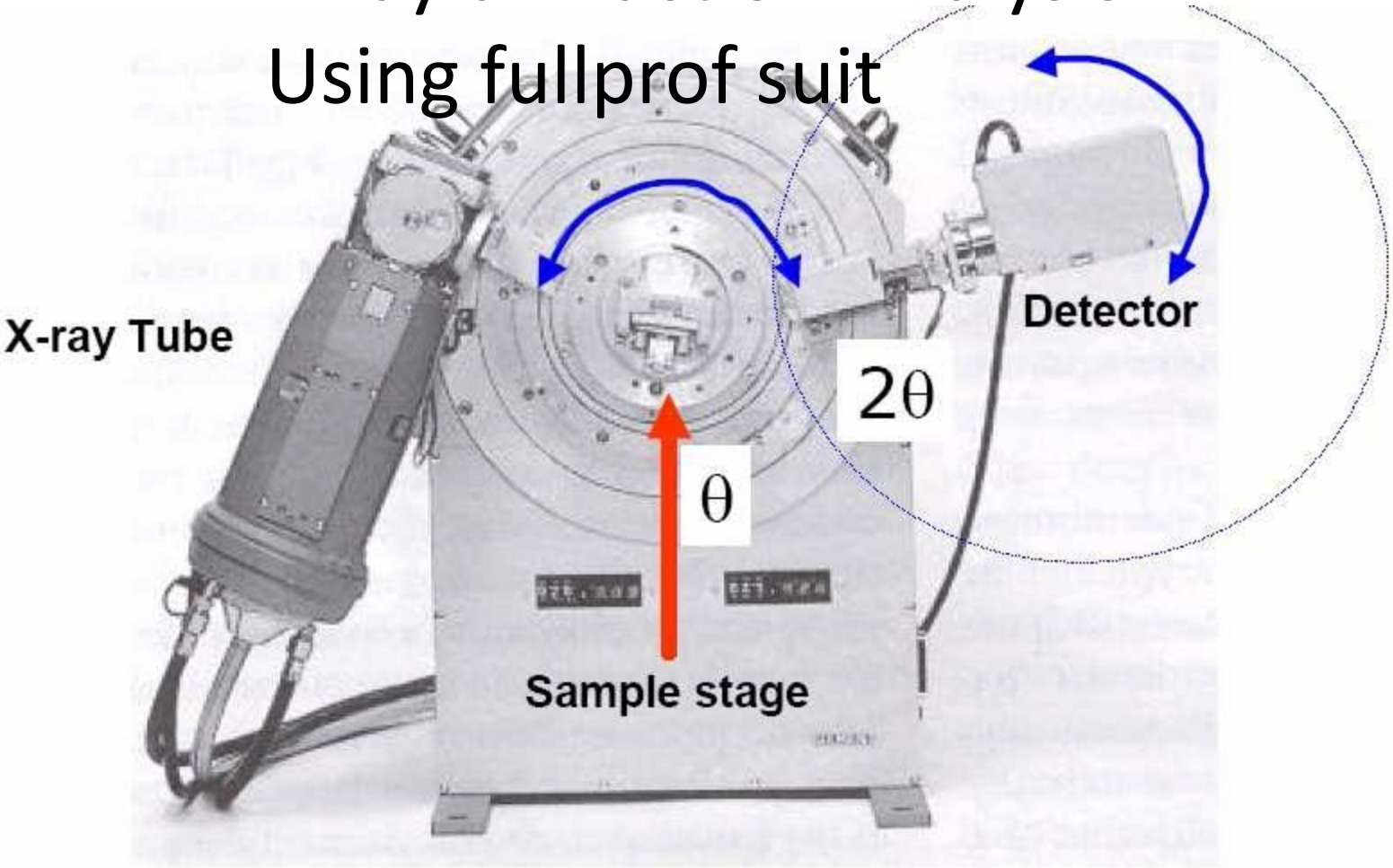
X-ray diffraction Analysis Using fullprof suit



Presented By
Geeta Ray

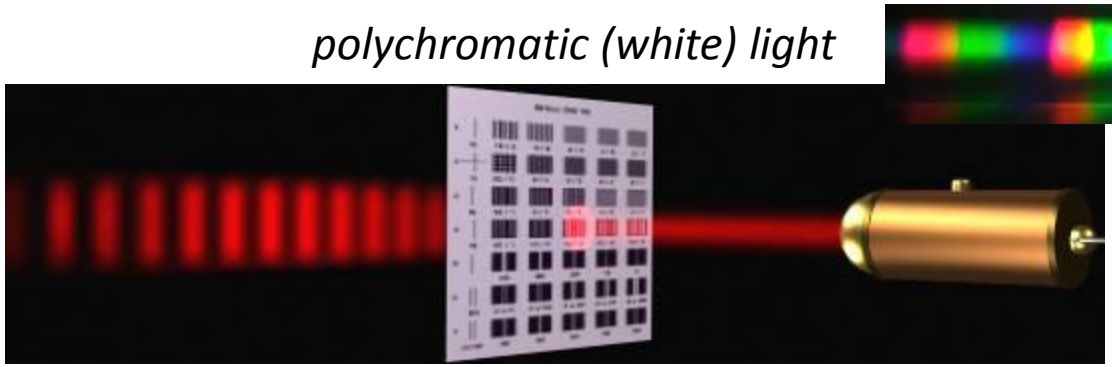

D8

X-Ray Diffraction

What is diffraction?

- incident radiation (e.g., light, X-rays) scatters as it passes through a finely spaced periodic array (e.g., grating, crystal lattice)

polychromatic (white) light



monochromatic light (e.g., laser)

- where beams of scattered radiation emerge from slit "in phase", constructive interference produces "diffraction maxima"

- position and intensity of maxima depends on spacing of array and integral number of λ contributing to signal (nλ)

WHY X-RAYS?

- For electromagnetic radiation to be diffracted the spacing in the grating should be of the same order as the wavelength

- In crystals the typical interatomic spacing ~ 2-3 Å so the suitable radiation is X-rays

- Hence, X-rays can be used for the study of crystal structures

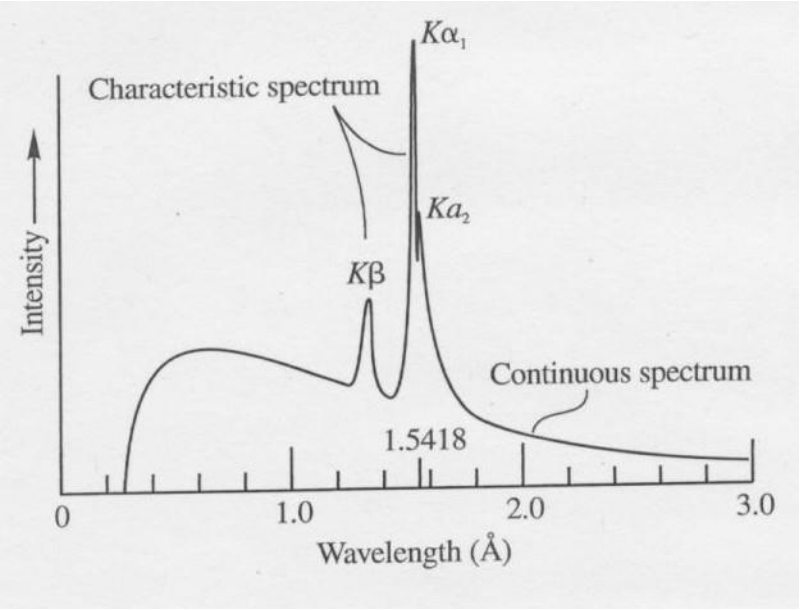


Table 8.1 Characteristic Wavelengths (Å) of Metals Commonly Used as Targets in X-Ray Tubes[a]

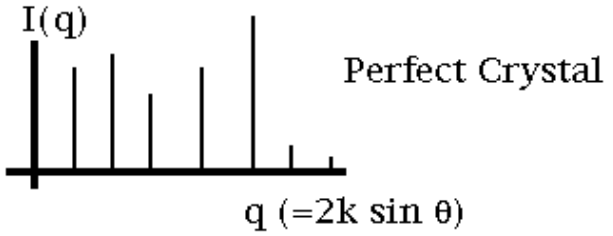
	Metal				
	Mo	**Cu**	**Co**	**Fe**	**Cr**
$K\beta$	0.63225	1.38217	1.62073	1.75653	2.08479
$K\alpha_1$	0.70926	1.54051	1.78892	1.93597	2.28962
$K\alpha_2$	0.71354	1.54433	1.79279	1.93991	2.29351
$K\bar{\alpha}$	0.7107	1.5418	1.7902	1.9373	2.2909

[a] $K\bar{\alpha}$ is the weighted average of $K\alpha_1$ and $K\alpha_2$.

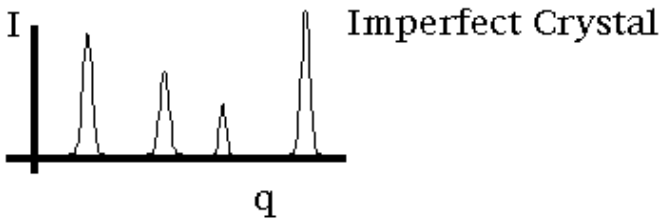
X-Ray Diffraction

What is X-ray diffraction (XRD) crystallography?

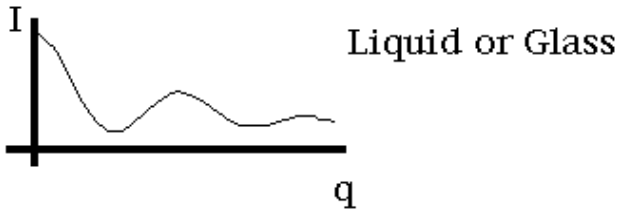
- periodic atomic arrays in crystal lattice act like 3-D diffraction gratings
- for practical purposes, diffraction can be treated like reflection from multiple equivalent lattice planes (*hkl*)



sharp peaks



broad peaks



diffuse, continuous spectrum

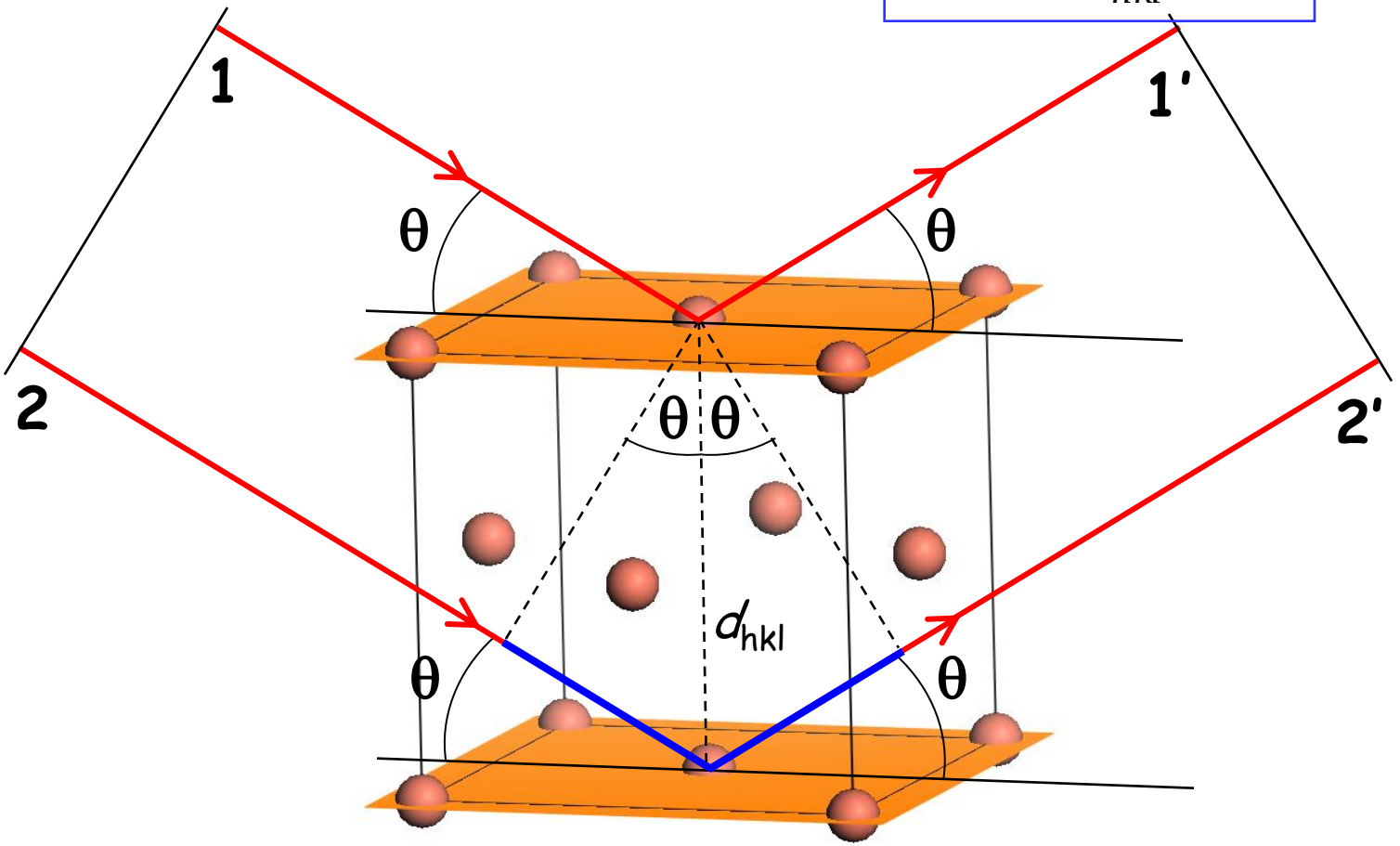
BRAGG VIEW OF DIFFRACTION

X-rays that hit the crystal are elastically scattered by the sets of (hkl) planes

