PIXE – principle and main aspects

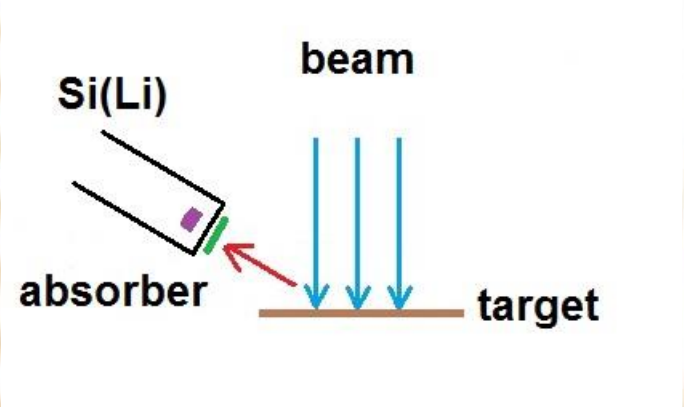
target chamber in Ion Beam Lab

# IBA AT FNSPE CTU

Ion Beams Laboratory, Department of Physical Electronics, Faculty of Nuclear Sciences and Physical Engineering

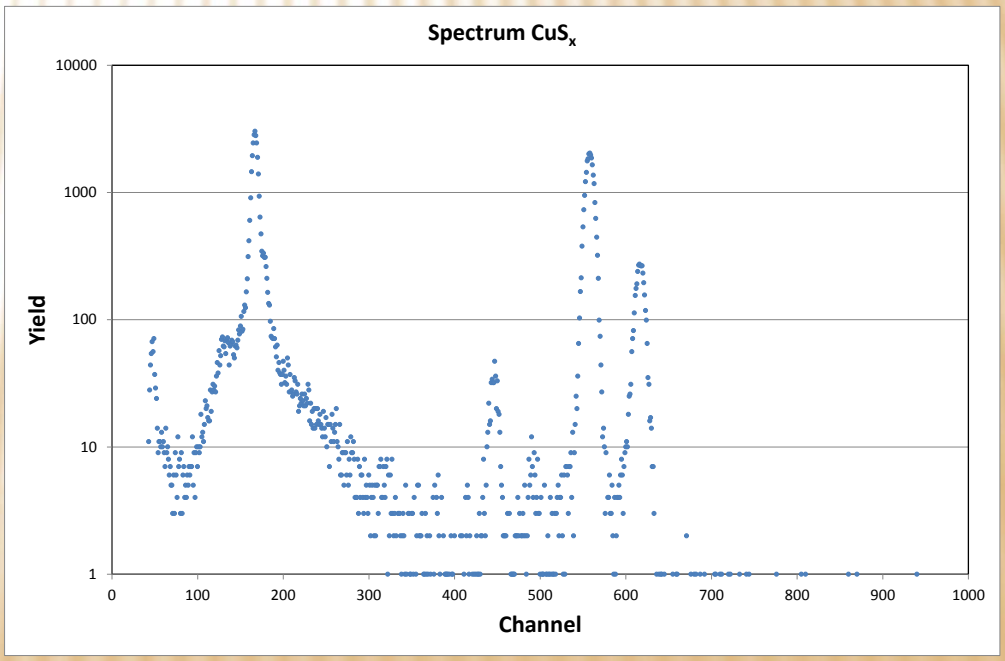
# PIXE - PRINCIPLE

Proton (Particle) Induced X-ray Analysis

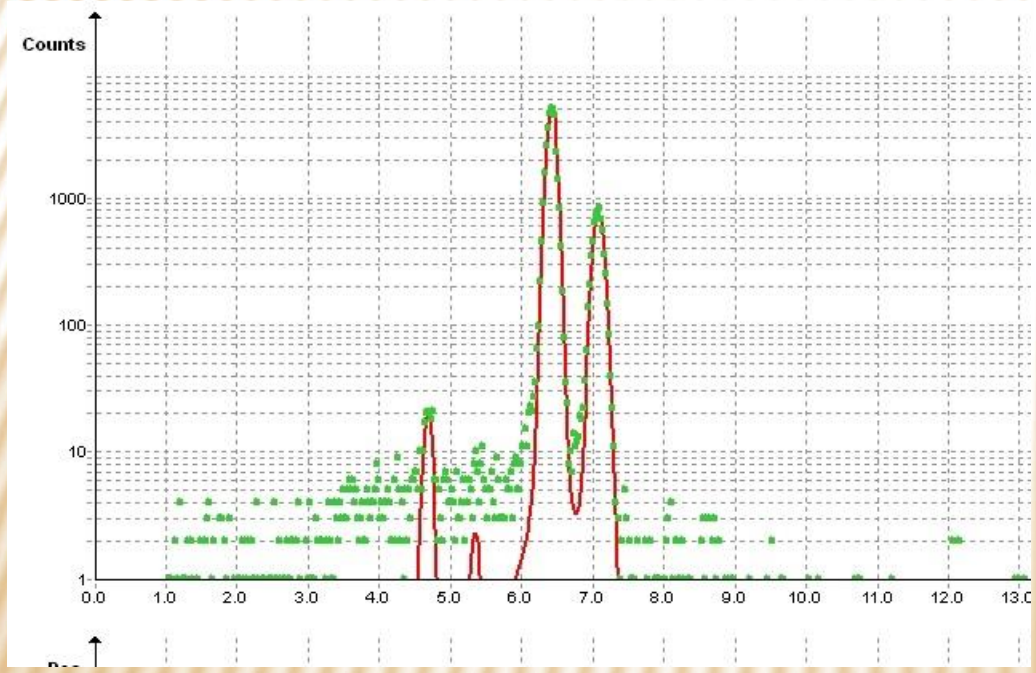



typicaly protons energy of MeV

energies of X-ray photons are registrated by multichannel analyzer



# PIXE - EVALUATION




Energy calibration of the horizontal axis => determination of the elements

Calculation of the peak area

$$c_e \sim \frac{S_e}{Q}$$

$S_e$ – *response (peak area)*
$Q$ – *beam dose*

in principle concentration of the element may be calculated from all known values (angels, cross sections….), in practice calibration by means of standards

