HECUS
X-RAY SYSTEMS GMBH GRAZ

SAXS COURSE

2003

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and

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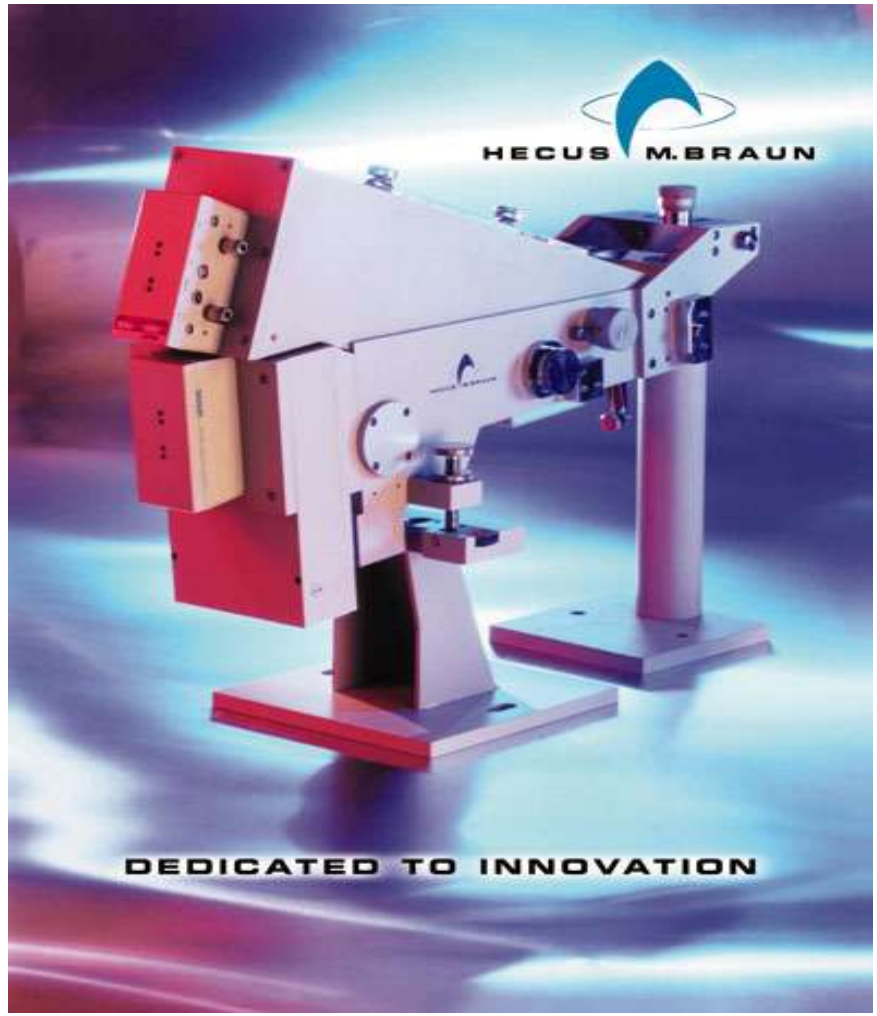
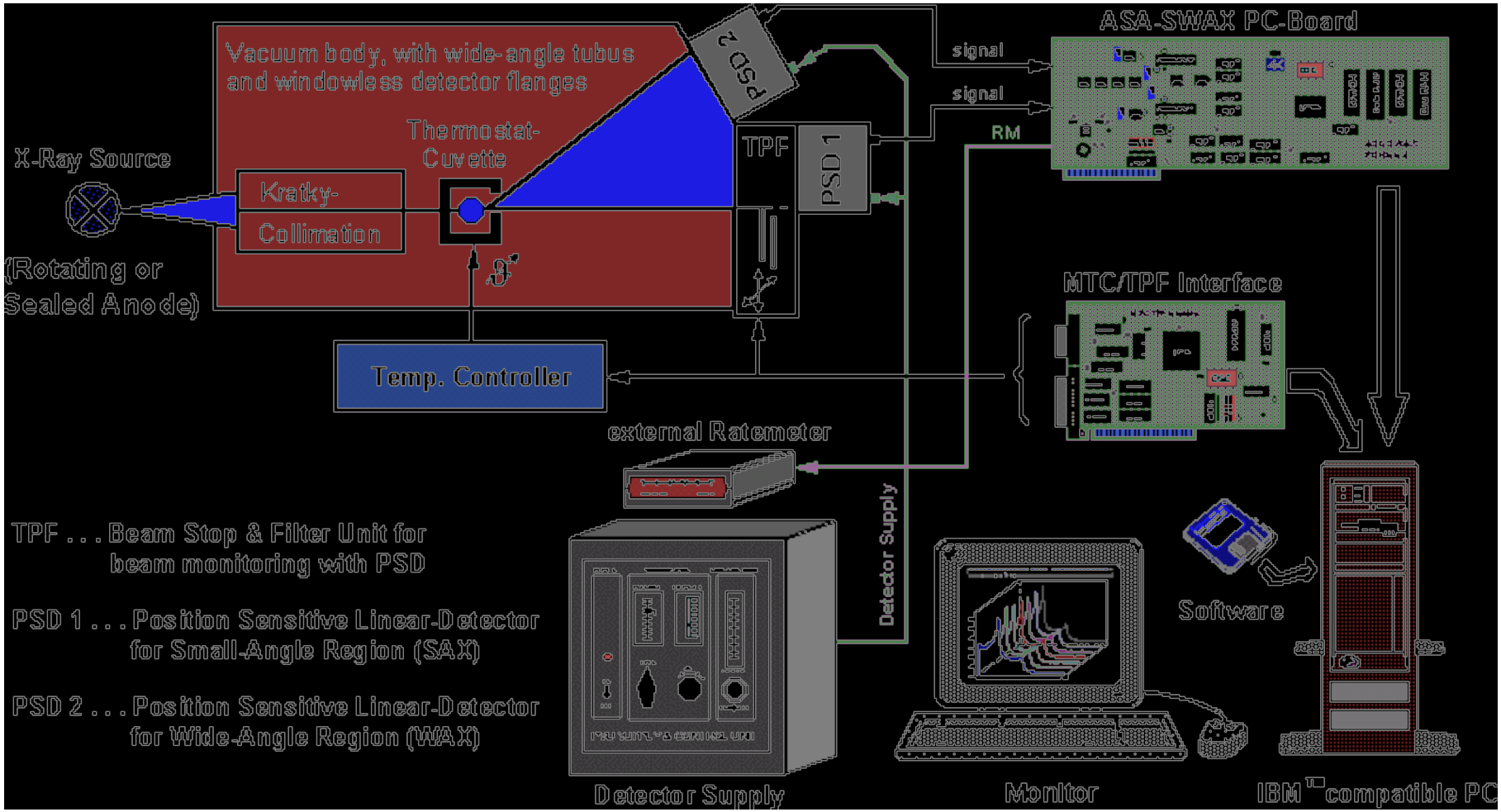
The SWAXS Instrument



