



# **SAXS COURSE**

**2003**

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**and**

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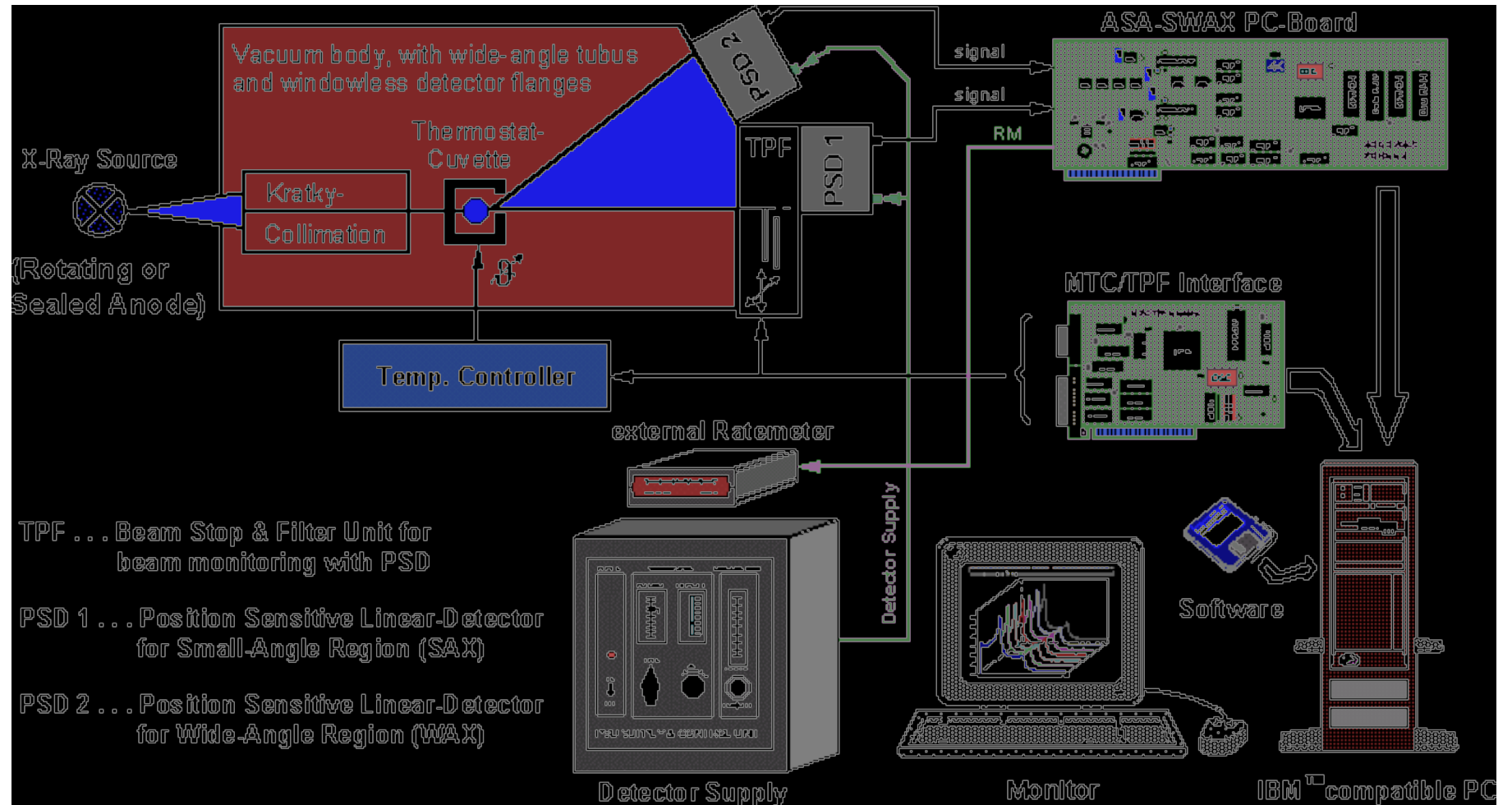
**Graz, Austria**



# The SWAXS Instrument



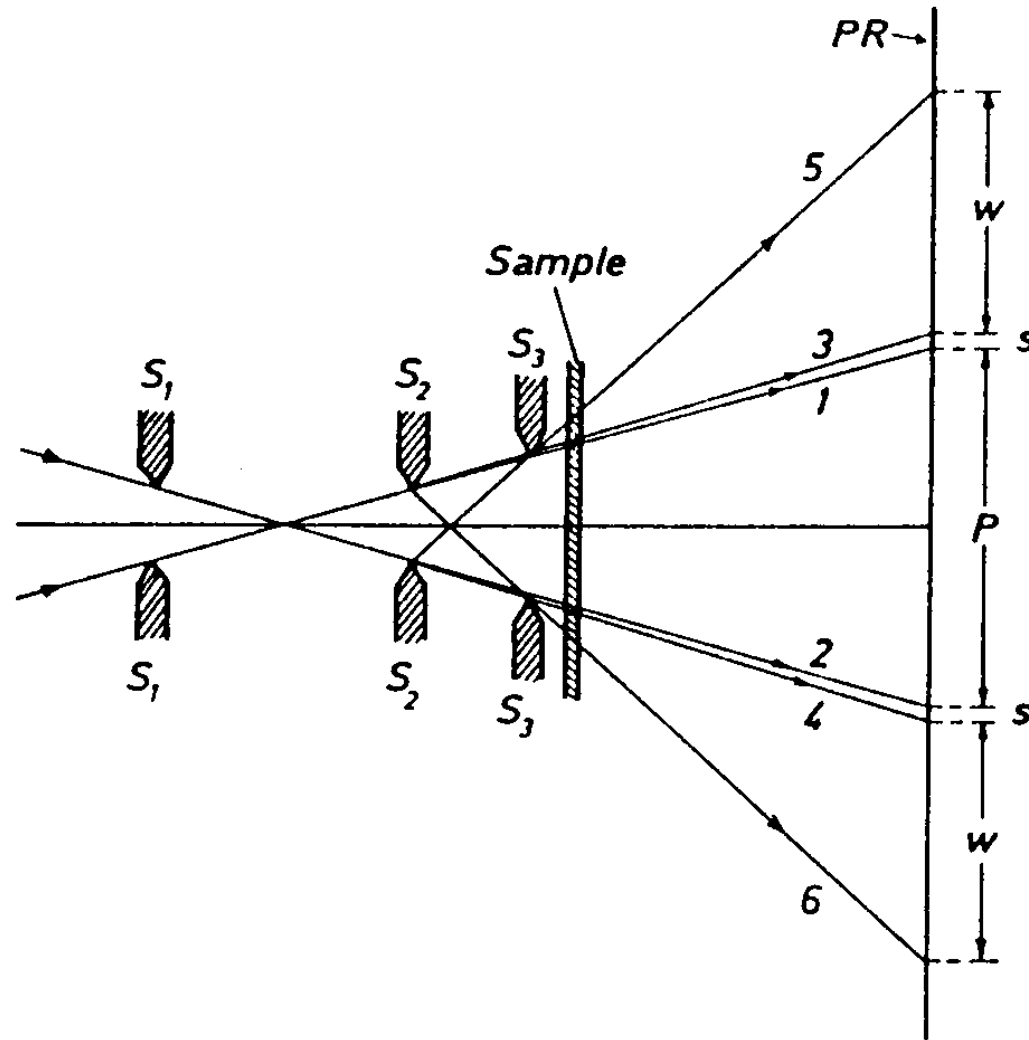
# Scheme of the HECUS-MBraun SWAXS- System