# ENERGY OF X-RAYS

| Z | Sym | Kα | Kβ | L1 | Lα | Ln | Lβ | | Lγ | | Mα |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 6 | C | 0,28 | | | | | | | | | |
| 7 | N | 0,39 | | | | | | | | | |
| 8 | O | 0,52 | | | | | | | | | |
| 9 | F | 0,68 | | | | | | | | | |
| 10 | Ne | 0,85 | | | | | | | | | |
| 11 | Na | 1,04 | | | | | | | | | |
| 12 | Mg | 1,25 | | | | | | | | | |
| 13 | Al | 1,49 | 1,56 | | | | | | | | |
| 14 | Si | 1,74 | 1,84 | | | | | | | | |
| 15 | P | 2,01 | 2,14 | | | | | | | | |
| 16 | S | 2,31 | 2,46 | | | | | | | | |
| 17 | Cl | 2,62 | 2,82 | | | | | | | | |
| 18 | Ar | 2,95 | 3,19 | | | | | | | | |
| 19 | K | 3,31 | 3,59 | | | | | | | | |
| 20 | Ca | 3,69 | 4,01 | | | | | | | | |
| 21 | Sc | 4,09 | 4,46 | | | | | | | | |
| 22 | Ti | 4,51 | 4,93 | 0,4 | 0 | 0,4 | 0 | 0,53 | 0 | 0 | |
| 23 | V | 4,95 | 5,43 | 0,45 | 0 | 0,45 | 0 | 0,59 | 0 | 0 | |
| 24 | Cr | 5,41 | 5,95 | 0,5 | 0 | 0,51 | 0 | 0,65 | 0 | 0 | |
| 25 | Mn | 5,9 | 6,49 | 0,56 | 0 | 0,57 | 0 | 0,72 | 0 | 0 | |
| 26 | Fe | 6,4 | 7,06 | 0,62 | 0,7 | 0,63 | 0,72 | 0,79 | 0 | 0 | |
| 27 | Co | 6,93 | 7,65 | 0,68 | 0,78 | 0,69 | 0,79 | 0,87 | 0 | 0 | |
| 28 | Ni | 7,48 | 8,26 | 0,74 | 0,85 | 0,76 | 0,87 | 0,94 | 0 | 0 | |
| 29 | Cu | 8,05 | 8,91 | 0,81 | 0,93 | 0,83 | 0,95 | 1,02 | 0 | 0 | |
| 30 | Zn | 8,64 | 9,57 | 0,88 | 1,01 | 0,91 | 1,03 | 1,11 | 0 | 0 | |
| 31 | Ga | 9,25 | 10,26 | 0,96 | 1,1 | 0,98 | 1,12 | 1,19 | 0 | 0 | |
| 32 | Ge | 9,89 | 10,98 | 1,04 | 1,19 | 1,07 | 1,22 | 1,29 | 0 | 0 | |
| 33 | As | 10,54 | 11,73 | 1,12 | 1,28 | 1,16 | 1,32 | 1,39 | 0 | 0 | |
| 34 | Se | 11,22 | 12,5 | 1,2 | 1,38 | 1,24 | 1,42 | 1,49 | 0 | 0 | |
| 35 | Br | 11,92 | 13,29 | 1,29 | 1,48 | 1,34 | 1,53 | 1,6 | 0 | 0 | |
| 36 | Kr | 12,65 | 14,11 | 1,38 | 1,58 | 1,44 | 1,63 | 1,71 | 0 | 0 | |
| 37 | Rb | 13,4 | 14,96 | 1,48 | 1,69 | 1,54 | 1,75 | 1,83 | 0 | 0 | |

