state fix all structural parameters and introduce the magnetic contribution as a new phase using the Le Bail fit mode and putting **More**=1 and **Jvi**=11
- 4- The above step produces an output file *.int that can be used as input data for Simulated Annealing runs



Tb^{3+}/Tb^{4+} Charge ordering in $K Tb_3 F_{12}$: magnetic frustration in the Tb^{3+} sublattice

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Magnetic properties of a mixed-valence (III/IV) terbium fluoride $K Tb_3 F_{12}$

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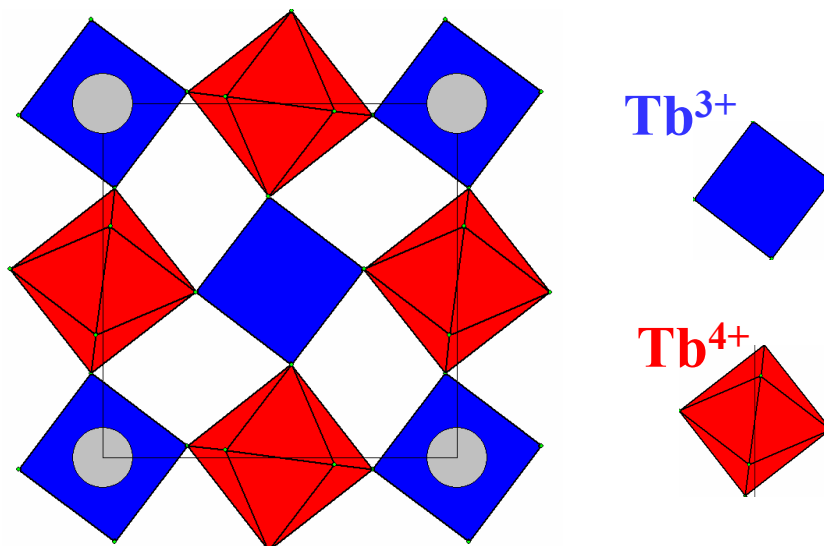
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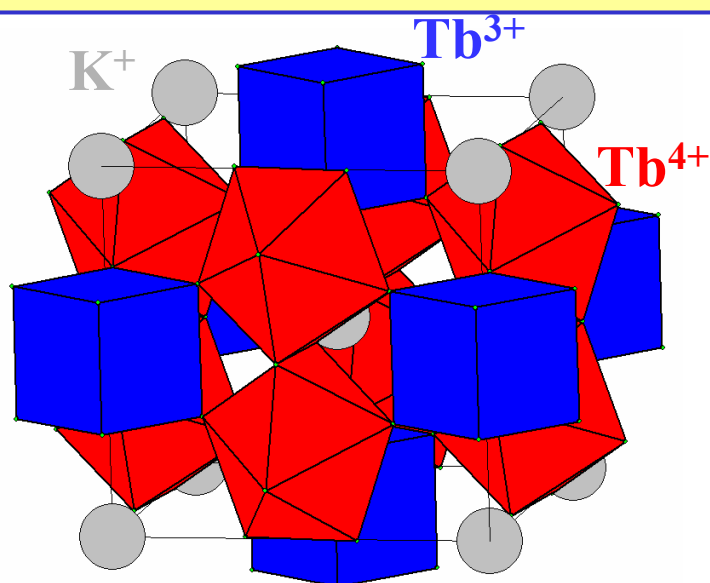


Structure of $\text{KTb}^{3+}\text{Tb}_2^{4+}\text{F}_{12}$



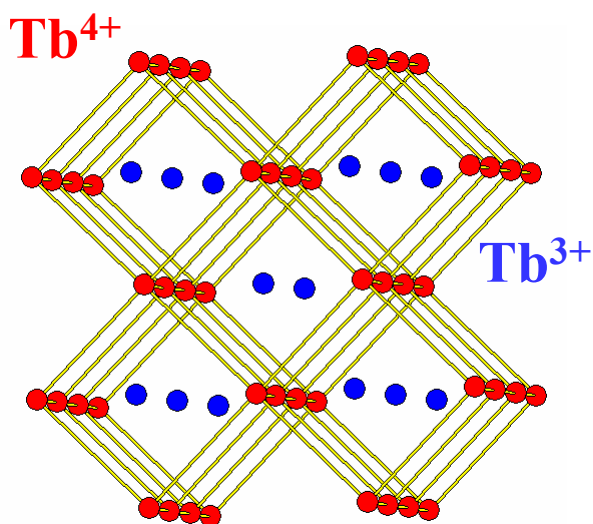
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Structure of $\text{KTb}^{3+}\text{Tb}_2^{4+}\text{F}_{12}$



