أكاديمية البحث العلمي والتكنولوجيا
Academy of Scientific Research
and Technology

FLNP

Crystal and magnetic structure of advanced metal oxides: neutron diffraction studies.

Presented by:

- **Mohamed Sami** – MSc Student, Institute of Graduate Studies and Research
- **Mostafa M. Elkady** – MSc Student, South Valley University
- **Sanaa Ismail** – BSc Graduate Student, Suez University

Project Supervisors:

- **Nadezhda Belozerova** – Junior Researcher, Condensed Matter Department, Frank Laboratory of Neutron Physics, JINR
- **Dr. Sergey Kichanov** – Leader Researcher, Condensed Matter Department, Frank Laboratory of Neutron Physics, JINR

Presentation Outline

What are neutrons?

Discovery of neutrons.

Fast pulsed reactor IBR-2.

Neutron diffraction and it's advantages.

Neutron Vs. X-ray diffraction.

Scientific software for data refinement.

Relation between temperature and the volume of the unit cell.

Neutron diffraction patterns for $Zn_{0.3}Cu_{0.7}Fe_{1.5}Ga_{0.5}O_4$ measured at different temperatures.

Relation between temperature and unit cell volume.

Relation between temperature and bond length.

Relation between temperature and bond angle.

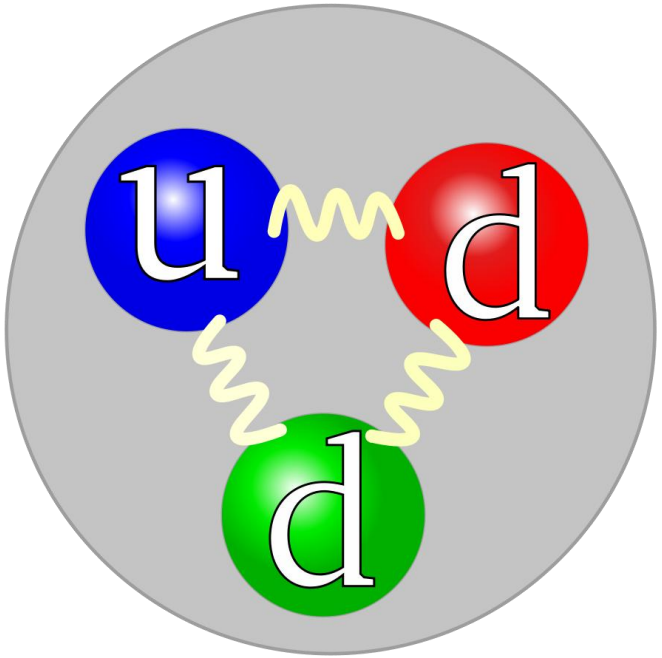
Temperature dependences of the magnetic moments of the atoms inside the spinel ferrite structure.

What are neutrons?

- Neutrons are subatomic particle with no net electric charge.
- Free neutrons are unstable; they under β decay, lifetime ~ 885.6 ± 0.8 sec.
- They can't be stored for long free;

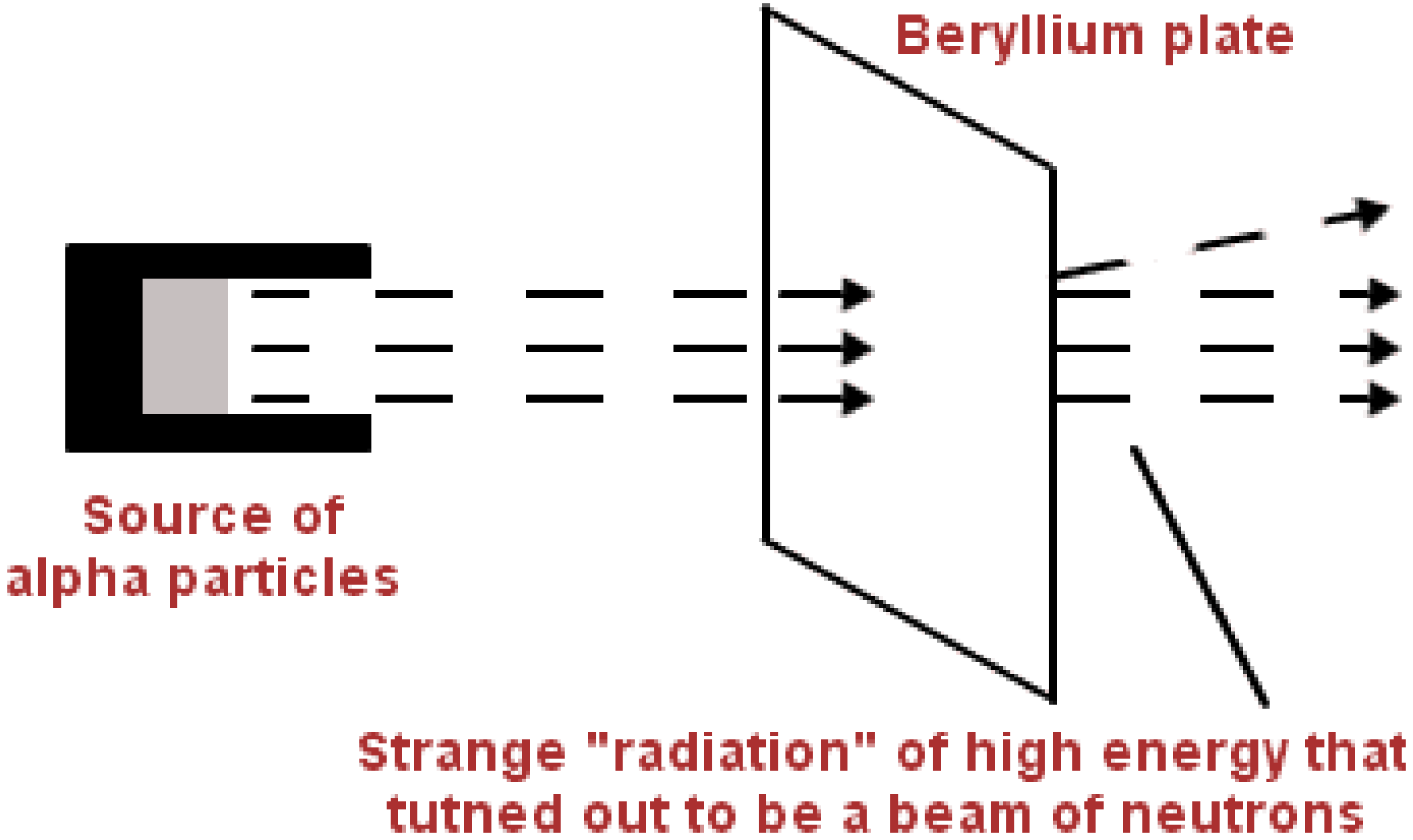
$$n \rightarrow p^{+} + e^{-} + \bar{\nu}_{e}$$

- It has Particle wave.
- Mass, Spin 1/2, Magnetic dipole moment.
- Neutrons interact with the nucleus.



schematic illustration for the neutron

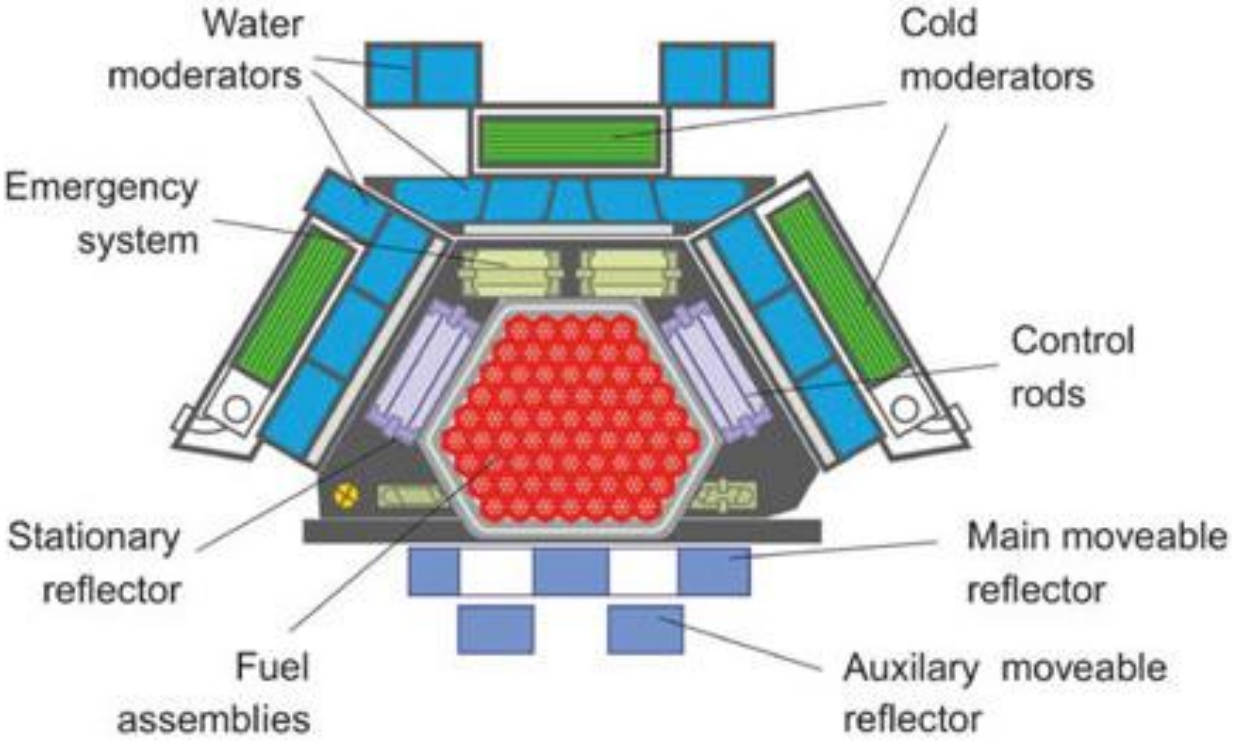
Discovery of neutrons

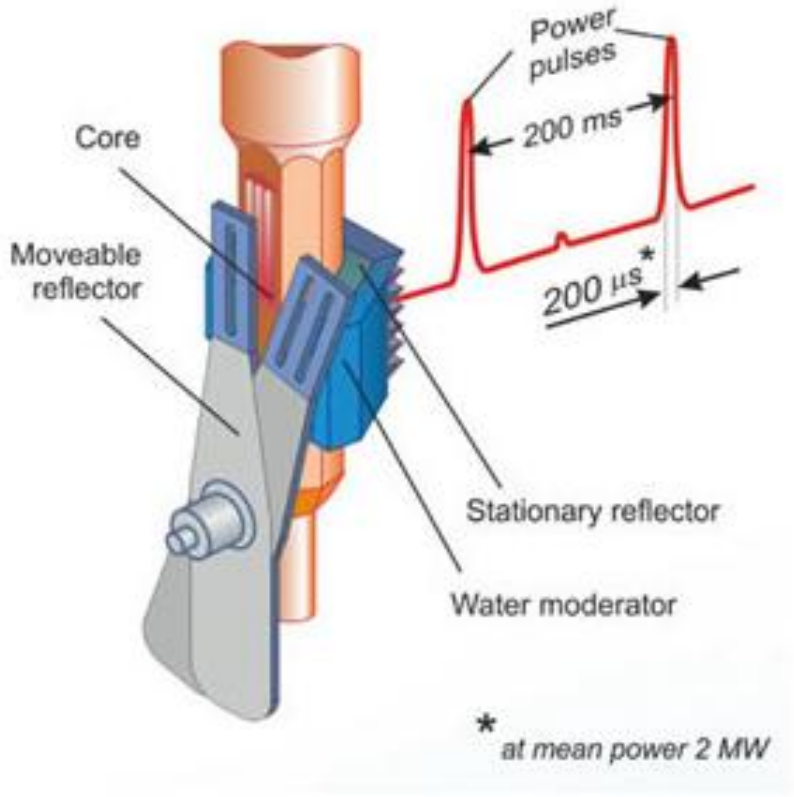


Neutron sources

- Small-scale neutron sources.
- Fission reaction.
- Neutron sources based on accelerators:
  I. Synthesis reaction
  II. Photonuclear reaction
  III. Spallation reaction