# IONIZATION CROSS SECTION

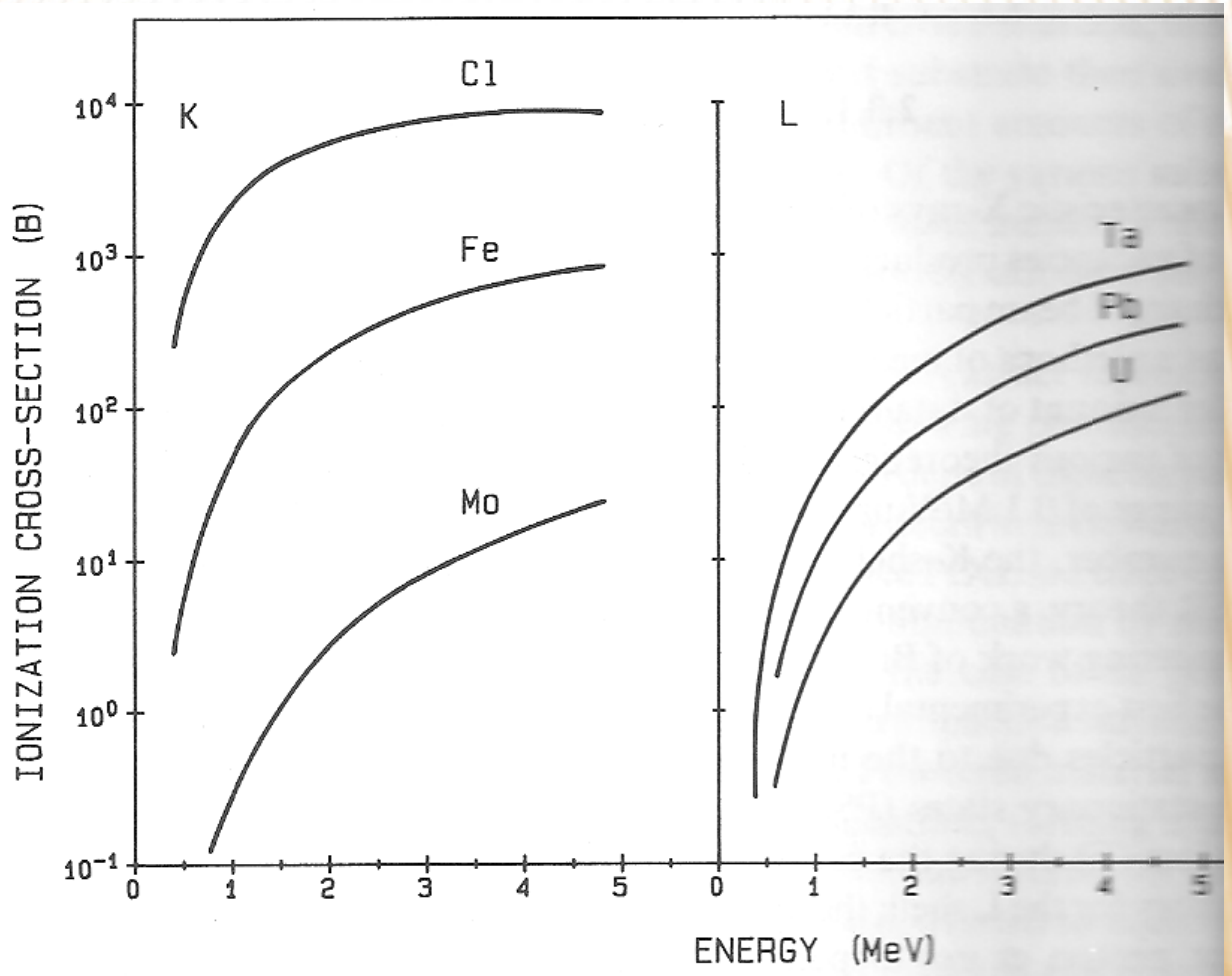



**Figure 2.3.** Representative ionization cross sections for protons. Units are 1 B = 1
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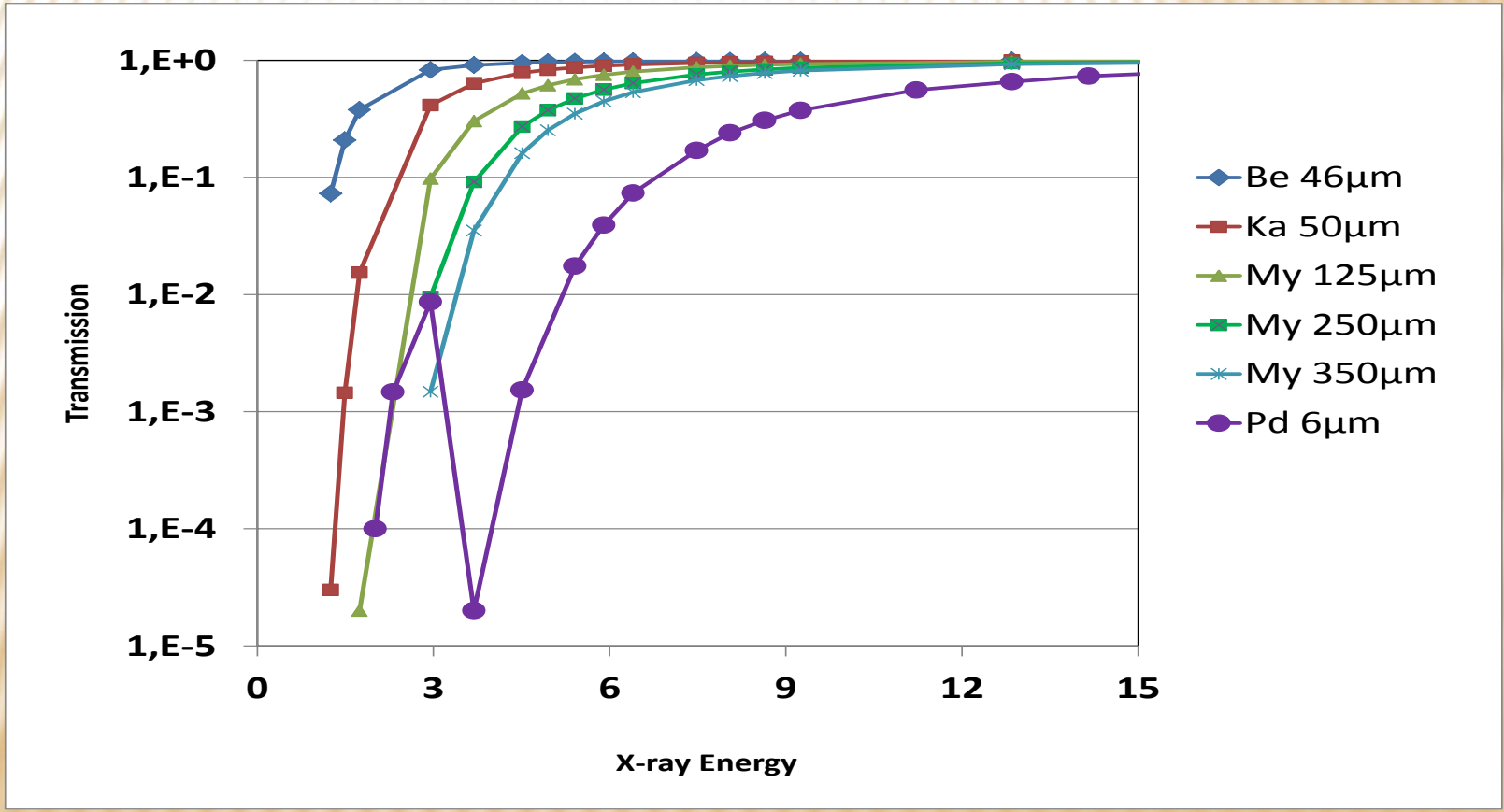
# BE WINDOW + ABSORBER

suppression of the low energy nois or the response from light elements (specific part of the spectra)