The path difference for rays 1 and 2 equals to the length of two blue lines:

$$\Delta(1\text{-}2) = 2d_{hkl}\sin\theta$$

$$n\lambda = 2d_{hkl}\sin\theta$$



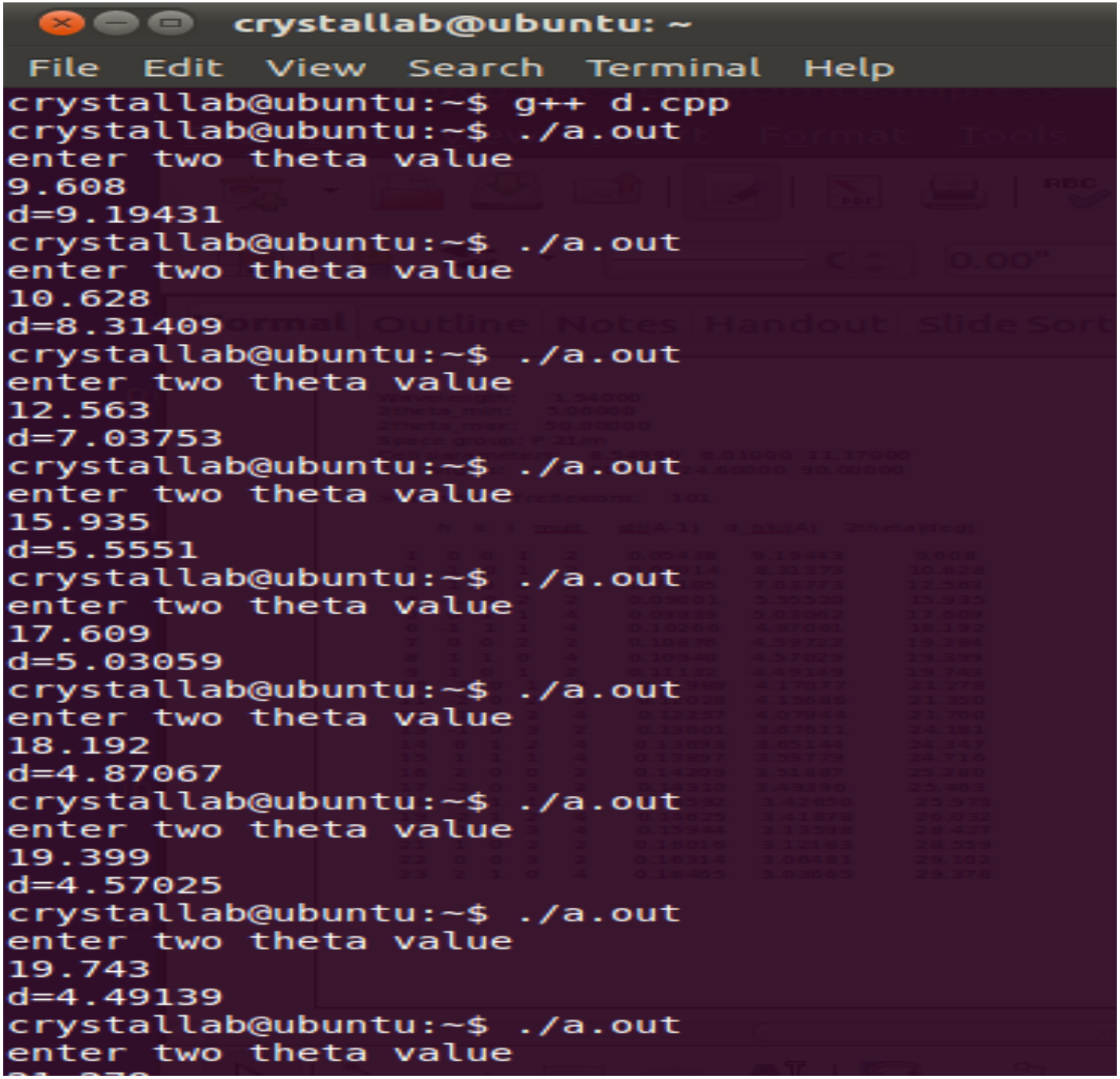
d.cpp (~) - gedit

File Edit View Search Tools Documents Help

Open Save Undo

d.cpp

```
1 #include<iostream>
2 #include<math.h>
3 using namespace std;
4  main()
5 {
6 float twotheta,d,s,l,pi;
7 cout<<"enter two theta value\n";
8 cin>>twotheta;
9 pi=4*atan(1);
10 l=twotheta*pi/360;
11
12 s=2*sin(l);
13 d=1.54/s;
14
15 cout<<"d="<< d<<"\n";
16 }
```


crystallab@ubuntu: ~
File Edit View Search Terminal Help
crystallab@ubuntu:~$ g++ d.cpp
crystallab@ubuntu:~$ ./a.out
enter two theta value
9.608
d=9.19431
crystallab@ubuntu:~$ ./a.out
enter two theta value
10.628
d=8.31409
crystallab@ubuntu:~$ ./a.out
enter two theta value
12.563
d=7.03753
crystallab@ubuntu:~$ ./a.out
enter two theta value
15.935
d=5.5551
crystallab@ubuntu:~$ ./a.out
enter two theta value
17.609
d=5.03059
crystallab@ubuntu:~$ ./a.out
enter two theta value
18.192
d=4.87067
crystallab@ubuntu:~$ ./a.out
enter two theta value
19.399
d=4.57025
crystallab@ubuntu:~$ ./a.out
enter two theta value
19.743
d=4.49139
crystallab@ubuntu:~$ ./a.out
enter two theta value

System	Lattice Parameters
Triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma \neq 90$
Monoclinic	$a \neq b \neq c$ $\alpha = \gamma = 90$, $\beta \neq 90$
Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90$
Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90$
Hexagonal	$a = b \neq c$ $\alpha = \beta = 90$, $\gamma = 120$
Rhombohedral (Trigonal)	$a = b = c$ $\alpha = \beta = \gamma \neq 90$
Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90$

Cubic:
$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Tetragonal:
$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$$

Hexagonal:
$$\frac{1}{d^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2}$$

Rhombohedral:
$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)\sin^2\alpha + 2(hk + kl + hl)(\cos^2\alpha - \cos\alpha)}{a^2(1 - 3\cos^2\alpha + 2\cos^3\alpha)}$$

Orthorhombic:
$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

Monoclinic:
$$\frac{1}{d^2} = \frac{1}{\sin^2\beta}\left(\frac{h^2}{a^2} + \frac{k^2\sin^2\beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl\cos\beta}{ac}\right)$$

Triclinic:
$$\frac{1}{d^2} = \frac{1}{V^2}(S_{11}h^2 + S_{22}k^2 + S_{33}l^2 + 2S_{12}hk + 2S_{23}kl + 2S_{13}hl)$$

In the equation for triclinic crystals,

V = volume of unit cell (see below),

$S_{11} = b^2c^2\sin^2\alpha$,

$S_{22} = a^2c^2\sin^2\beta$,

$S_{33} = a^2b^2\sin^2\gamma$,

$S_{12} = abc^2(\cos\alpha\cos\beta - \cos\gamma)$,

$S_{23} = a^2bc(\cos\beta\cos\gamma - \cos\alpha)$,

$S_{13} = ab^2c(\cos\gamma\cos\alpha - \cos\beta)$.

Possible space groups For monoclinic system	Systematic absences
Pm, P2/m	hkl: none h0l: none 0k0: none
$P2^1$, $P2^1/m$	hkl: none h0l: none 0k0: k = 2n + 1
Pc, P2/c	hkl: none h0l: l = 2n + 1 0k0: none
$P2^1/c$	hkl: none h0l: l = 2n + 1 0k0: k = 2n + 1
C2, Cm, C2/m	C2, Cm, C2/m h0l: (h = 2n + 1) 0k0: (k = 2n + 1)

MILLER PLANES

Atoms form periodically arranged planes

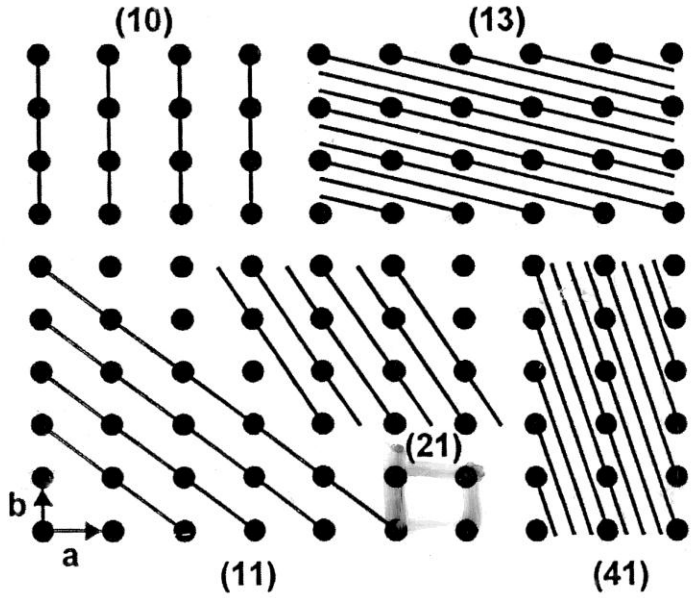
Any set of planes is characterized by:

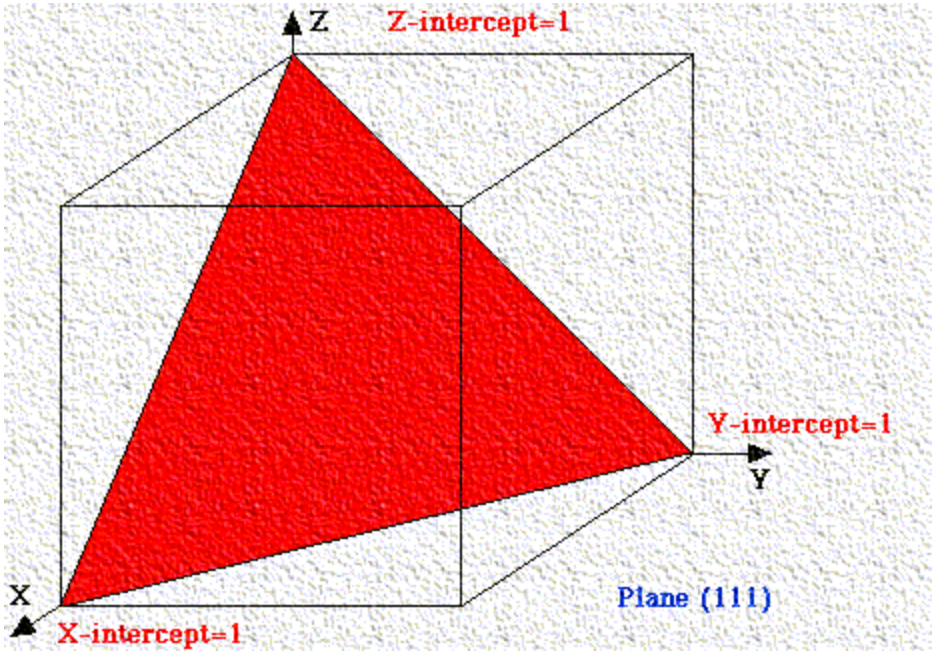
(1) their orientation in the crystal (hkl) – Miller indices
(2) their *d*-spacing (d_{hkl}) – distance between the planes