Fast pulsed reactor IBR-2





Neutron Deacceleration

Materials for deaccelerating neutrons

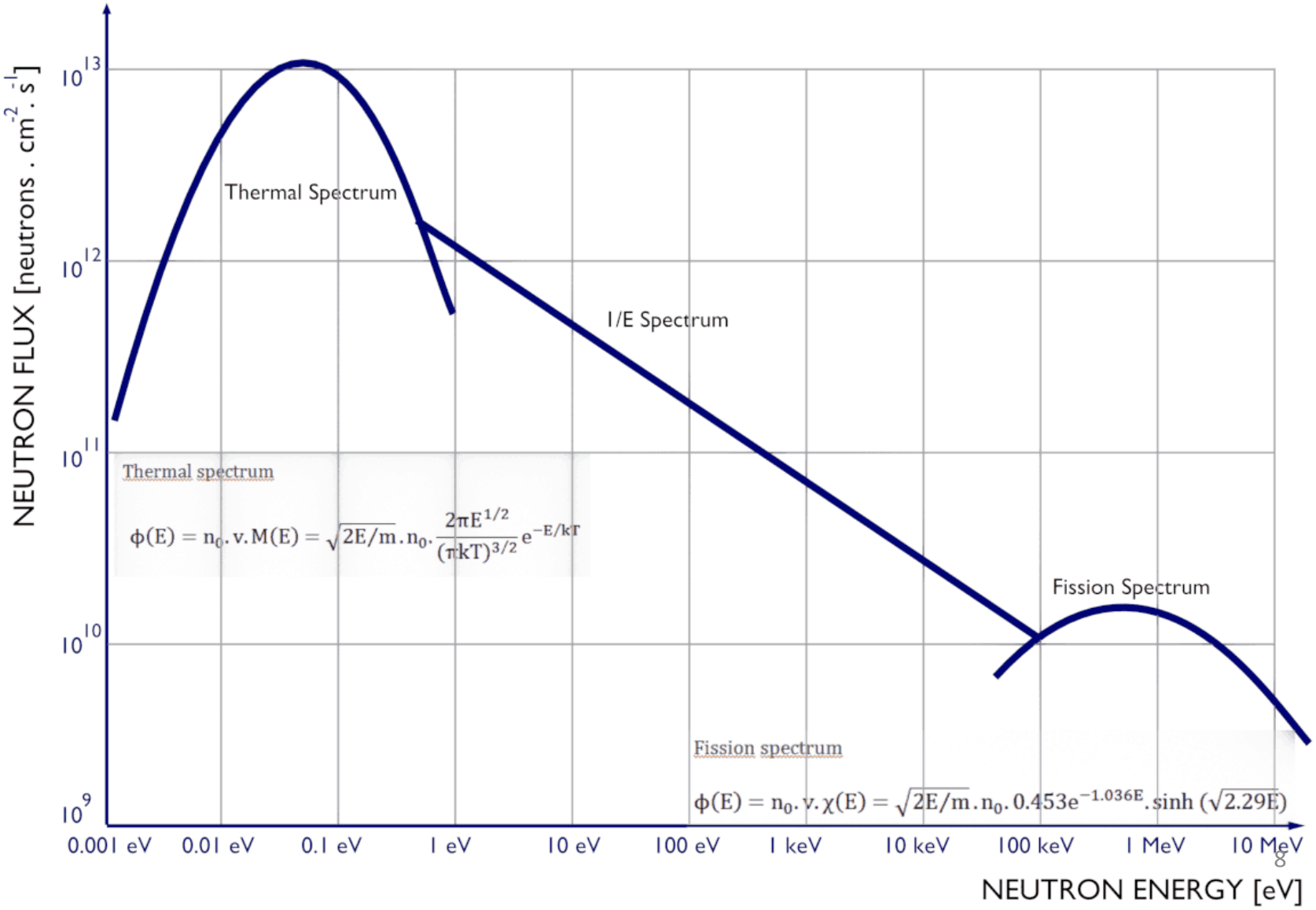
H_2O

D_2O

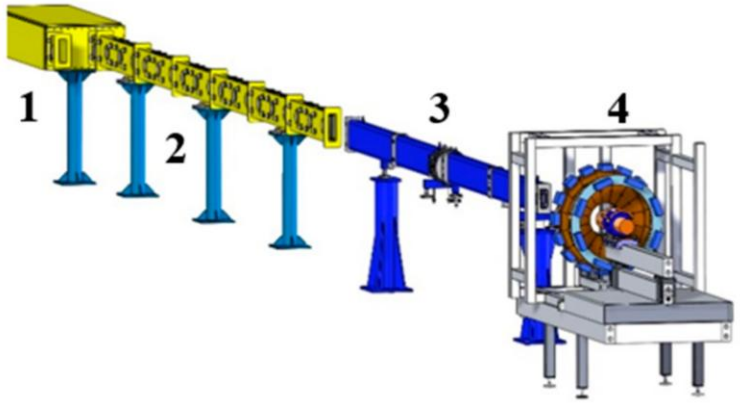
Be

BeO

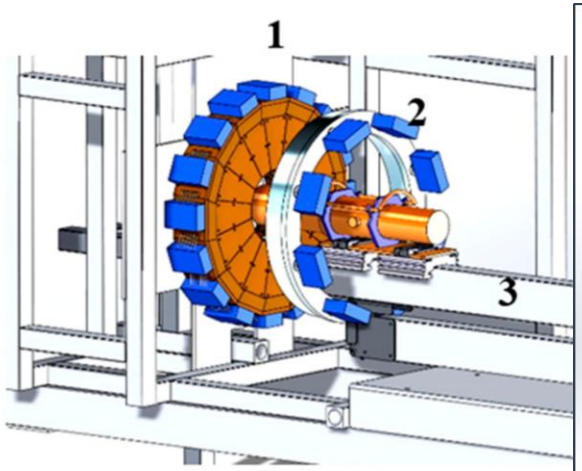
Graphite



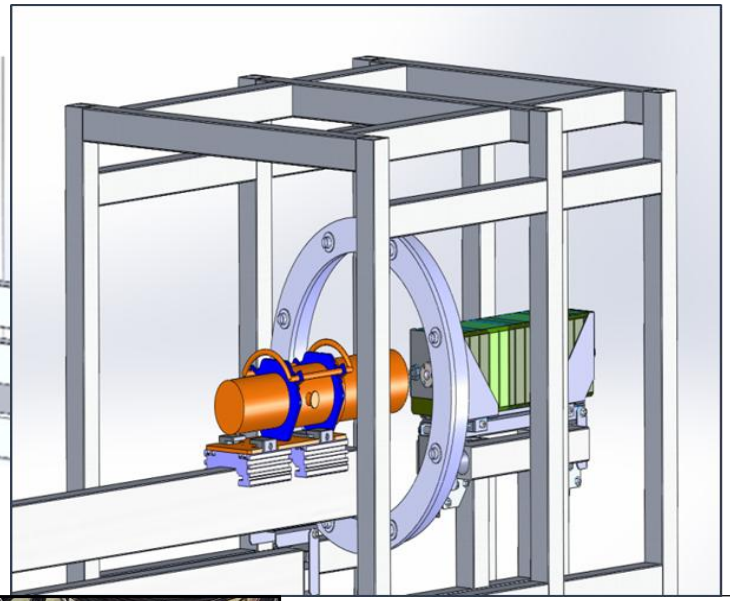
Neutron spectrometer

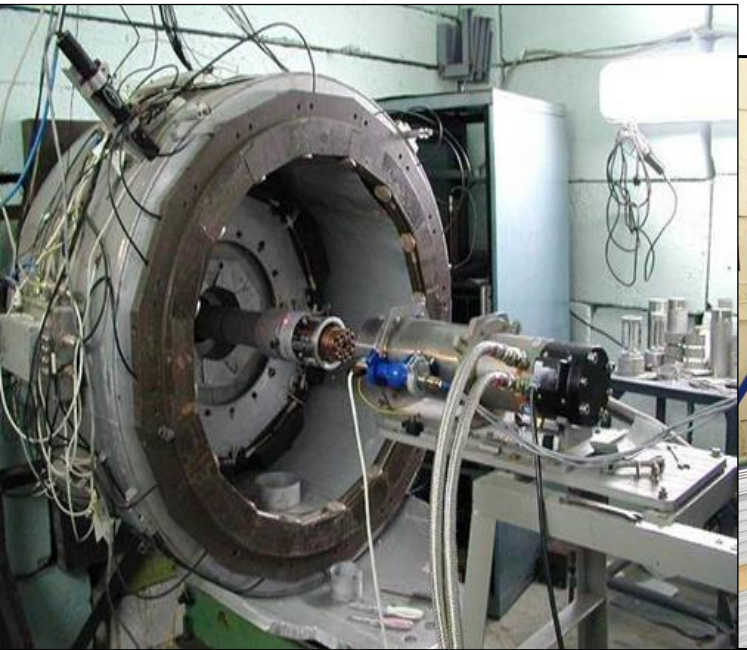


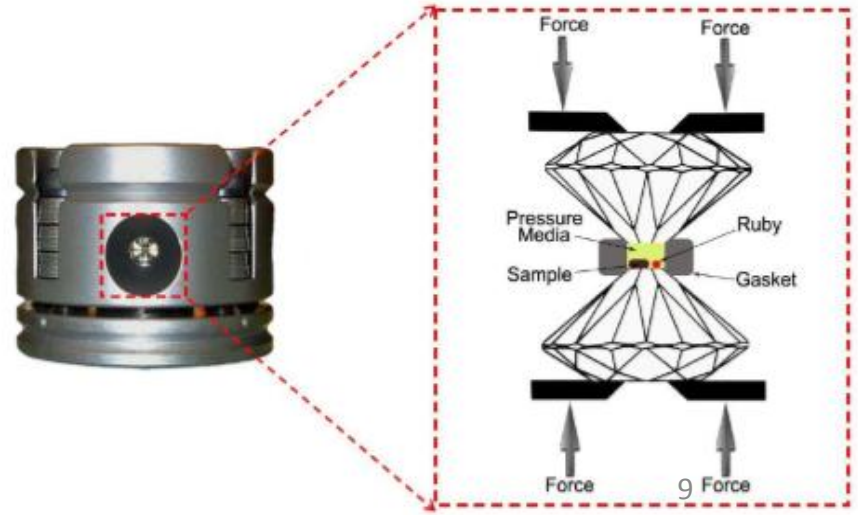
(a)