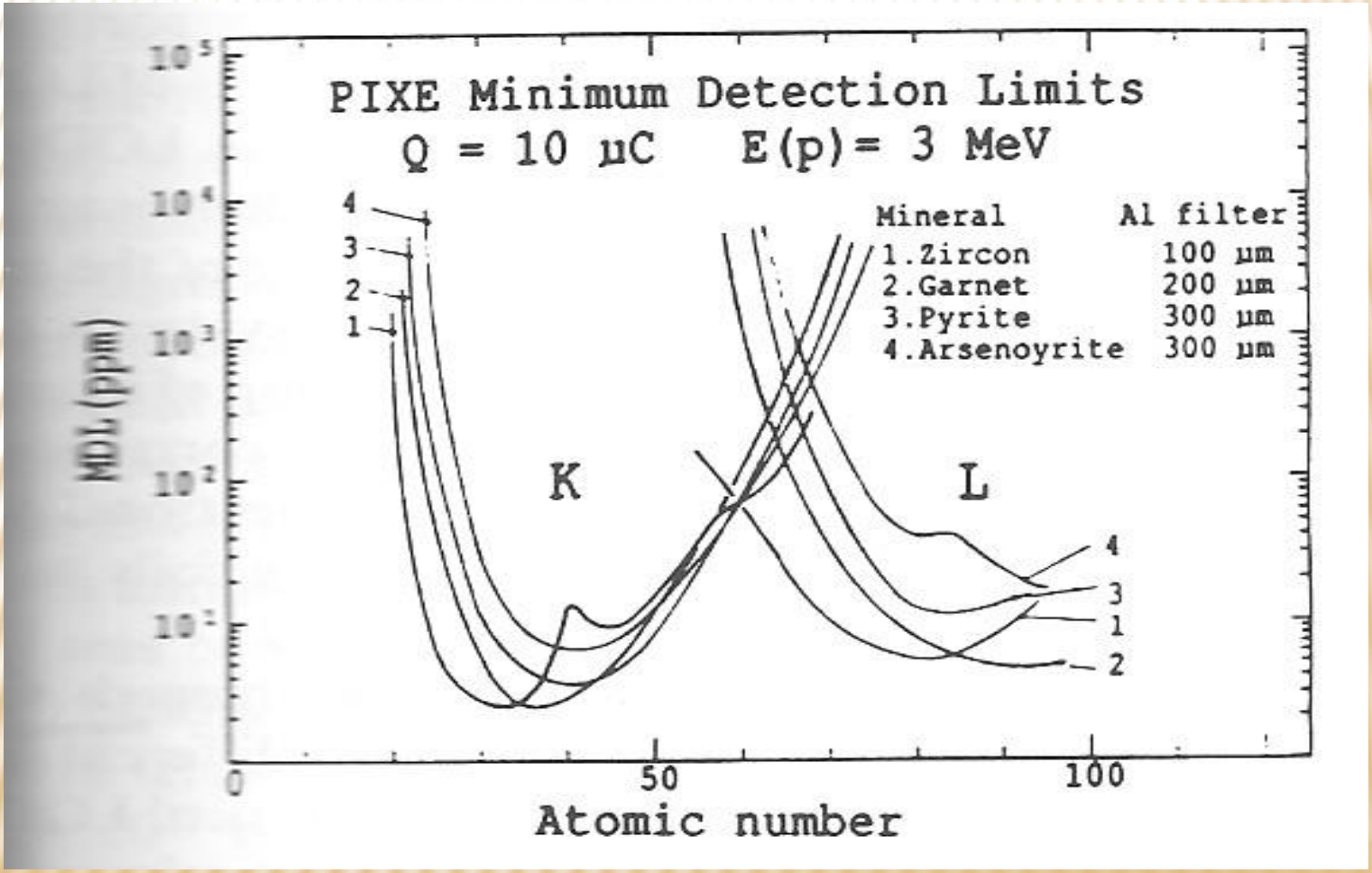


# ABSORBER - FUNNY

“Funny filter” - two layers, one has opening

e.g. Ka50µm + My250µm with opening ø1,5 mm [Si(Li) crystal is 5x5mm]