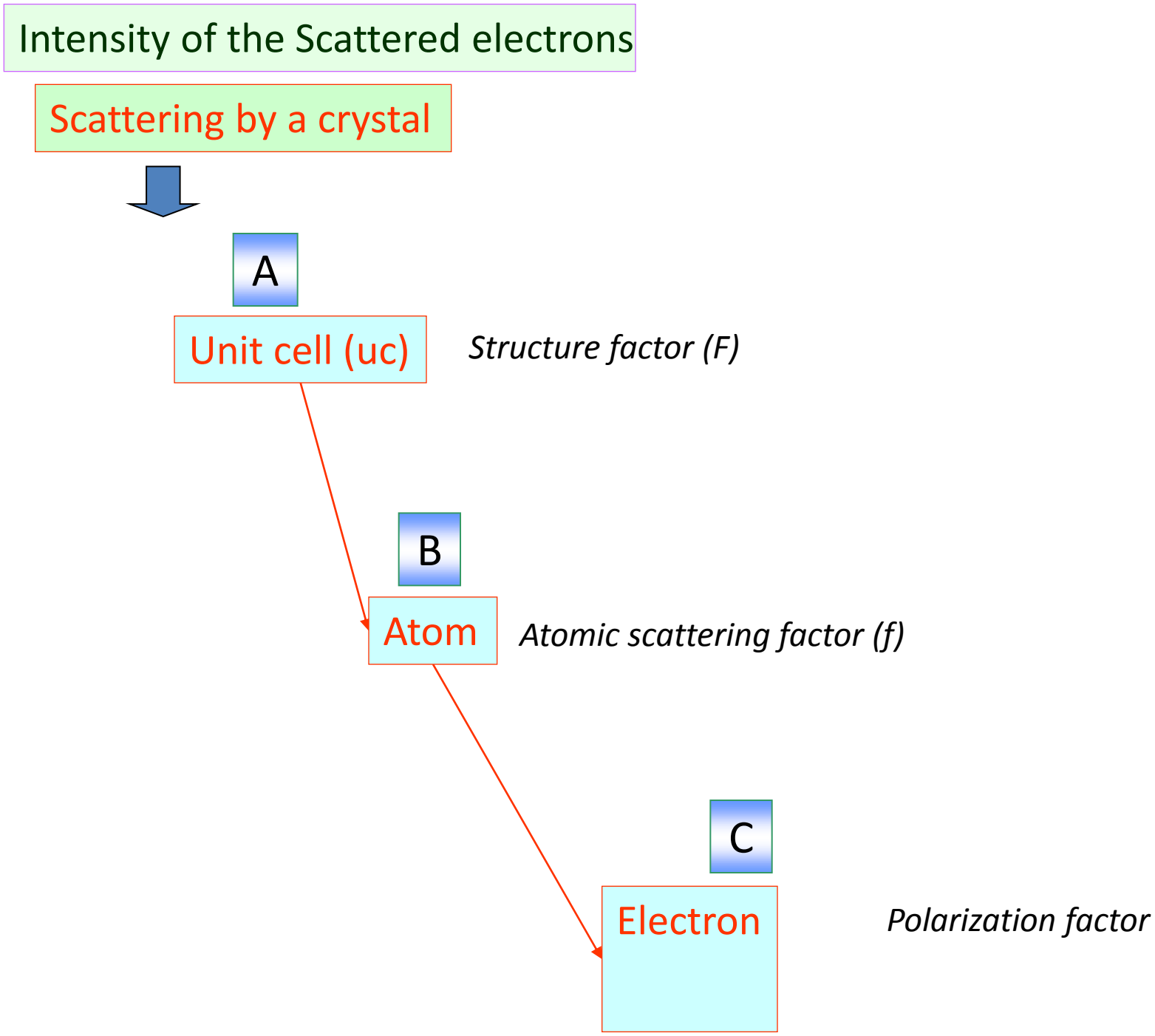
h, k, l correspond to the number of segments in which the a, b, c axes, respectively, are cut by the set of planes

On average, the higher (hkl), the closer is the interplanar distance, d_{hkl}

2-D Examples




Z
Z-intercept=1
Y-intercept=1
Y
X
X-intercept=1
Plane (111)


Intensity of the Scattered electrons
Scattering by a crystal
A
Unit cell (uc)
Structure factor (F)
B
Atom
Atomic scattering factor (f)
C
Electron
Polarization factor

Diffracted Beam Intensity

- Structure factor
- Polarization factor
- Lorentz factor
- Multiplicity factor
- Temperature factor
- Absorption factor

$$I(q) \propto \left|F\left(q\right)\right|^2$$

$$I_C(q) = mALpK\left|F(q)\right|^2 + I_b$$

The Structure Factor

$$F_{hkl} = \sum_{1}^{N} f_n e^{2\pi i\left(hu_n + kv_n + lw_n\right)}$$

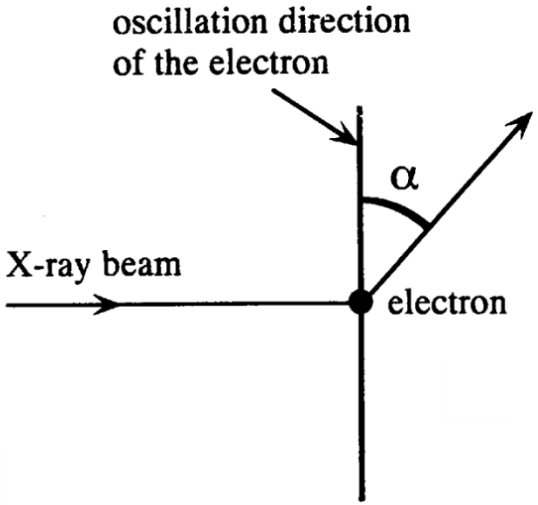
$$F_{hkl} = \frac{amplitude\ \ scattered\ \ by\ \ all\ \ atoms\ \ in\ a\ unit\ cell}{amplitude\ \ scattered\ \ by\ \ a\ single\ \ electron}$$

- **The structure factor contains the information regarding the types (*f*) and locations (*u, v, w*) of atoms within a unit cell.**

- **A comparison of the observed and calculated structure factors is a common goal of X-ray structural analysis.**

The Polarization Factor

- The polarization factor *p* arises from the fact that an electron does not scatter along its direction of vibration
- In other directions electrons radiate with an intensity proportional to $(\sin \alpha)^2$:



The polarization factor (assuming that the incident beam is unpolarized):

$$p = \frac{1 + \cos^2 2\theta}{2}$$

The Lorentz - Polarization Factor

- The Lorenz factor L depends on the measurement technique used and, for the Diffractometer data obtained by the usual θ-2θ scans, it can be written as

$$L = \frac{1}{\sin 2\theta}$$

- The combination of geometric corrections are lumped together into a single Lorentz-polarization (Lp) factor:

$$Lp = \frac{1+\cos^2 2\theta}{\sin 2\theta}$$

The effect of the Lp factor is to decrease the intensity at intermediate angles and increase the intensity in the forward and backwards directions

The Temperature Factor

- As atoms vibrate about their equilibrium positions in a crystal, the electron density is spread out over a larger volume.
- This causes the atomic scattering factor to decrease with $\sin\theta/\lambda$ (or $|S| = 4\pi\sin\theta/\lambda$) more rapidly than it would normally.

The temperature factor is given by:

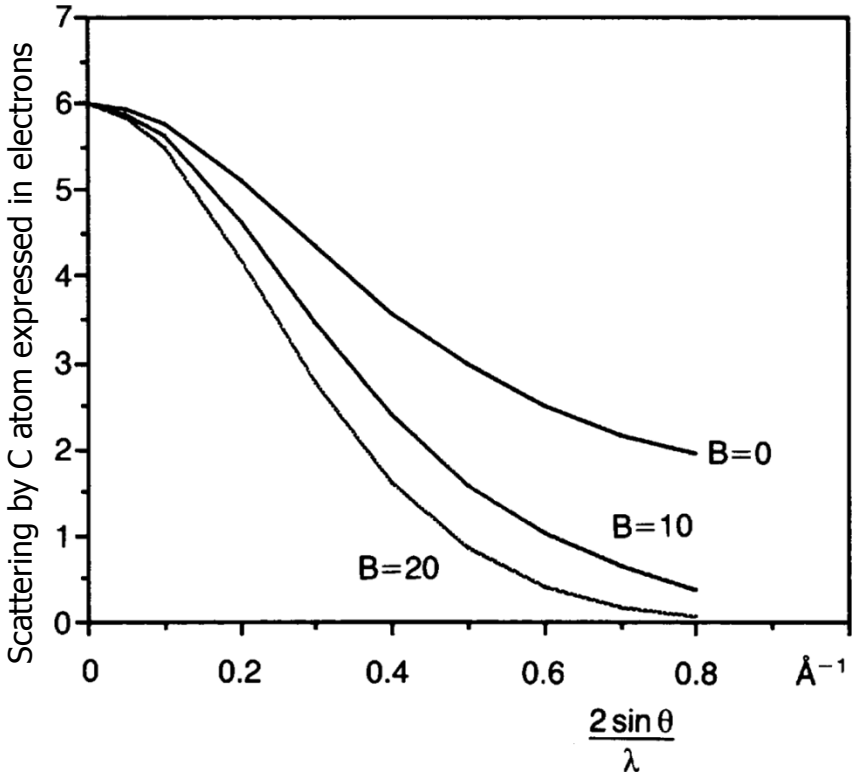
$$\exp\left[-B\frac{\sin^2\theta}{\lambda^2}\right]$$

where the thermal factor B is related to the mean square displacement of the atomic vibration:

$$B = 8\pi^2 \times \overline{u^2}$$

This is incorporated into the atomic scattering factor:

$$f \rightarrow f_0 e^{-M} \Rightarrow f^2 \sim e^{-2M}$$



The Multiplicity Factor

- The multiplicity factor arises from the fact that in general there will be several sets of *hkl* -planes having different orientations in a crystal but with the same d and F^2 values
- Evaluated by finding the number of variations in position and sign in $\pm h$, $\pm k$ and $\pm l$ and have planes with the same d and F^2
- The value depends on *hkl* and crystal symmetry
- For the highest cubic symmetry we have:

$100, \bar{1}00, 010, 0\bar{1}0, 001, 00\bar{1}$ $\quad p_{100} = 6$

$110, \bar{1}10, 1\bar{1}0, \bar{1}\,\bar{1}0, 101, 10\bar{1}, \bar{1}0\bar{1}, \bar{1}01, 011, 0\bar{1}1, 01\bar{1}, 0\bar{1}\,\bar{1}$ $\quad p_{110} = 12$

$111, 11\bar{1}, 1\bar{1}1, \bar{1}11, 1\bar{1}\,\bar{1}, \bar{1}1\bar{1}, \bar{1}\,\bar{1}1, \bar{1}\,\bar{1}\,\bar{1}$ $\quad p_{111}=8$

The Absorption Factor

- Angle-dependent absorption within the sample itself will modify the observed intensity