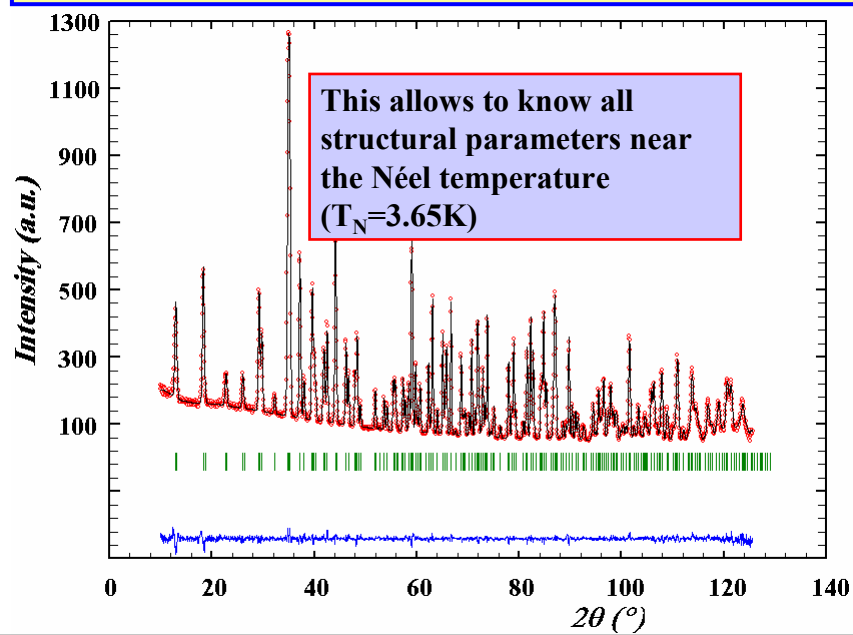
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Topology of Magnetic ions in $\text{KTb}^{3+}\text{Tb}_2^{4+}\text{F}_{12}$



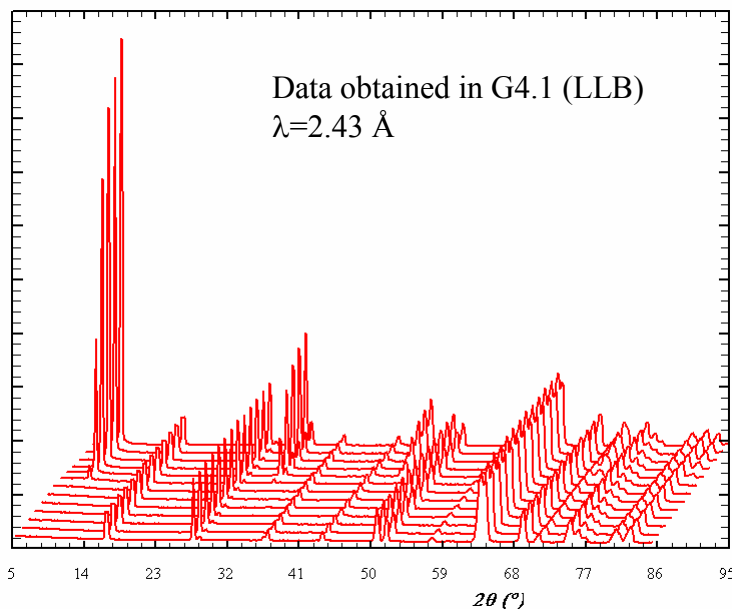
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Refinement of the crystal structure of: $\text{KTb}_3\text{F}_{14}$ at 5K



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Powder diffraction patterns of $\text{KTb}_3\text{F}_{14}$