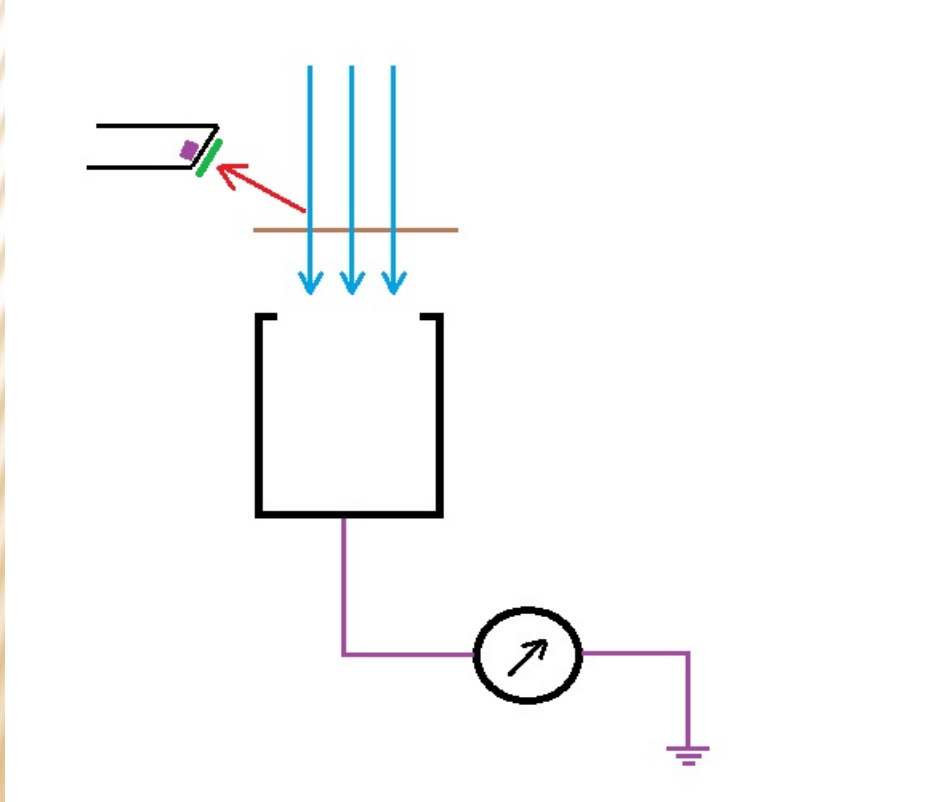
# DETECTION LIMITS




Johansson at all.: PIXE

# PIXE ANALYSIS – THIN SAMPLE

Beam penetrates with negligible loss of energy of protons



$$c_e = H(E_X).\frac{S_e}{Q}$$

*$S_e$ – response (peak area)*
*Q – beam dose*

*H(E) - determined by standard samples*

# PIXE THICK SAMPLE

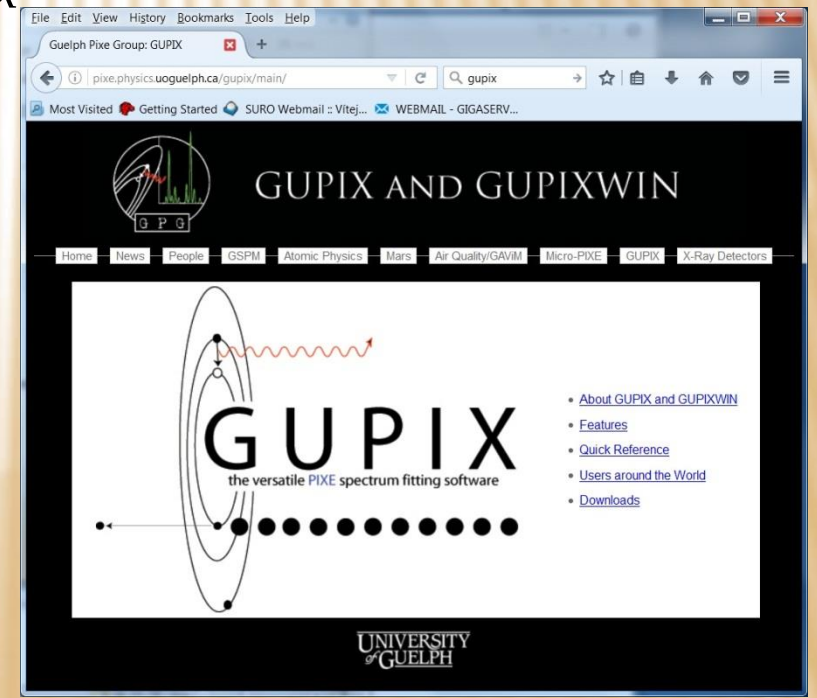
Interactions are taking place in some depth

*Reduced proton energy*

*Attenuation of X-ray photons*

*Special cases – trace elements in known matrix*

Computer codes, e.g. Gupix



# ION BEAM LABORATORY

# DEPARTMENT OF PHYSICAL ELECTRONICS

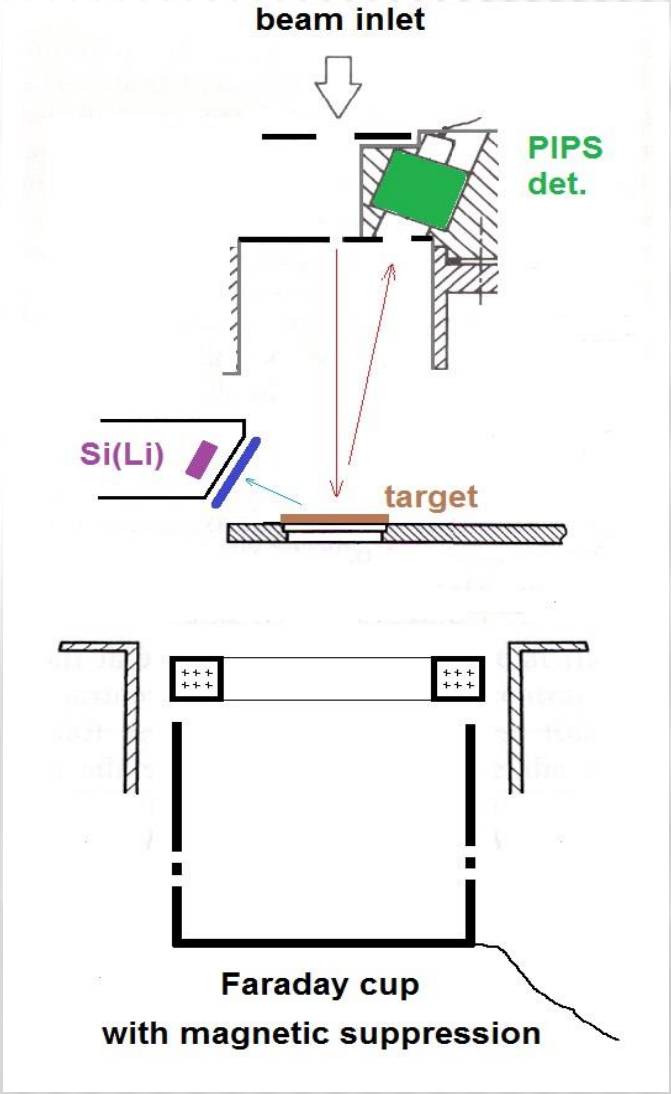
# FACULTY OF NUCLEAR SCIENCES AND PHYSICAL ENGINEERIG

# CTU IN PRAGUE

Vertical beam from Van de Graaff accelerator: http://www.utef.cvut.cz/about-vdg-lab