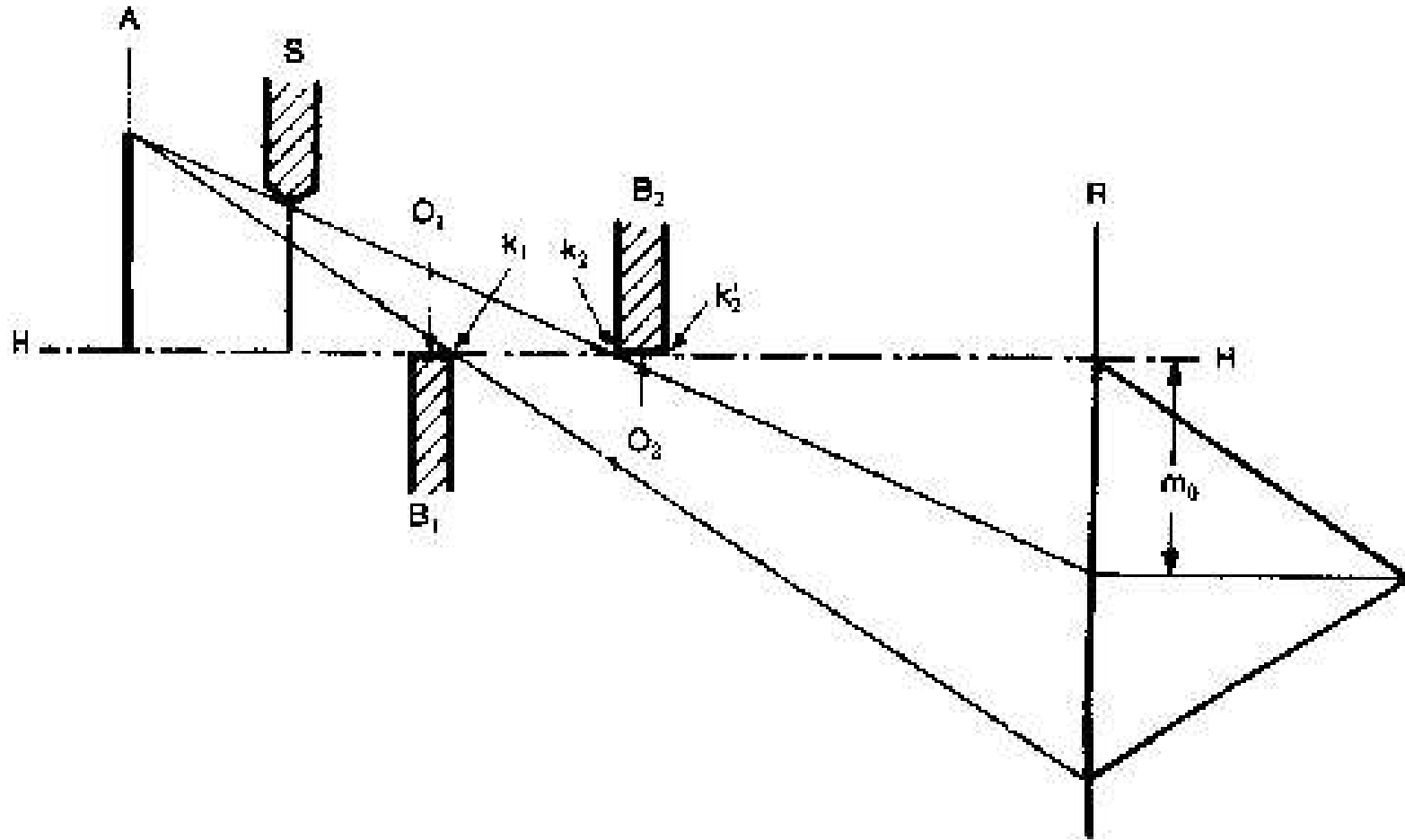
# X-ray Source

- **Sealed tube or rotating anode with copper target**
- **Line shaped focus**
- **Monochromatisation by pulse height discriminator  
combined with Ni-filter**

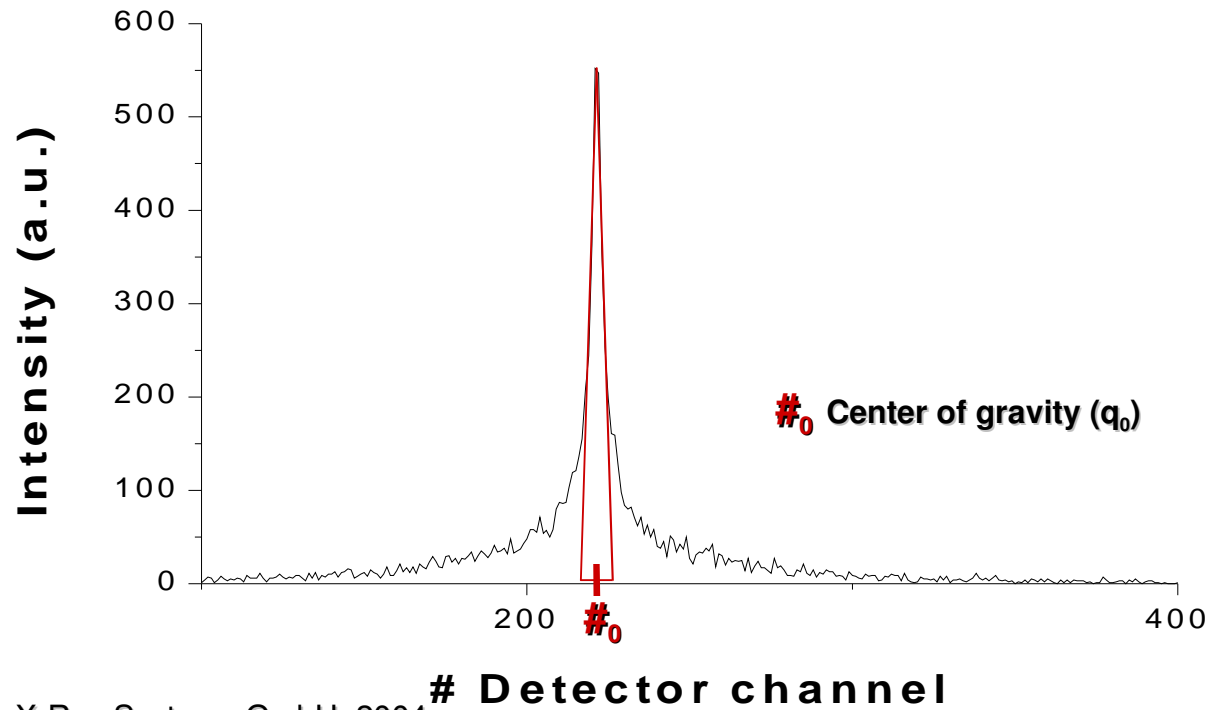
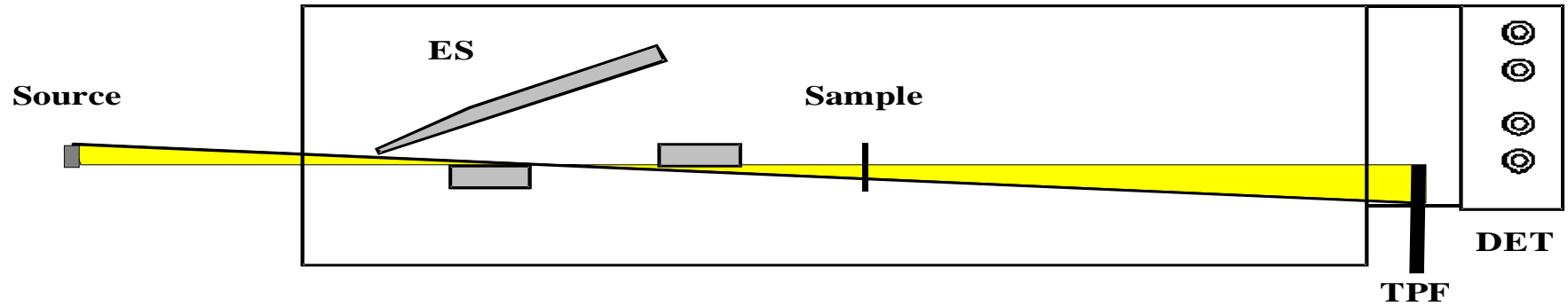
# Slit Collimation



