Introduction to Diffraction analysis

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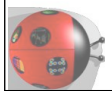
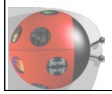


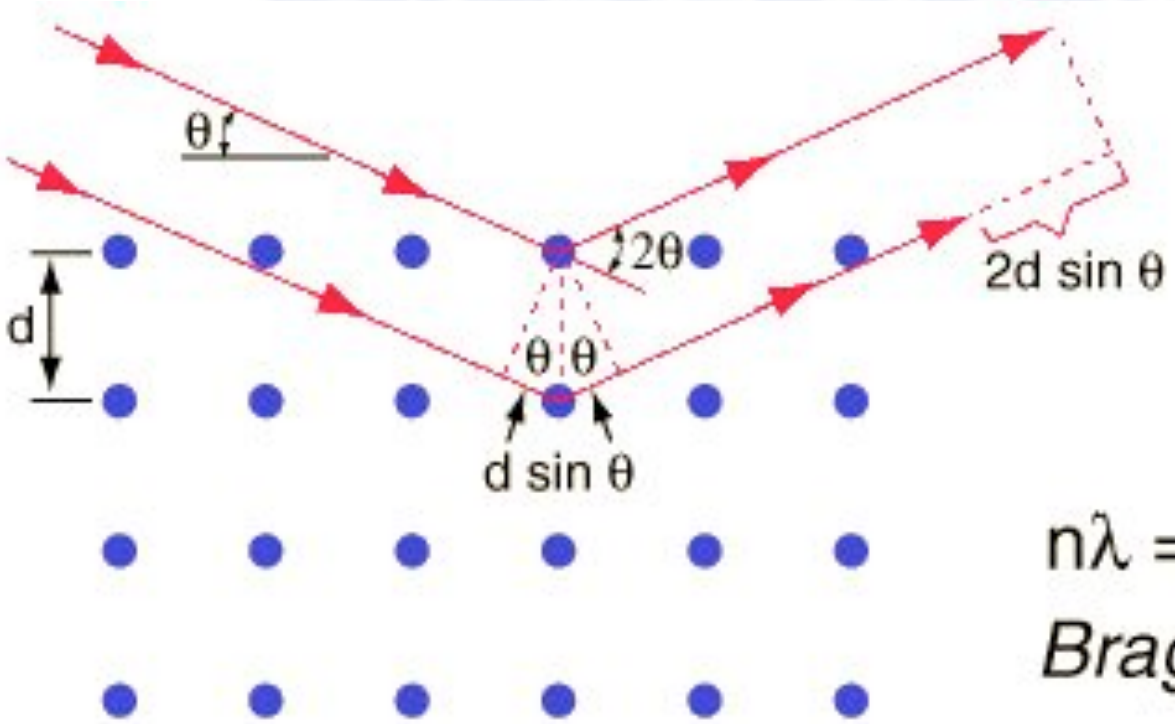
Outline: basic concepts

- The Bragg law
- The intensity of the diffraction
- Powder diffraction and instrumentation
  - Bragg-Brentano
  - Texture goniometers
  - Residual stress measurements
- Diffraction analyses



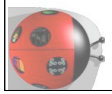
The Bragg law

- *Constructive interference and interplanar spacing:*



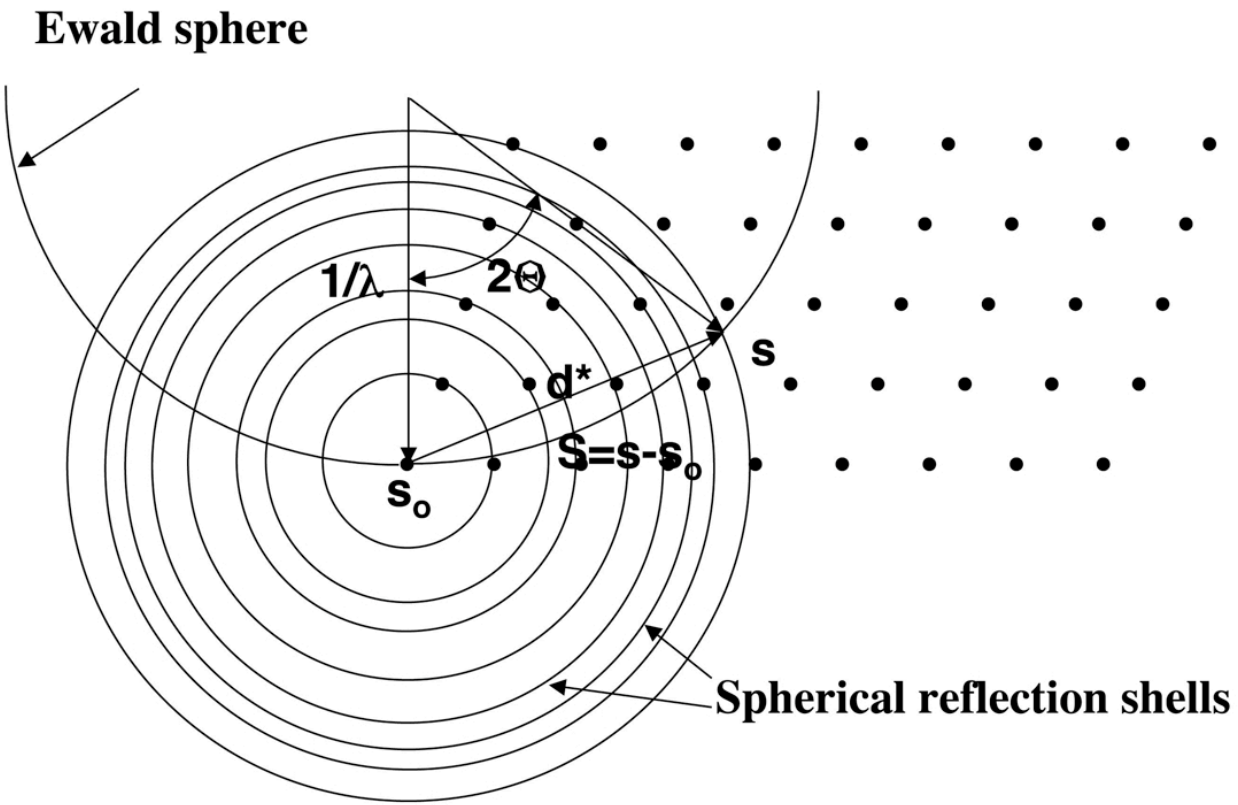
$n\lambda = 2d \sin \theta$

Bragg's Law

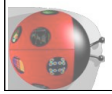


Reciprocal space

Again get Bragg's Law $d^*/2 = \sin\Theta/\lambda$

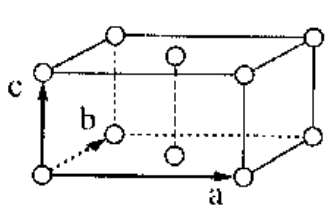


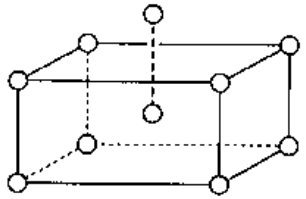
$$d_{hkl} = \frac{V_C}{\sqrt{s_{11}h^2 + s_{22}k^2 + s_{33}l^2 + 2s_{12}hk + 2s_{13}hl + 2s_{23}kl}}$$

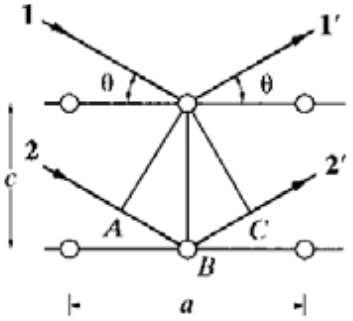


The Bragg law and the intensities

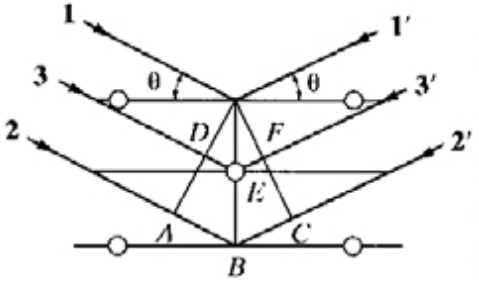
- *Consider diffraction from the (001) plane*