# BASIC ARRANGEMENT FOR PIXE/RBS

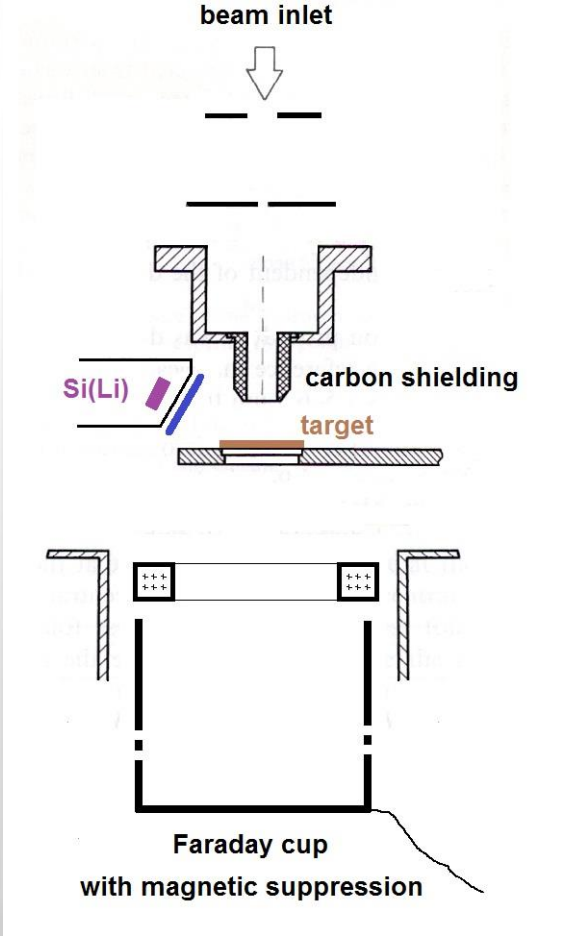
**protons or $He^+$ 0,7 – 2,2 MeV**
**beam max. Ø 6 (8) mm**




in case of the thin sample (beam penetrates through the sample) beam dose is measured directly

# PIXE ANALYSIS, THIN SAMPLE

**protons 0,7 – 2,2 MeV**
**beam max. Ø 6 (8) mm**

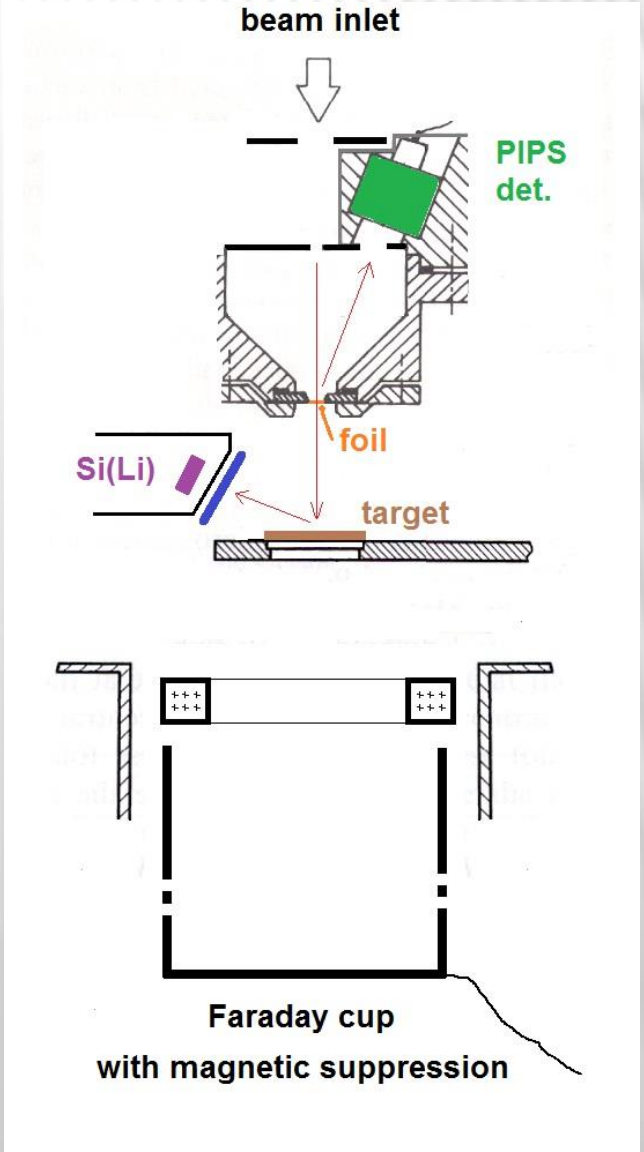



energy loss in the target is negligible, beam dose is measured by Faraday cup, evaluation is simple