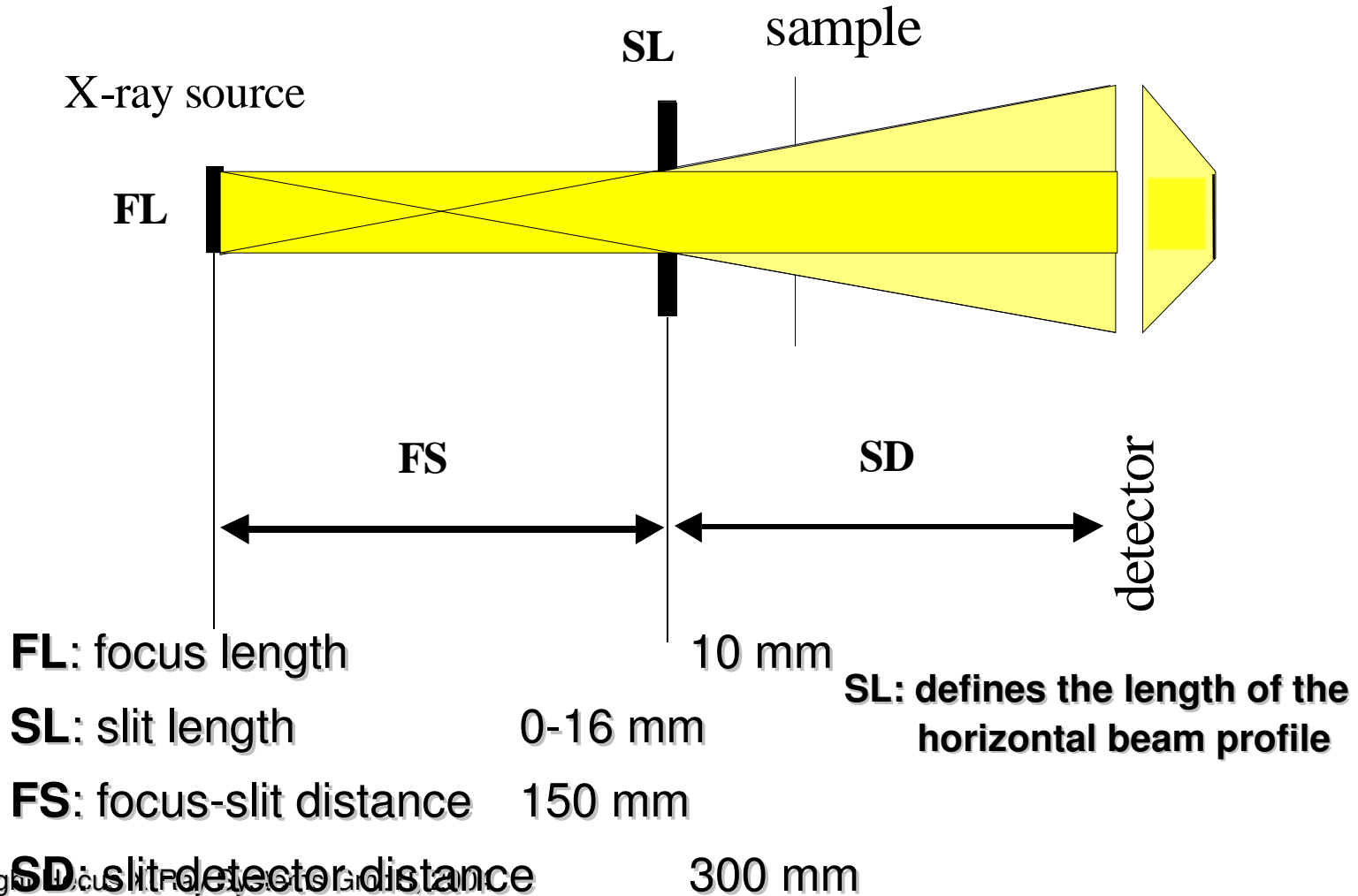
# Kratky-Type Collimation System



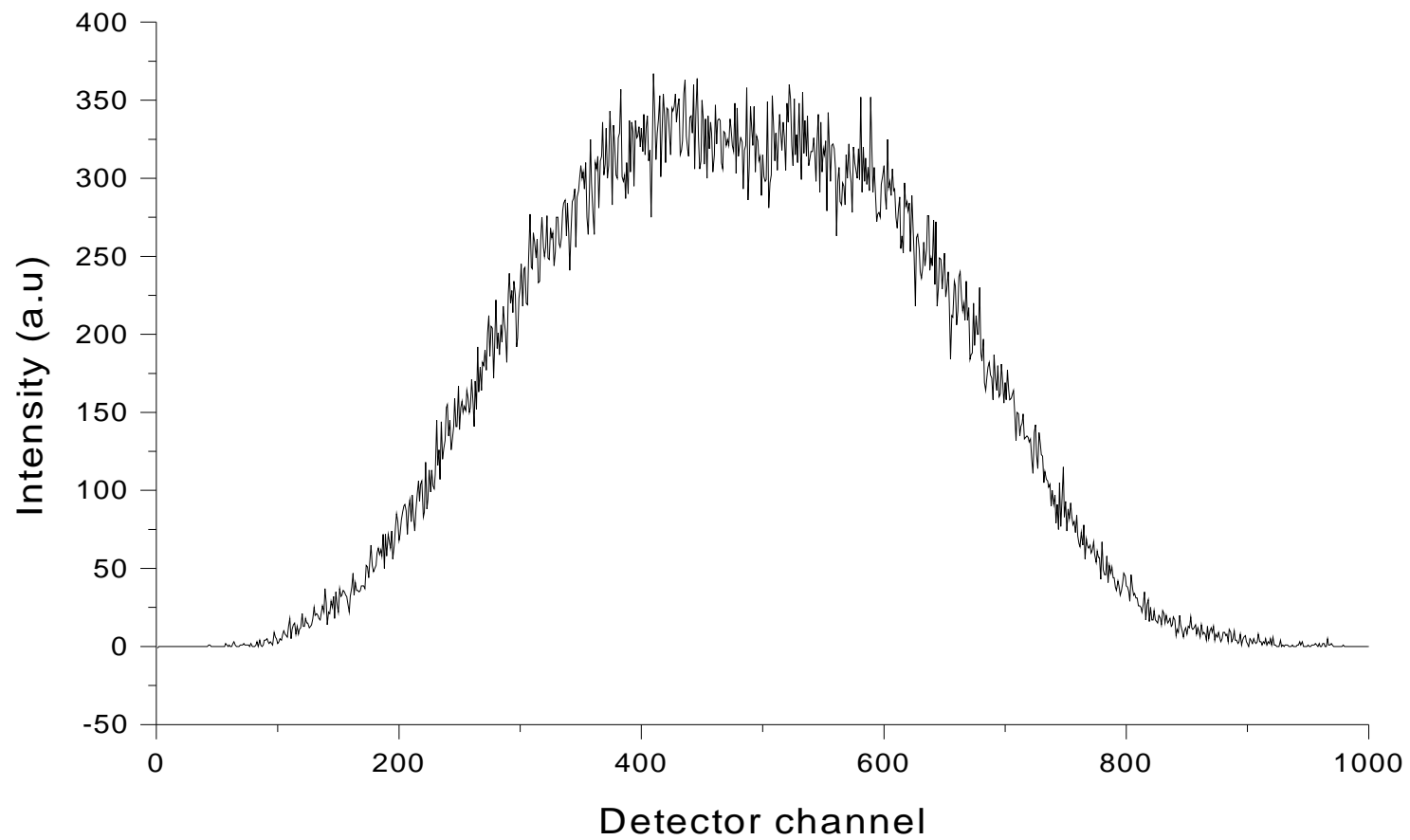
# Vertical Beam Profile



# Horizontal Beam Profile







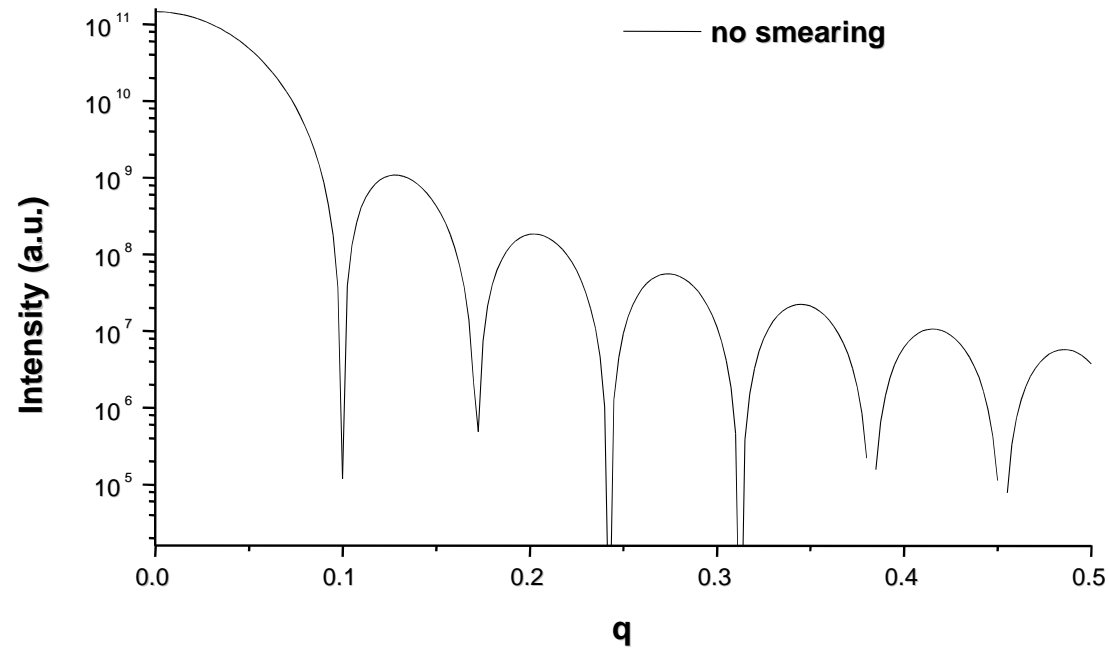
# **Advantages of Line-Collimation for SAXS**

- **Higher Intensity**
- **Higher Resolution**
- **Shorter Measuring Times**
- **Inexpensive X-ray Generators**
- **Easy Alignment**