Absorption factor for thin films is given by:

$$A = 1 - \exp\left(-\frac{2\mu\tau}{\sin\theta}\right)$$

where μ is the absorption coefficient, τ is the total thickness of the film

Diffracted Beam Intensity

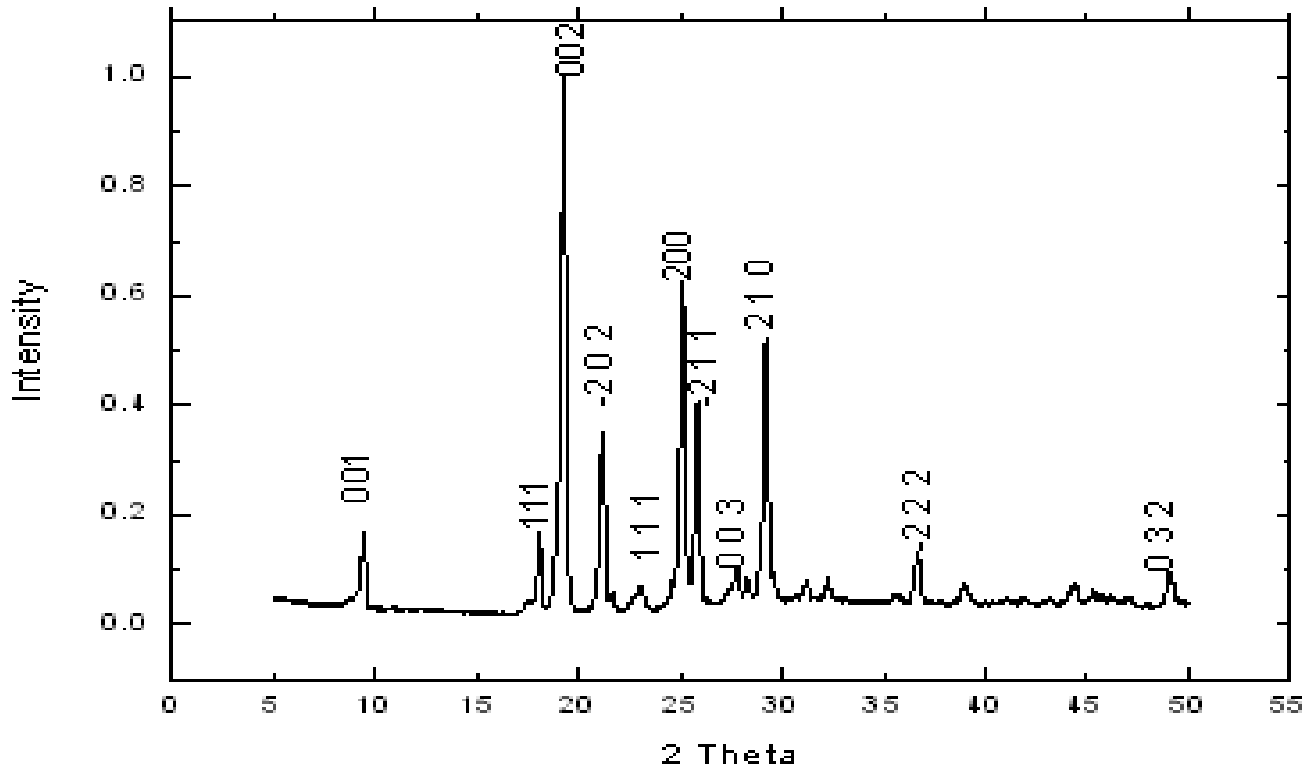
$$I \propto F_{hkl} F_{hkl}^{*} = \left| F_{hkl} \right|^2$$

$$I_C(q) = Ap(Lp)K\left|F(q)\right|^2 + I_b$$

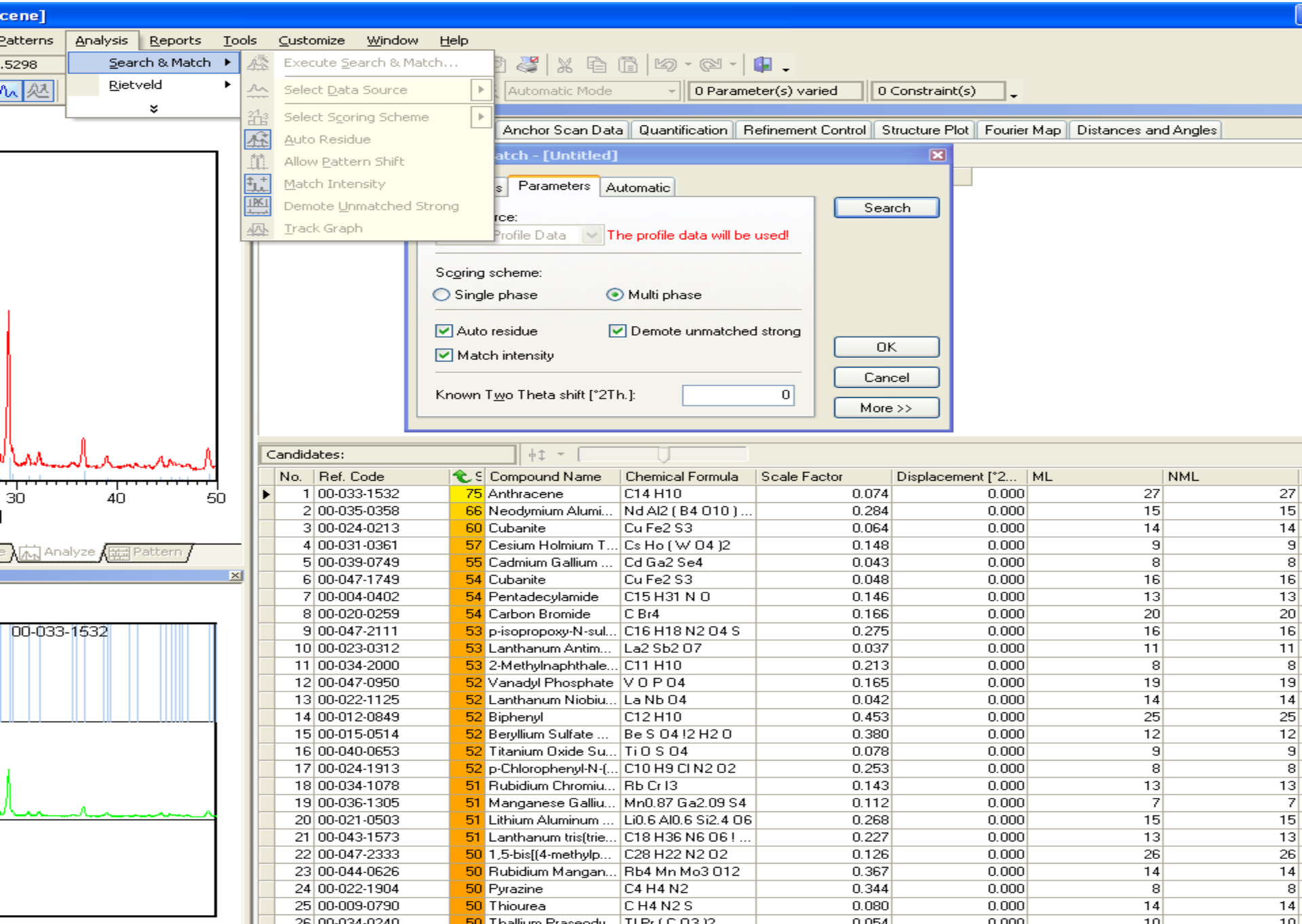
where K is the scaling factor, I_b is the background intensity, $q = 4\sin\vartheta/\lambda$ is the scattering vector for x-rays of wavelength λ

$$I_C(q) = \left[1 - \exp\left(-\frac{2\mu\tau}{\sin\theta}\right)\right]\frac{1+\cos^2 2\theta}{\sin 2\theta} K\left|F(q)\right|^2 + I_b$$

X-ray Diffraction Graph Of Pure Anthracene using ORIGIN



X’Pert Highscore


cene]
Patterns
Analysis
Reports
Tools
Customize
Window
Help
Search & Match
Rietveld
Execute Search & Match...
Select Data Source
Select Scoring Scheme
Auto Residue
Allow Pattern Shift
Match Intensity
Demote Unmatched Strong
Track Graph
Automatic Mode
0 Parameter(s) varied
0 Constraint(s)
Anchor Scan Data
Quantification
Refinement Control
Structure Plot
Fourier Map
Distances and Angles
atch - [Untitled]
Parameters
Automatic
Search
rce:
Profile Data
The profile data will be used!
Scoring scheme:
Single phase
Multi phase
Auto residue
Demote unmatched strong
Match intensity
OK
Cancel
Known Two Theta shift [°2Th.]:
0
More >>
30
40
50
Analyze
Pattern
00-033-1532
Candidates:
No. | Ref. Code | S | Compound Name | Chemical Formula | Scale Factor | Displacement [°2... | ML | NML
1 | 00-033-1532 | 75 | Anthracene | C14 H10 | 0.074 | 0.000 | 27 | 27
2 | 00-035-0358 | 66 | Neodymium Alumi... | Nd Al2 (B4 O10) ... | 0.284 | 0.000 | 15 | 15
3 | 00-024-0213 | 60 | Cubanite | Cu Fe2 S3 | 0.064 | 0.000 | 14 | 14
4 | 00-031-0361 | 57 | Cesium Holmium T... | Cs Ho (W O4)2 | 0.148 | 0.000 | 9 | 9
5 | 00-039-0749 | 55 | Cadmium Gallium ... | Cd Ga2 Se4 | 0.043 | 0.000 | 8 | 8
6 | 00-047-1749 | 54 | Cubanite | Cu Fe2 S3 | 0.048 | 0.000 | 16 | 16
7 | 00-004-0402 | 54 | Pentadecylamide | C15 H31 N O | 0.146 | 0.000 | 13 | 13
8 | 00-020-0259 | 54 | Carbon Bromide | C Br4 | 0.166 | 0.000 | 20 | 20
9 | 00-047-2111 | 53 | p-isopropoxy-N-sul... | C16 H18 N2 O4 S | 0.275 | 0.000 | 16 | 16
10 | 00-023-0312 | 53 | Lanthanum Antim... | La2 Sb2 O7 | 0.037 | 0.000 | 11 | 11
11 | 00-034-2000 | 53 | 2-Methylnaphthale... | C11 H10 | 0.213 | 0.000 | 8 | 8
12 | 00-047-0950 | 52 | Vanadyl Phosphate | V O P O4 | 0.165 | 0.000 | 19 | 19
13 | 00-022-1125 | 52 | Lanthanum Niobiu... | La Nb O4 | 0.042 | 0.000 | 14 | 14
14 | 00-012-0849 | 52 | Biphenyl | C12 H10 | 0.453 | 0.000 | 25 | 25
15 | 00-015-0514 | 52 | Beryllium Sulfate ... | Be S O4 I2 H2 O | 0.380 | 0.000 | 12 | 12
16 | 00-040-0653 | 52 | Titanium Oxide Su... | Ti O S O4 | 0.078 | 0.000 | 9 | 9
17 | 00-024-1913 | 52 | p-Chlorophenyl-N-(... | C10 H9 Cl N2 O2 | 0.253 | 0.000 | 8 | 8
18 | 00-034-1078 | 51 | Rubidium Chromiu... | Rb Cr I3 | 0.143 | 0.000 | 13 | 13
19 | 00-036-1305 | 51 | Manganese Galliu... | Mn0.87 Ga2.09 S4 | 0.112 | 0.000 | 7 | 7
20 | 00-021-0503 | 51 | Lithium Aluminum ... | Li0.6 Al0.6 Si2.4 O6 | 0.268 | 0.000 | 15 | 15
21 | 00-043-1573 | 51 | Lanthanum tris(trie... | C18 H36 N6 O6 ! ... | 0.227 | 0.000 | 13 | 13
22 | 00-047-2333 | 50 | 1,5-bis[(4-methylp... | C28 H22 N2 O2 | 0.126 | 0.000 | 26 | 26
23 | 00-044-0626 | 50 | Rubidium Mangan... | Rb4 Mn Mo3 O12 | 0.367 | 0.000 | 14 | 14
24 | 00-022-1904 | 50 | Pyrazine | C4 H4 N2 | 0.344 | 0.000 | 8 | 8
25 | 00-009-0790 | 50 | Thiourea | C H4 N2 S | 0.080 | 0.000 | 14 | 14

USING FULLPROF SUIT

FullProf has been mainly developed for Rietveld analysis (structure profile refinement) of neutron (nuclear and magnetic scattering) or X-ray powder diffraction data collected at constant or variable step in scattering angle 2θ. The program can be also used as a Profile Matching without the knowledge of the structure.