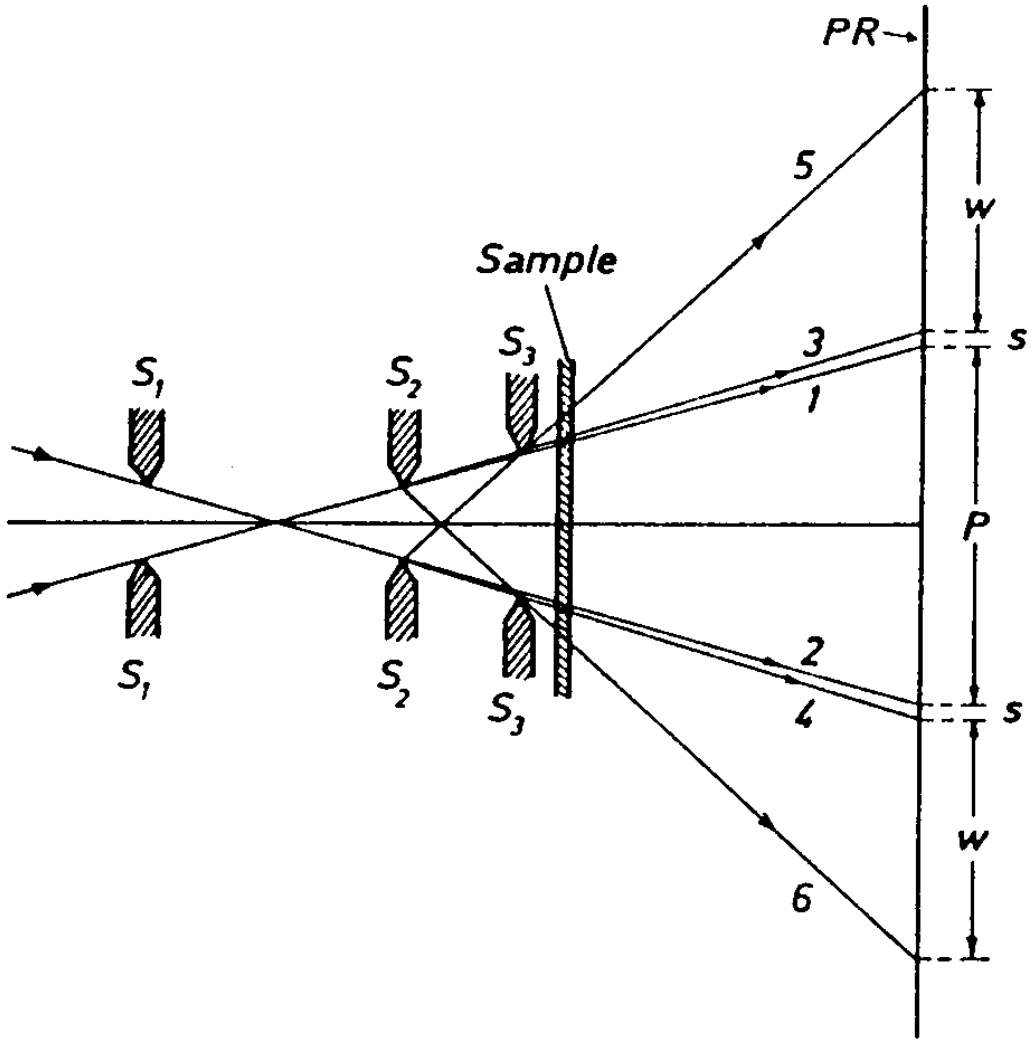
Scheme of the HECUS-MBraun SWAXS- System