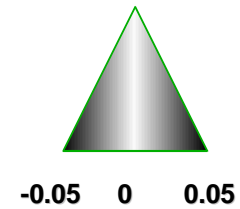
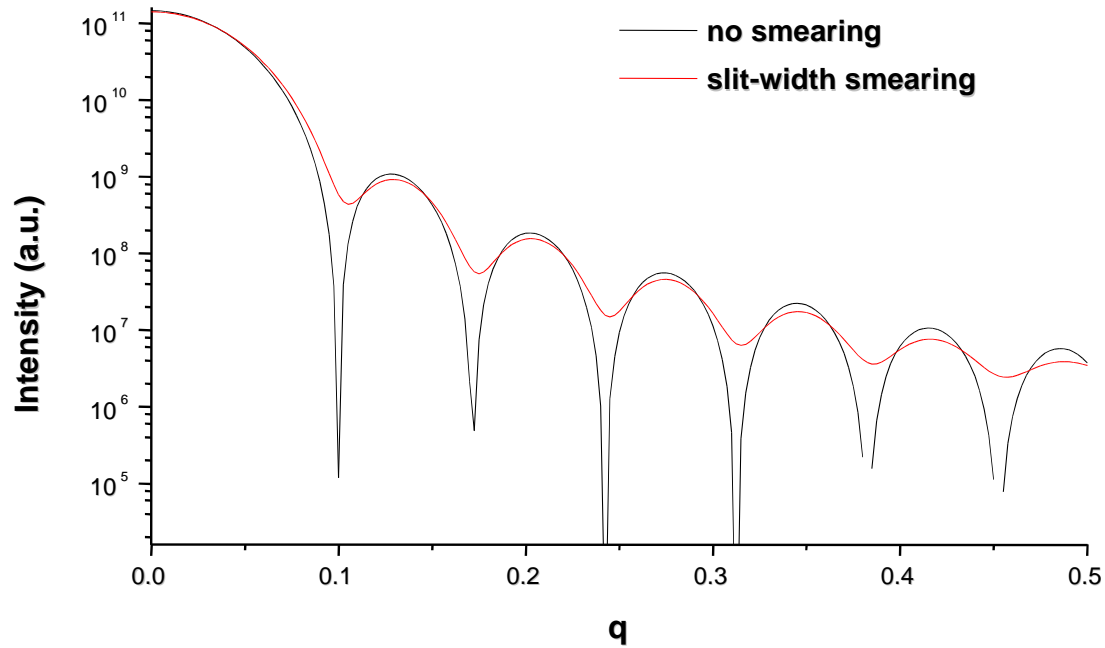
# Disadvantages of Line-Collimation

- **'Smearing' by slit-width and slit-length profiles**

# 'Smearing' by slit-width and slit-length profiles

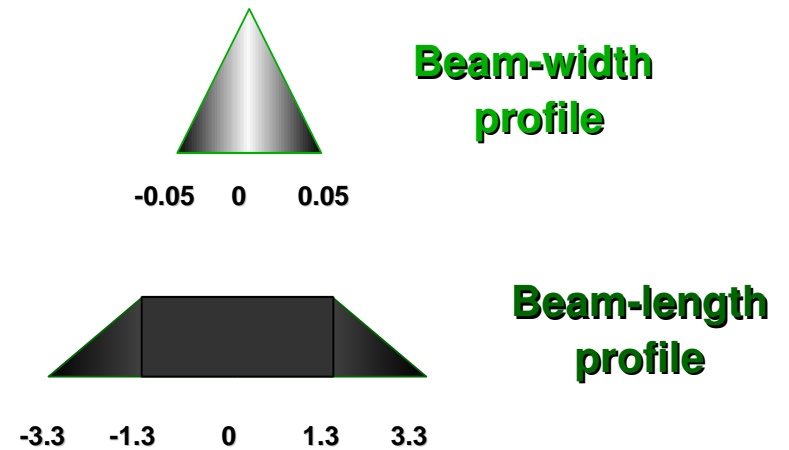
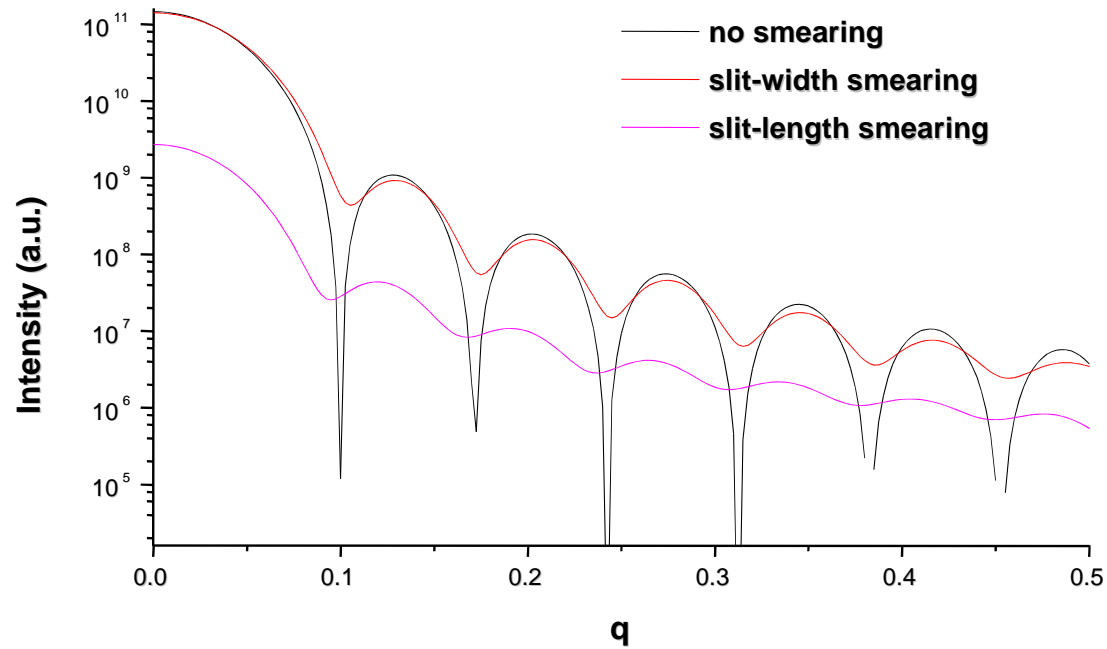


# 'Smearing' by slit-width and slit-length profiles

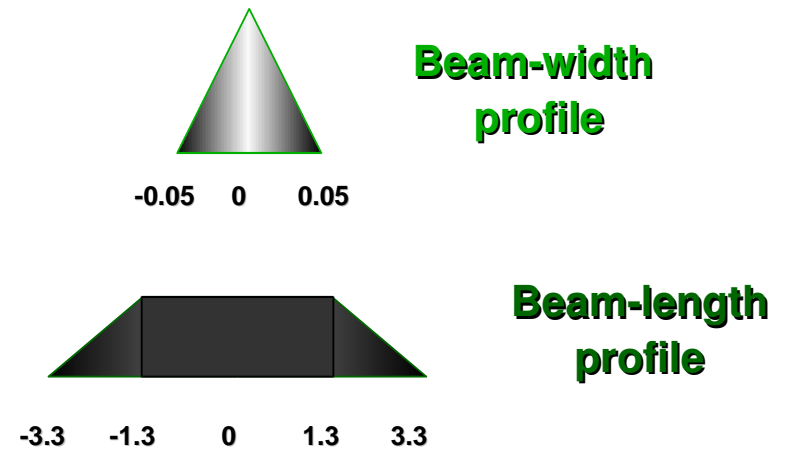
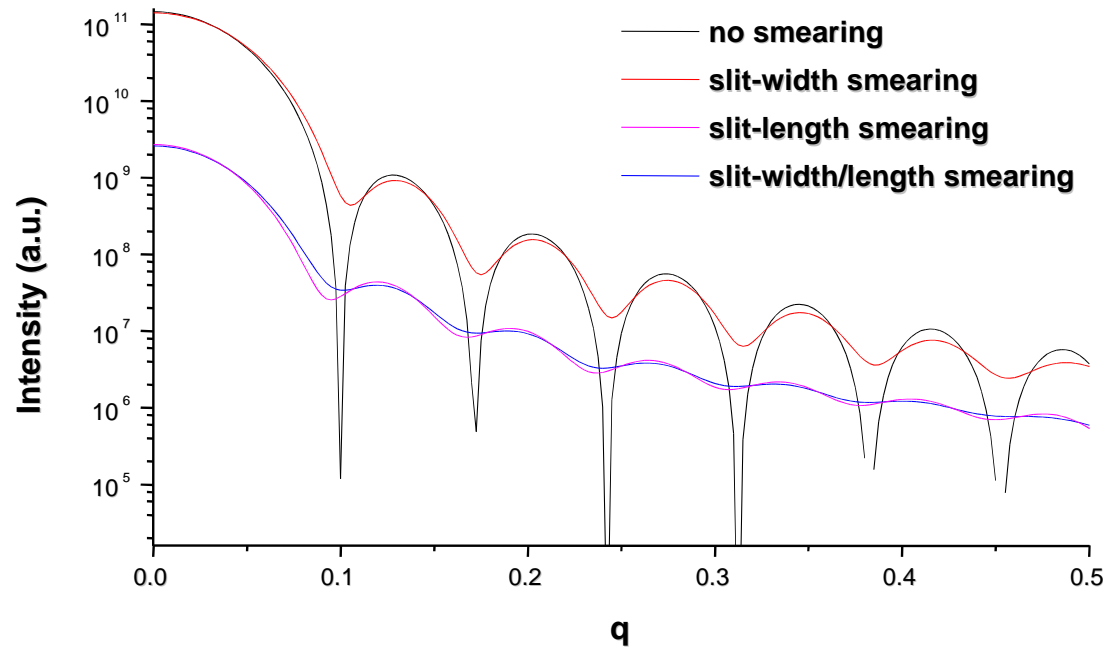