(a) 


(b)

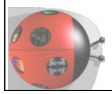


(a)



(b)

If the path length between rays 1 and 2 differs by λ, the path length between rays 1 and 3 will differ by λ/2 and destructive interference in (b) will lead to no diffracted intensity



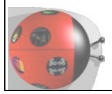
Diffraction intensities

- **The intensity in a powder diffractometer**

$$I_i^{calc} = S_F \sum_{j=1}^{Nphases} \frac{f_j}{V_j^2} \sum_{k=1}^{Npeaks} L_k \left| F_{k,j} \right|^2 S_j \left(2\theta_i - 2\theta_{k,j} \right) P_{k,j} A_j + bkg_i$$

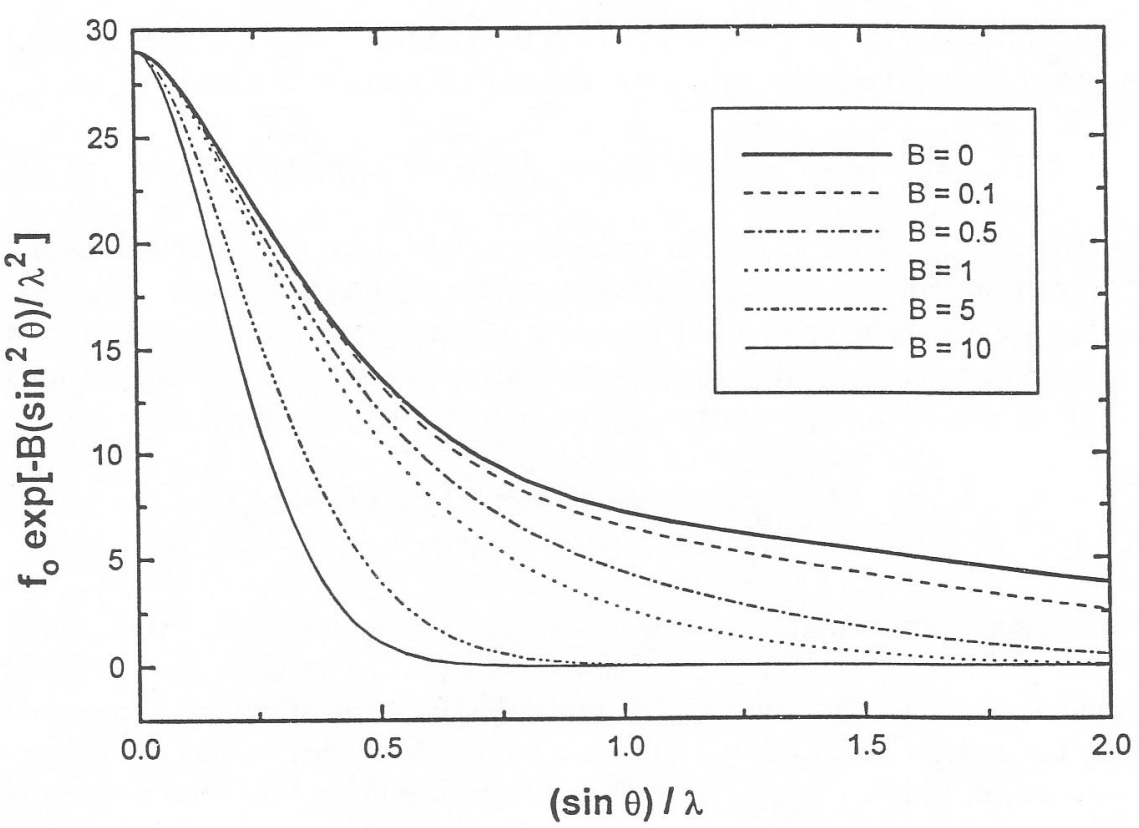
- **The structure factor:**

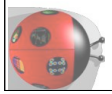
$$\left| F_{k,j} \right|^2 = m_k \left| \sum_{n=1}^{N} f_n e^{-B_n \frac{\sin^2 \theta}{\lambda^2}} \left(e^{2\pi i \left(hx_n + ky_n + lz_n \right)} \right) \right|^2$$



Atomic scattering factor and Debye-Waller

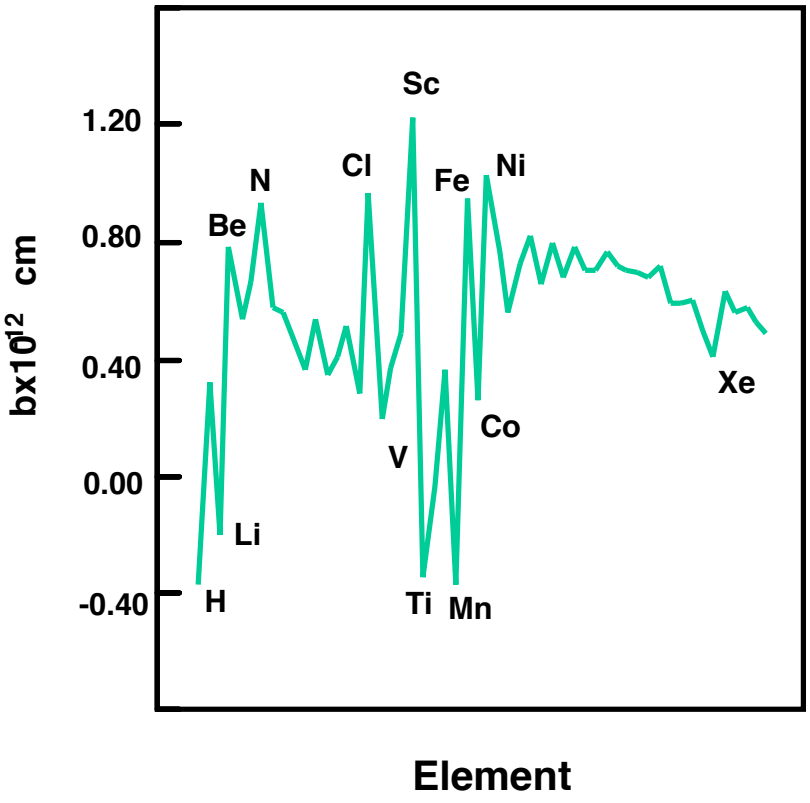
- *The atomic scattering factor for X-ray decreases with the diffraction angle and is proportional to the number of electrons. For neutron is not correlated to the atomic number.*

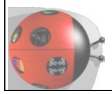




Neutron scattering factors

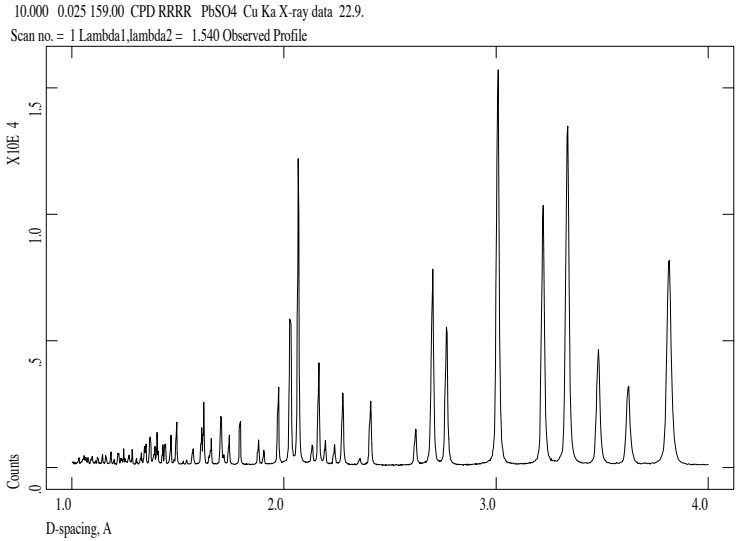
- *For light atoms neutron scattering has some advantages*
- *For atoms very close in the periodic table, neutron scattering may help distinguish them.*