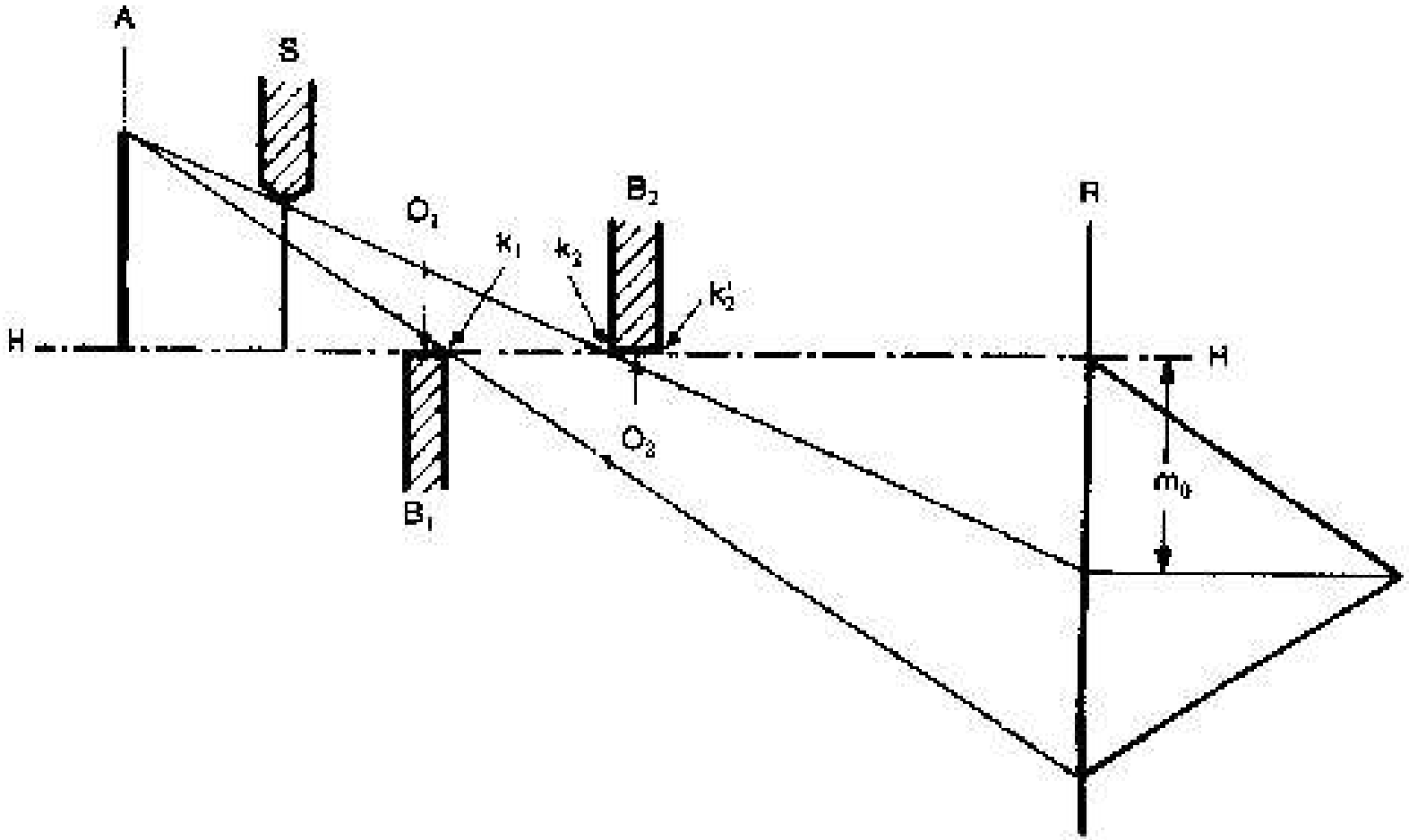
X-ray Source

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

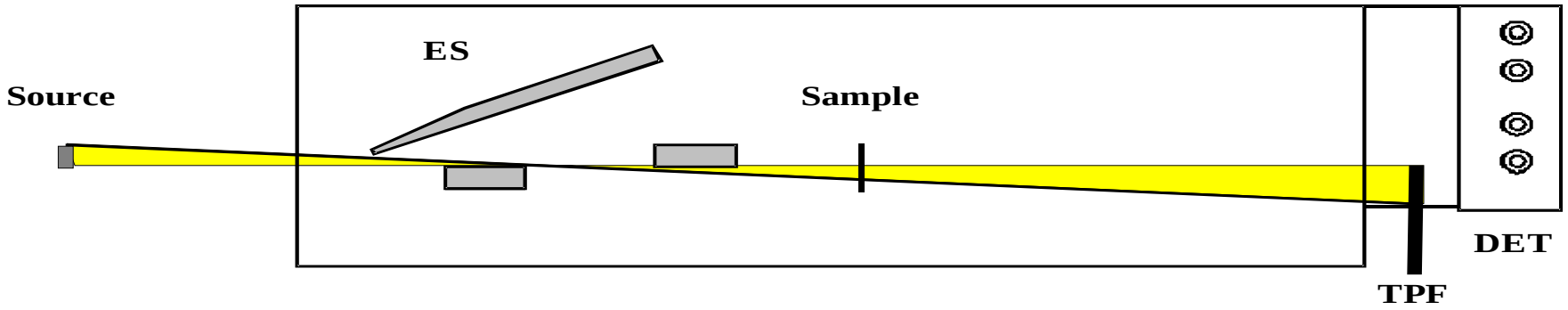
Slit Collimation

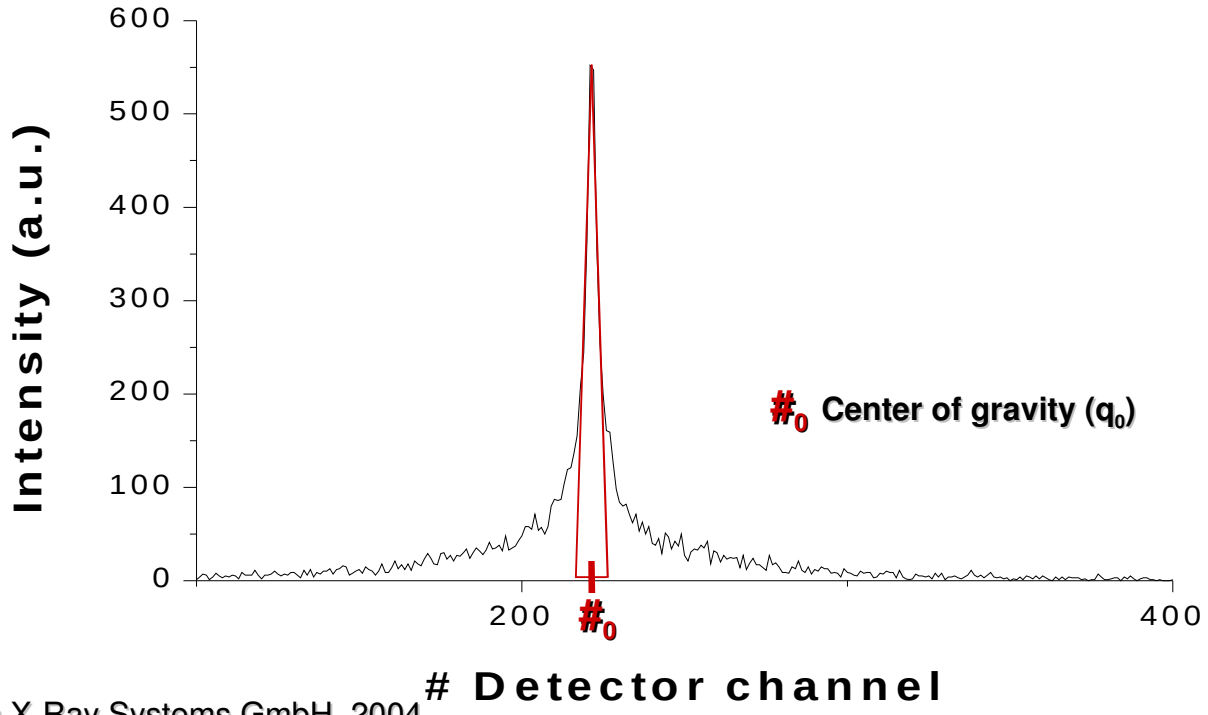


Kratky-Type Collimation System