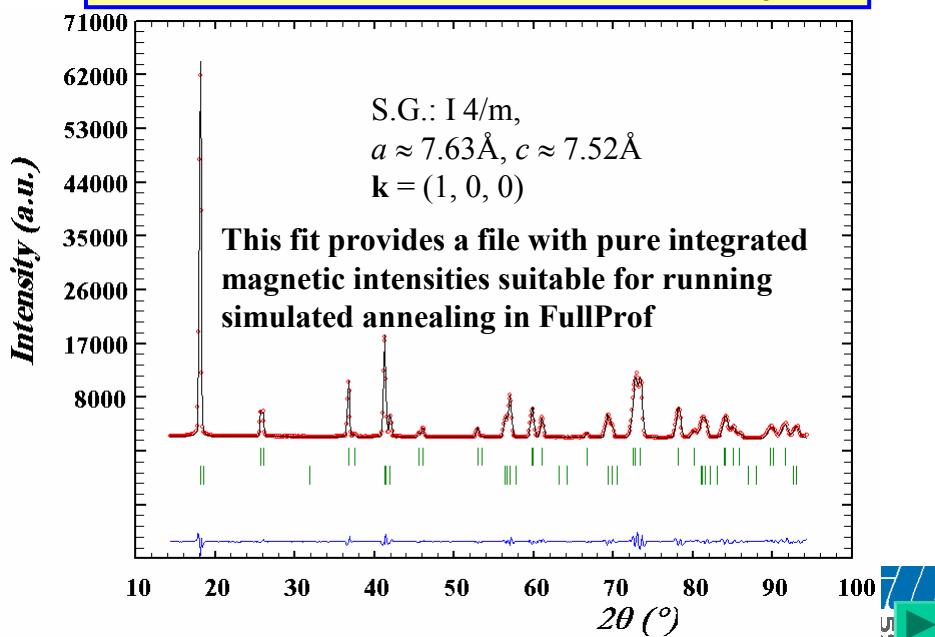
```

!-----
! Data for PHASE number: 2 ==> Current R_Bragg for Pattern# 1: 0.45
!-----
KTb3F12-M
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth ATZ Nvk Npr More
0 0 0 0.0 0.0 1.0 2 -1 0 0 0 0.000 1 7 1
!
!Jvi Jdi Hel Sol Mom Ter Brind RMua RMub RMuc Jtyp Nsp_Ref
11 0 0 0 0 0 1.0000 0.0000 0.0000 0.0000 1 0
!
I -1 <--Space group symbol
!-----> Profile Parameters for Pattern # 1
! Scale Shape1 Bov Str1 Str2 Str3 Strain-Model
4.3356 0.00000 0.00000 0.00000 0.00000 0.00000 0
0.00000 0.000 0.000 0.000 0.000 0.000
! U V W X Y GauSiz LorSiz
0.794074 -0.280000 0.080000 0.000000 0.045159 0.000000 0.000000
61.000 0.000 0.000 0.000 71.000 0.000 0.000
! a b c alpha beta gamma
7.695388 7.695388 7.540170 90.000000 90.000000 90.000000
31.00000 31.00000 41.00000 0.00000 0.00000 0.00000
! Pref1 Pref2 Asy1 Asy2 Asy3 Asy4 S_L D_L
1.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000 0.00000
0.00 0.00 0.00 0.00 0.00 0.00 0.00 0.00
! Propagation vectors:
1.0000000 0.0000000 0.0000000 Propagation Vector 1
0.000000 0.000000 0.000000

```



Profile matching (Le Bail) fit: $\text{KTb}_3\text{F}_{14}$



Example of *.int file generated by *FullProf* using **More=1, Jvi=11**

```
Phase No: 2 KTh3F12-M
(4i4,2f12.2,i4,3f14.4)
2.4260 0 2
1
1 1.00000 0.00000 0.00000
0 0 0 1 -1.00 47.42 2
-1 1 0 1 18893.78 67.06 2
-1 0 1 1 0.00 1.00 2
-2 -1 1 1 -1.00 0.00 2
-2 1 1 1 -1.00 0.00 2
0 -1 1 1 -1.00 0.00 2
0 1 1 1 0.00 0.00 2
0 2 0 1 -1.00 7.41 2
0 -2 0 1 -1.00 1.00 2
1 -1 0 1 -1.00 7.41 2
1 1 0 1 -1.00 7.41 2
-3 0 1 1 -1.00 0.42 2
-1 -2 1 1 -1.00 0.42 2
-1 2 1 1 -1.00 0.42 2
1 0 1 1 6566.70 14.85 2
-2 0 2 1 -1.00 2.24 2
-1 -1 2 1 -1.00 2.24 2
-1 1 2 1 -1.00 2.24 2
0 0 2 1 1327.23 4.48 2
. . . . .
```

Overlapped reflections re-grouped
 <- Format of h,k,l,iv, Int, sigma, multip.
 <- Wavelength, type of data, powder ind.
 <- Number of propagation vectors
 <- Propagation vector

Negative intensity means that the reflection contributes to the next positive observation

How to prepare a Simulating Annealing PCR file?