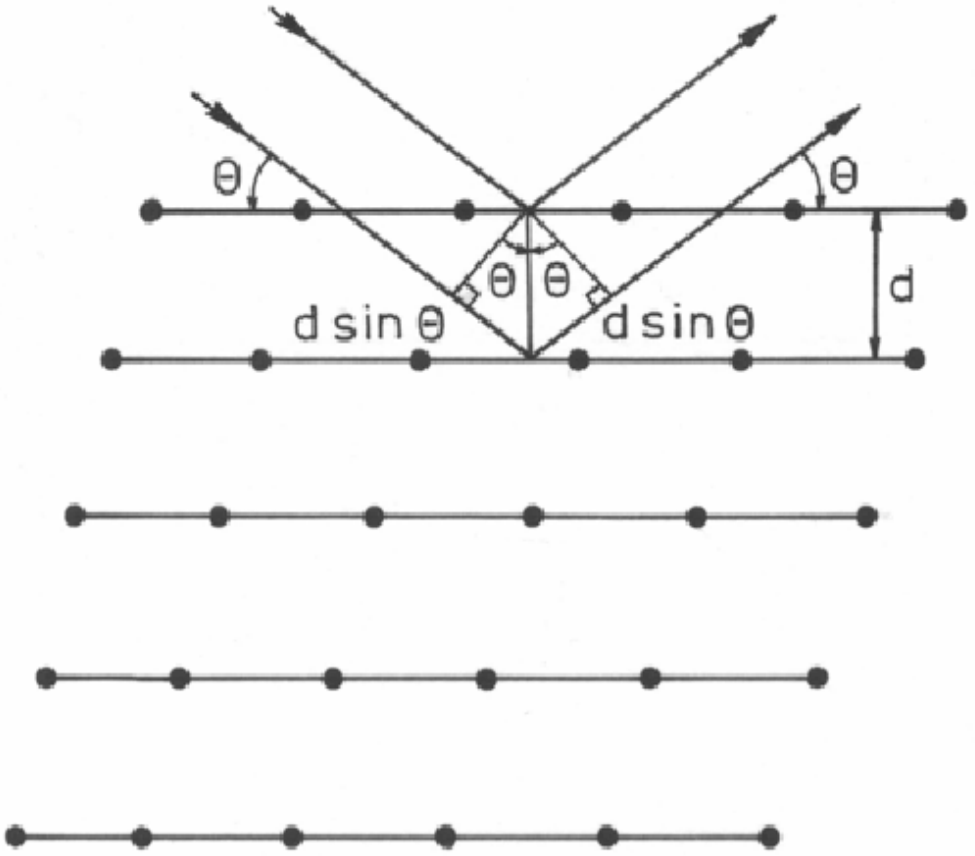
(b)



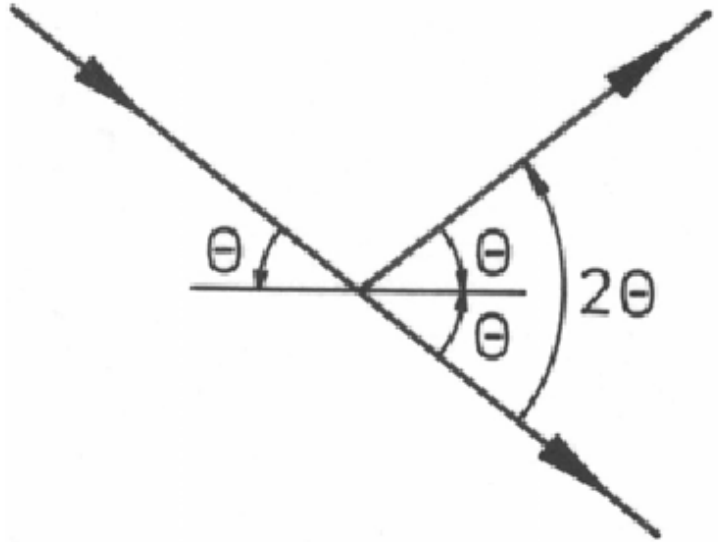




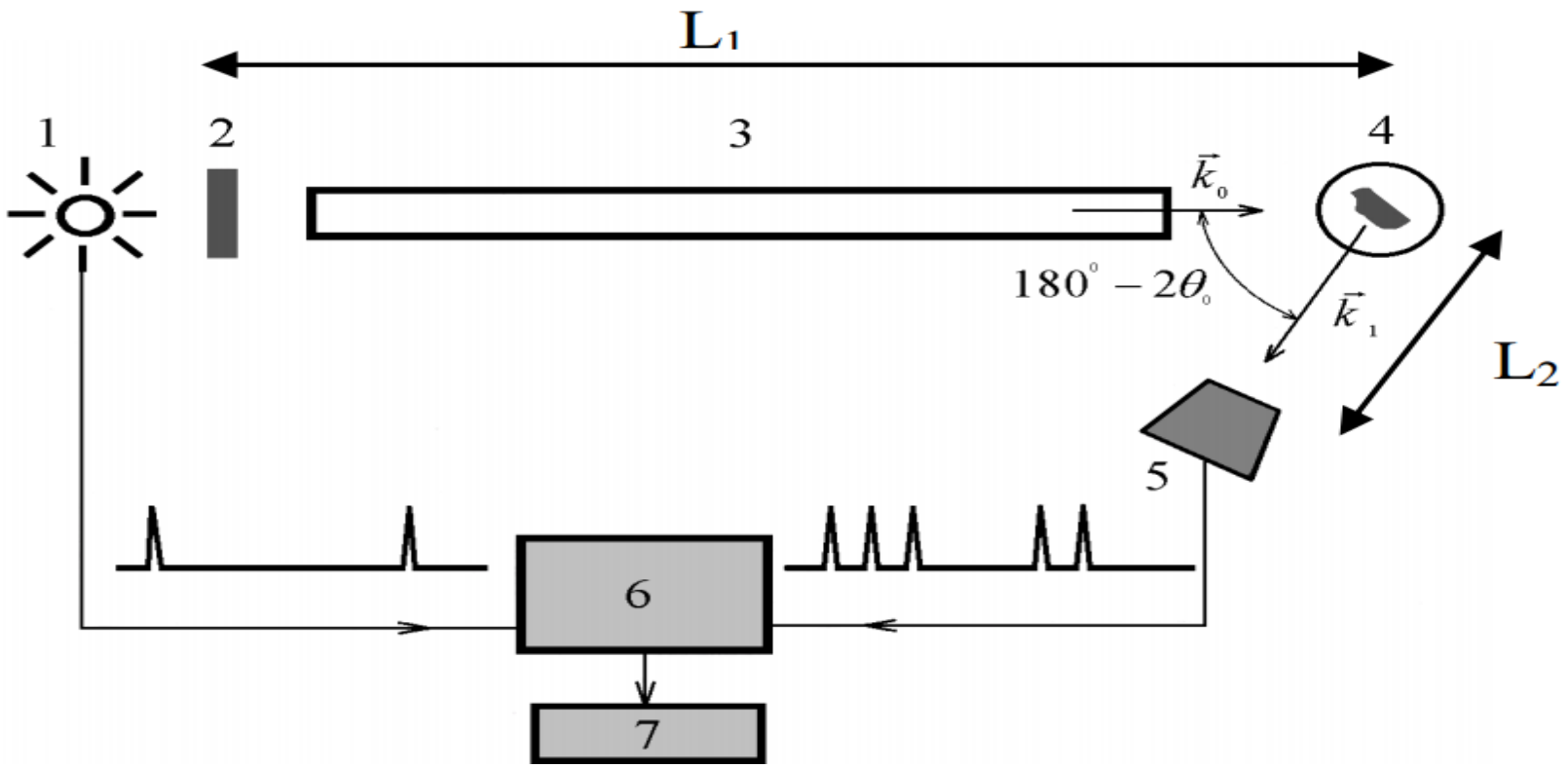
Neutron diffraction



$$2d_{hkl}sin\theta_0 = \lambda$$



Time of fly method



The lay-out of a TOF diffraction experiment:
1 – pulsed neutron source. 2 – moderator. 3 – flight path of the primary beam.
4 – sample. 5 – detector. 6 – time analyzer.
7 – RW memory