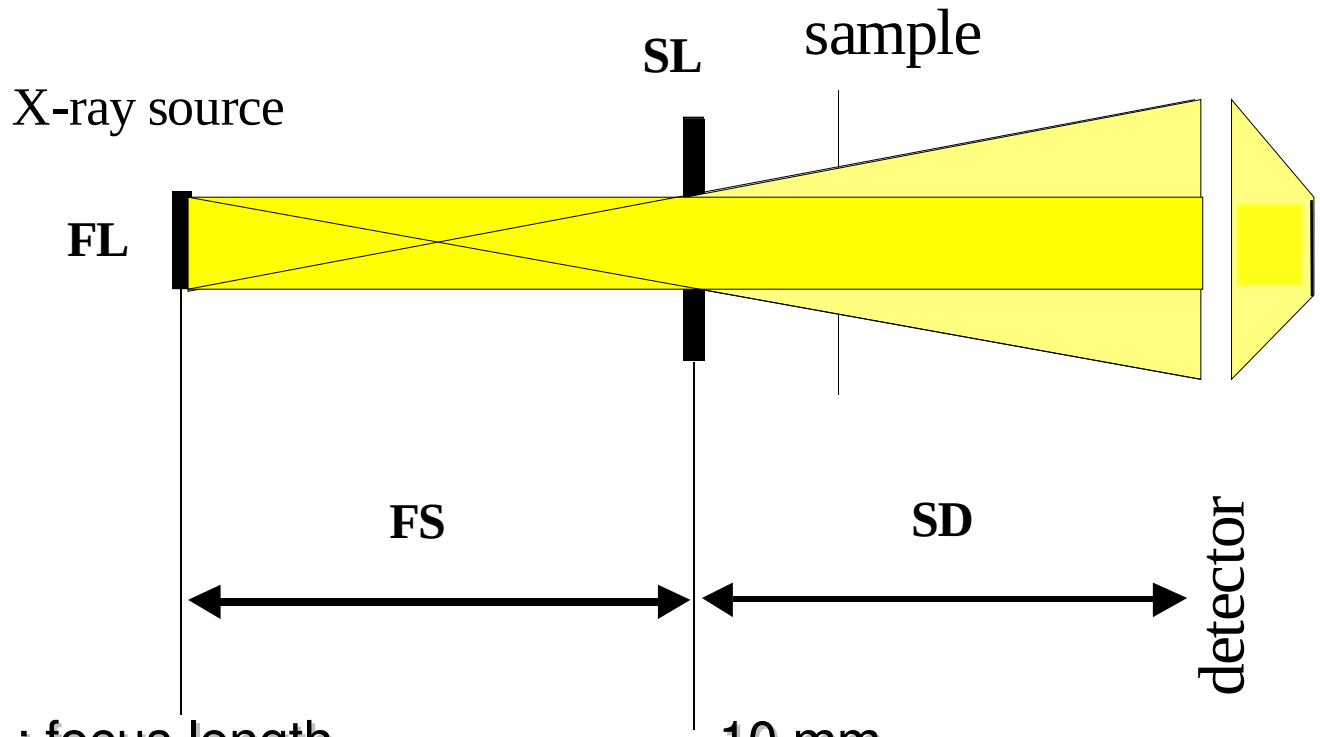


Vertical Beam Profile





Horizontal Beam Profile



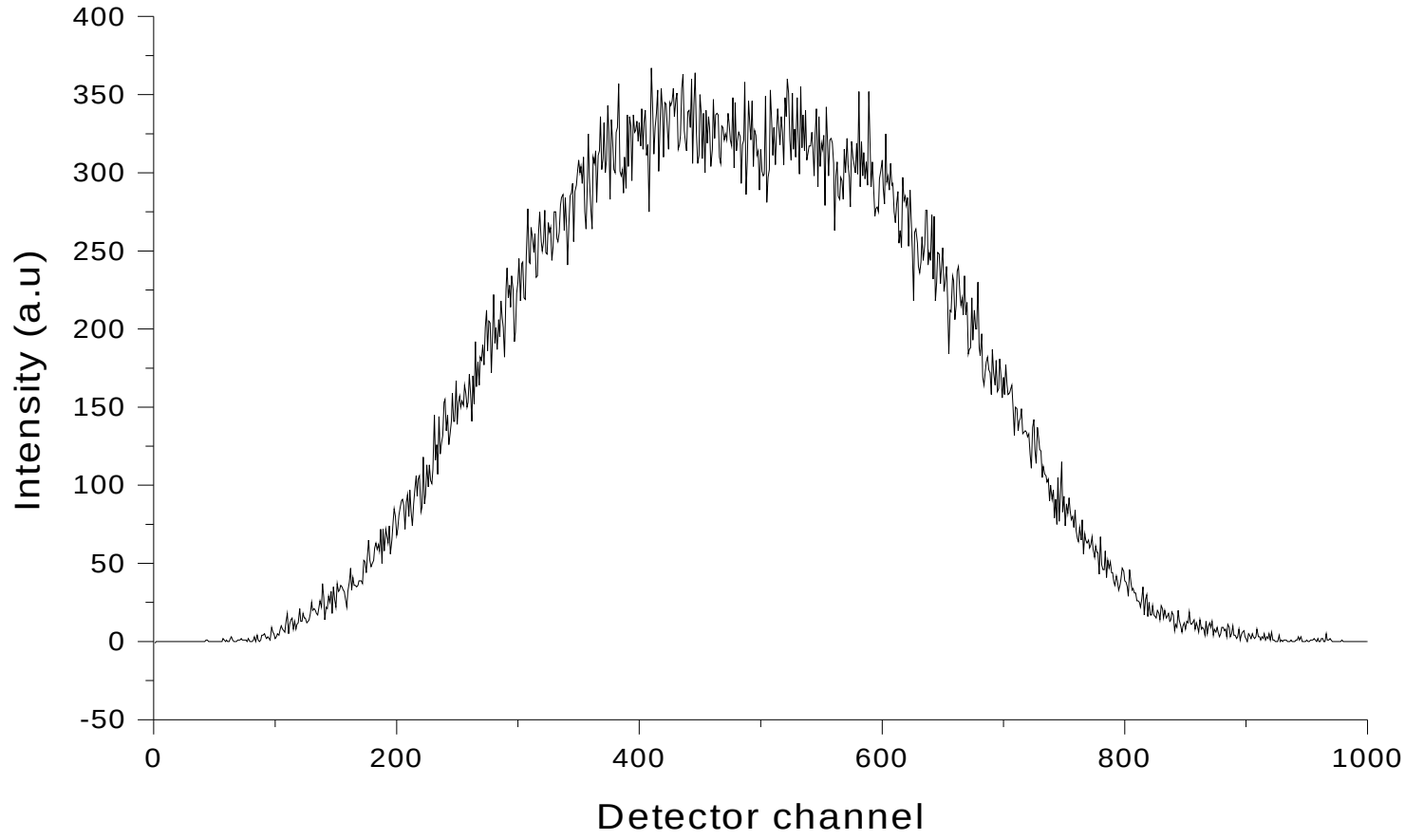
FL: focus length 10 mm

SL: slit length 0-16 mm

FS: focus-slit distance 150 mm