Cry=3 tells the program to use the Simulated annealing mode
Nre=8 number of free parameters with box constraints

```

COMM Ktb3F12 - T=1.4K - G4.1
! Files =>
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
  1  0  1  0  0  0  0  0  0  0  0  0  0  8  3  0  0  0  1
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
  0  0  1  0  1  0  0  0  0  3  5  0  0  0  0  0  0  0
!
!NCY Eps R_at R_an R_pr R_gl Thmin Step Thmax PSD Sent0
  1  0.10 1.00 1.00 1.00 1.00 15.0000 0.100000 94.9000 0.000 0.000
!
!
      8      !Number of refined parameters
!-----
! Data for PHASE number:  1 ==> Current R_Bragg for Pattern#  1:    2.03
!-----
Ktb3F12-M
!
. . . . .

```



How to prepare a Simulating Annealing PCR file?

```

!Nat Dis Mom Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth      ATZ      Nvk Npr More
  3  0  0  0.0 0.0 1.0 -1  4 -1  0  0      0.000  1  0  0
!
I -1                                <--Space group symbol
!Nsym Cen Laue MagMat
  1  1  1  1
!
SYMM  x,y,z
MSYM  u,v,w,0.0
!
!Atom Typ  Mag Vek  X      Y      Z      Biso  Occ      Rm      Rphi  Rtheta
!      Im  Iphi  Itheta beta11 beta22 beta33 MagPh
T3_1 JTB3  1  0  0.00000 0.00000 0.50000 0.06775 1.00000 0.09  0.976  0.000
      0.00  0.00  0.00  0.00  0.00  0.00  0.00 11.00 31.00 41.00
      0.000 0.000 0.000 0.000 0.000 0.000 0.00000
      0.00  0.00  0.00  0.00  0.00  0.00  0.00
T4_1 JTB3  1  0  0.00000 0.50000 0.25000 0.03425 1.00000 1.909  1.927  0.00
      0.00  0.00  0.00  0.00  0.00  0.00  0.00 21.00 51.00 61.00
      0.000 0.000 0.000 0.000 0.000 0.000 0.00000
      0.00  0.00  0.00  0.00  0.00  0.00  0.00
T4_2 JTB3  1  0  0.00000 0.50000 0.75000 0.03425 1.00000 1.909  0.561  0.00
      0.00  0.00  0.00  0.00  0.00  0.00  0.00 21.00 71.00 81.00
      0.000 0.000 0.000 0.000 0.000 0.000 0.00000
      0.00  0.00  0.00  0.00  0.00  0.00  0.00

```

No symmetry constraints:
Spherical components, $m_{Tb^{4+}(1)} = m_{Tb^{4+}(2)}$



How to prepare a Simulating Annealing PCR file?

No profile parameters,
part of the file similar to single crystal format

```
! Scale Factors
! Sc1      Sc2      Sc3      Sc4      Sc5      Sc6
  4.336    0.000    0.000    0.000    0.000    0.000
    0.00    0.00    0.00    0.00    0.00    0.00
! Extinction Parameters
! Ext1      Ext2      Ext3      Ext4      Ext5      Ext6      Ext7      Ext-Model
  0.000    0.000    0.000    0.000    0.000    0.000    0.000    0
    0.00    0.00    0.00    0.00    0.00    0.00    0.00    0.00
!      a      b      c      alpha      beta      gamma
  7.695388  7.695388  7.540171  90.000000  90.000000  90.000000
  0.000000  0.000000  0.000000  0.000000  0.000000  0.000000
! x-Lambda/2 +      Not yet used parameters
  0.000000  0.000000  0.000000  0.000000  0.000000
    0.00    0.00    0.00    0.00    0.00
! Propagation vectors:
  1.0000000  0.0000000  0.0000000      Propagation Vector 1
  0.0000000  0.0000000  0.0000000
```



How to prepare a Simulating Annealing PCR file?