FullProf Suite ToolBar

File Programs Settings FP Dimensions Run a Script Help

Working Directory: C:\Documents and Settings\Raman\Desktop\ Code File: Type: Date: 16/04/2012

WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]

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

Format of the data file

Format of data file:

- ( ) X,Y data + INSTRM=10
- ( ) INSTRM=0: Free F.(Ti,step,Tf)
- ( ) INSTRM=1: Old D1A
- ( ) INSTRM=3: D1B (ILL)
- ( ) INSTRM=4: Brookhaven(Synchr.)
- ( ) INSTRM=5: G4.1
- ( ) INSTRM=6: D2B/3T2/G4.2
- ( ) INSTRM=8: HRPT/DMC (PSI)
- ( ) INSTRM=9: RX (Socabim)
- ( ) INSTRM=11: Variable Time step
- (•) GSAS data
- ( ) CPI (Xrays)
- ( ) PANalytical formats
- ( ) INSTRM=14: ISIS normalized data
- ( ) 15. Rigaku RINT

OK Cancel

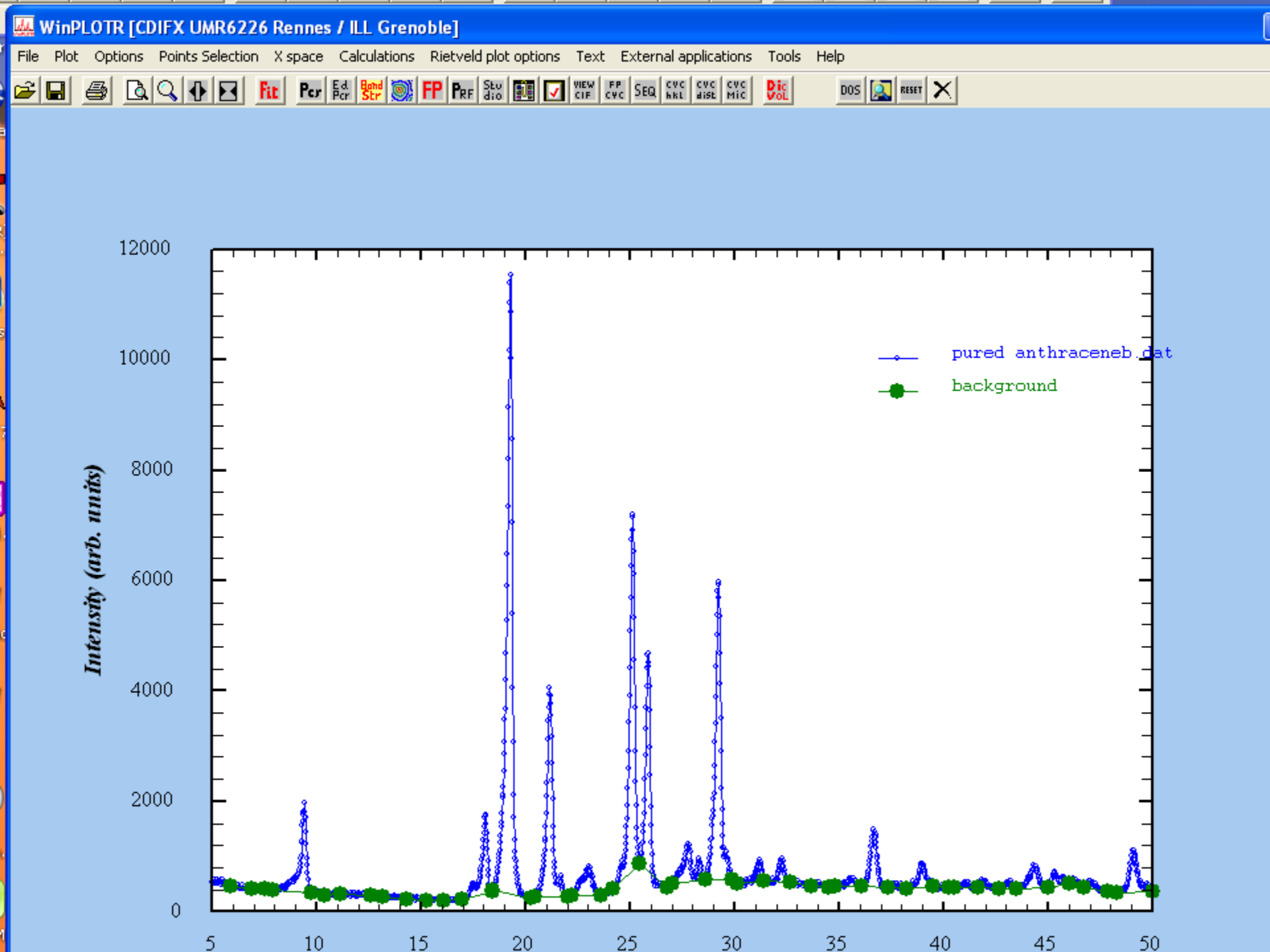
pured anthraceneb.dat

2000

WinPLOTR

WinPLOTR is a software to plot and analyse powder diffraction patterns
It can be used to plot raw or normalized data files coming from neutron
And x-ray diffractometers as well as Rietveld files created by several
Rietveld type refinement program.

WinPLOTR has also been developed to be preferential graphic interface for
The Rietveld type FullProf program : edition of PCR input file ,plot Rietveld
Type plots.


WinPLOTR [CDIFX UMR6226 Rennes / ILL Grenoble]
File Plot Options Points Selection X space Calculations Rietveld plot options Text External applications Tools Help
pured anthraceneb.dat
background
Intensity (arb. units)
12000
10000
8000
6000
4000
2000
0
5
10
15
20
25
30
35
40
45
50

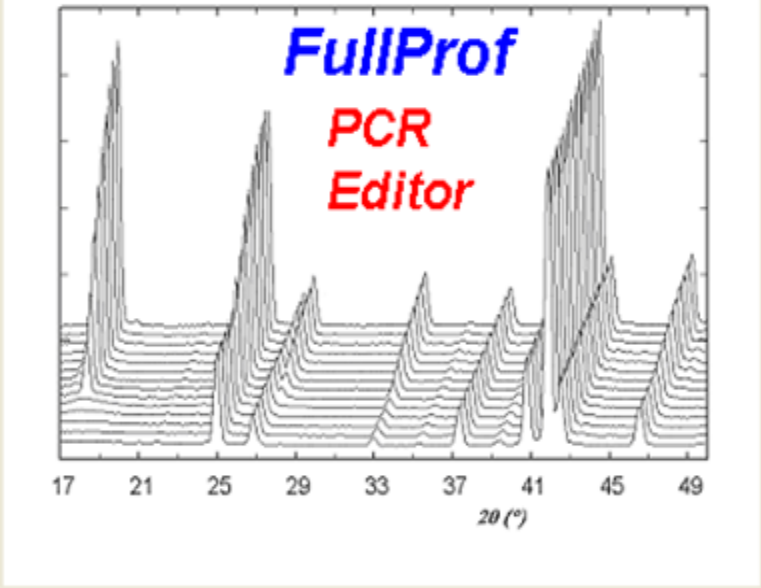
FullProf Suite ToolBar

File Programs Settings FP Dimensions Run a Script Help

Working Directory: C:\Documents and Settings\Raman\Desktop\ Code File: Type: Date: 09/04/2012

Editor of PCR Files

File Editor Tools Templates Help Exit



Information

Title, type of job: Rietveld, Integrated Intensities, Simulated Annealing, ... General

Type of Patterns, profile, background, diffraction geometry, user-given scattering factors ... Patterns

Phase name, type of calculations (JBT), ATZ, contribution to patterns, symmetry, ... Phases

Number of cycles, relaxation factors, access to patterns and phases (atoms and profile) Refinement

Constraints definitions, adding, deleting, modifying... Constraints

Fixing range of parameters, distances, angles, magnetic moments and linear restraints Box/Restraints

Output options for patterns and phases: Reflection lists, Fourier, distances, BVS... Output

pantra Profiles: 1 Phases: 1 6/ 3/2012 14:55:17

The main window of **EdPCR** program contains a menu bar and a toolbar with the usual buttons. A brief information is obtained when you left the mouse on a button of the toolbar.

The information of the *PCR* file is distributed in seven buttons:

General
Define a general information as title, type of job: Rietveld, Profile Matching, Simulating Annealing.

Patterns
Define patterns information: types of profile, background, geometry aspects...

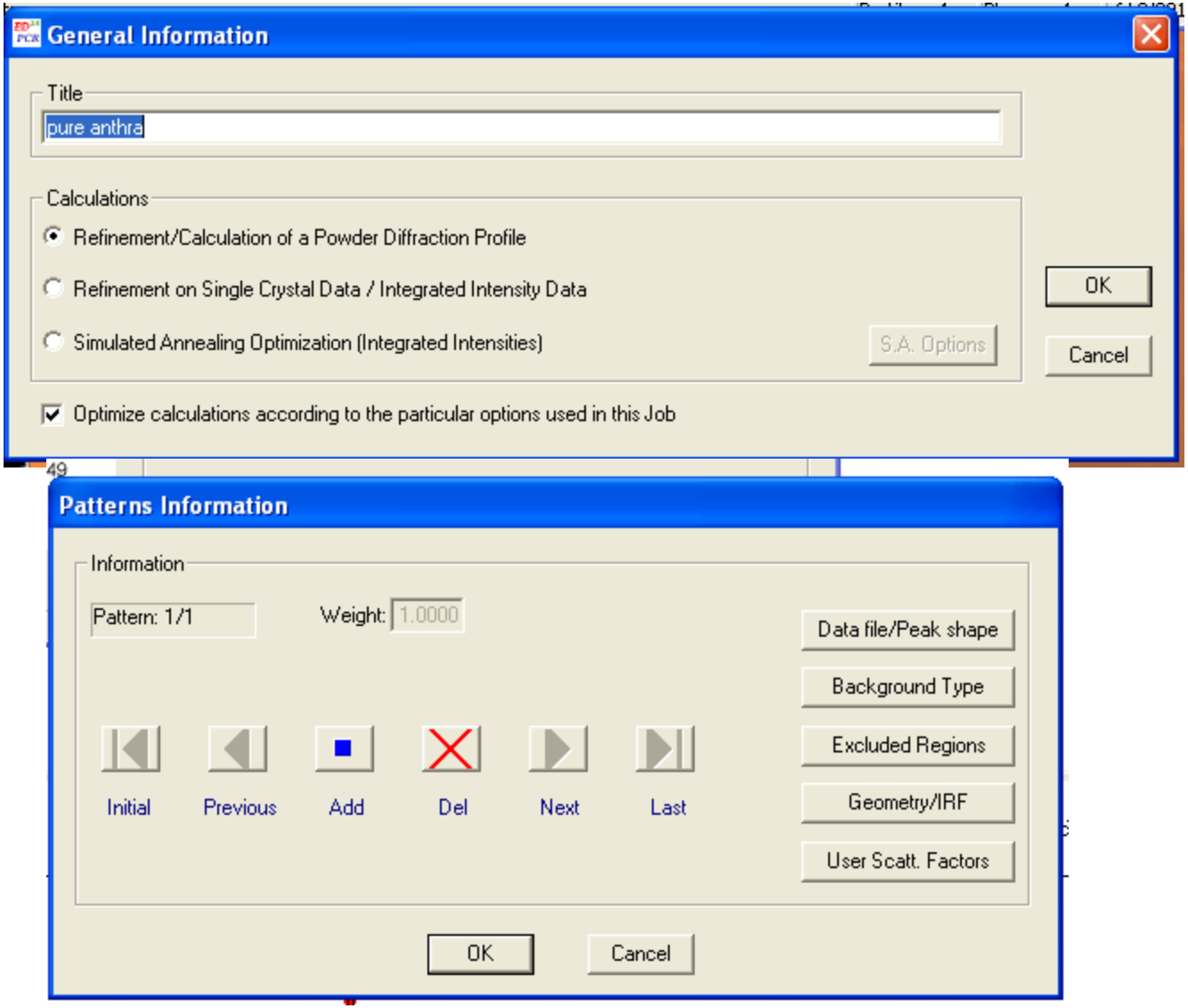
Phases
Define Phase information: Names, contribution to patterns, symmetry…