SD: slit-detector distance 300 mm

SL: defines the length of the horizontal beam profile



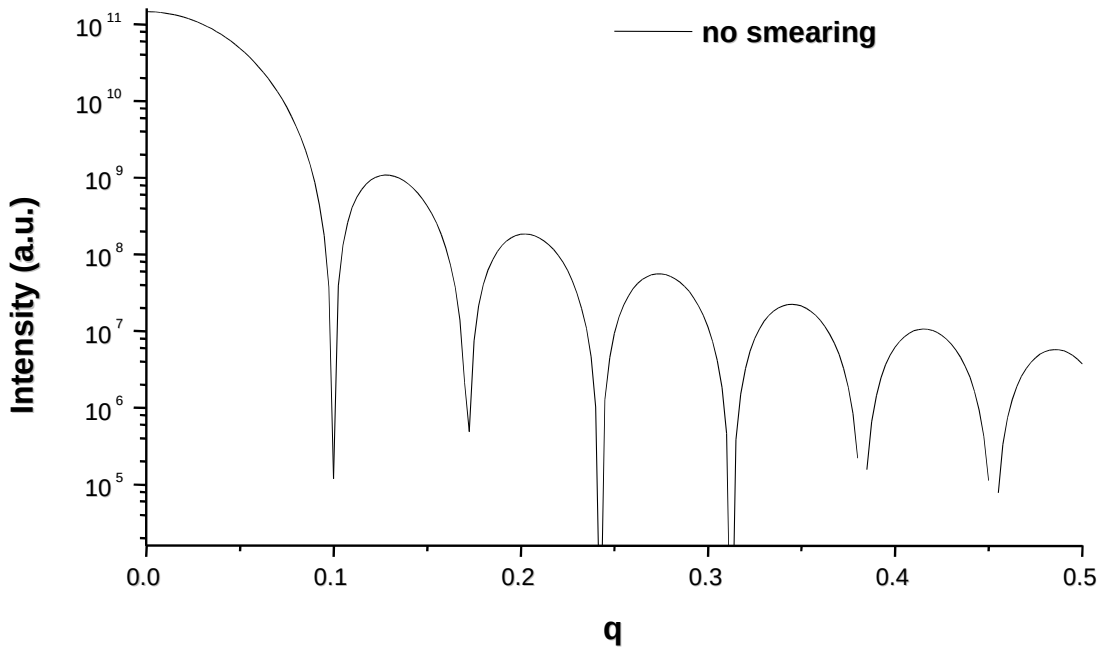
Advantages of Line-Collimation for SAXS

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