Parameter number **Ranges and steps** **boundary conditions**

```
! Limits for selected parameters (+ steps & BoundCond for SA):
1  0.0000  9.0000  0.5831  0 Rmom_T3_1
2  0.0000  9.0000  0.0582  0 Rmom_T4_1
3  0.0000  360.0000  2.0000  1 RPhi_T3_1
4  0.0000  180.0000  2.0000  0 RThet_T3_1
5  0.0000  360.0000  2.0000  1 RPhi_T4_1
6  0.0000  180.0000  2.0000  0 RThet_T4_1
7  0.0000  360.0000  2.0000  1 RPhi_T4_2
8  0.0000  180.0000  2.0000  0 RThet_T4_2

!
! T_ini  Anneal  Accept  NumTemps  NumThCyc  InitConf
  5.000  0.900  0.050  45  0  0
! NCyclM  Nsolu  Num_Ref  Nscalef  NAlgor
  150  1  110  0  0
```

Number of reflections to consider

Automatic treatment(1) or fixed(0) scale factor

Corana algorithm Initial step = range

Random initial configuration



```

FullProf.2k_Multi_Pattern
=> *****
=> ** PROGRAM FullProf.2k (Version 2.45 - Jul2003-LIB 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
=> (Multi_Pattern: Windows-version)

=> START Date:19/08/2003 Time => 01:48:24.458
=> Reading control file *.PCR ...
=> End of preliminary calculations !

=> **** SIMULATED ANNEALING SEARCH FOR STARTING CONFIGURATION ****

=> Initial configuration cost: 80.12
=> Initial configuration state vector:
=> Rnom_T3_ Rnom_T4_ RPhi_T3_ RThet_T3 RPhi_T4_ RThet_T4 RPhi_T4_ RThet_T4
=> 1 2 3 4 5 6 7 8
=> 1.6457 5.2475 2.7474 40.2139 87.8963 67.3143 109.4662 150.3001

=> NT: 1 Temp: 10.00 (%Acc): 62.58 <Step>:204.7500 <R-factor>: 43.2703
=> NT: 2 Temp: 9.00 (%Acc): 59.00 <Step>:204.5625 <R-factor>: 24.5563
=> NT: 3 Temp: 8.10 (%Acc): 60.08 <Step>:184.1281 <R-factor>: 19.2916
=> NT: 4 Temp: 7.29 (%Acc): 61.83 <Step>:177.4144 <R-factor>: 27.5773
=> NT: 5 Temp: 6.56 (%Acc): 63.58 <Step>:172.6875 <R-factor>: 16.4234
=> NT: 6 Temp: 5.90 (%Acc): 62.25 <Step>:171.5513 <R-factor>: 18.6187
=> NT: 7 Temp: 5.31 (%Acc): 61.50 <Step>:169.7019 <R-factor>: 14.4648
=> NT: 8 Temp: 4.78 (%Acc): 66.83 <Step>:167.7481 <R-factor>: 14.2252
=> NT: 9 Temp: 4.30 (%Acc): 64.67 <Step>:167.7481 <R-factor>: 13.8968
=> NT: 10 Temp: 3.87 (%Acc): 64.58 <Step>:166.6603 <R-factor>: 12.1475
=> NT: 11 Temp: 3.49 (%Acc): 63.17 <Step>:166.2180 <R-factor>: 10.8446
=> NT: 12 Temp: 3.14 (%Acc): 64.08 <Step>:166.1077 <R-factor>: 6.9079
=> NT: 13 Temp: 2.82 (%Acc): 60.42 <Step>:165.7257 <R-factor>: 7.5311
=> NT: 14 Temp: 2.54 (%Acc): 63.50 <Step>:165.0035 <R-factor>: 7.4506
=> NT: 15 Temp: 2.29 (%Acc): 63.83 <Step>:163.9018 <R-factor>: 6.9362
=> NT: 16 Temp: 2.06 (%Acc): 65.42 <Step>:163.6537 <R-factor>: 5.8735
=> NT: 17 Temp: 1.85 (%Acc): 62.92 <Step>:163.6499 <R-factor>: 6.0264
=> NT: 18 Temp: 1.67 (%Acc): 64.92 <Step>:163.4573 <R-factor>: 6.4474
=> NT: 19 Temp: 1.50 (%Acc): 64.33 <Step>:163.4573 <R-factor>: 4.6377
=> NT: 20 Temp: 1.35 (%Acc): 64.08 <Step>:163.4573 <R-factor>: 4.8564
=> NT: 21 Temp: 1.22 (%Acc): 63.67 <Step>:163.3963 <R-factor>: 4.2360
=> NT: 22 Temp: 1.09 (%Acc): 63.50 <Step>:162.9754 <R-factor>: 4.2860
=> NT: 23 Temp: 0.98 (%Acc): 63.17 <Step>:162.3272 <R-factor>: 4.0311
=> NT: 24 Temp: 0.89 (%Acc): 59.42 <Step>:162.2940 <R-factor>: 4.1985
=> NT: 25 Temp: 0.80 (%Acc): 55.50 <Step>:162.1390 <R-factor>: 3.9900
=> NT: 26 Temp: 0.72 (%Acc): 62.17 <Step>:161.9370 <R-factor>: 3.3584
=> NT: 27 Temp: 0.65 (%Acc): 63.67 <Step>:161.7196 <R-factor>: 2.9223