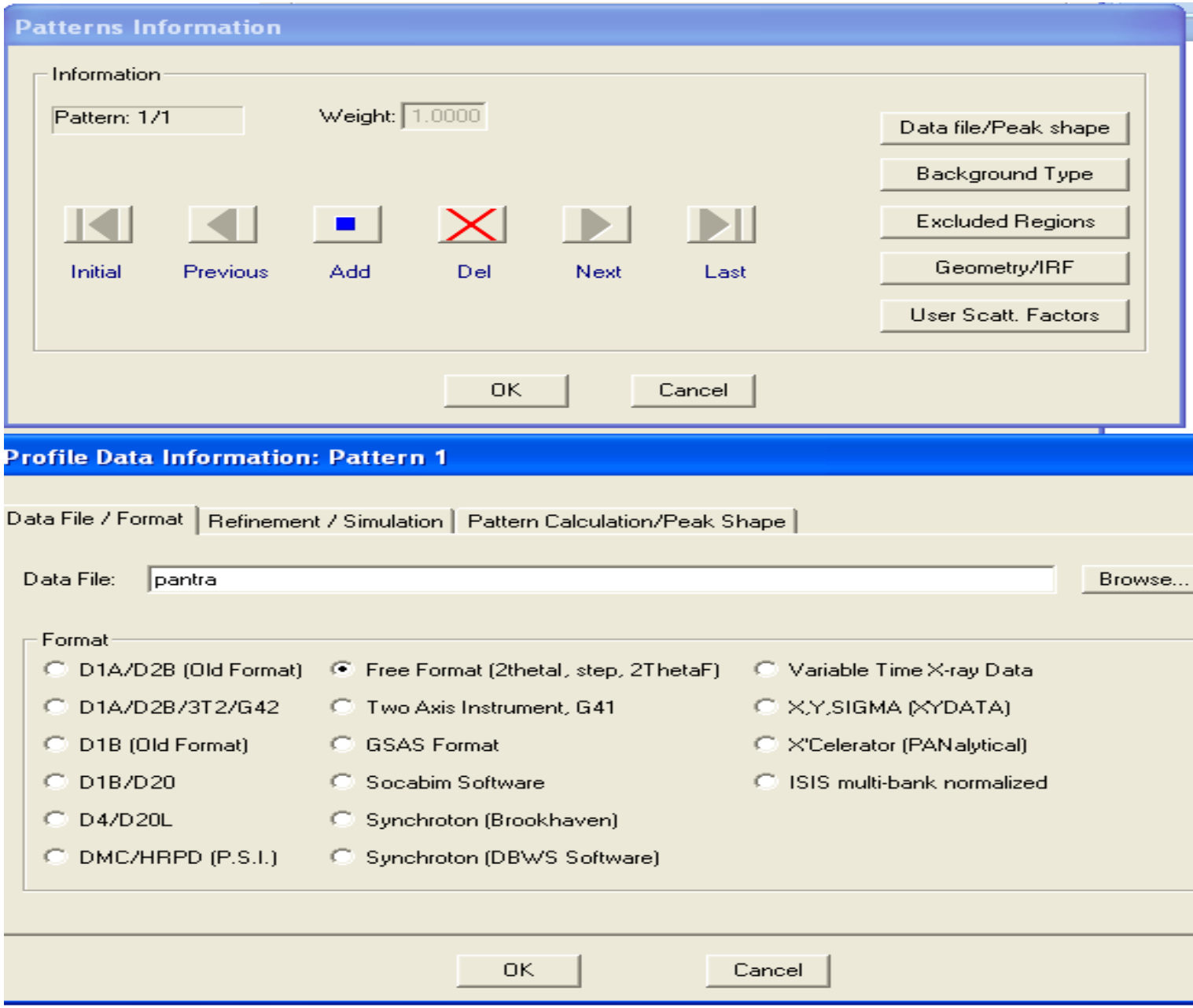
Refinement
This button is the access to the most important part of EdPCR: editing structural and profile parameters and conditions of refinement. Atom positions, profile shape parameters, magnetic moments, micro structural parameters, etc … are accessible through this button.

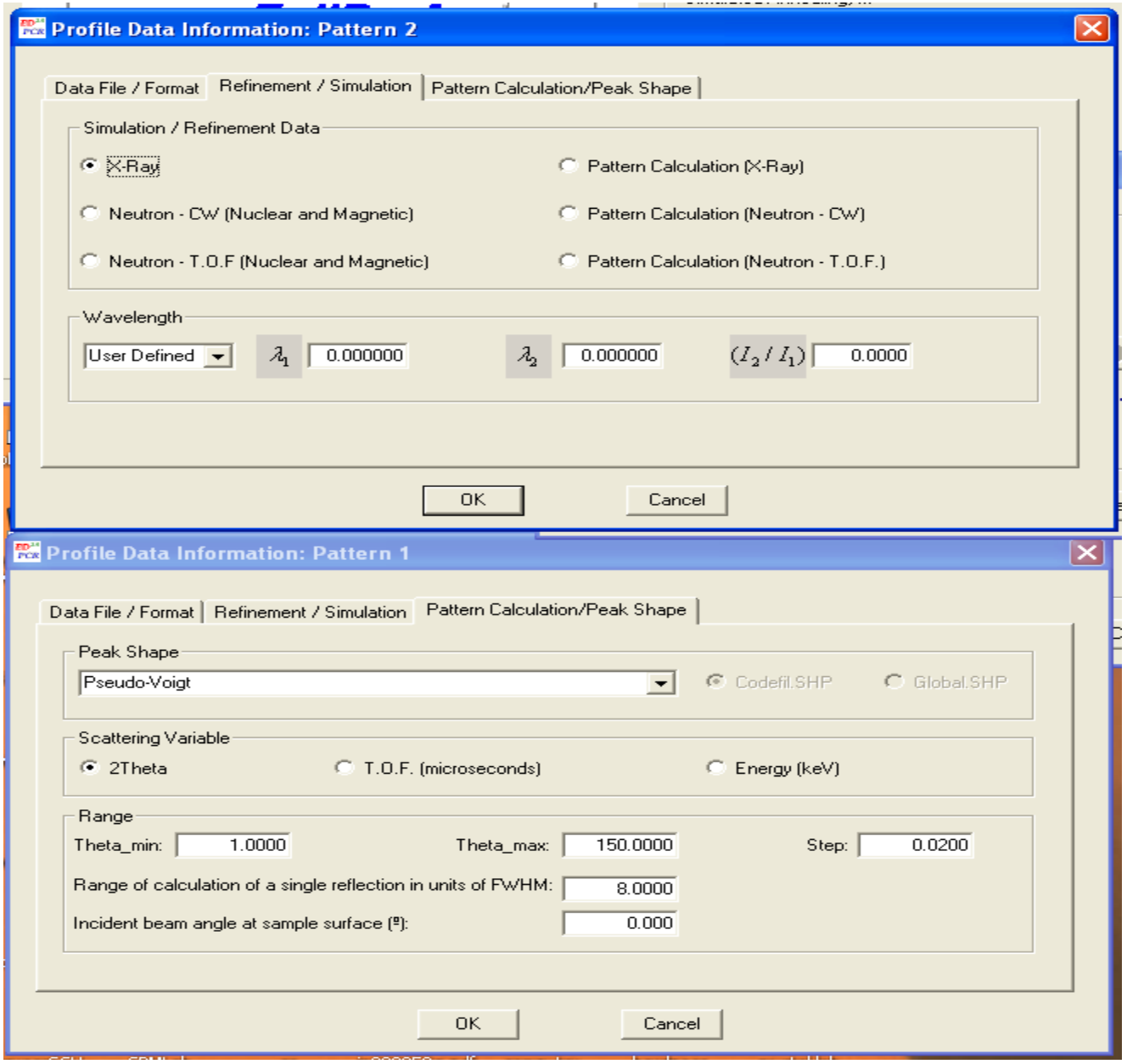
Constraints
Define constrains for refinable parameters. You can modify, add and delete constrains relations easily by using mouse selection and clicks.

.

Output
Access to the selection of output options for each phase and pattern. This allows selecting output files: Fourier, hkl-lists, files for other programs, etc.


General Information
Title
pure anthra
Calculations
Refinement/Calculation of a Powder Diffraction Profile
Refinement on Single Crystal Data / Integrated Intensity Data
Simulated Annealing Optimization (Integrated Intensities)
S.A. Options
OK
Cancel
Optimize calculations according to the particular options used in this Job
Patterns Information
Information
Pattern: 1/1
Weight: 1.0000
Data file/Peak shape
Background Type
Excluded Regions
Geometry/IRF
User Scatt. Factors
Initial
Previous
Add
Del
Next
Last
OK
Cancel


Patterns Information
Information
Pattern: 1/1
Weight: 1.0000
Data file/Peak shape
Background Type
Excluded Regions
Geometry/IRF
User Scatt. Factors
Initial
Previous
Add
Del
Next
Last
OK
Cancel
Profile Data Information: Pattern 1
Data File / Format
Refinement / Simulation
Pattern Calculation/Peak Shape
Data File:
pantra
Browse...
Format
D1A/D2B (Old Format)
Free Format (2thetal, step, 2ThetaF)
Variable Time X-ray Data
D1A/D2B/3T2/G42
Two Axis Instrument, G41
X,Y,SIGMA (XYDATA)
D1B (Old Format)
GSAS Format
X'Celerator (PANalytical)
D1B/D20
Socabim Software
ISIS multi-bank normalized
D4/D20L
Synchroton (Brookhaven)
DMC/HRPD (P.S.I.)
Synchroton (DBWS Software)
OK
Cancel

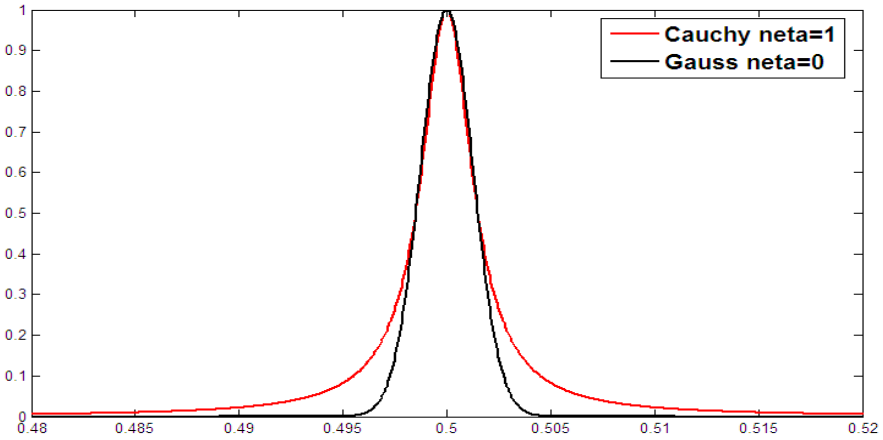

Profile Data Information: Pattern 2
Data File / Format
Refinement / Simulation
Pattern Calculation/Peak Shape
Simulation / Refinement Data
X-Ray
Neutron - CW (Nuclear and Magnetic)
Neutron - T.O.F (Nuclear and Magnetic)
Pattern Calculation (X-Ray)
Pattern Calculation (Neutron - CW)
Pattern Calculation (Neutron - T.O.F.)
Wavelength
User Defined
λ1
0.000000
λ2
0.000000
(I2 / I1)
0.0000
OK
Cancel
Profile Data Information: Pattern 1
Data File / Format
Refinement / Simulation
Pattern Calculation/Peak Shape
Peak Shape
Pseudo-Voigt
Codefil.SHP
Global.SHP
Scattering Variable
2Theta
T.O.F. (microseconds)
Energy (keV)
Range
Theta_min:
1.0000
Theta_max:
150.0000
Step:
0.0200
Range of calculation of a single reflection in units of FWHM:
8.0000
Incident beam angle at sample surface (º):
0.000
OK
Cancel