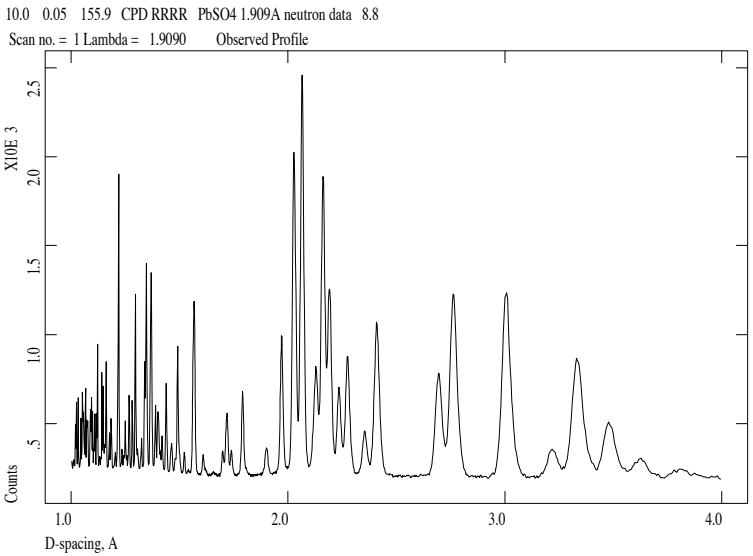




X-ray and neutron diffraction

X-ray Diffraction - CuKa
Phillips PW1710
- **Higher resolution**
- **Intensity falloff at small d spacings**
- **Better at resolving small lattice distortions**

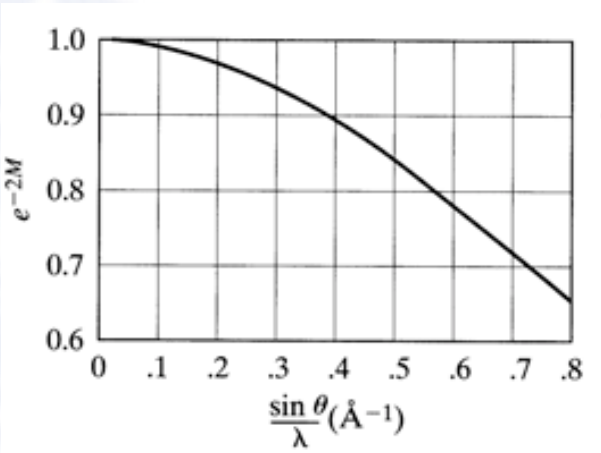


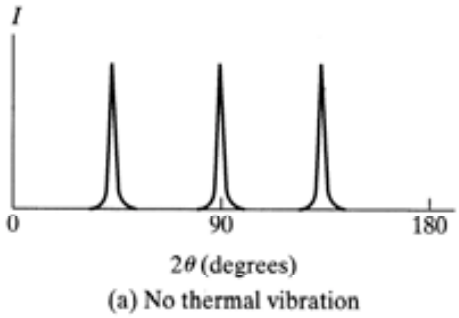


Neutron Diffraction - D1a, ILL
λ=1.909 Å
- **Lower resolution**
- **Much higher intensity at small d-spacings**
- **Better atomic positions/thermal parameters**

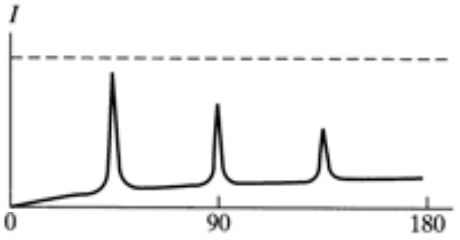
Thermal or Debye-Waller factor

- *It causes a decrease of the intensities at high angle*
- *It is proportional to the thermal vibrations*
- *Intensities decrease increasing the temperature*
- *From the Debye-Waller it is possible to estimate the Debye temperature*