```



Simulated Annealing run of *FullProf*

```

FullProf.2k_Multi_Pattern
=> NT: 73 Temp: 0.01 (%Acc): 41.42 <Step>: 14.1507 <R-factor>: 1.7957
=> NT: 74 Temp: 0.00 (%Acc): 38.75 <Step>: 13.1795 <R-factor>: 1.7943
=> NT: 75 Temp: 0.00 (%Acc): 41.92 <Step>: 11.1802 <R-factor>: 1.7894
=> NT: 76 Temp: 0.00 (%Acc): 40.67 <Step>: 10.9559 <R-factor>: 1.7851
=> NT: 77 Temp: 0.00 (%Acc): 43.25 <Step>: 10.5303 <R-factor>: 1.7886
=> NT: 78 Temp: 0.00 (%Acc): 39.67 <Step>: 9.9443 <R-factor>: 1.7834
=> NT: 79 Temp: 0.00 (%Acc): 40.50 <Step>: 9.4706 <R-factor>: 1.7828
=> NT: 80 Temp: 0.00 (%Acc): 42.33 <Step>: 9.1180 <R-factor>: 1.7809
=> NT: 81 Temp: 0.00 (%Acc): 43.25 <Step>: 8.7911 <R-factor>: 1.7823
=> NT: 82 Temp: 0.00 (%Acc): 41.42 <Step>: 8.7285 <R-factor>: 1.7799
=> NT: 83 Temp: 0.00 (%Acc): 38.58 <Step>: 8.5013 <R-factor>: 1.7779
=> NT: 84 Temp: 0.00 (%Acc): 39.75 <Step>: 7.7504 <R-factor>: 1.7774
=> NT: 85 Temp: 0.00 (%Acc): 45.50 <Step>: 6.3718 <R-factor>: 1.7776

=> BEST CONFIGURATIONS FOUND BY Simulated Annealing FOR PHASE: 1
=> -> Configuration parameters ( 110 reflections):

=> Sol#: 1 RF2= 1.772 :
=> Rnom_T3_ Rnom_T4_ RPhi_T3_ RThet_T3 RPhi_T4_ RThet_T4 RPhi_T4_ RThet_T4
=> 1 2 3 4 5 6 7 8
=> 0.6694 6.8896 133.9639 89.7299 47.1463 7.8380 46.9585 7.8030

=> CPU Time: 89.018 seconds
=> 1.484 minutes

=> END Date:19/08/2003 Time => 01:49:53.476

=> Data Files :
=> - 1p4-pm2
=> PCR File : simann

```



Symmetry analysis for magnetic structure determination of $\text{KTb}_3\text{F}_{12}$

Space group: $I4/m$

Cell parameters: $a=7.695 \text{ \AA}$, $c=7.540 \text{ \AA}$

Propagation vector $\mathbf{k} = (1, 0, 0)$

Results of *BasIreps*:

$G_{\mathbf{k}} = I4/m$ (invariant vector), $\mathbf{k} \equiv -\mathbf{k}$

8 irreducible representations of dimension 1 (some complex!)