**Beam-width  
profile**

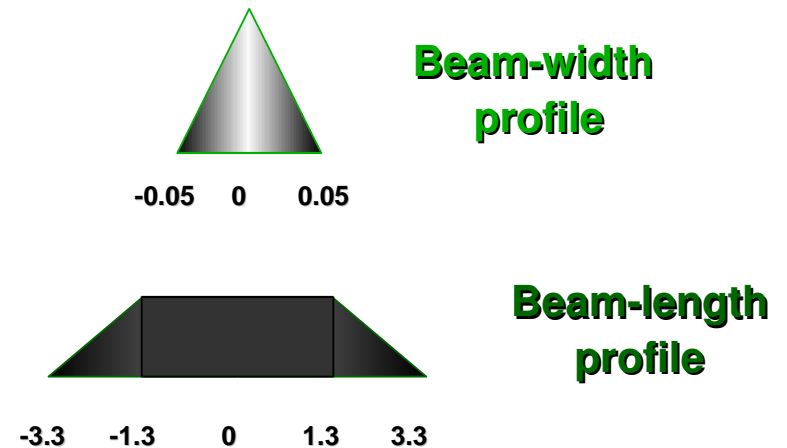
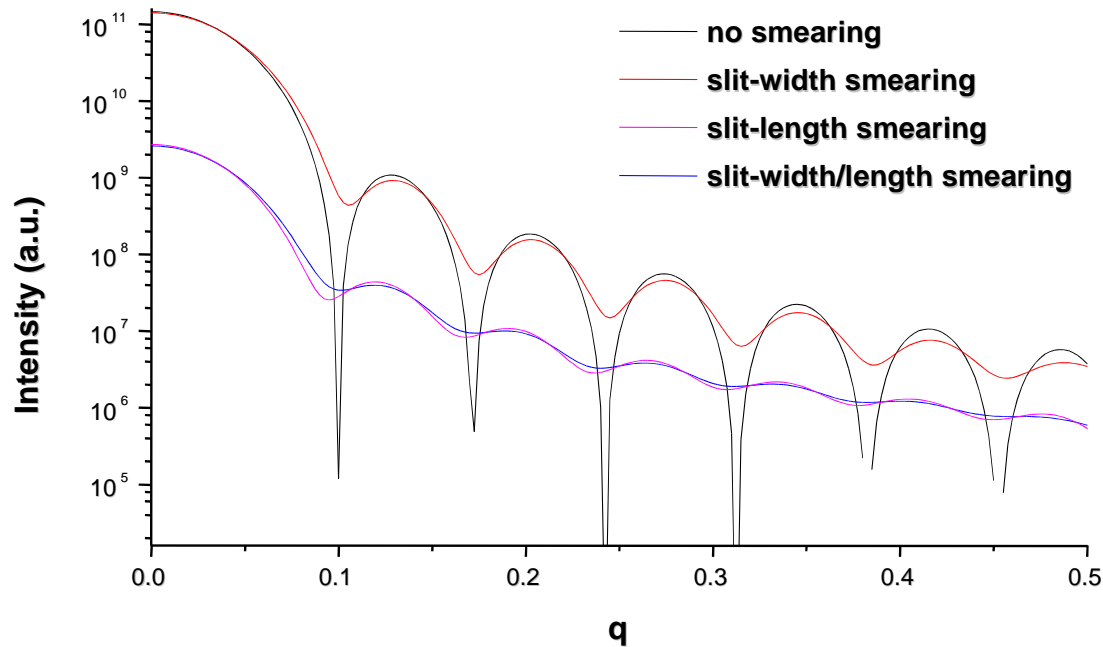
# 'Smearing' by slit-width and slit-length profiles



# 'Smearing' by slit-width and slit-length profiles



# 'Smearing' by slit-width and slit-length profiles



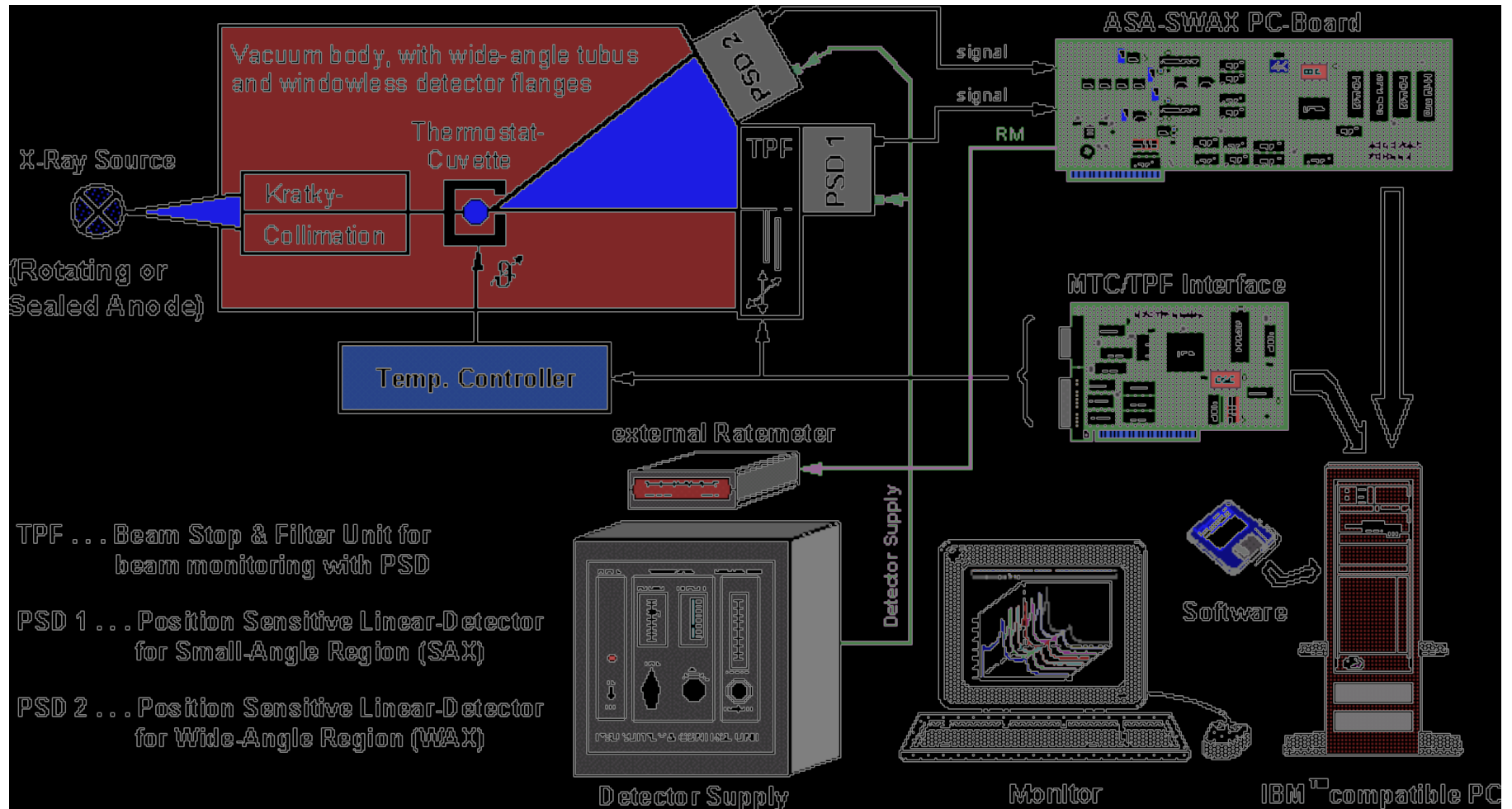
Further smearing effects by wavelength distribution and polydispersity.