$$p = mv = \frac{h}{\lambda}$$

$$\lambda = \frac{h}{mv}$$

$$v = \frac{L_0 - L_1}{t}$$

$$\lambda = \frac{ht}{m(L_0 + L_1)}$$

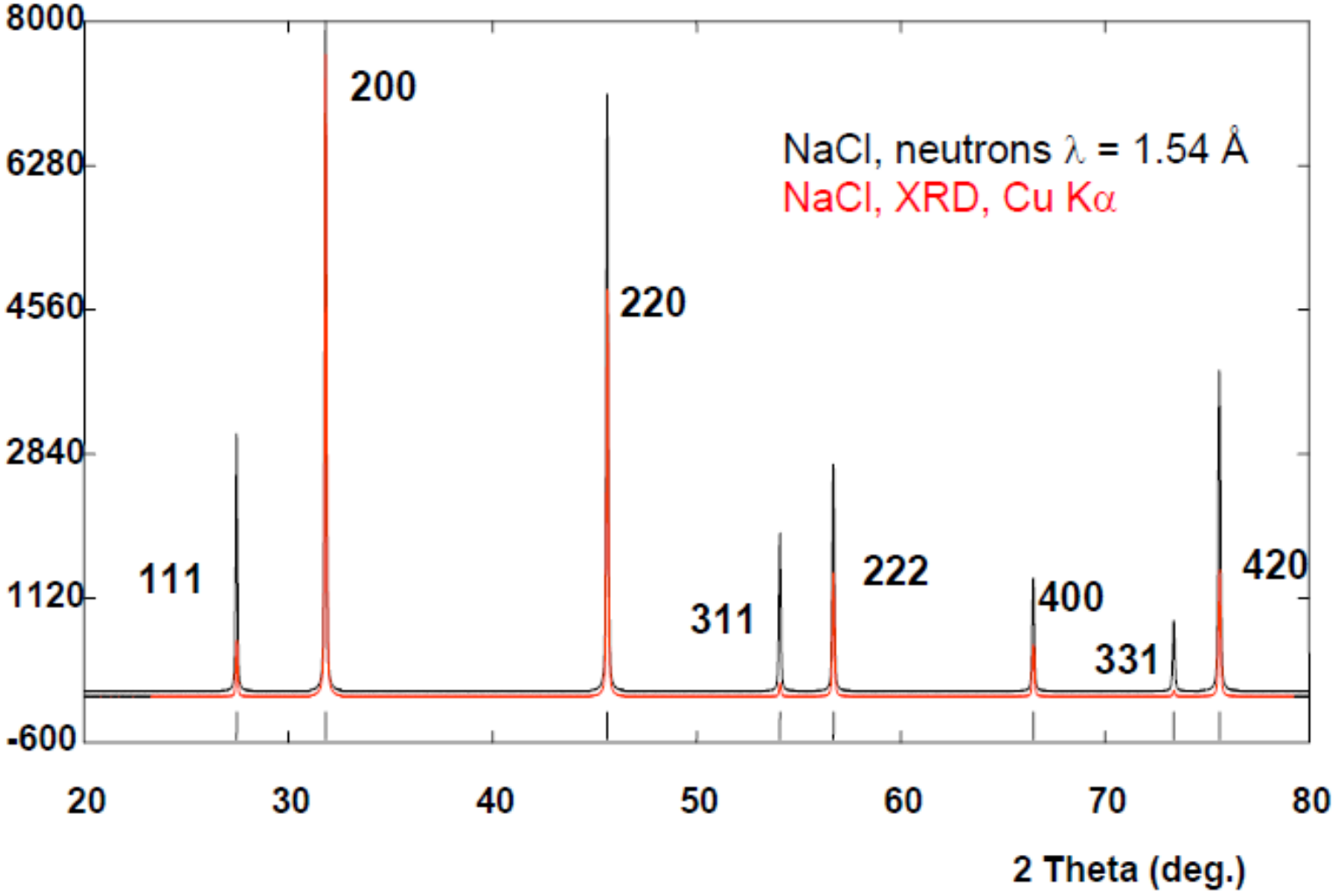
$$2d_{hkl}sin\theta_0 = \lambda$$

$$d_{hkl} = \frac{ht}{2m(L_0+L_1)sin\theta_0}$$

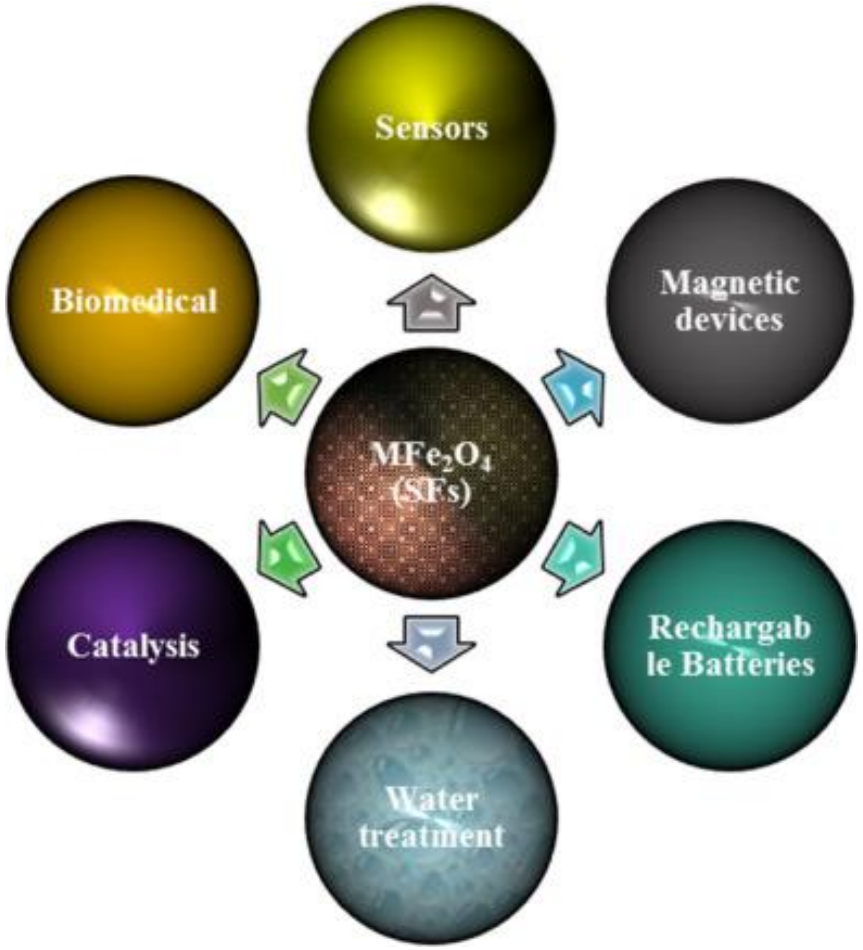
Neutron Vs. X-ray diffraction

Neutron diffraction	X-ray diffraction
Lower absorption	Stronger absorption
Large amounts of sample needed	Lower amounts of sample needed
Neighbors and isotopes can be discriminated	Neighbors and isotopes cannot be discriminated
Light elements can be seen	Light elements hard to detect
Low availability (nuclear reactor)	High availability (lab instrument)
Magnetic structures can be investigated	
Magnetic structures can be Investigated	

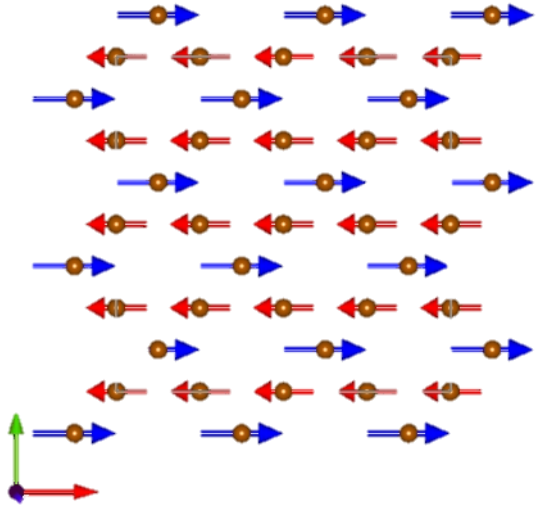
Example; Neutron Vs. XRD pattern



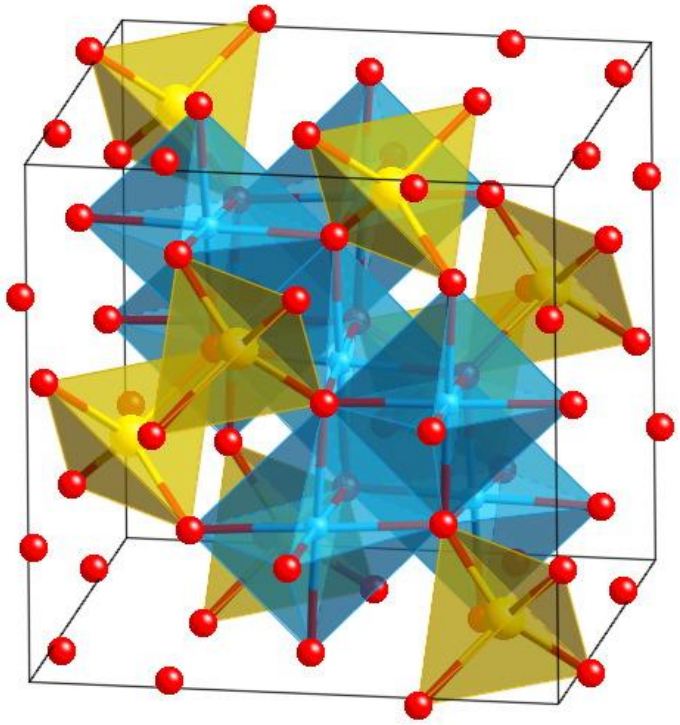
$Zn_{0.3}Cu_{0.7}Fe_{1.5}Ga_{0.5}O_4$ spinel ferrites



Application fields



Ferrimagnetic structure



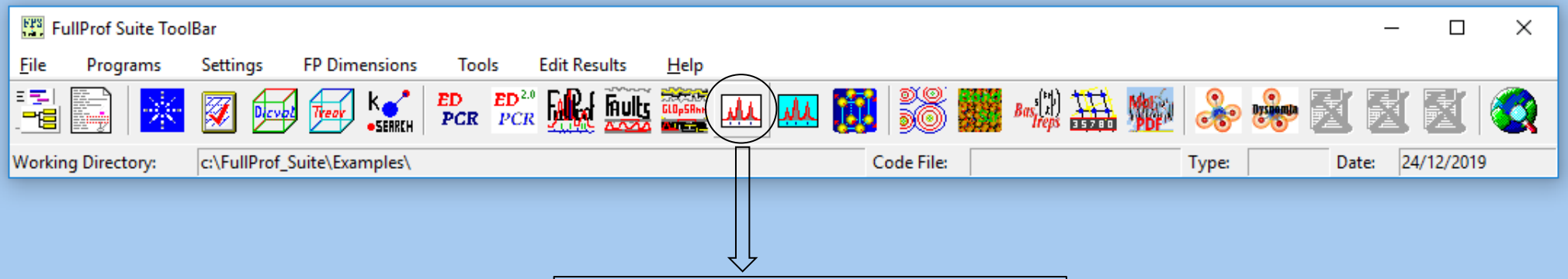
Cubic structure

Scientific software for data refinement

- Determine Lattice parameters, position of the atoms inside the unit cell, and the value of the magnetic dipole moment of those atoms.

- a schematic illustration for the unit cell using the obtained parameter from FullProf software and calculating the angles and distances between atoms

- Drawing the relation between obtained parameter and the temperature of the samples