# PIXE ANALYSIS, THICK SAMPLE

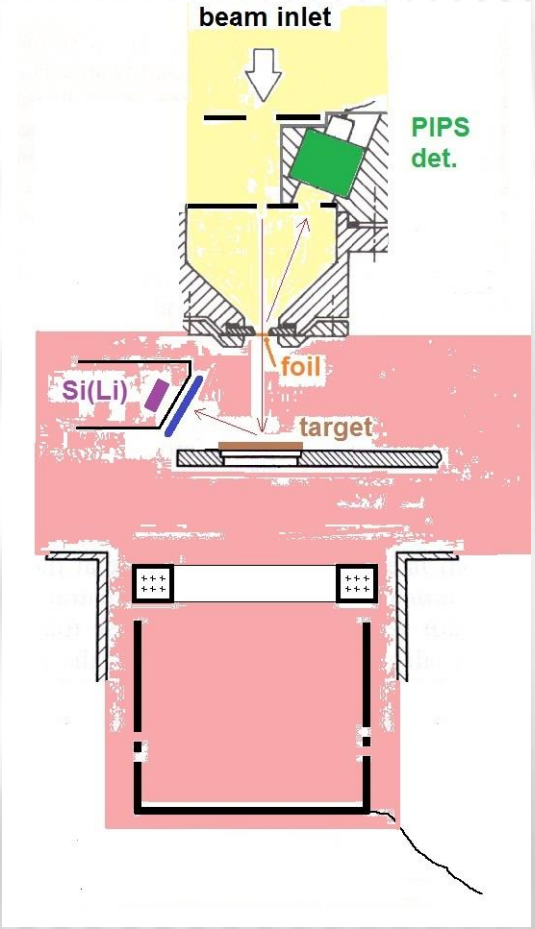
**protons 0,7 – 2,2 MeV**
**beam max. Ø 6 mm**

1. without target - calibration of the RBS spectra vs. beam charge
2. PIXE analysis simultaneously with RBS spectra from the foil
3. recalculation of the beam charge



# PIXE ANALYSIS NON-CONDUCTIVE SAMPLES

**protons 0,7 – 2,2 MeV**
**beam max. Ø 4 mm**




3 – 10 Pa $N_2$

# CALIBRATION

Thin XRF standards MicroMatter$^{TM}$ – one or two elements, typically 50μg/cm$^2$ ±5%

NIST – multielemental

# LIMITATIONS

Pulse rate – max. ~ 1000 fotons/s, in other case pile-up occures => sometimes long time for acquisition of the spectrum