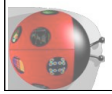




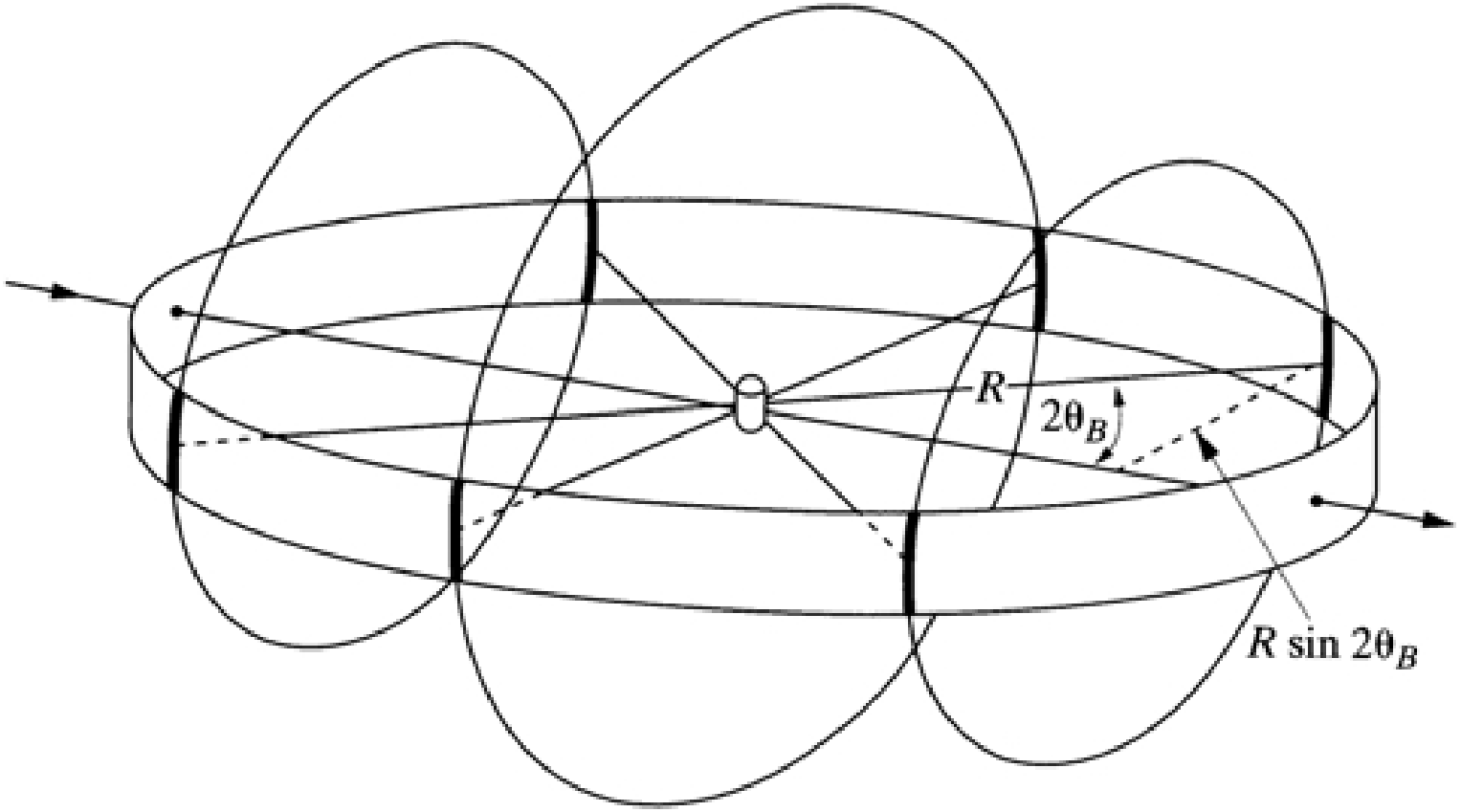
(a) No thermal vibration



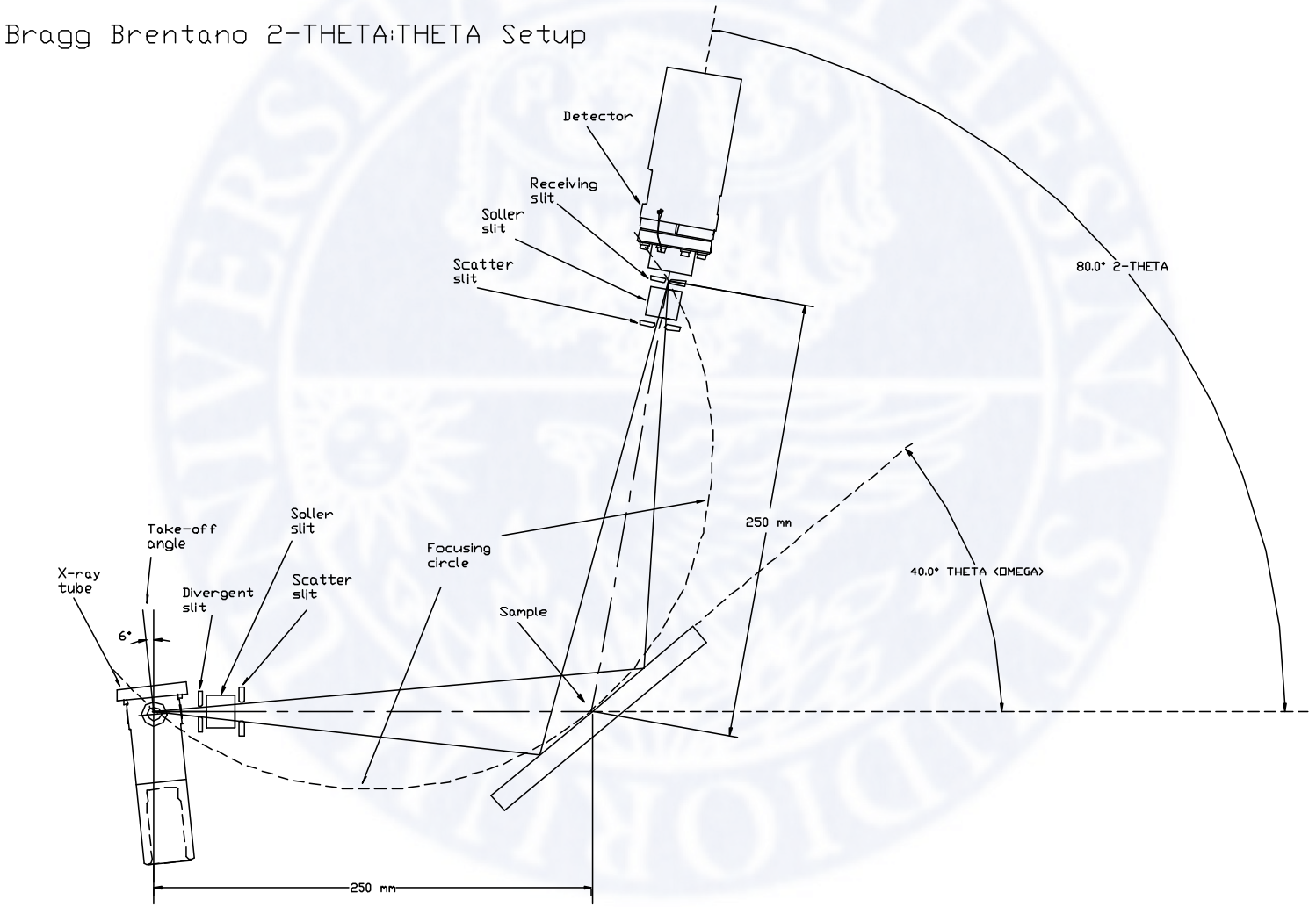
(b) Thermal vibration



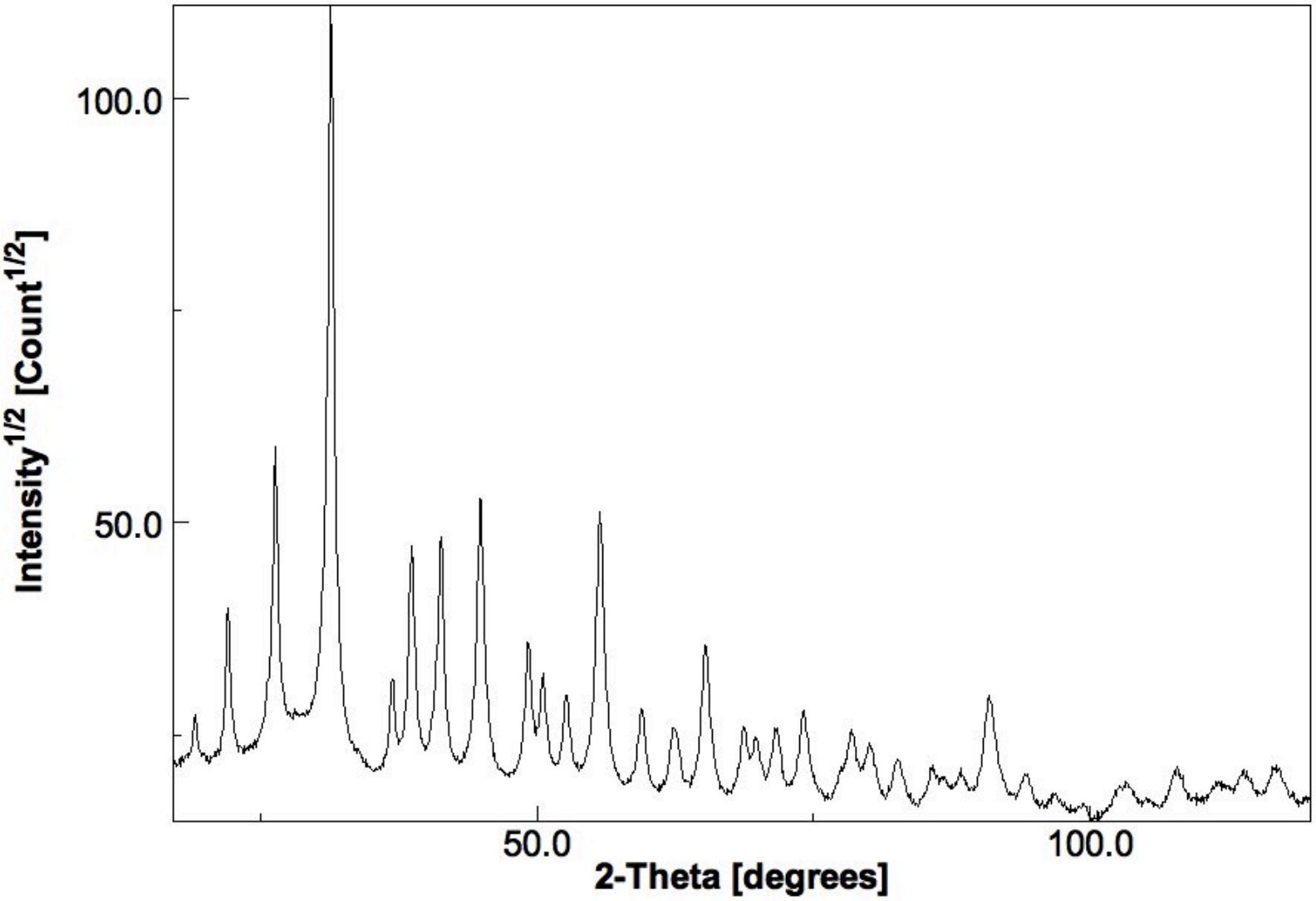
Powder diffraction and Debye-Scherrer camera