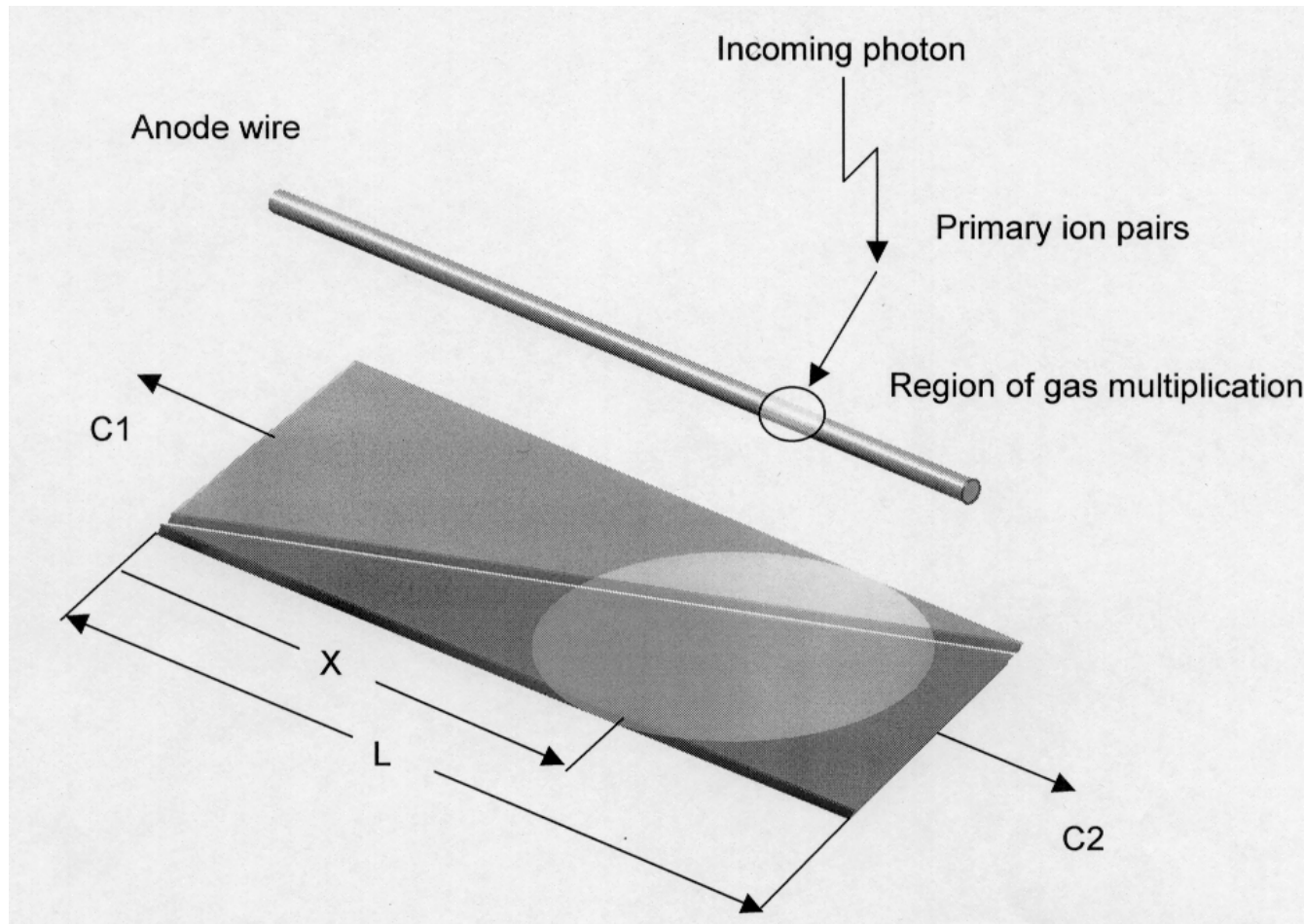
# Flexible Sample Environment

- **Temperature controlled sample holder KPR (0-70° C)**
- **Temperature controlled sample holder KHR (25-300° C)**
- **Quartz capillary sample cuvette for SAXS or SWAXS**
- **Rotating capillary**
- **Flow-through cell** (manual or with Sampler)
- **Sample holder for pastes and powders**
- **Time-temperature-programmer MTC**
- **Relative humidity cell** (in development)