WinPLOTR,

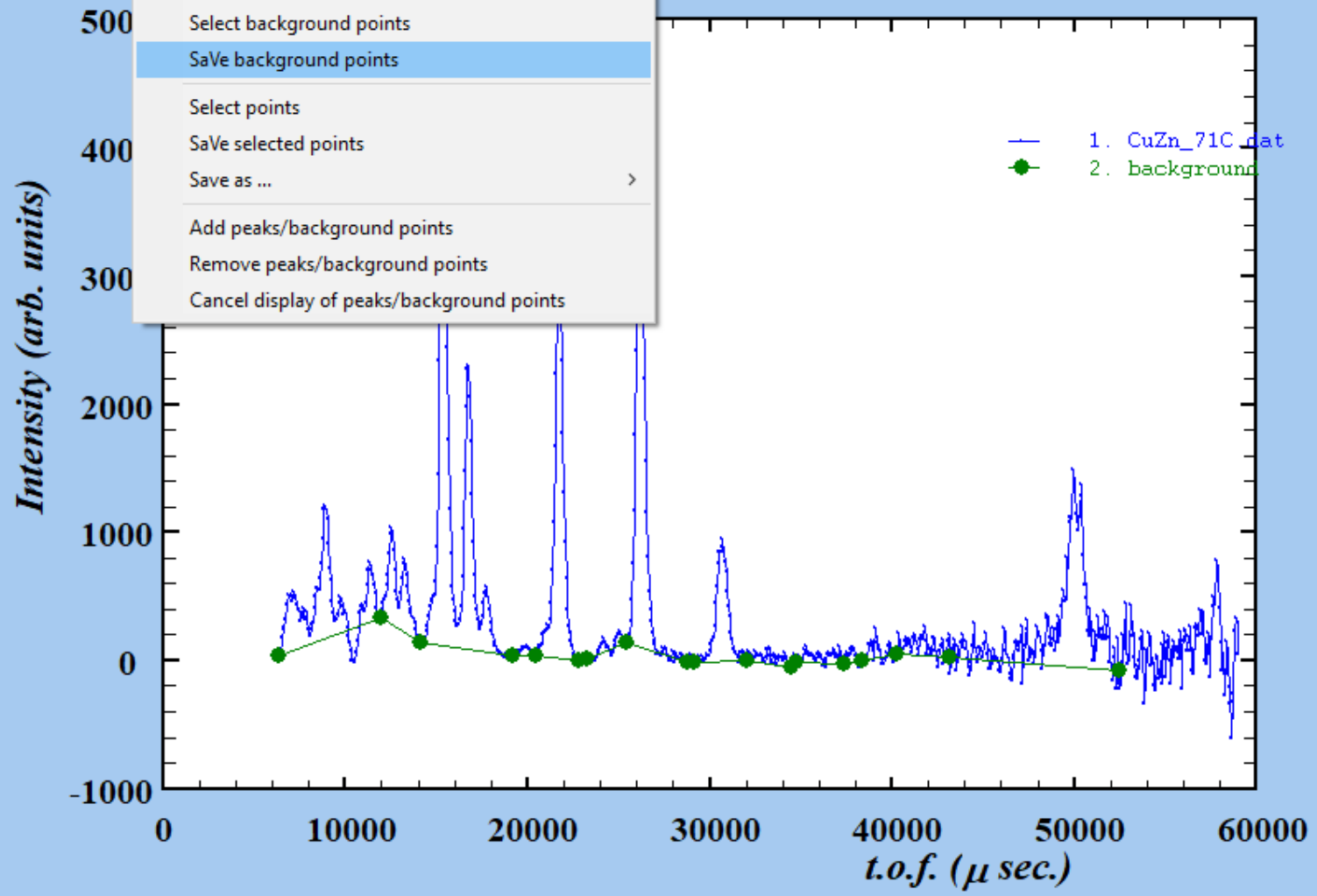
a graphic tool for powder diffraction

[Version: April 2019]

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]

File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help

- Automatic peak search
- Automatic satellites search (on .PRF file)
- Save peaks
- Save peaks as .PRN file
- Automatic background
- Select background points
- SaVe background points
- Select points
- SaVe selected points
- Save as ... ›
- Add peaks/background points
- Remove peaks/background points
- Cancel display of peaks/background points



Save background selected points

Background points:

	background_position	background_value
1	6336.00000	34.78909
2	11968.00000	333.46371
3	14144.00000	146.54225
4	19200.00000	31.68526
5	20416.00000	38.12156
6	22784.00000	2.07193
7	23296.00000	5.68234
8	25408.00000	141.97520
9	28736.00000	-12.76212
10	29184.00000	-10.94326
11	32000.00000	2.37467
12	34432.00000	-49.35867
13	34752.00000	-13.48343
14	37376.00000	-23.91998
15	38336.00000	-6.34380
16	40256.00000	48.16691
17	43200.00000	27.04206
18	52480.00000	-76.13683

CuZn_48C - Notepad

File Edit Format View Help

```
Fe3O4
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ    Nvk Npr More
   6   0   0 1.0 0.0 0.0   1   0  -1   0   0      2404.580   0   7   0
!
!
F -1                    <--Space group symbol for hkl generation
!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      Rx      Ry      Rz
!     Ix     Iy     Iz    beta11  beta22  beta33    MagPh
FeT1   MFE3  1  0  0.12500 0.12500 0.12500 0.90514  2.36250   3.877   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00    0.00    0.00    0.0
     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
FeT1   MFE3  1  0  0.87500 0.87500 0.87500 0.90514  2.36250   3.877   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00    0.00    0.00    0.0
     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
FeO1   MFE3  1  0  0.50000 0.50000 0.50000 0.90514  3.37500   1.400   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00   21.00    0.00    0.0
     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
FeO2   MFE3  1  0  0.25000 0.75000 0.00000 0.90514  3.37500   1.400   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00   21.00    0.00    0.0
     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
FeO3   MFE3  1  0  0.75000 0.00000 0.25000 0.90514  3.37500   1.400   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00   21.00    0.00    0.0
     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
FeO4   MFE3  1  0  0.00000 0.25000 0.75000 0.90514  3.37500   1.400   0.000   0.00
                      0.00    0.00    0.00    0.00     0.00   21.00    0.00    0.0
     0.000   0.000   0.000   0.000   0.000   0.000  0.00000
```

CuZn_48C - Notepad

File Edit Format View Help

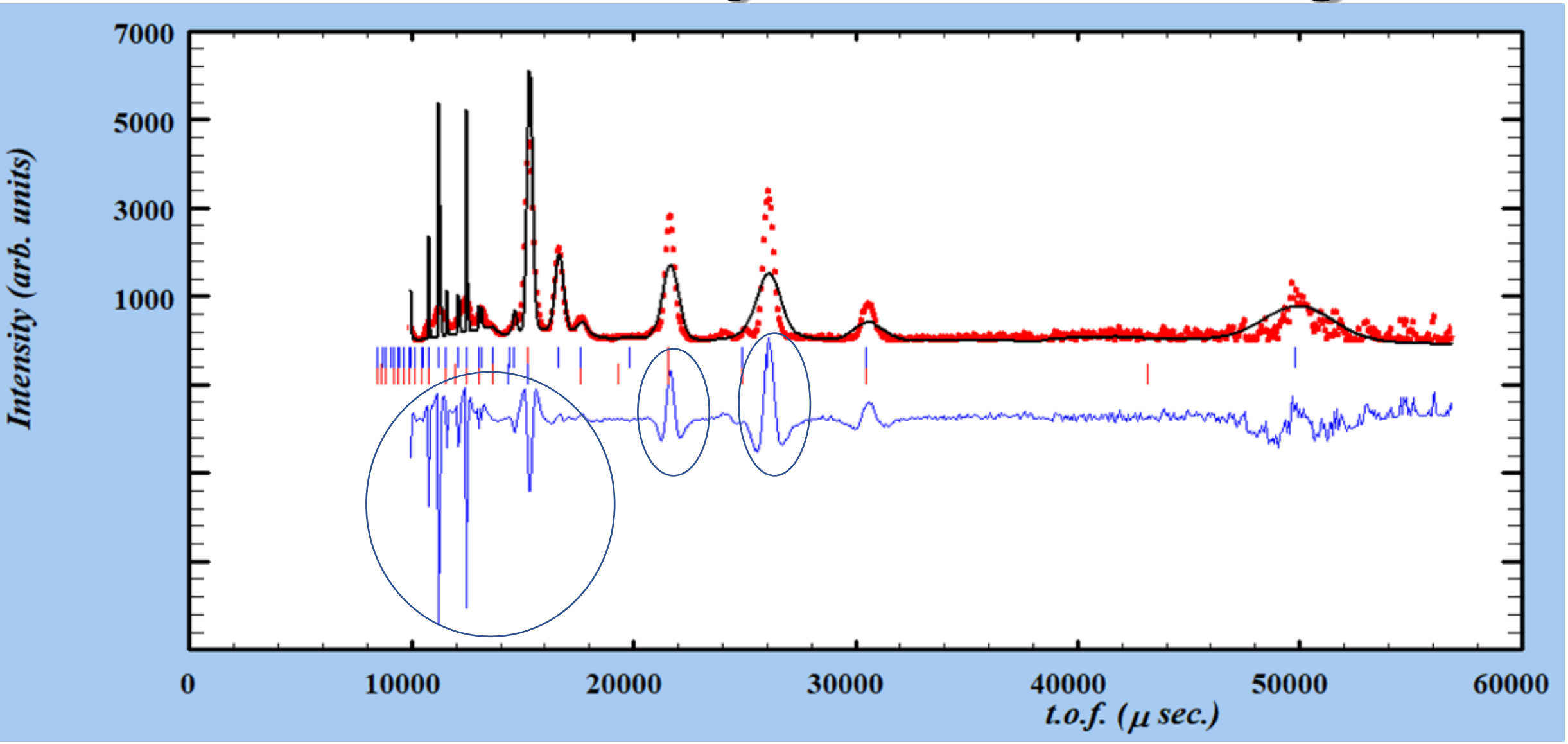
```
COMM  LPCM-70 , T= 48C
! Current global Chi2 (Bragg contrib.) =     0.1212E+05
! Files => DAT-file: cuzn_48c.dat,  PCR-file: cuzn_48c
!Job Npr Nph Nba Nex Nsc Nor Dum Iwg Ilo Ias Res Ste Nre Cry Uni Cor Opt Aut
  -1   9   3  65   2   0   0   1   0   0   1   0   0   0   0   1   0   0   1
!
!Ipr Ppl Ioc Mat Pcr Ls1 Ls2 Ls3 NLI Prf Ins Rpa Sym Hkl Fou Sho Ana
   1   0   1   0   1   1   4   0   0   3  10   1   1   1   0   0   1
!
!  Bkpos        Wdt    Iabscor for Pattern#  1
   15000.000    3.20     1
!NCY  Eps  R_at  R_an  R_pr  R_gl    TOF-min      <Step>       TOF-max
200  0.10  0.20  0.20  0.20  0.20    6656.0000     64.0782  59072.0000
!
```