Overlapping of the spectral lines

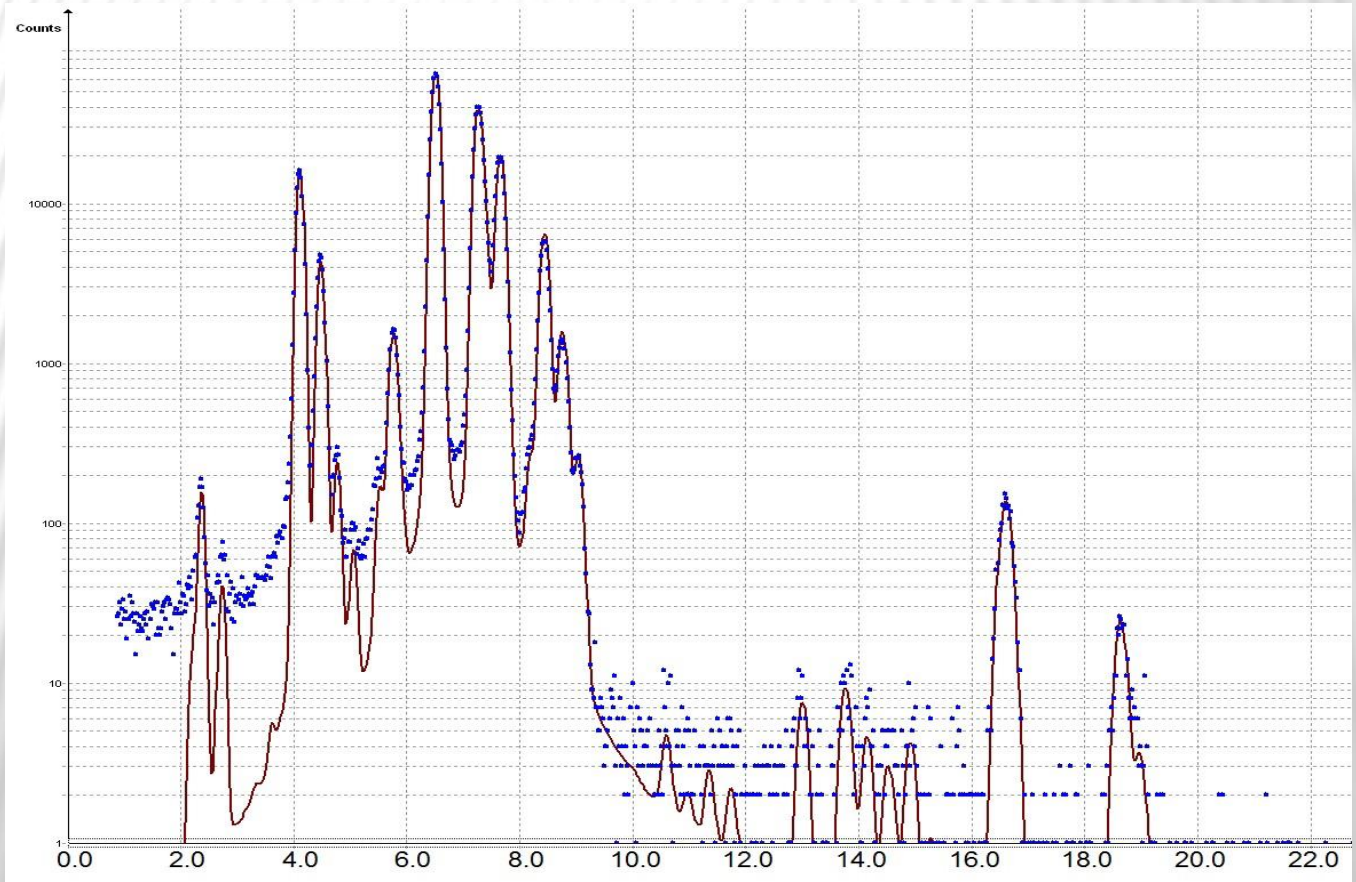
Charging of the non-conducting targets

Thermal or radiational load

colouring

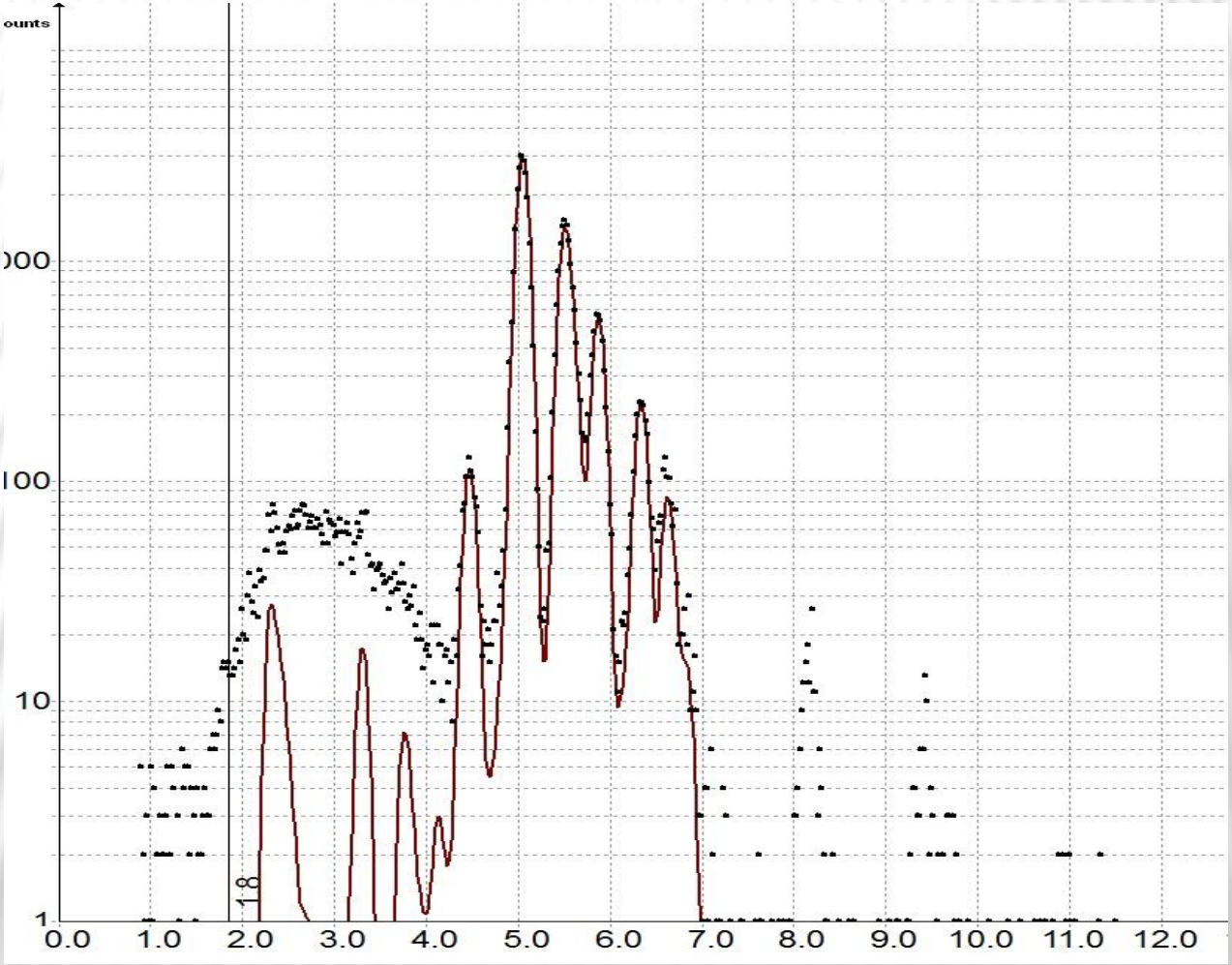
# EXAMPLE

Layer $NaNbO_3$ with 1% Pr, approximately 100nm thick on $DyScO_3$
- very difficult



# EXAMPLE

**Pr only**



# EXAMPLE

Listing from Gupix

```
File:sc018.pix   Sec: 2026.  uC:  0.391  nA:  0.193  PUcor:1.0007
 The last column is a decision on the presence of that element in the spectrum.
   Y: present at level of quantization,   N: not present at limit of detection
   ?: may be present near LOD levels (user must decide)  H or uC Corr[F]: 1.000
 Det Res(eV): 141.8                                    Chi**2:    2.743 (    8.678)
       Layer          H   Yield Det. Filter
 Element     Area  value  /uC/ Eff. Trans.     Conc.  %Stat.  %Fit      LOD
  Z Sym  #  counts ( -4)   ppm (-3)  (-5)       ppm    Error  Error     ppm
  - ---  -  ------ ----- ----- ---- ------     -----   -----  -----    -----  -
 41 NbK  1  1093.5   340  2.74  960  97500   32072.5    2.82   3.16    511.1  Y
 41 NbLB *    70.3   130 310.3  842      3 1540488.9   33.12  28.55 999999.0  ?
 59 PrLA 1   367.5   195 92.62  975  39201    1360.2   10.54   9.72    222.8  Y
 21 ScK  2 67967.1    84 435.2  958  17277  287126.3    0.32   0.46    204.9  Y
 66 DyLA 2  439397   267 134.5  985  65035  488064.1    0.15   0.19    129.8  Y
(A "*" in the Layer# column indicates that elmt NOT used in matrix iteration)
--------------------------------------------------------------------------------
```

# REFERENCES

Johansson, Campbell, Malmqvist: Particle Induced X-Ray Emission Spectrometry (PIXE), John Wiley & sons, 1995