# Position Sensitive Detector PSD



# Principle of Operation



## Operating parameters:

High voltage	3.6-3.8 KV
Gas pressure	7.5-8 bar
Fill gas	9/1 Ar/CH4

## Position:

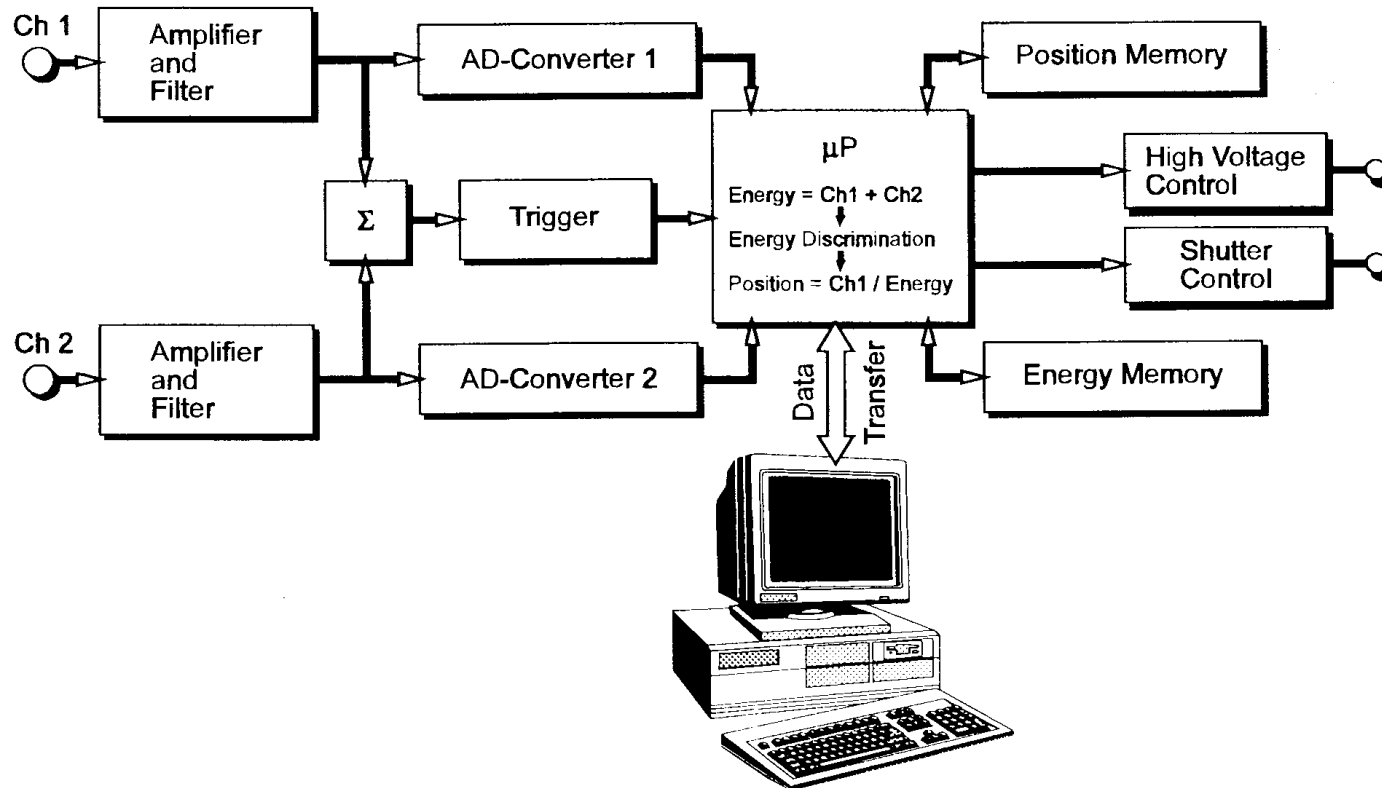
$$X = L * C2 / (C1 + C2)$$

## Energie:

$$E = C1 + C2$$

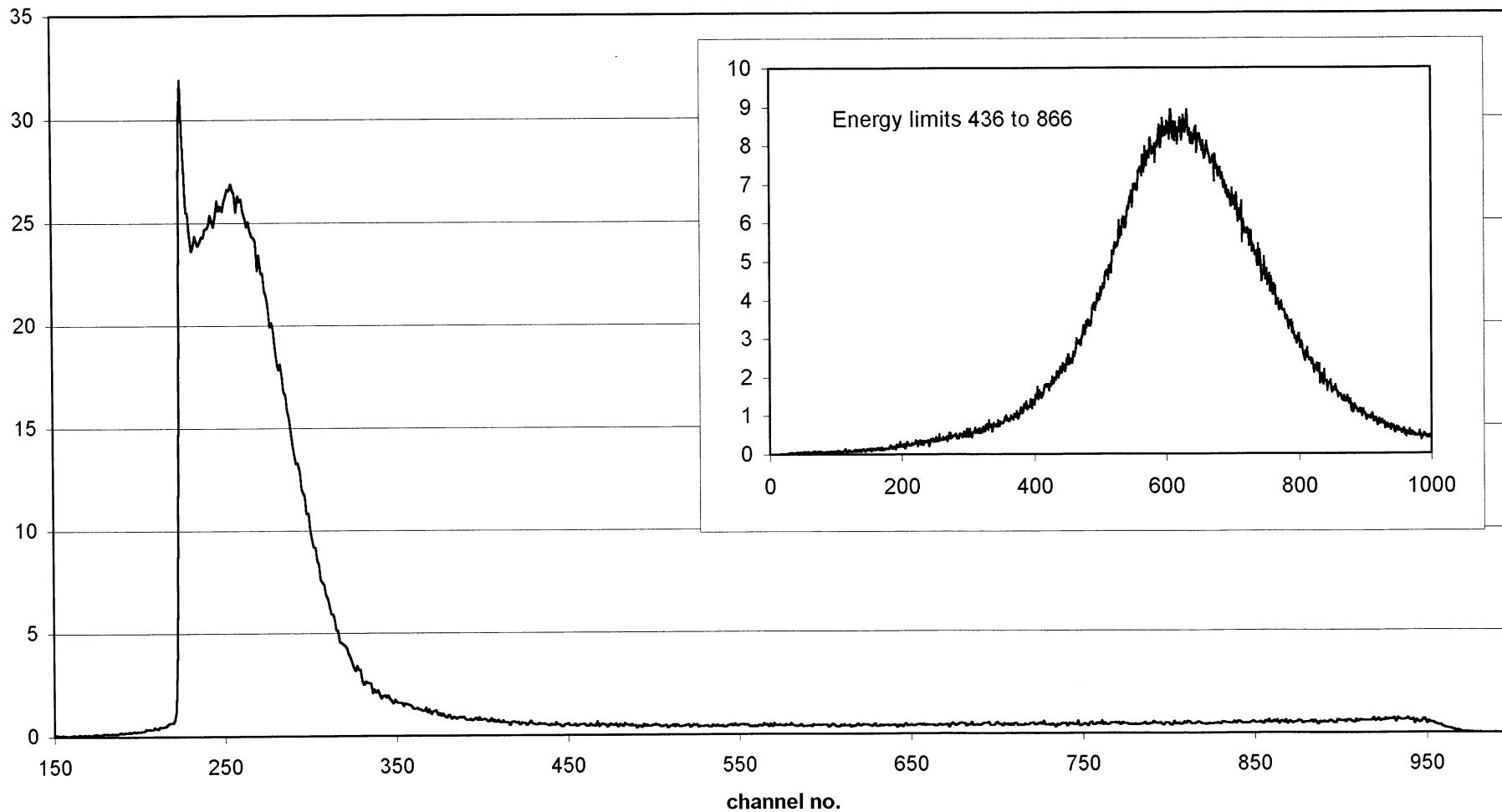
C1, C2 integrated cathode signals  
L active length of the detector  
X position of incoming photon