CuZn_48C - Notepad

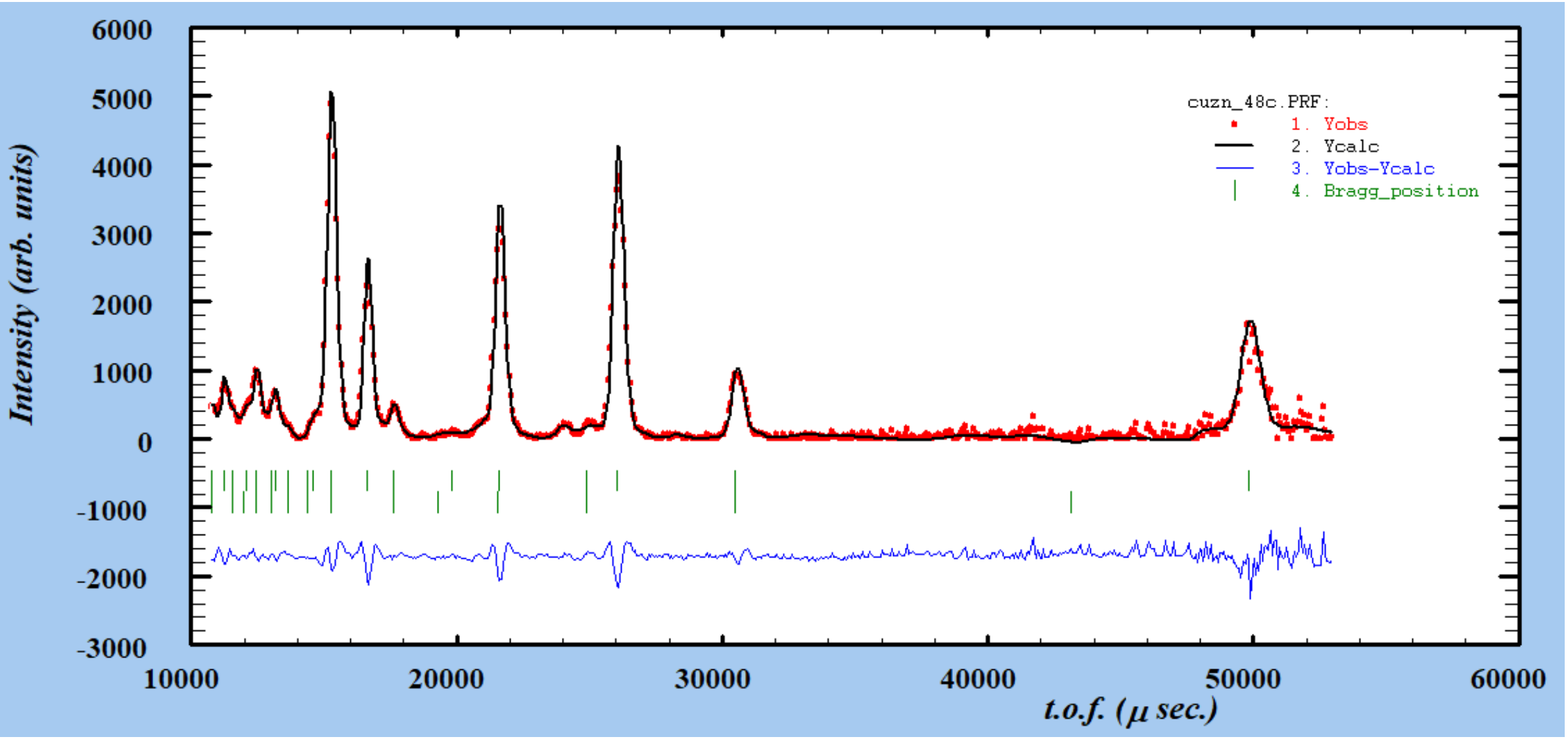
File Edit Format View Help

```
!  Data for PHASE number:   1  ==> Current R_Bragg for Pattern#  1:  11.2005
!-------------------------------------------------------------------------------
LPCM-75 (30% O-18); DN-6
!
!Nat Dis Ang Pr1 Pr2 Pr3 Jbt Irf Isy Str Furth       ATZ    Nvk Npr More
   7   0   0 0.0 0.0 1.0   0   0   0   0   0      50134.535   0   9   0
!
!
F d -3 m                <--Space group symbol
!Atom   Typ       X        Y        Z      Biso      Occ     In Fin N_t Spc /Codes
Zn     ZN      0.12500  0.12500  0.12500  1.94223  0.11250   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
GaA    Ga      0.12500  0.12500  0.12500  1.94223  0.00000   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
FeA    FE      0.12500  0.12500  0.12500  1.94223  0.26250   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
Cu     CU      0.50000  0.50000  0.50000 -1.41956  0.26250   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
FeB    FE      0.50000  0.50000  0.50000 -1.41956  0.37500   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
GaB    GA      0.50000  0.50000  0.50000 -1.41956  0.18750   0   0   0    0
                  0.00     0.00     0.00     0.00     0.00
O      O       0.25888  0.25888  0.25888  1.93015  1.50000   0   0   0    0
```

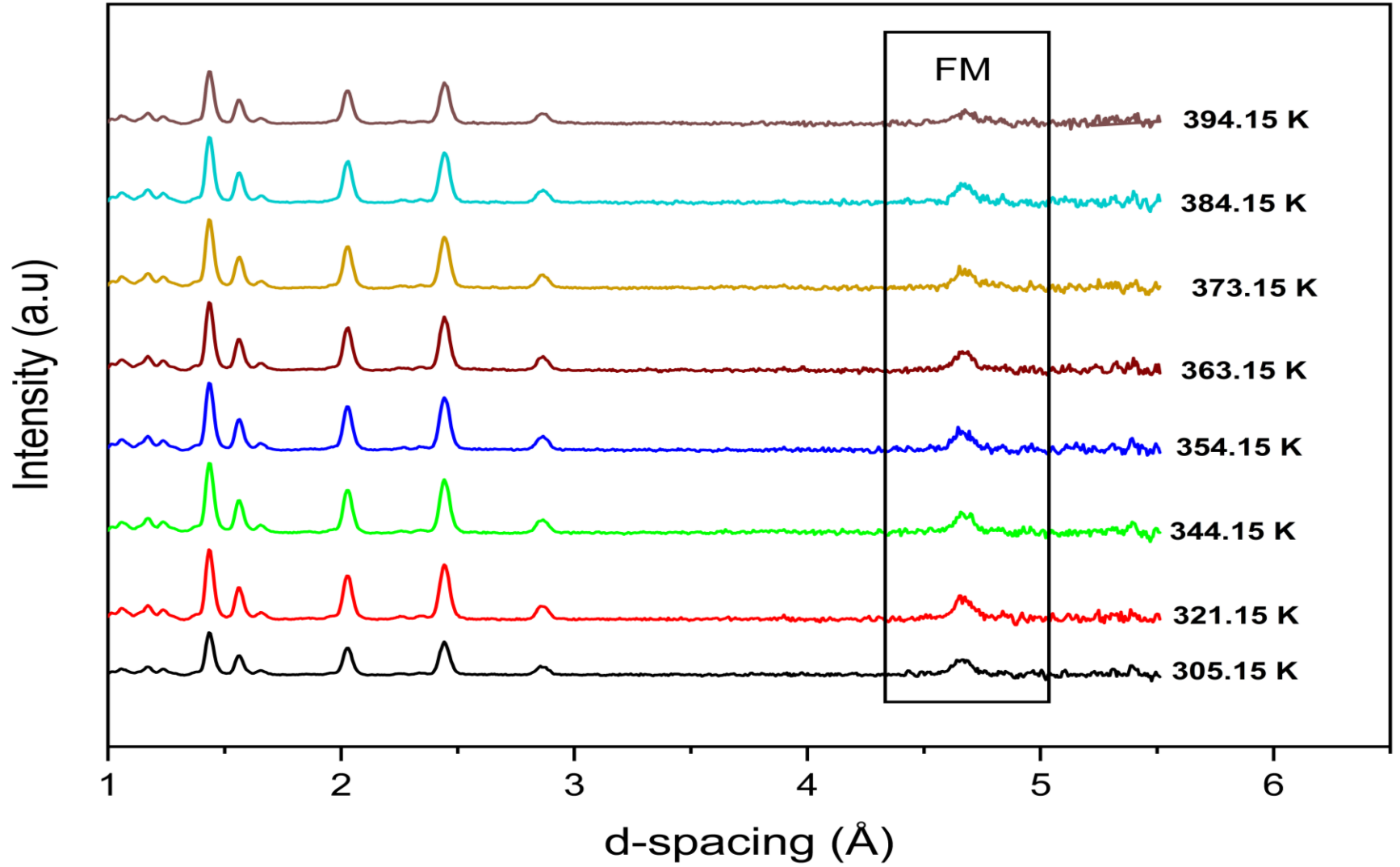
Our first try of data fitting



Better data fitting



Neutron diffraction patterns for $Zn_{0.3}Cu_{0.7}Fe_{1.5}Ga_{0.5}O_4$ measured at different temperatures.



Diamond - [Diamond1]

File Edit View Structure Picture Build Objects Move Tools Window Help

Diamond1

Title	Code	Formula sum	HM symbol	SGR no.	Cell parameters
Diamond1					

Pictures

No structure data sets defined for Diam

(You can create a data set and type in structure data with "
or insert structures from a file with "Structure/Ins

Tiles Details

New Structure

Atomic parameters

The atomic parameters use fractional coordinates for a crystal structure but orthogonal coordinates (in Angstroem units) for a "molecular structure".

Here you can define element (with oxidation number) as well as x, y, and z for every atom. For mixed or defect sites, standard uncertainties, and displacement parameters, use the "Atomic Parameters" dialog ("Structure" menu) instead.

Atom: O x/a: 0.2588 y/b: 0.2588 z/c: 0.2588 Add

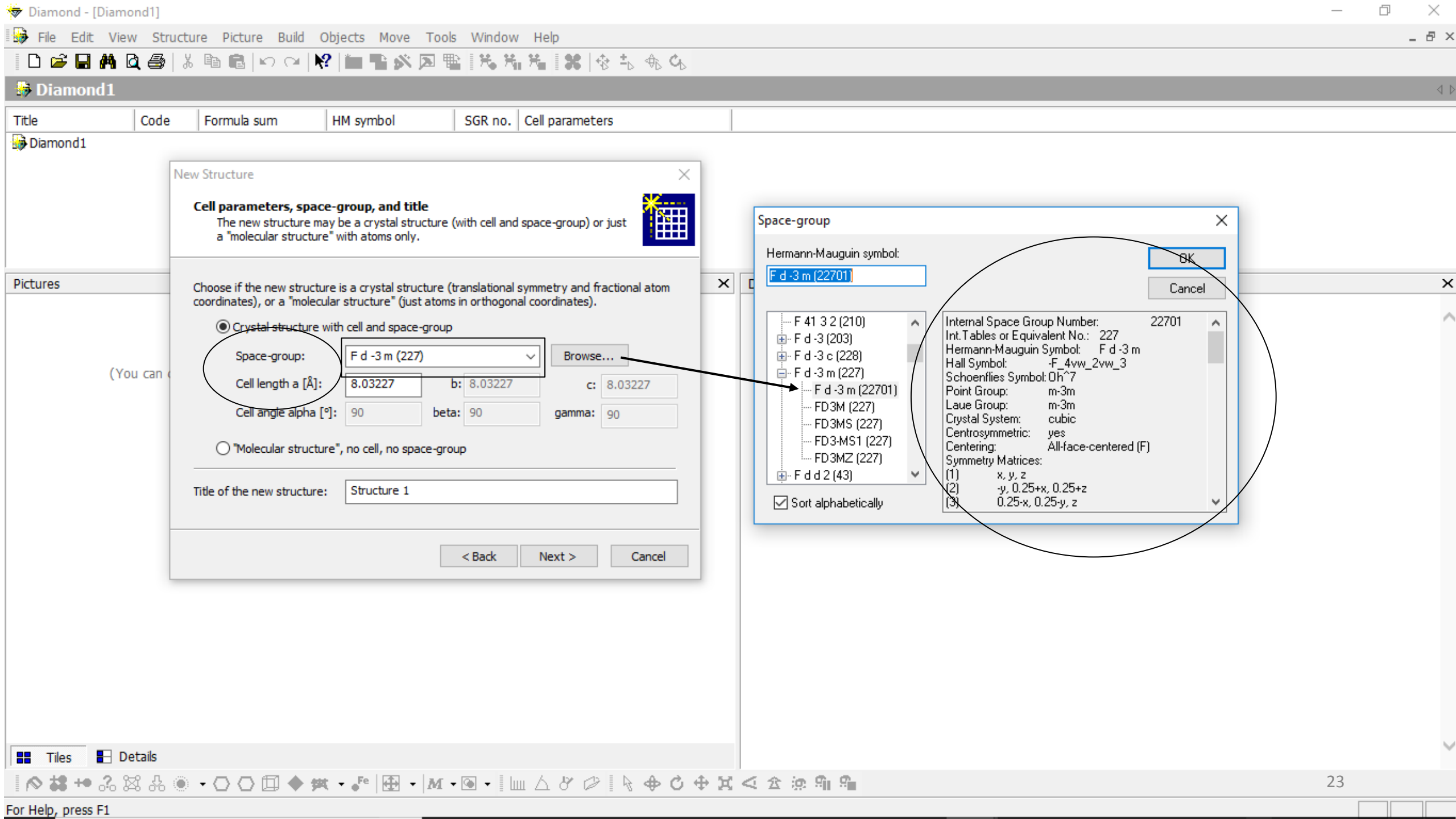
Atoms in the asymmetric unit:

Atom	x	y	z
FeA	0.125	0.125	0.125
FeB	0.5	0.5	0.5
O	0.2588	0.2588	0.2588

Delete

< Back Next > Cancel

For Help, press F1

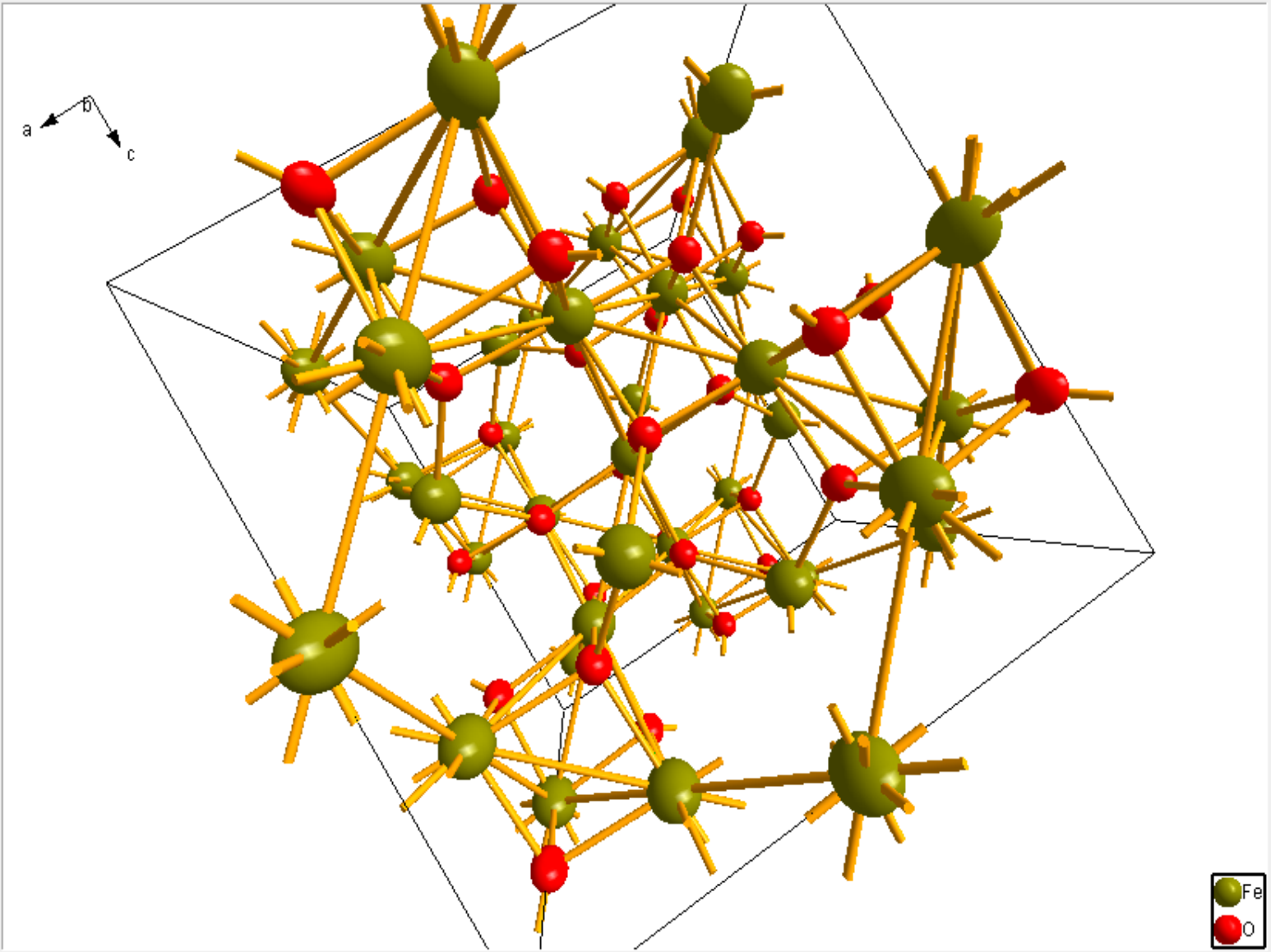

Diamond - [Diamond1]
File Edit View Structure Picture Build Objects Move Tools Window Help
Diamond1
Title Code Formula sum HM symbol SGR no. Cell parameters
Diamond1
New Structure
Cell parameters, space-group, and title
The new structure may be a crystal structure (with cell and space-group) or just a "molecular structure" with atoms only.
Choose if the new structure is a crystal structure (translational symmetry and fractional atom coordinates), or a "molecular structure" (just atoms in orthogonal coordinates).
Crystal structure with cell and space-group
Space-group: F d -3 m (227)
Browse...
Cell length a [Å]: 8.03227
b: 8.03227
c: 8.03227
Cell angle alpha [°]: 90
beta: 90
gamma: 90
"Molecular structure", no cell, no space-group
Title of the new structure: Structure 1
< Back
Next >
Cancel
Space-group
Hermann-Mauguin symbol:
F d -3 m (22701)
OK
Cancel
F 41 3 2 (210)
F d -3 (203)
F d -3 c (228)
F d -3 m (227)
F d -3 m (22701)
FD3M (227)
FD3MS (227)
FD3-MS1 (227)
FD3MZ (227)
F d d 2 (43)
Sort alphabetically
Internal Space Group Number: 22701
Int.Tables or Equivalent No.: 227
Hermann-Mauguin Symbol: F d -3 m
Hall Symbol: -F_4vw_2vw_3
Schoenflies Symbol: Oh^7
Point Group: m-3m
Laue Group: m-3m
Crystal System: cubic
Centrosymmetric: yes
Centering: All-face-centered (F)
Symmetry Matrices:
(1) x, y, z
(2) -y, 0.25+x, 0.25+z
(3) 0.25-x, 0.25-y, z
Pictures
Tiles
Details
23
For Help, press F1

Diamond - [Diamond3.diamdoc]

File Edit View Structure Picture Build Objects Move Tools Window Help

Diamond3.diamdoc > temp 90 > Picture 1

Data sheet | Distances/angles | Powder pattern



Data brief

General

Bibliographic data

Phase data

Space-group	F d -3 m (227) - cubic
Cell	a=8.0332 Å V=518.40 Å^3

Atomic parameters

Atom	Ox.	Wyck.	Site	S.O.F.	x/a	y/b	z/c	U [Å^2]
Fe1		8a	-43m		1/8	1/8	1/8	
Fe2		16d	.-3m		1/2	1/2	1/2	
O1		32e	.3m		0.26127	0.26127	0.26127	