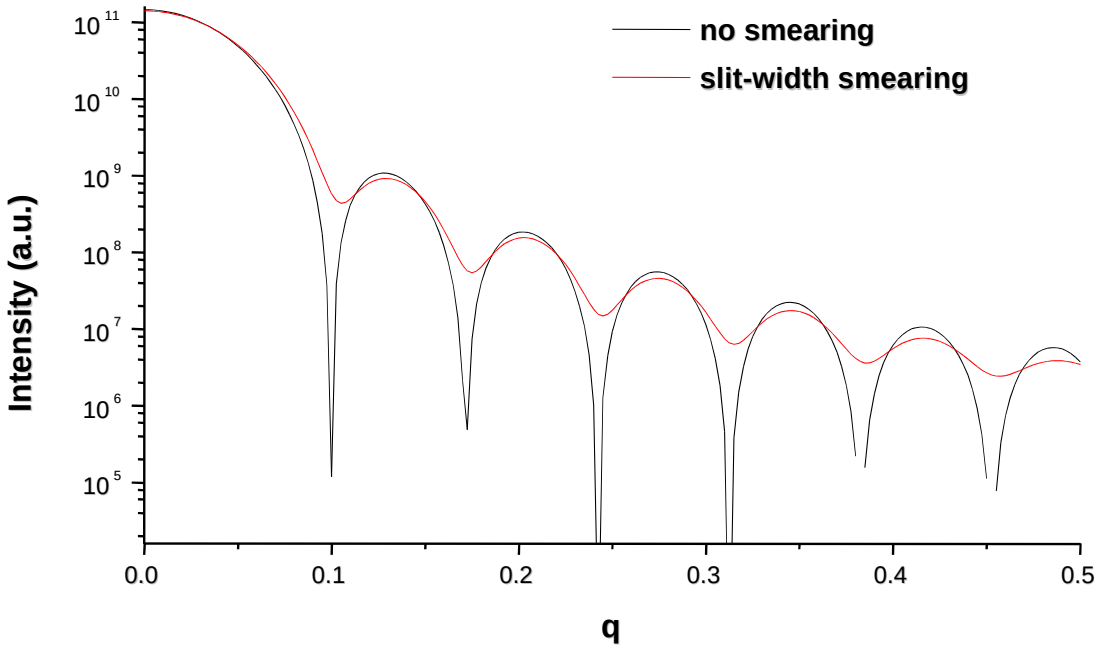
Disadvantages of Line-Collimation

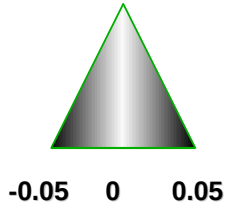
- 'Smearing' by slit-width and slit-length profiles

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