# Amplitude Spectra Analyser (ASA)



# Lupolen

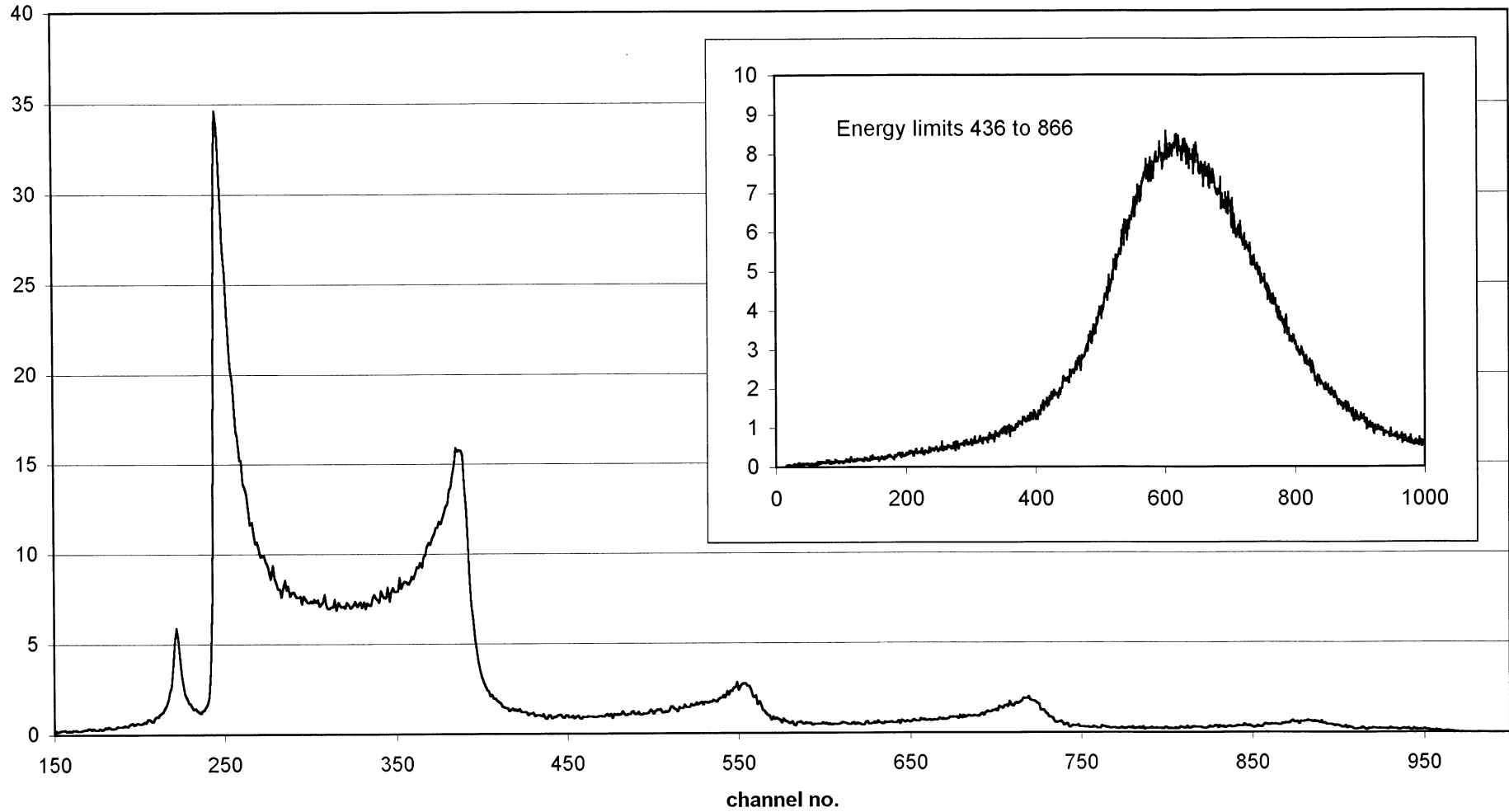
counts / s



Total rate: 2500 cps

# Silverstearate

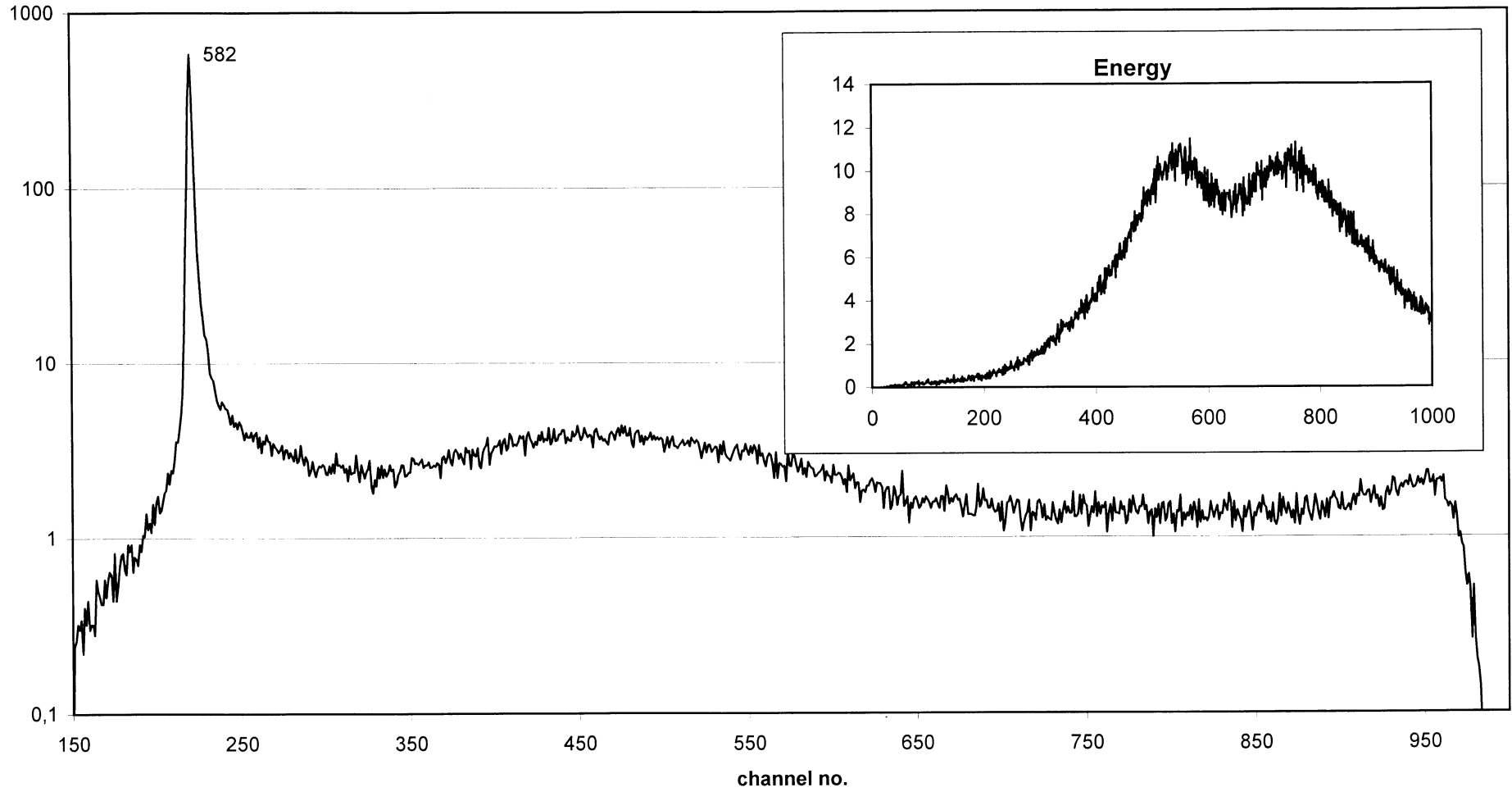
counts / s



Total rate: 2500 cps

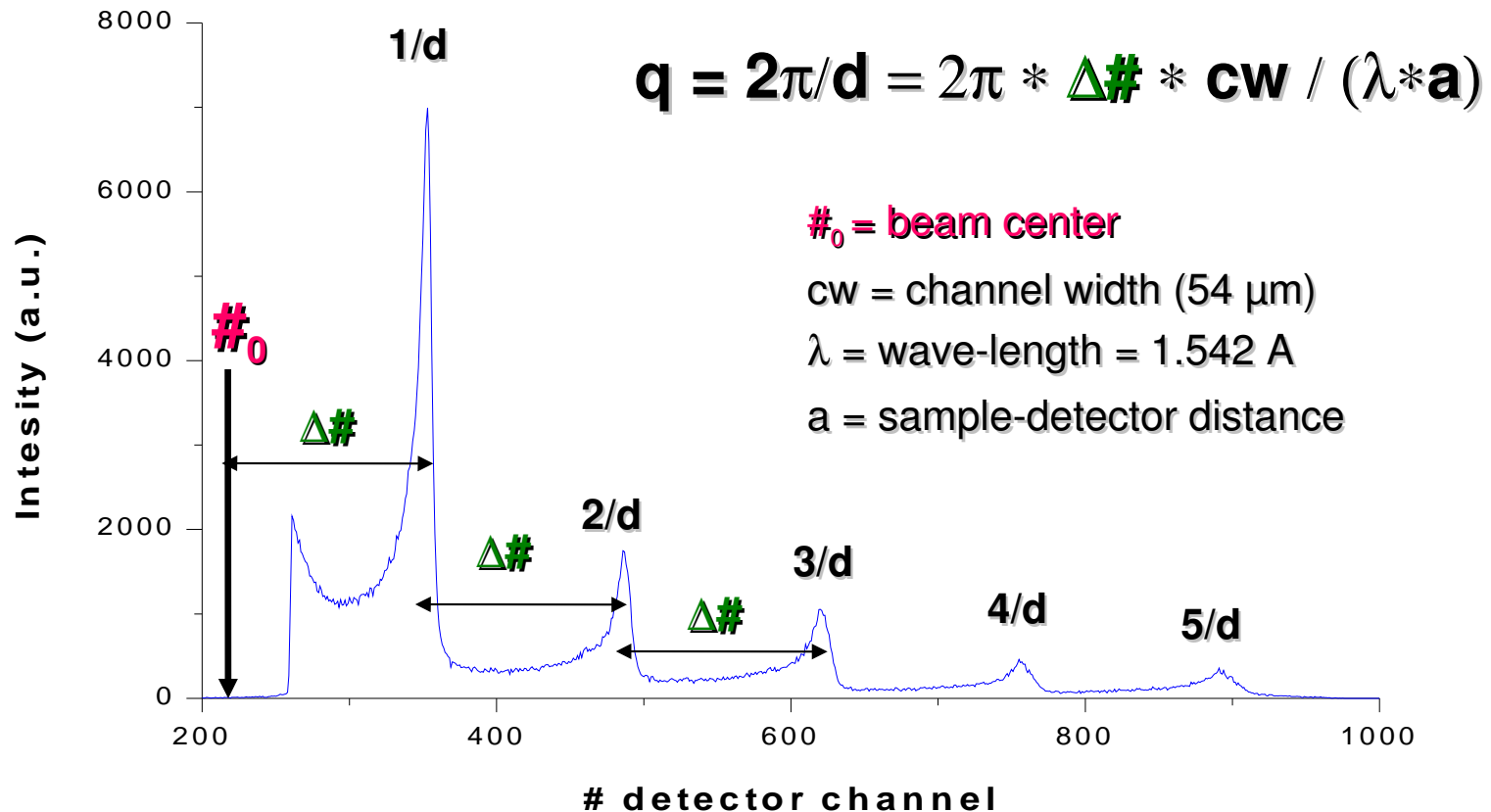
# 'burn in' effect on the wire

counts / s



Total rate: 5200 cps

# Calibration of the q-scale (SAXS) with Ag-behenate powder: Lamellar d-spacing: $d = 58.38 \text{ \AA}$

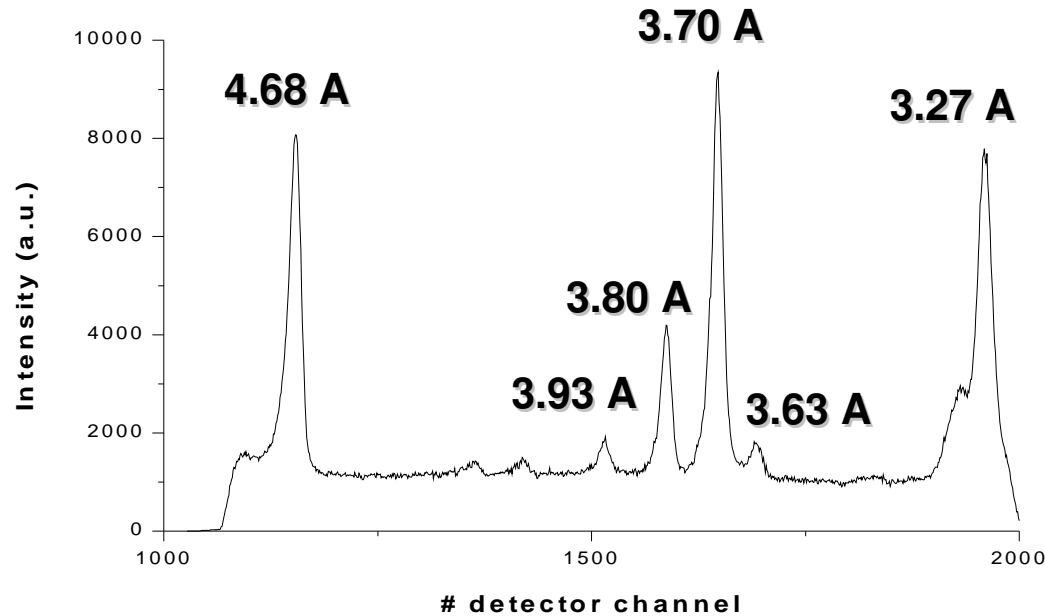




# **Influences on the Gas Multiplication Factor**

- **Gas pressure**
- **Impurities within the gas**
- **Count rate**
- **Aging effects**
- **High voltage**

# Calibration of the q-scale (WAXS) with p-Br-BA powder:



#-1024	d (A)	1/d
124	4.68	0.2137
490	3.93	0.2545
557	3.80	0.2630
618	3.70	0.2700
657	3.63	0.2755
927	3.27	0.3058

$$1/d = 2\pi/q = a + b \cdot \#$$

$$d = 1 / (a + b \cdot \#)$$

## Main features:

- superior intensity and resolution ( $D_{\max}$ : 150 nm )
- real-time detector readout – no delays in TR-SWAX
- minimal noise level (vacuum throughout beam-path)
- on-line beam monitoring (semitransparent beamstop)
- PC - automatization
- high-throughput capability
- versatile sample environment : flow-through, rotation, powder and paste cells, all *in-vacuo* and thermostated