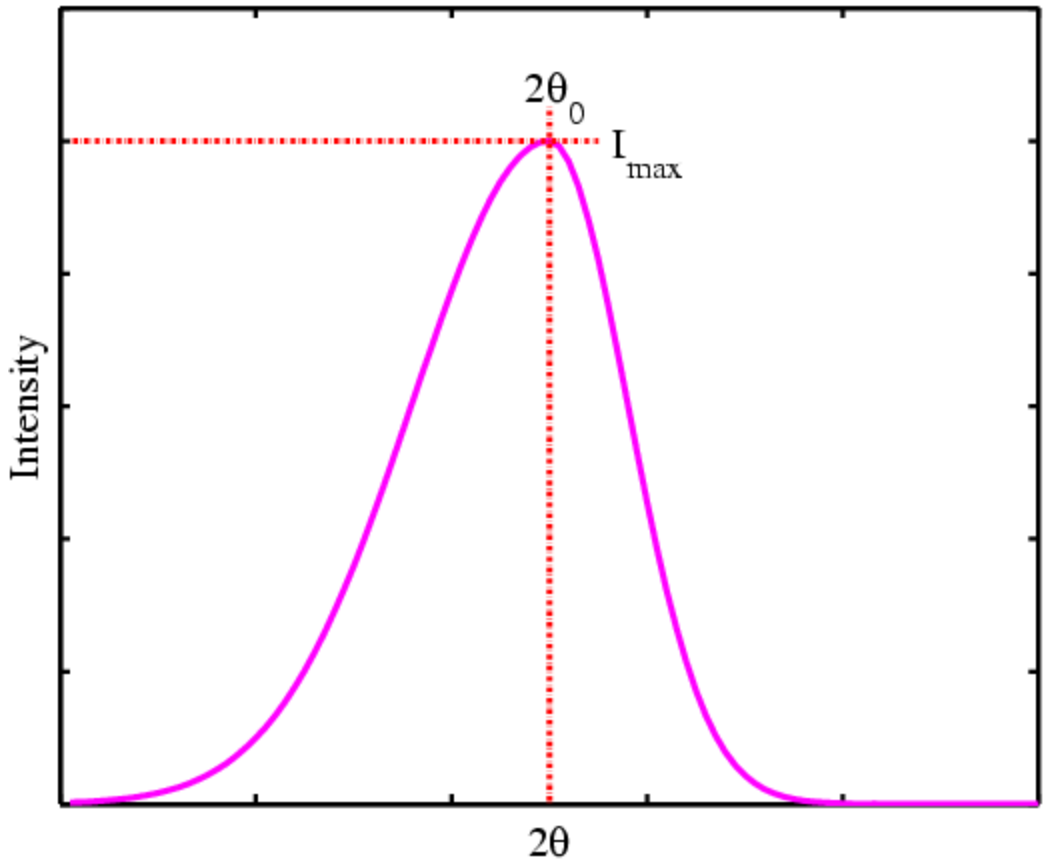
The pseudo-Voigt function

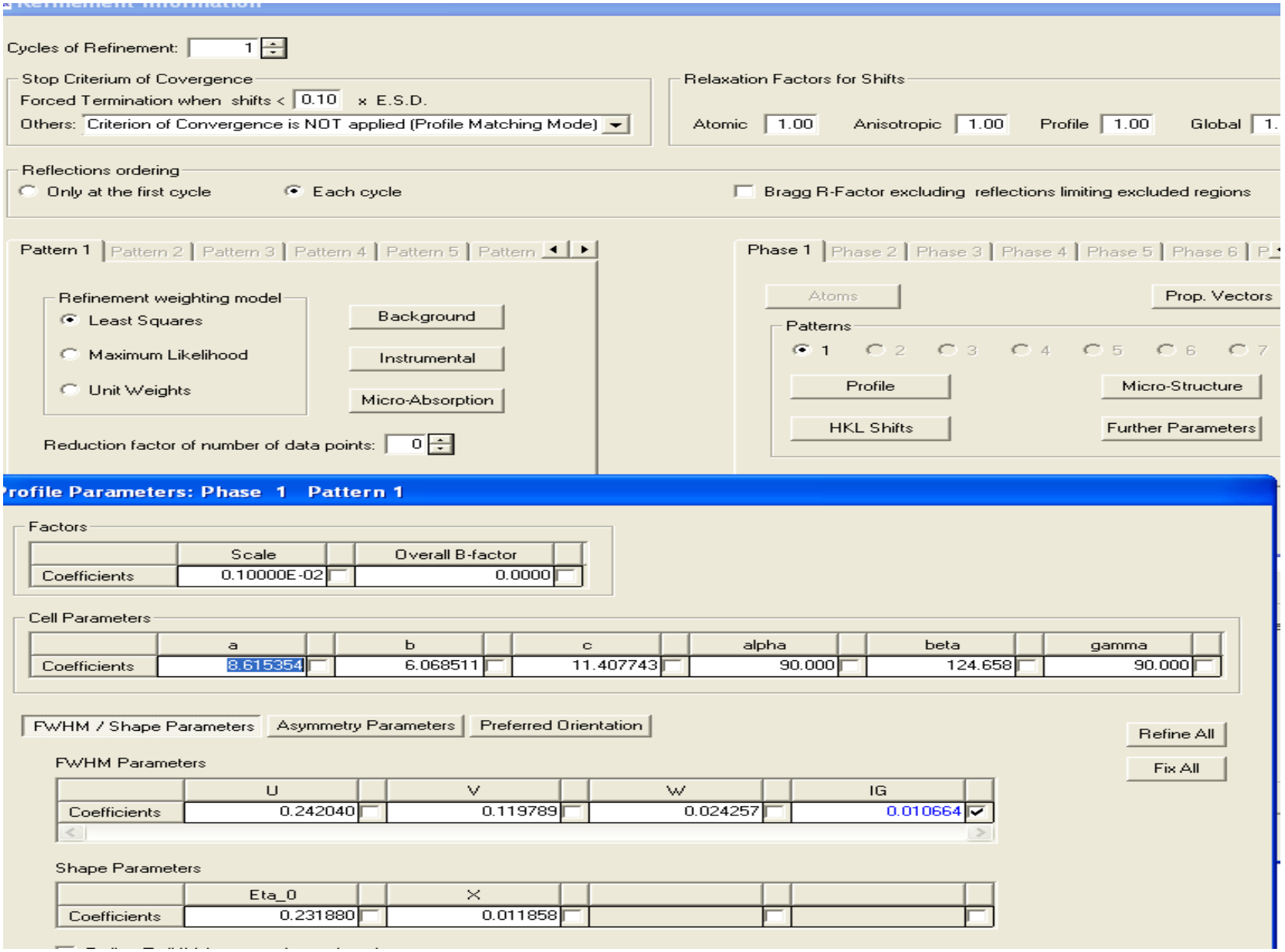
The pseudo-Voigt function has been shown to provide a good approximation to most peaks.
The pseudo-Voigt can be given by the following equation:

$I(2\theta) = I_{hkl} [\eta \mathbf{L} (2\theta - 2\theta_0) + (1 - \eta) \mathbf{G} (2\theta - 2\theta_0)]$

where, respectively, $\mathbf{L} (2\theta - 2\theta_0)$ and $\mathbf{G} (2\theta - 2\theta_0)$ represent suitably normalised Lorentz and Gaussian functions,.




2θ0
Imax
Intensity
2θ


Cycles of Refinement: 1
Stop Criterium of Covergence
Forced Termination when shifts < 0.10 x E.S.D.
Others: Criterion of Convergence is NOT applied (Profile Matching Mode)
Relaxation Factors for Shifts
Atomic 1.00 Anisotropic 1.00 Profile 1.00 Global 1.
Reflections ordering
Only at the first cycle
Each cycle
Bragg R-Factor excluding reflections limiting excluded regions
Pattern 1 Pattern 2 Pattern 3 Pattern 4 Pattern 5 Pattern
Refinement weighting model
Least Squares
Maximum Likelihood
Unit Weights
Background
Instrumental
Micro-Absorption
Reduction factor of number of data points: 0
Phase 1 Phase 2 Phase 3 Phase 4 Phase 5 Phase 6
Atoms
Prop. Vectors
Patterns
1 2 3 4 5 6 7
Profile
Micro-Structure
HKL Shifts
Further Parameters
rofile Parameters: Phase 1 Pattern 1
Factors
Scale
Overall B-factor
Coefficients 0.10000E-02 0.0000
Cell Parameters
a b c alpha beta gamma
Coefficients 8.615354 6.068511 11.407743 90.000 124.658 90.000
FWHM / Shape Parameters
Asymmetry Parameters
Preferred Orientation
Refine All
Fix All
FWHM Parameters
U V W IG
Coefficients 0.242040 0.119789 0.024257 0.010664
Shape Parameters
Eta_0 X
Coefficients 0.231880 0.011858

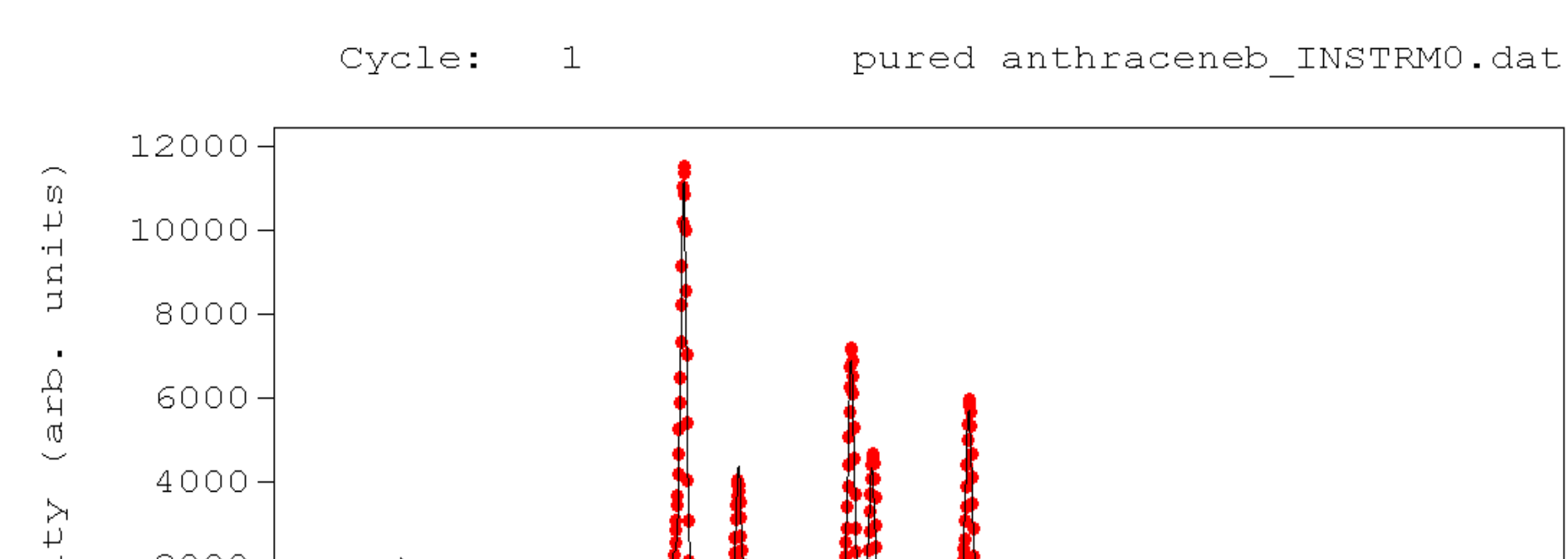
FullProf Program

Load Edit PCR Mode Run Exit

```
=> Solving L.S. equations...
=> Writing results for cycle       1
=> R-Factors:  10.6     14.4     Chi2:  14.4     DW-Stat.:  0.1862    Patt#:  1
=> Expected :           3.79                               1.8698
=> Conventional Rietveld R-factors for Pattern:       1
=> Rp: 23.9     Rwp: 24.4     Rexp:    6.43                        Chi2:  14.4
   => Global user-weigthed Chi2 (Bragg contrib.):   17.09
   => ---------> Pattern#       1
   => Phase:       1
   =>    Bragg R-factor:  0.2845E-03
   =>    RF-factor     :  0.7207E-01
=> Normal end, final calculations and writing...


=>              CPU Time:     0.172 seconds
=>                            0.003 minutes

=> END   Date:16/04/2012  Time => 11:57:09.484
```



$$R_F = \frac{\sum \left| I_K('obs')^{1/2} - I_K('calc')^{1/2} \right|}{\sum I_K('obs')^{1/2}}$$

R-structure factor

$$R_B = \frac{\sum \left| I_K('obs') - I_K('calc') \right|}{\sum I_K('obs')}$$

R-Bragg factor

$$R_p = \frac{\sum \left| y_i(obs) - y_i(calc) \right|}{\sum y_i(obs)}$$

R-pattern

$$R_{wp} = \left\{ \frac{\sum w_i (y_i(obs) - y_i(calc))^2}{\sum w_i (y_i(obs))^2} \right\}^{1/2}$$

R-weighted pattern

The function that is minimised is the chi-square χ^2:

$$\chi^2 = \frac{\sum_i w_i * \left| Y^i_{obs} - Y^i_{calc} \right|^2}{N - P}$$

where:

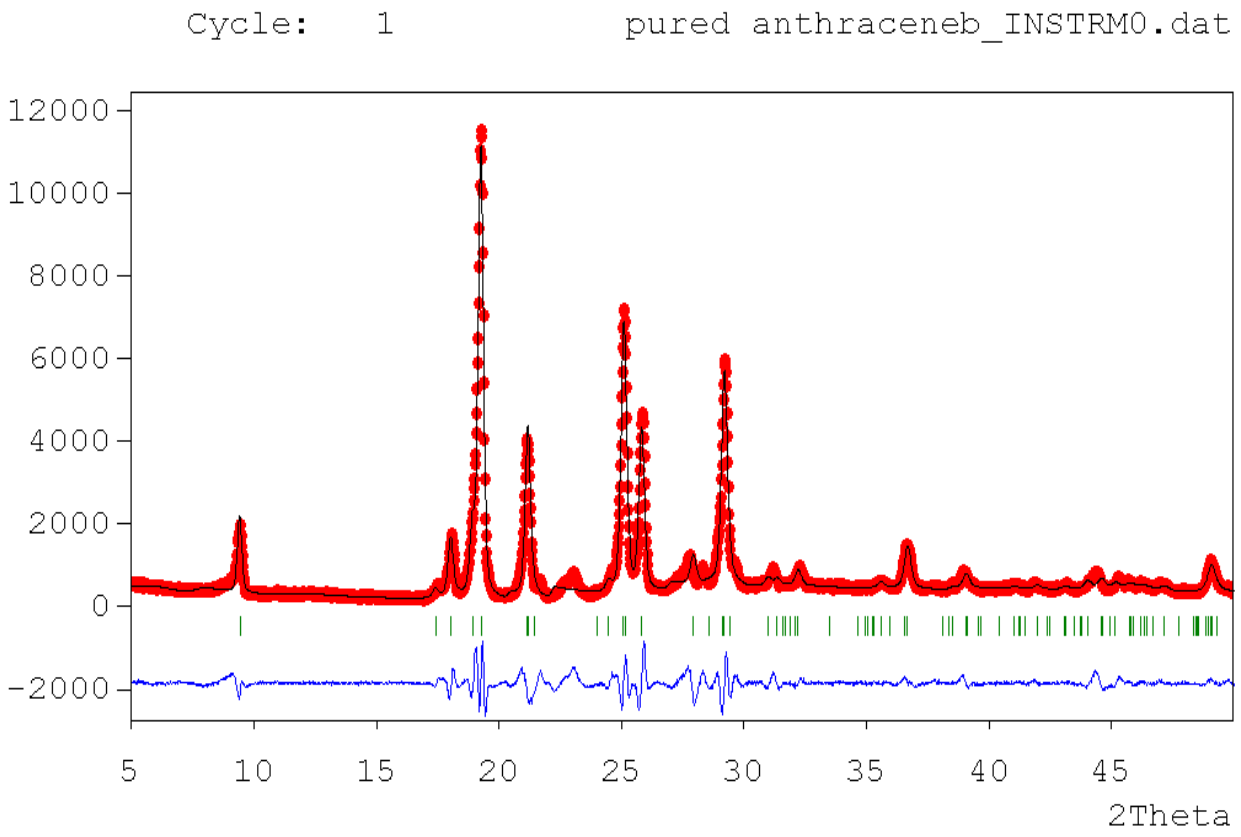
$\sum_i$: summation over the N points of the fitted region.

w_i : weighting factor ($w_i = \frac{1}{\sigma(Y^i_{obs})}$)

Y^i_{obs} : observed counts

Y^i_{calc} : calculated counts

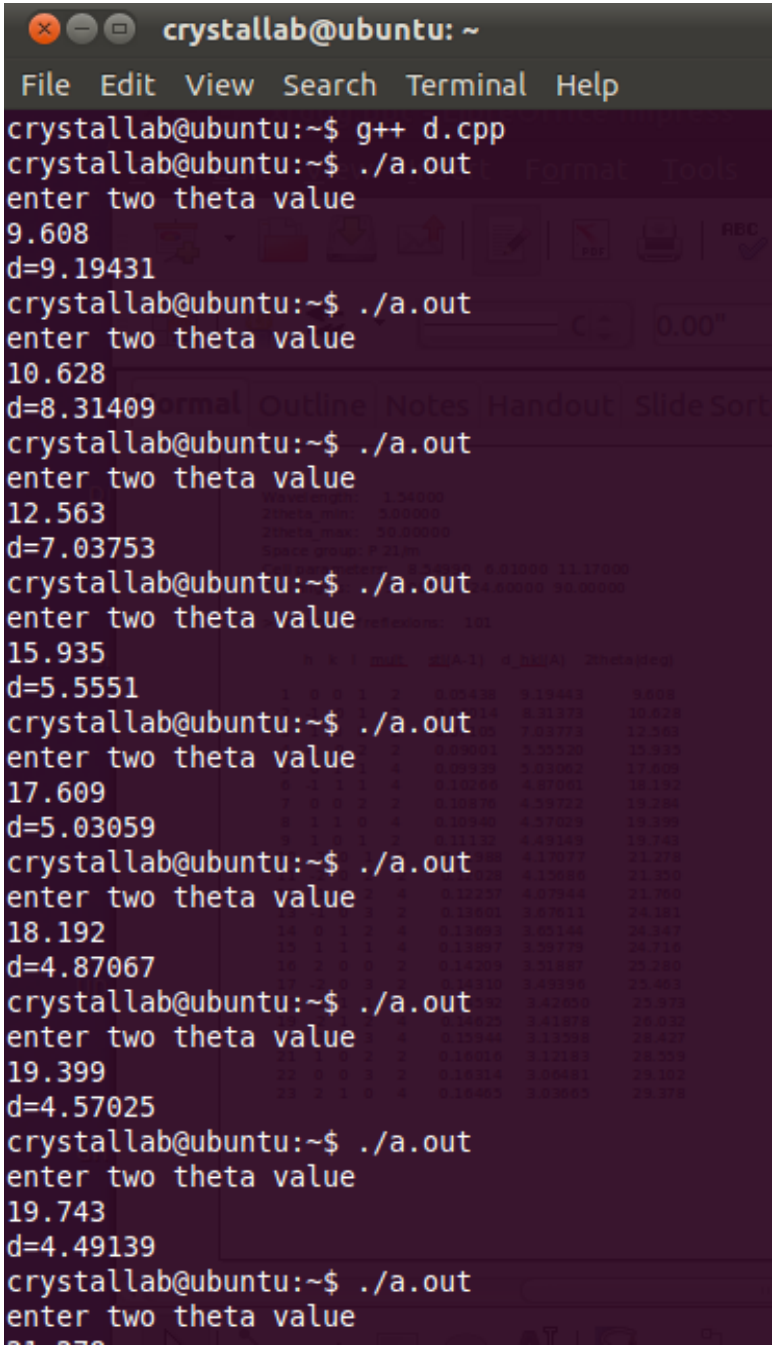
P : number of refined parameters.


Cycle: 1
pured anthraceneb_INSTRM0.dat
12000
10000
8000
6000
4000
2000
0
-2000
5
10
15
20
25
30
35
40
45
2Theta

Wavelength: 1.54000
2theta_min: 5.00000
2theta_max: 50.00000
Space group: P 21/m
Cell parameters: 8.54990 6.01000 11.17000
Cell angles: 90.00000 124.60000 90.00000

> Number of reflexions: 101

	h	k	l	mult	stl(A-1)	d_hkl(A)	2theta(deg)
1	0	0	1	2	0.05438	9.19443	9.608
2	-1	0	1	2	0.06014	8.31373	10.628
3	1	0	0	2	0.07105	7.03773	12.563
4	-1	0	2	2	0.09001	5.55520	15.935
5	0	1	1	4	0.09939	5.03062	17.609
6	-1	1	1	4	0.10266	4.87061	18.192
7	0	0	2	2	0.10876	4.59722	19.284
8	1	1	0	4	0.10940	4.57029	19.399
9	1	0	1	2	0.11132	4.49149	19.743
10	-2	0	1	2	0.11988	4.17077	21.278
11	-2	0	2	2	0.12028	4.15686	21.350
12	-1	1	2	4	0.12257	4.07944	21.760
13	-1	0	3	2	0.13601	3.67611	24.181
14	0	1	2	4	0.13693	3.65144	24.347
15	1	1	1	4	0.13897	3.59779	24.716
16	2	0	0	2	0.14209	3.51887	25.280
17	-2	0	3	2	0.14310	3.49396	25.463
18	-2	1	1	4	0.14592	3.42650	25.973
19	-2	1	2	4	0.14625	3.41878	26.032
20	-1	1	3	4	0.15944	3.13598	28.427
21	1	0	2	2	0.16016	3.12183	28.559
22	0	0	3	2	0.16314	3.06481	29.102
23	2	1	0	4	0.16465	3.03665	29.378



24	-2	1	3	4	0.16553	3.02060	29.537
25	0	2	0	2	0.16639	3.00500	29.694
26	0	2	1	4	0.17505	2.85632	31.278
27	-3	0	2	2	0.17587	2.84303	31.428
28	-1	2	1	4	0.17692	2.82606	31.622
29	2	0	1	2	0.17867	2.79847	31.942
30	-2	0	4	2	0.18001	2.77760	32.188
31	-3	0	3	2	0.18042	2.77124	32.264
32	1	1	2	4	0.18048	2.77038	32.275
33	1	2	0	4	0.18092	2.76362	32.356
34	0	1	3	4	0.18313	2.73030	32.762
35	-1	0	4	2	0.18658	2.67979	33.397
36	-3	0	1	2	0.18767	2.66420	33.598
37	-1	2	2	4	0.18917	2.64308	33.875
38	-3	1	2	4	0.19455	2.56998	34.869
39	2	1	1	4	0.19709	2.53693	35.338
40	-2	1	4	4	0.19831	2.52135	35.564
41	-3	1	3	4	0.19868	2.51659	35.633
42	0	2	2	4	0.19878	2.51531	35.652
43	1	2	1	4	0.20019	2.49757	35.914
44	-3	0	4	2	0.20023	2.49717	35.919
45	-1	1	4	4	0.20429	2.44751	36.674
46	-2	2	1	4	0.20508	2.43809	36.821
47	-3	1	1	4	0.20529	2.43562	36.860
48	-2	2	2	4	0.20531	2.43531	36.864
49	1	0	3	2	0.21172	2.36159	38.05
51	-1	2	3	4	0.21491	2.32659	38.654
52	-3	1	4	4	0.21682	2.30603	39.012
53	0	0	4	2	0.21752	2.29861	39.143
54	2	2	0	4	0.21880	2.28515	39.383
55	-2	2	3	4	0.21946	2.27828	39.507
56	2	0	2	2	0.22264	2.24575	40.104
57	-2	0	5	2	0.22415	2.23063	40.387
58	1	1	3	4	0.22748	2.19799	41.014
59	3	1	0	4	0.22880	2.18533	41.262
60	1	2	2	4	0.23095	2.16498	41.668
61	-3	0	5	2	0.23139	2.16083	41.752
62	0	1	4	4	0.23289	2.14694	42.035
63	0	2	3	4	0.23303	2.14569	42.060
64	-4	0	3	2	0.23393	2.13741	42.231
65	2	1	2	4	0.23768	2.10368	42.941
66	-1	0	5	2	0.23883	2.09353	43.160
67	-2	1	5	4	0.23909	2.09124	43.210
68	-4	0	2	2	0.23976	2.08538	43.337
69	-4	0	4	2	0.24057	2.07843	43.489
70	-3	2	2	4	0.24211	2.06521	43.782

71	2	2	1	4	0.24415	2.04794	44.171
72	-2	2	4	4	0.24513	2.03972	44.358
73	-3	2	3	4	0.24544	2.03720	44.416
74	-3	1	5	4	0.24589	2.03339	44.504
75	3	0	1	2	0.24809	2.01541	44.922
76	-4	1	3	4	0.24828	2.01385	44.959
77	-1	2	4	4	0.25000	2.00003	45.287
78	-3	2	1	4	0.25081	1.99352	45.443
79	-1	1	5	4	0.25291	1.97702	45.844
80	-4	1	2	4	0.25379	1.97015	46.013
81	-4	1	4	4	0.25455	1.96429	46.158
82	0	3	1	4	0.25544	1.95741	46.330
83	-1	3	1	4	0.25673	1.94759	46.577
84	-4	0	1	2	0.25723	1.94381	46.673
85	-4	0	5	2	0.25872	1.93259	46.960
86	1	3	0	4	0.25950	1.92679	47.110
87	-3	2	4	4	0.26034	1.92058	47.272
88	3	1	1	4	0.26167	1.91083	47.527
89	1	0	4	2	0.26441	1.89098	48.058
90	-1	3	2	4	0.26532	1.88454	48.232
91	1	2	3	4	0.26928	1.85681	49.000
92	-3	0	6	2	0.27002	1.85173	49.143
93	-4	1	1	4	0.27035	1.84948	49.207
94	3	2	0	4	0.27039	1.84916	49.216
95	2	0	3	2	0.27043	1.84892	49.223
96	-4	1	5	4	0.27177	1.83981	49.483
97	0	0	5	2	0.27190	1.83889	49.509
98	-2	0	6	2	0.27203	1.83806	49.533
99	0	3	2	4	0.27225	1.83653	49.577
100	1	3	1	4	0.27328	1.82959	49.778
101	0	2	4	4	0.27386	1.82572	49.890

Application of XRD

XRD is a nondestructive technique. Some of the uses of x-ray diffraction are;

1. Differentiation between crystalline and amorphous materials;
2. Determination of the structure of crystalline materials;
3. Determination of electron distribution within the atoms, and throughout the unit cell;
4. Determination of the orientation of single crystals;
5. Determination of the texture of polygrained materials;
6. Measurement of strain and small grain size…..etc

Advantages and disadvantages of X-rays

Advantages;

- X-ray is the cheapest, the most convenient and widely used method.
- X-rays are not absorbed very much by air, so the specimen need not be in an evacuated chamber.

Disadvantage;

- They do not interact very strongly with lighter elements.

ThankYou