Site Tb^{3+} : 1 sublattice \Rightarrow 1: $(0,0,1/2)$

$$\Gamma_m = \Gamma_1 \oplus \Gamma_5 \oplus \Gamma_7$$

Site Tb^{4+} : 2 sublattices \Rightarrow 1: $(0,1/2,1/4)$ 2: $(0,1/2,3/4)$

$$\Gamma_m = \Gamma_2 \oplus \Gamma_3 \oplus \Gamma_5 \oplus \Gamma_6 \oplus \Gamma_7 \oplus \Gamma_8$$



Symmetry analysis for magnetic structure determination of $\text{KTb}_3\text{F}_{12}$

Basis functions of the Irreducible representations

Site Tb^{3+} : $\Gamma_1 : \mathbf{S}_{\mathbf{k}} = (0, 0, \nu)$

1 sublattice $(0,0,1/2)$ $\Gamma_5 : \mathbf{S}_{\mathbf{k}} = (\nu, 0, 0) + i(0, -\nu, 0)$

$\Gamma_7 : \mathbf{S}_{\mathbf{k}} = (\nu, 0, 0) + i(0, \nu, 0)$

Possible magnetic structures: $\Gamma_1, \Gamma_5 \oplus \Gamma_7$



Symmetry analysis for magnetic structure determination of $\text{KTb}_3\text{F}_{12}$

Site Tb^{4+} : 2 sublattices $(0,1/2,1/4)$, $(0,1/2,3/4)$

$$\Gamma_2 : \mathbf{S}_k(1) = (0, 0, u); \quad \mathbf{S}_k(2) = (0, 0, -u)$$

$$\Gamma_3 : \mathbf{S}_k(1) = (0, 0, u); \quad \mathbf{S}_k(2) = (0, 0, u)$$

$$\Gamma_5 : \mathbf{S}_k(1) = (u, 0, 0) + i(0, u, 0); \quad \mathbf{S}_k(2) = (u, 0, 0) + i(0, u, 0)$$

$$\Gamma_6 : \mathbf{S}_k(1) = (u, 0, 0) + i(0, -u, 0); \quad \mathbf{S}_k(2) = (-u, 0, 0) + i(0, u, 0)$$

$$\Gamma_7 : \mathbf{S}_k(1) = (u, 0, 0) + i(0, -u, 0); \quad \mathbf{S}_k(2) = (u, 0, 0) + i(0, -u, 0)$$

$$\Gamma_8 : \mathbf{S}_k(1) = (u, 0, 0) + i(0, u, 0); \quad \mathbf{S}_k(2) = (-u, 0, 0) + i(0, -u, 0)$$

Possible magnetic structures:

$$\Gamma_2, \quad \Gamma_3, \quad \Gamma_5 \oplus \Gamma_7, \quad \Gamma_6 \oplus \Gamma_8$$



Symmetry analysis for magnetic structure determination of $\text{KTb}_3\text{F}_{12}$

Site Tb^{3+} : 1 sublattice $(0,0,1/2)$

Site Tb^{4+} : 2 sublattices $(0,1/2,1/4)$, $(0,1/2,3/4)$,

Possible magnetic structures:

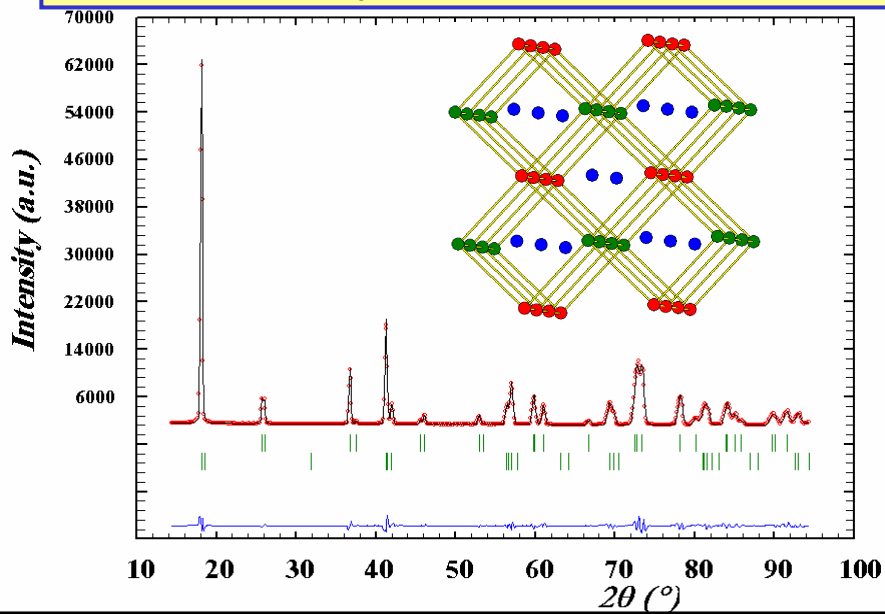
Only the mixed representation $\Gamma_5 \oplus \Gamma_7$ is possible for having magnetic moments in both sites. We have found experimentally that the Tb^{4+} orders according to the representation Γ_3 that is not allowed for the Tb^{3+} site, so this ion should not have a magnetic moment.