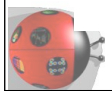


A modern diffractomer

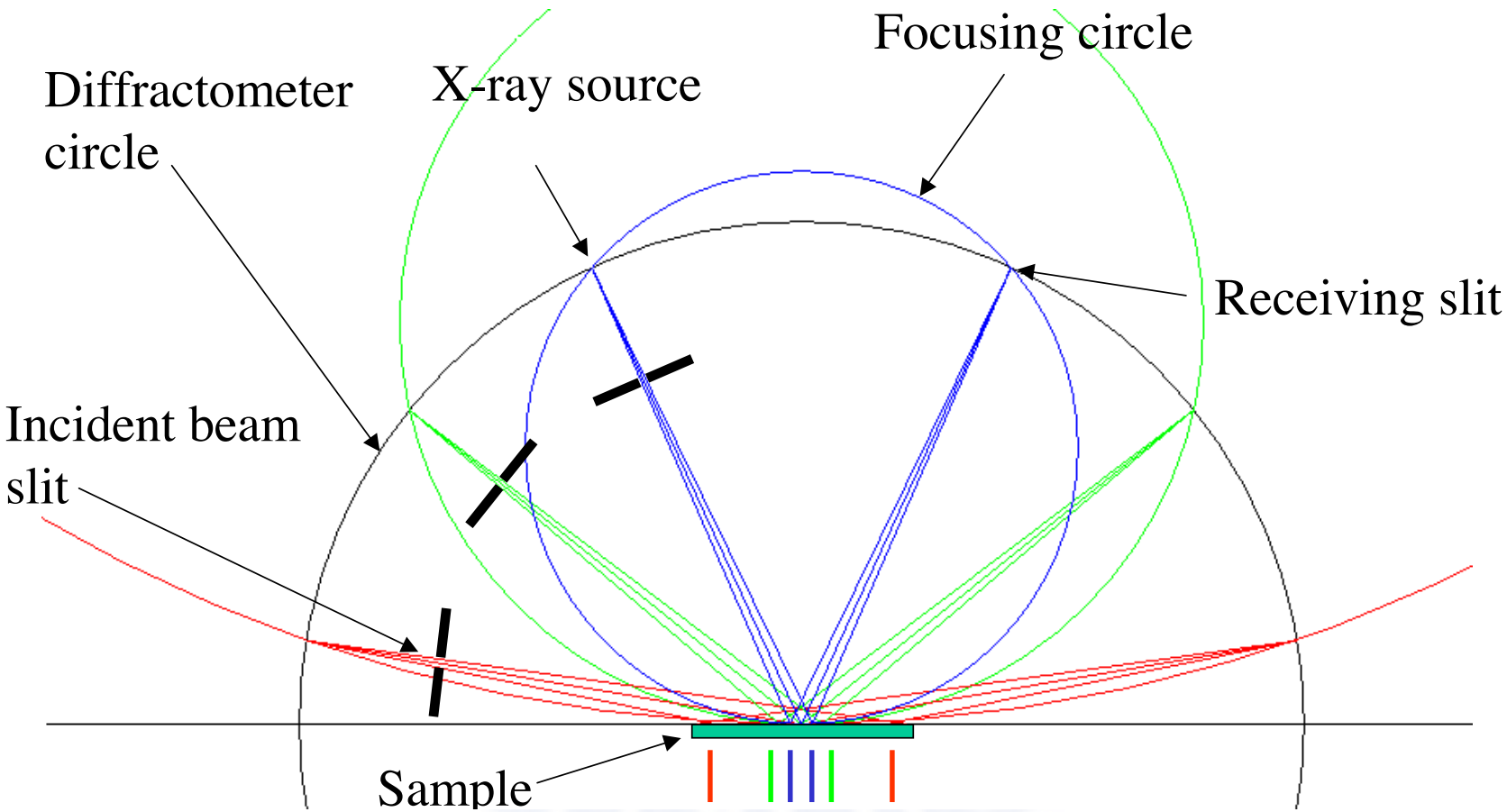


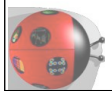
A typical spectrum



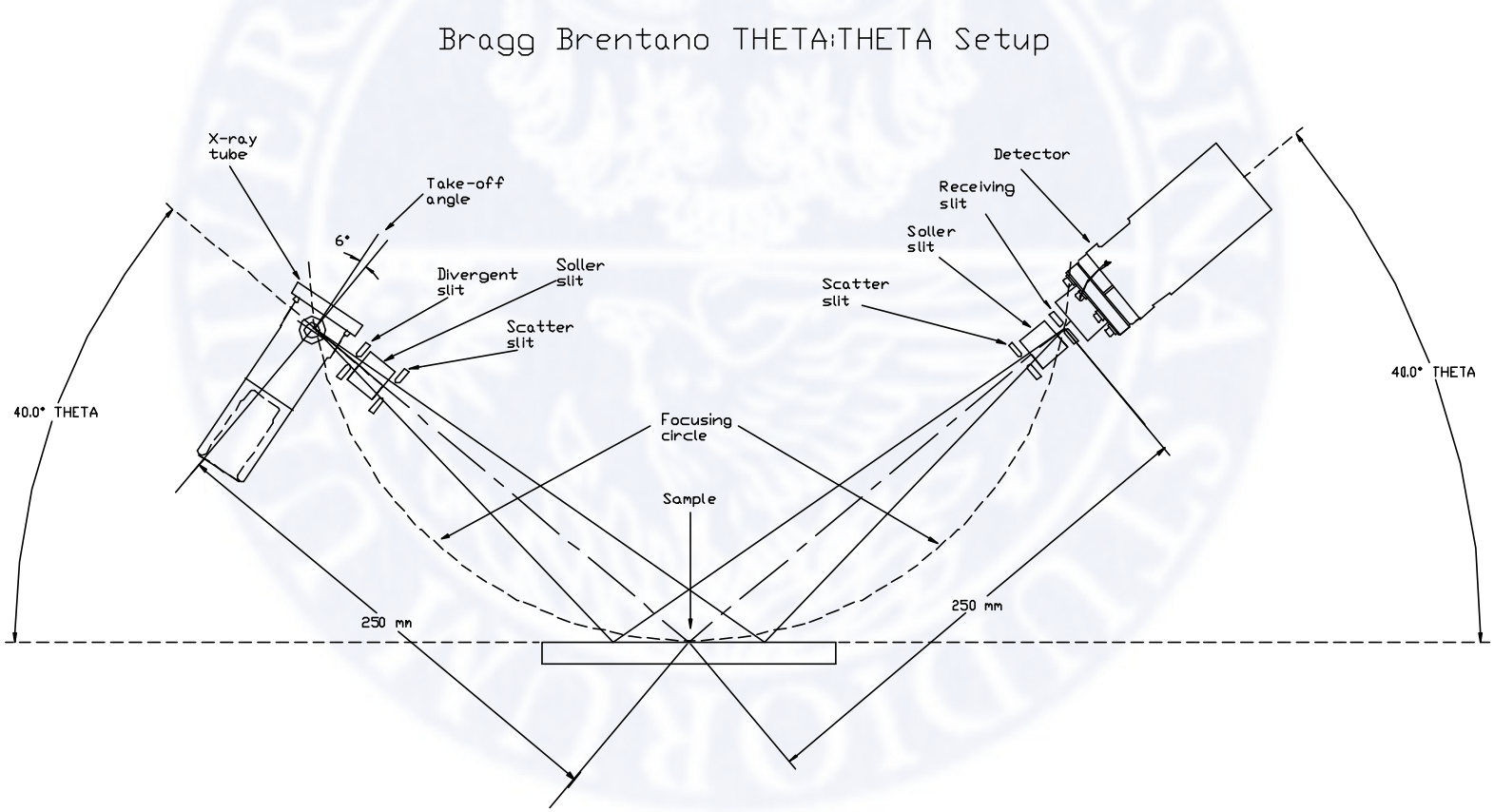


Parafocusing circle (Bragg-Brentano)