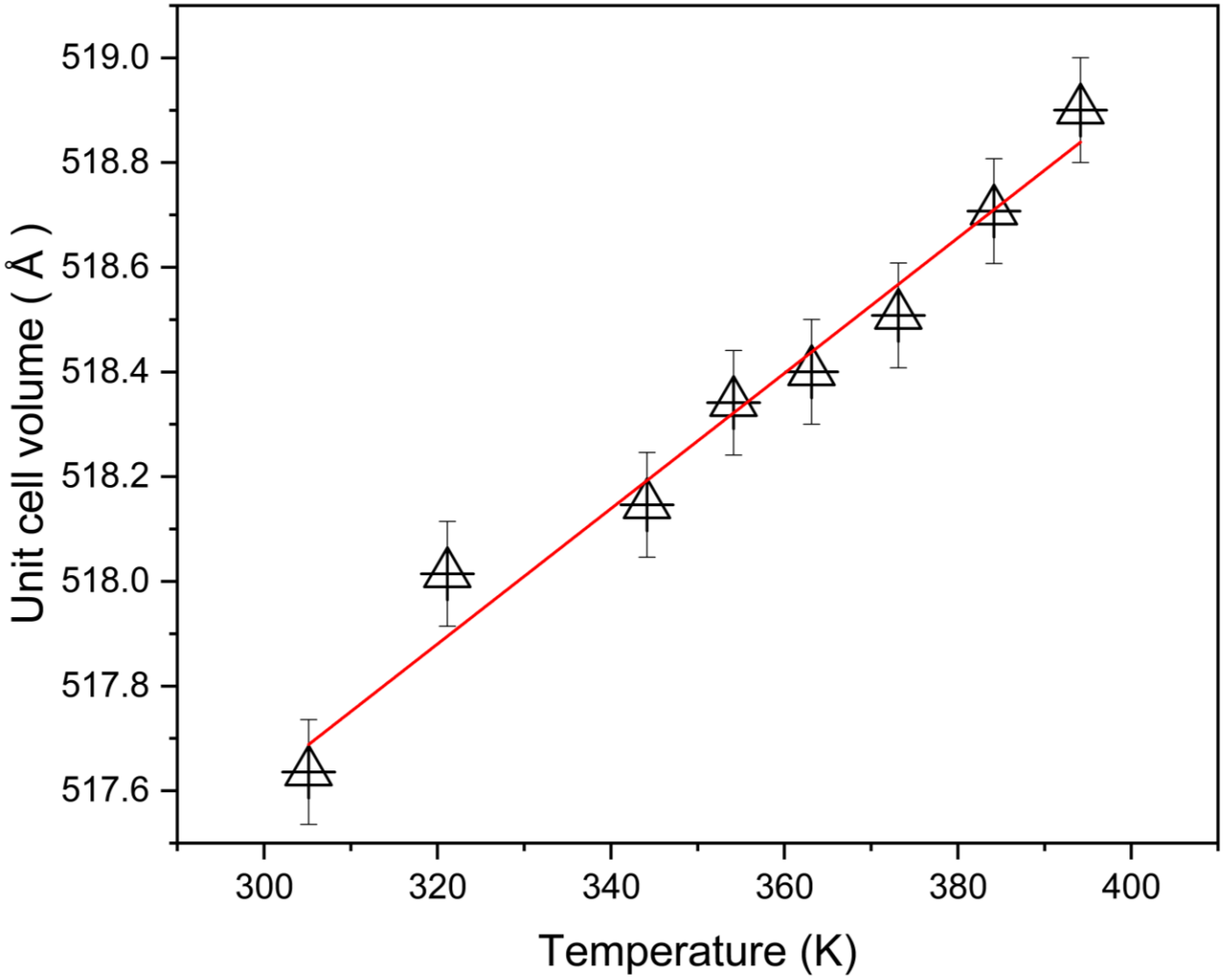
Properties

Structure picture contents

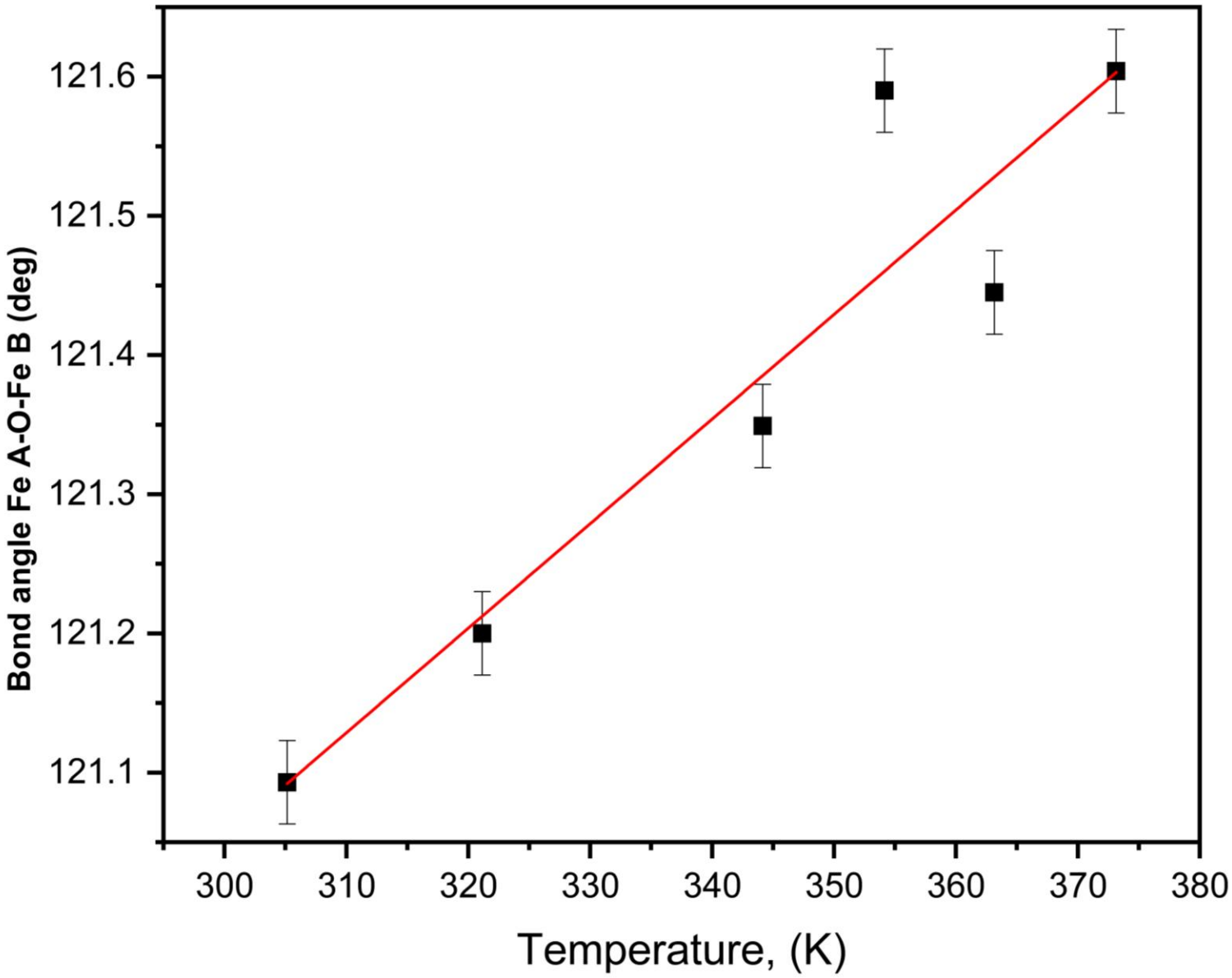
Atomic parameters	3
Symmetry records	48
Atoms in unit cell	56
Explicitely defined bonds	0
Created atoms	191
Created bonds	362
Cell corners	8
Cell edges	12

For Help, press F1

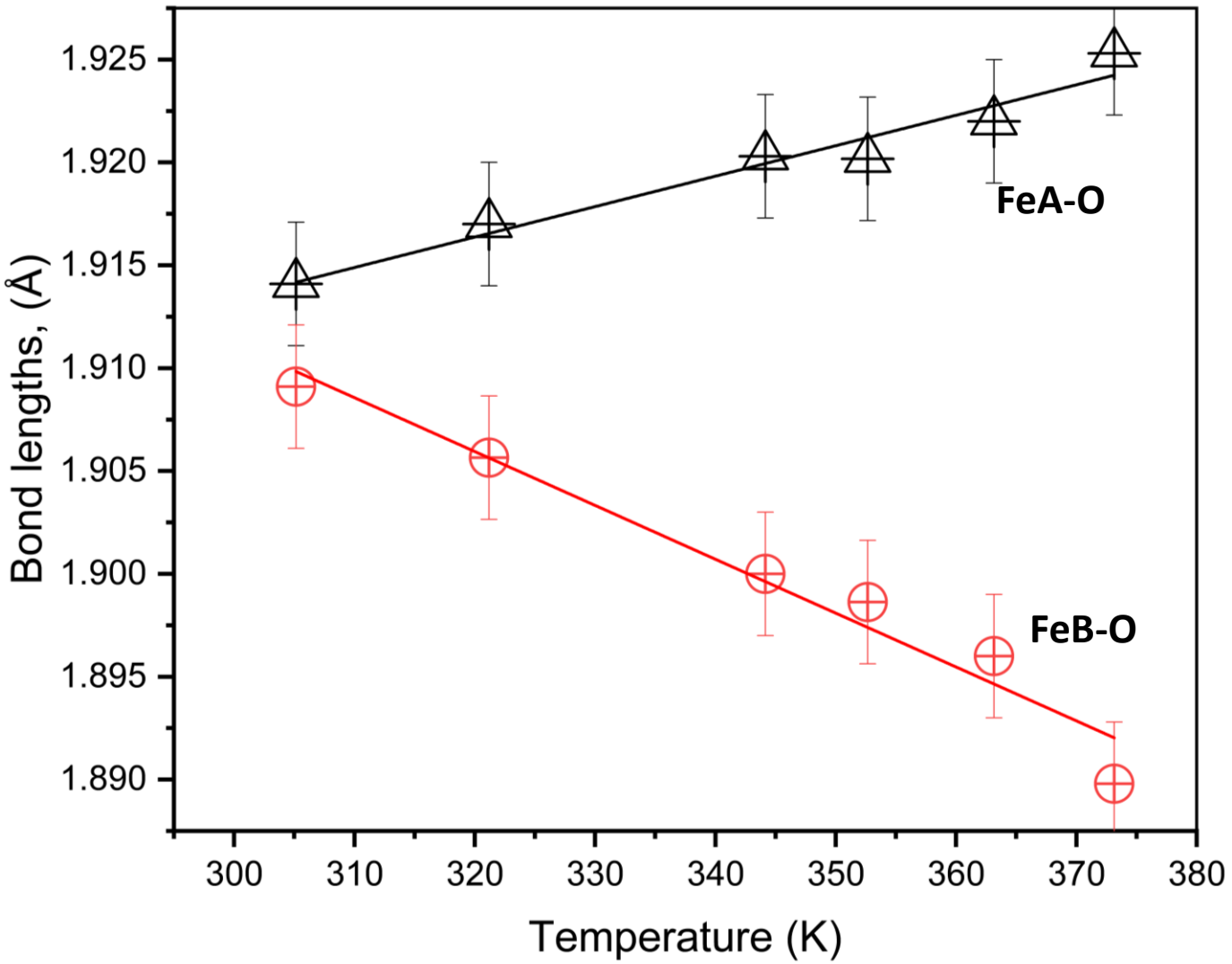
Relation between temperature and unit cell volume.



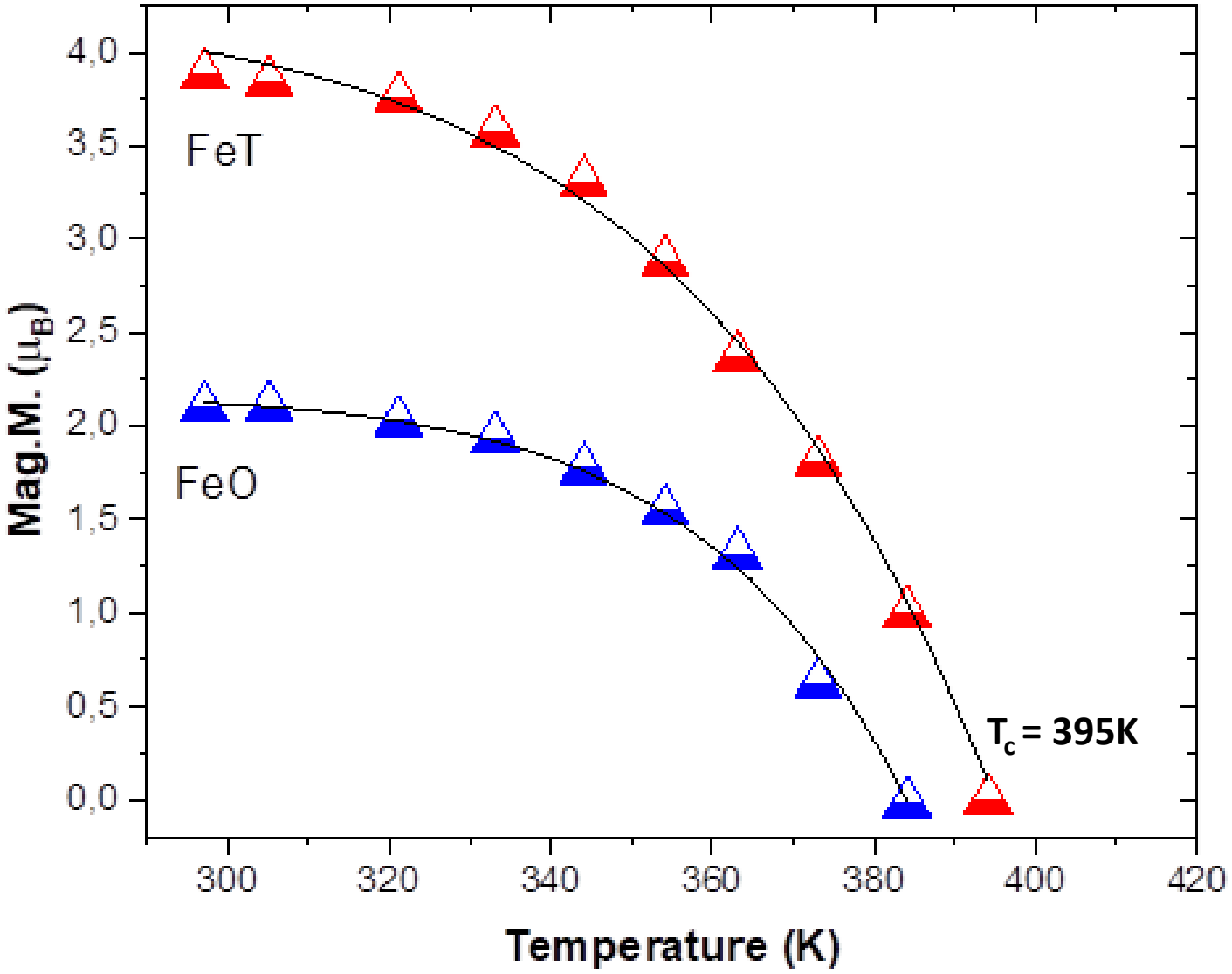
Relation between temperature and bond Angle



Relation between temperature and bond length



Temperature dependences of the magnetic moments of the atoms inside the spinel ferrite structure.



Thanks!