The representation Γ_3 is one dimensional, so there exist a magnetic Shubnikov group that is easily found realizing that for this representation the four-fold axis is primed (negative character) contrary to the mirror plane.

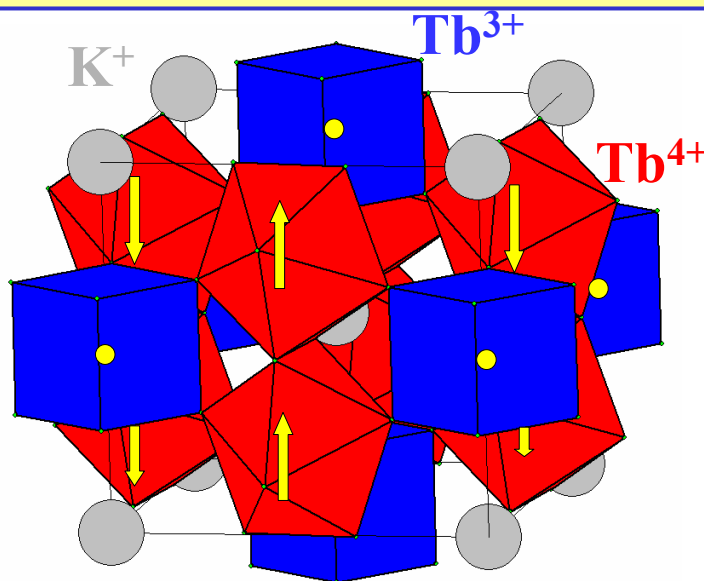
The Shubnikov group is: I_4'/m



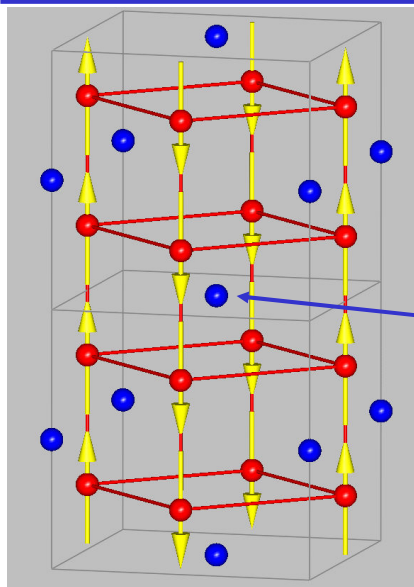
Rietveld refinement of the magnetic structure of $\text{KTb}_3\text{F}_{12}$ on G4.1 (LLB)



Magnetic Structure of $\text{KTb}^{3+}\text{Tb}_2^{4+}\text{F}_{12}$



Magnetic Structure of $\text{KTb}^{3+}\text{Tb}_2^{4+}\text{F}_{12}$



Only Tb^{4+} is ordered as F chains along c that are AF coupled in the basal plane

Tb^{3+} does not have static magnetic moment because the molecular field due to Tb^{4+} is exactly zero at its site. Tb^{3+} remains paramagnetic



Conclusions about the determination of the magnetic structure of $\text{KTb}_3\text{F}_{12}$

Simulated Annealing:

In this case, the information contained in the powder diffraction pattern is enough to obtain the magnetic structure without symmetry constraints.

Symmetry Analysis:

The irreducible representation involved in the magnetic phase transition, Γ_3 , is not allowed for the site of Tb^{3+} , so that this ion remains disordered (idle spin, “spin fou”).

One can verify that in the Shubnikov group $I_p 4'/m$ the magnetic point group of the site $2b(\text{Tb}^{3+})$, $4'/m$, is not admissible.



The end

Web site for the
FullProf Suite

<http://www.ill.fr/dif/Soft/fp/>

Go to the Diffraction Group web site: <http://www.ill.fr/dif>
Look for “Software”, “FullProf”...

The programs of the *FullProf Suite* are available for the
following platforms:

Windows and Linux
(a limited Mac OS version will be available soon)

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