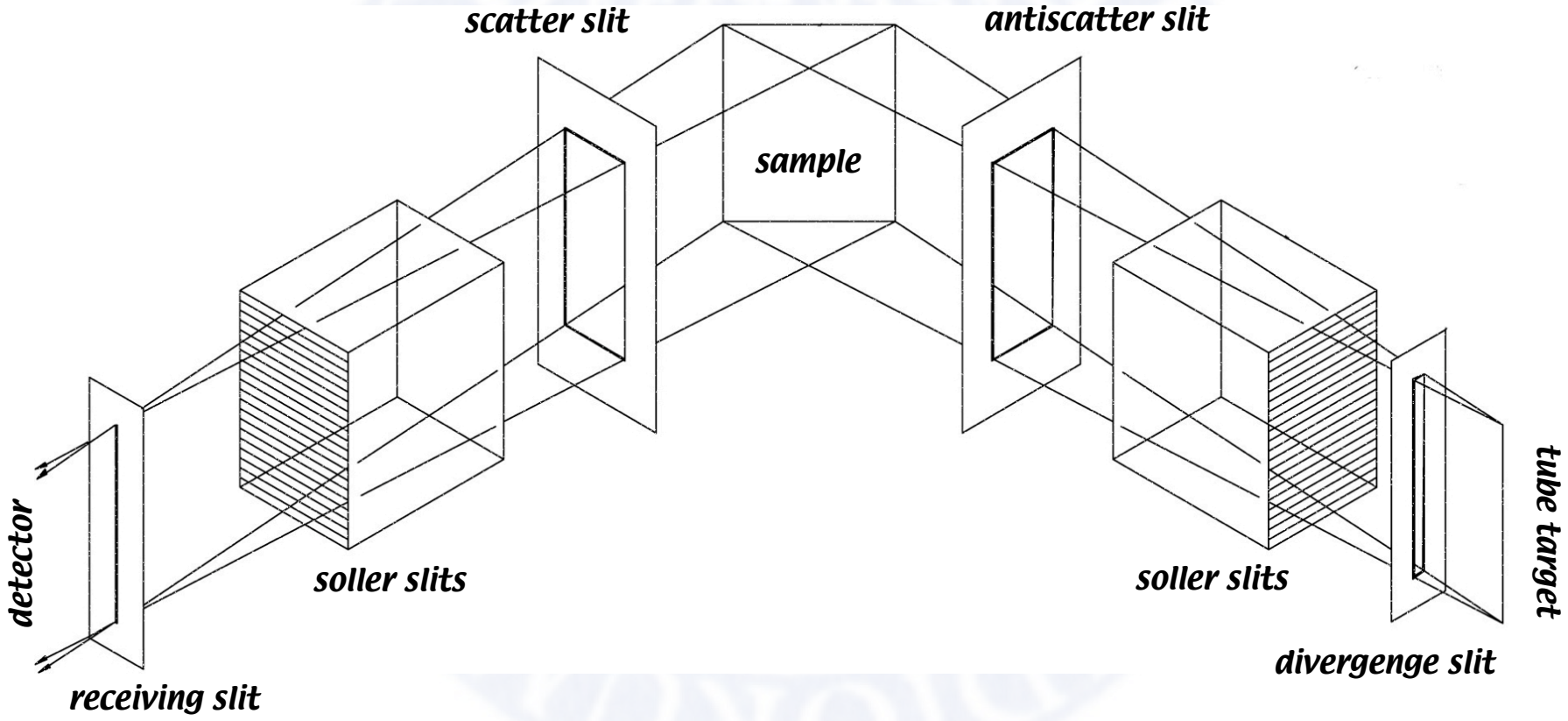




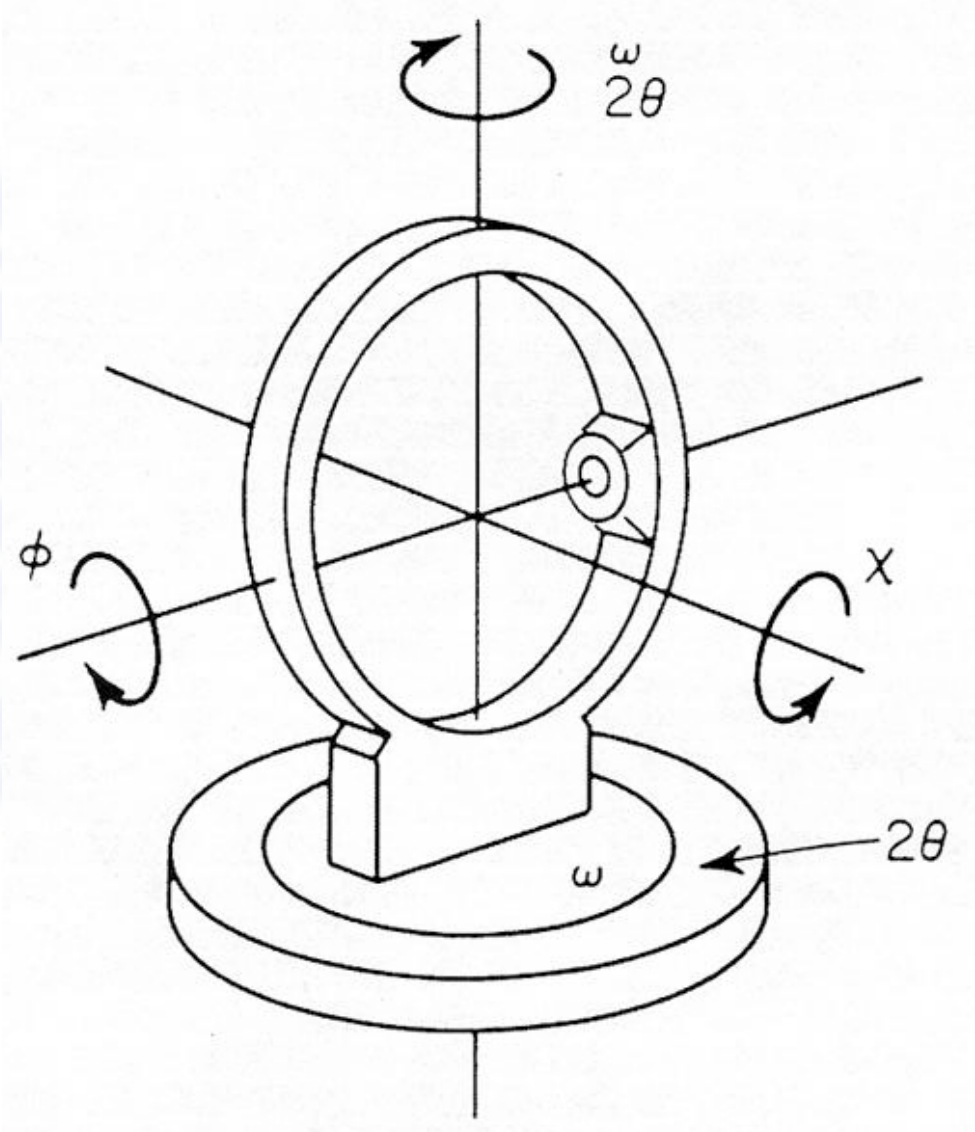
Theta-theta diffractometer



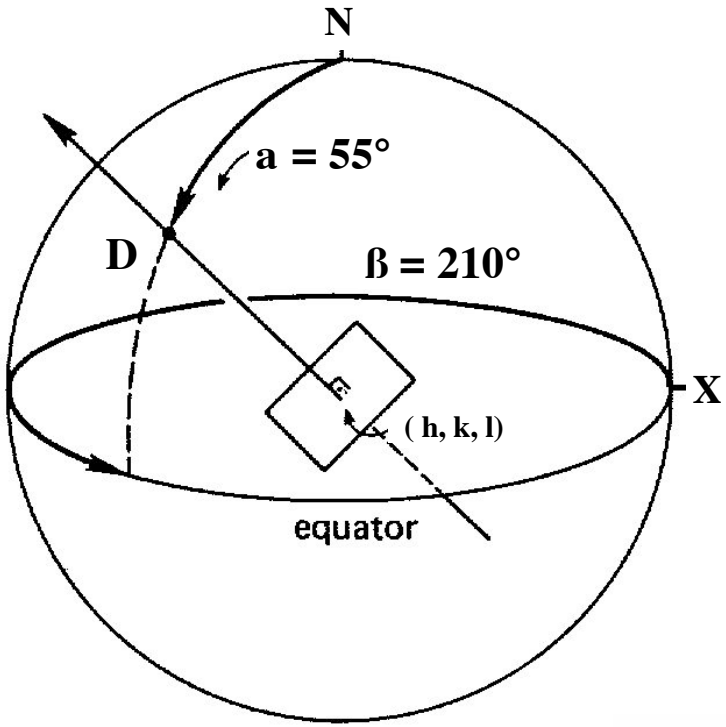
Slits system in Bragg-Brentano



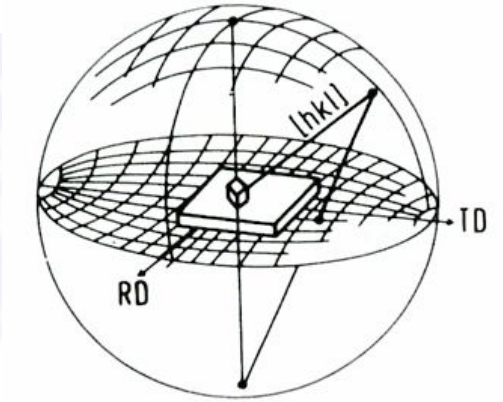
Texture goniometer

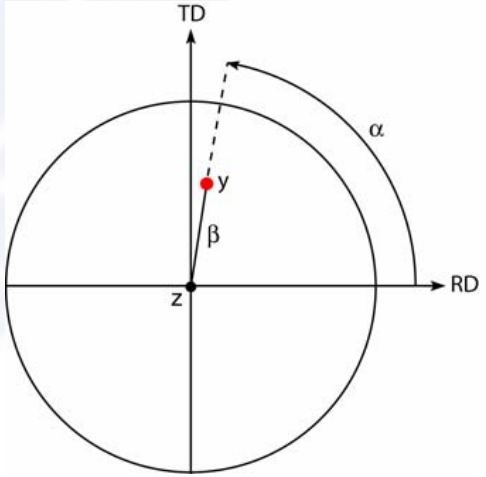


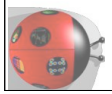
Texture orientations



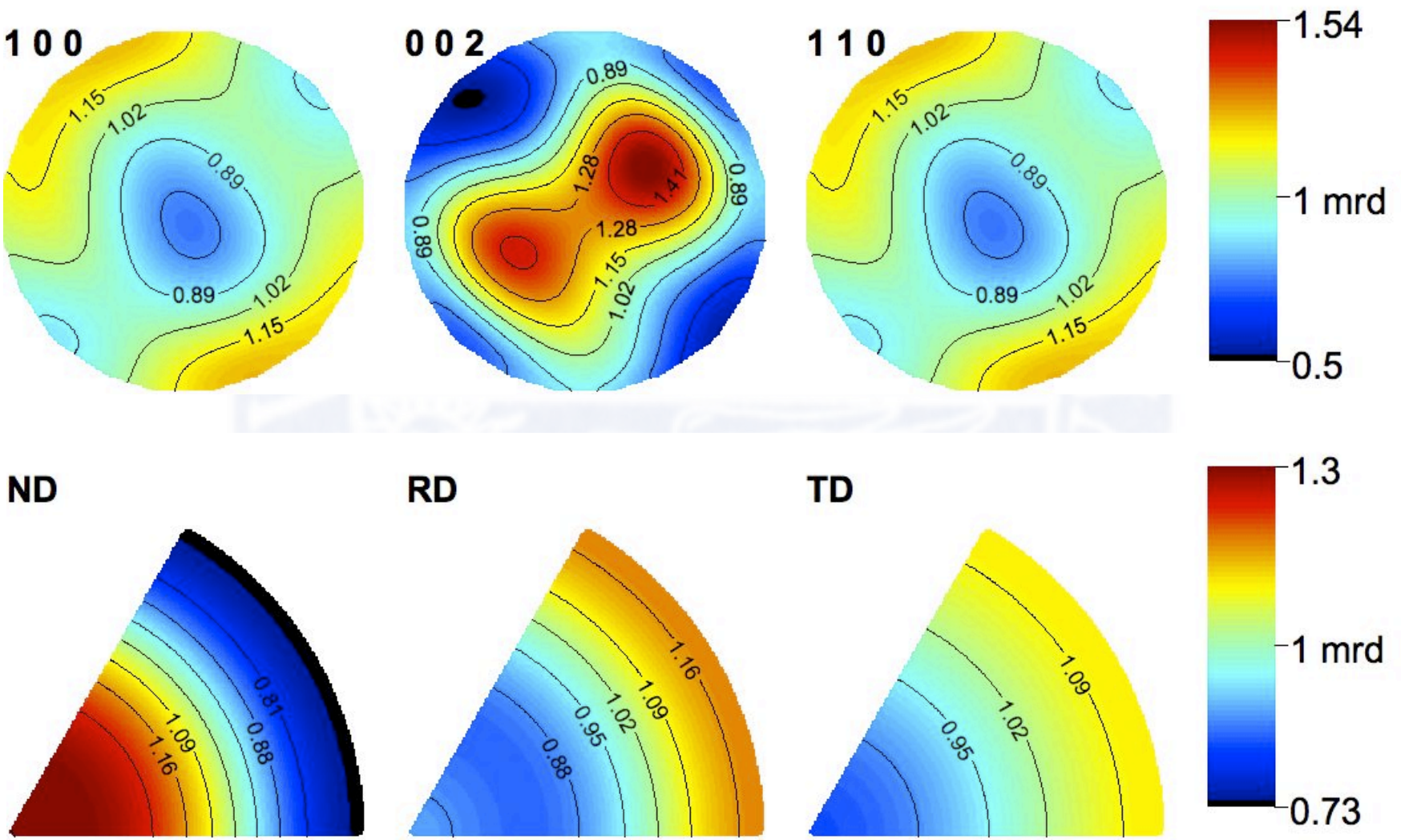
Pole figure representation



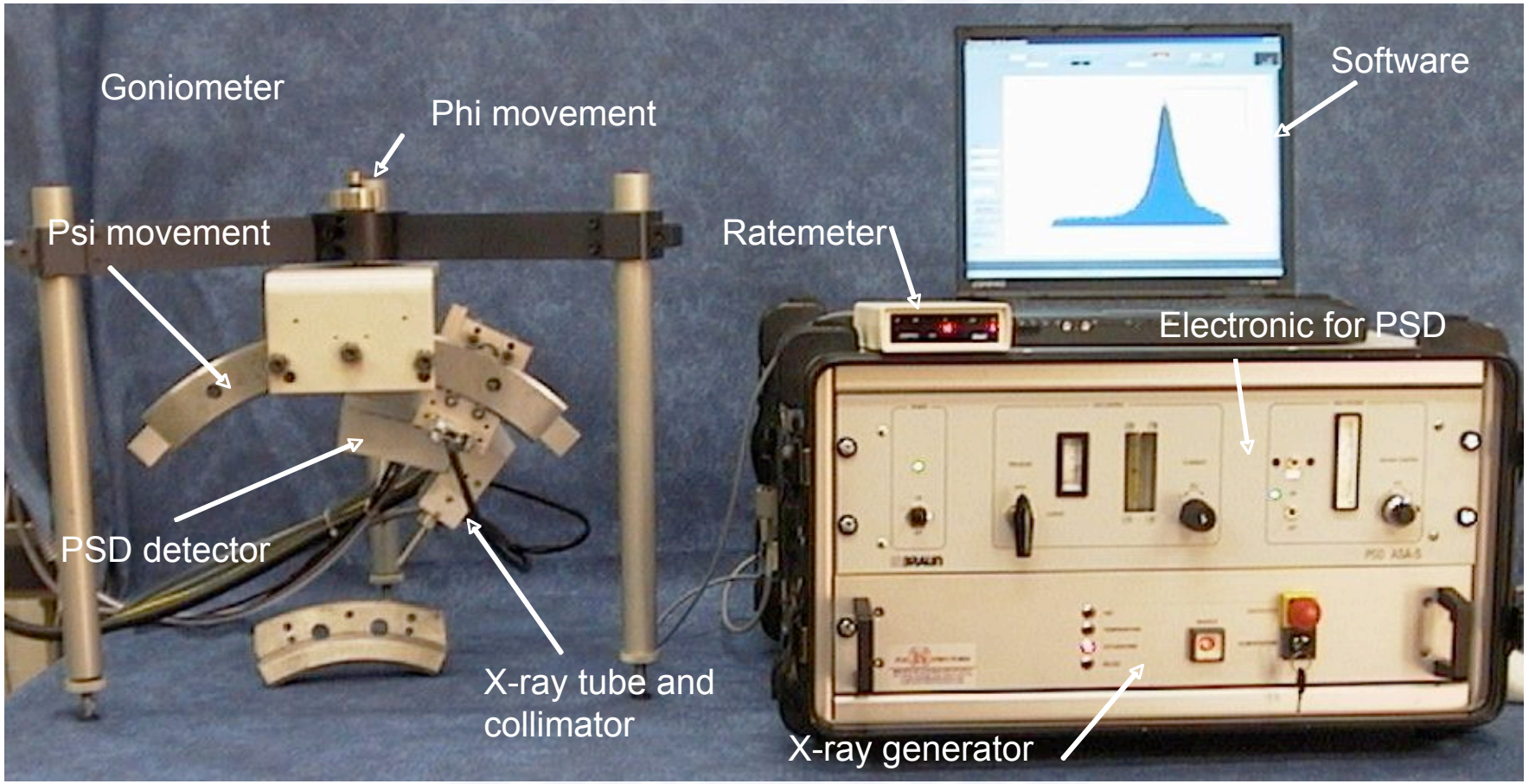




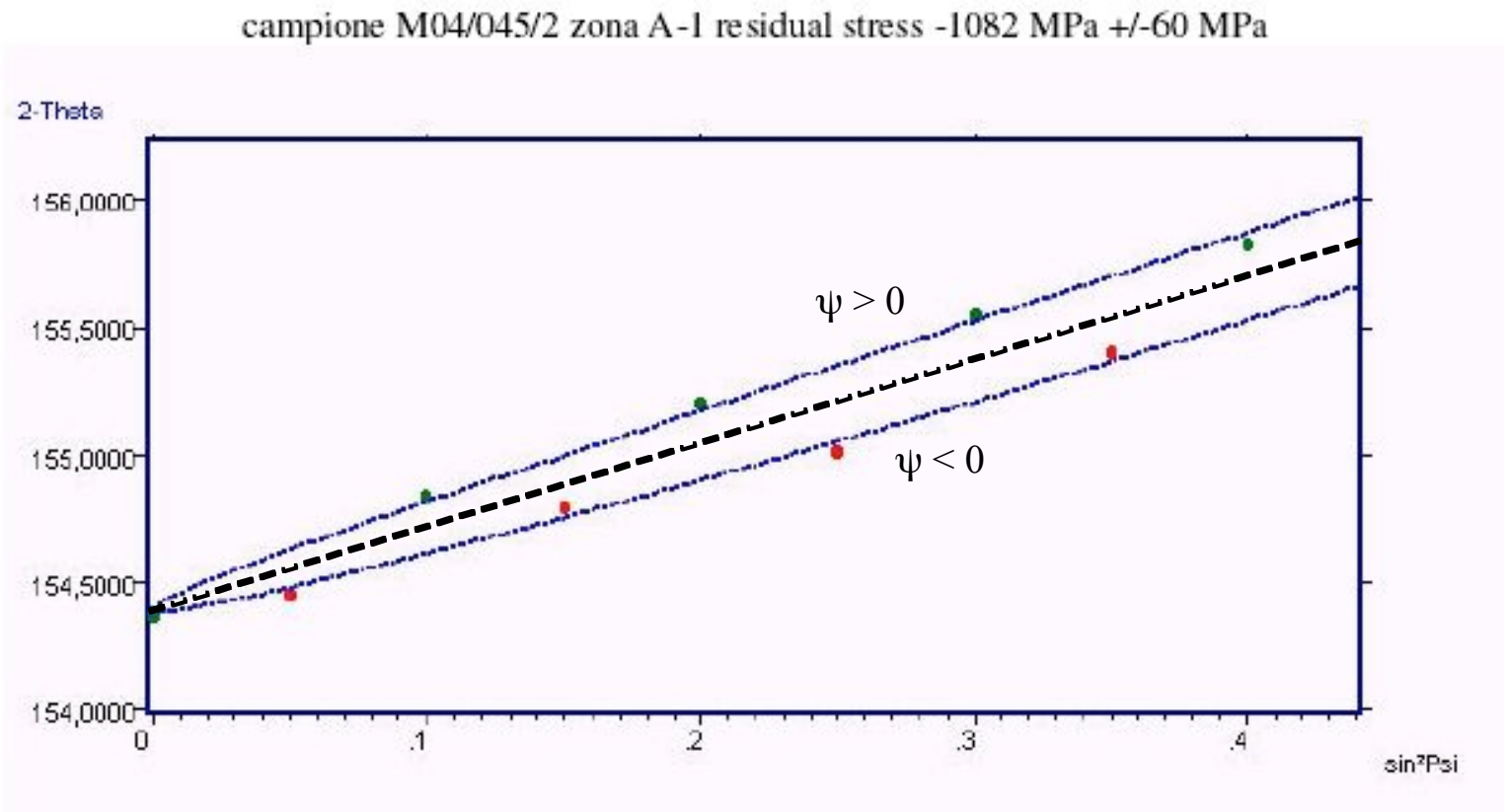
Pole figure projections (and inverse)



Residual stress measurement



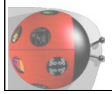
Residual stress analysis



When either or both of ε_{13}, ε_{23} are non-zero, d measured at positive and negative Psi will be different due to the argument $\sin^2\Psi$ associated with these terms causing a "split" in the d (2-theta) vs. $\sin^2$Psi data. This effect is termed "Psi-splitting".

Diffraction analyses

- *Phase identifications (crystalline and amorphous)*
- *Crystal structure determination*
- *Crystal structure refinements (cell parameters and atomic positions)*
- *Quantitative phase analysis (and crystallinity determination)*
- *Microstructural analyses (crystallite sizes - microstrain distributions etc.)*
- *Texture analysis*
- *Residual stress analysis*
- *Order-disorder transitions and compositional analyses*
- *Thin films*



Search-Match and the PDF system

PDF # 340412, Wavelength = 1.54178 (A)

34-0412 Quality: I

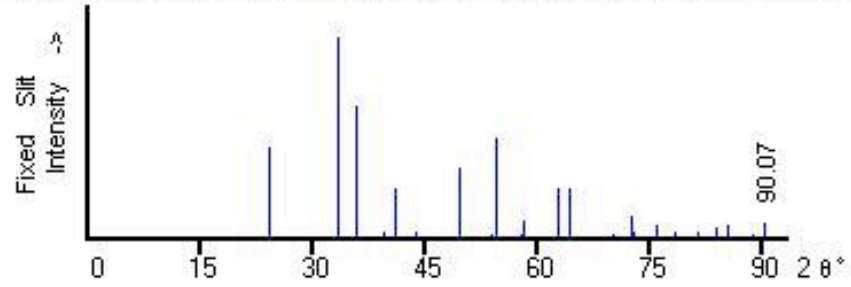
CAS Number:

Molecular Weight: 156.61
Volume[CD]: 297.39
Dx: 5.247 Dm:

Sys: Rhombohedral
Lattice: Rhomb-centered
S.G.: R
Cell Parameters:
a 5.017 b c 13.643
α β γ

SS/FOM: F27=57(.0125, 38)
I/Icor:
Rad: CuKa
Lambda: 1.54178
Filter:
d-sp:

(Fe0.6 Cr0.4)2 O3
Iron Chromium Oxide
Ref: Keller, L. et al., North Dakota State University, Fargo, North Dakota, USA, ICDD Grant-in-Aid, (1982)



2θ	Int-f	h	k	l	2θ	Int-f	h	k	l	2θ	Int-f	h	k	l
24.284	45	0	1	2	57.741	2	1	2	2	78.255	3	3	0	6
33.408	100	1	0	4	58.108	8	0	1	8	79.222	1	2	2	3
35.802	65	1	1	0	62.796	25	2	1	4	81.032	1	3	1	2
39.650	3	0	0	6	64.199	5	0	2	7	81.297	3	1	2	8
41.122	25	1	1	3	64.303	25	3	0	0	83.677	5	0	2	10
43.751	3	2	0	2	70.144	2	2	0	8	85.413	6	0	0	12
49.763	35	0	2	4	72.610	11	1	0	10	85.413	6	1	3	4
53.950	2	2	0	5	72.909	3	1	1	9	88.749	2	3	1	5
54.449	50	1	1	6	75.739	3	2	1	7	90.074	7			
56.435	1	2	1	1	75.817	6	2	2	0