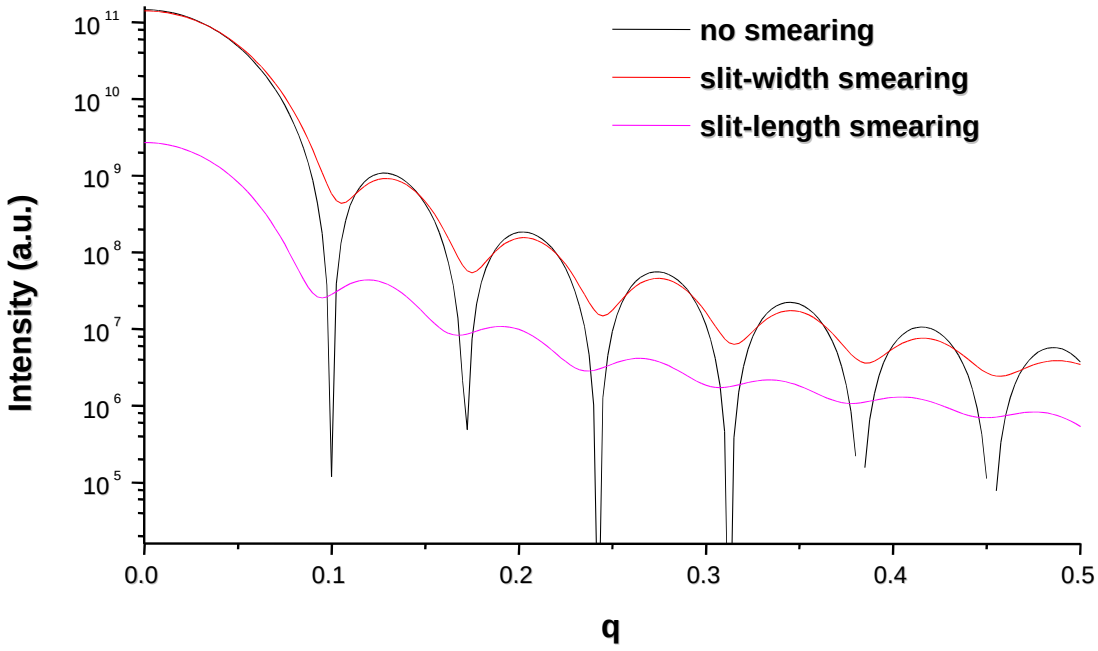
'Smearing' by slit-width and slit-length profiles

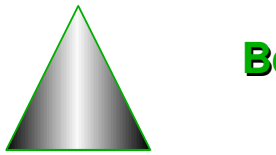




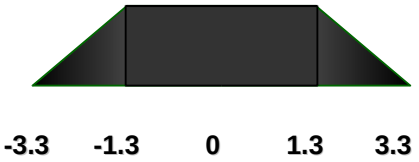
Beam-width profile

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



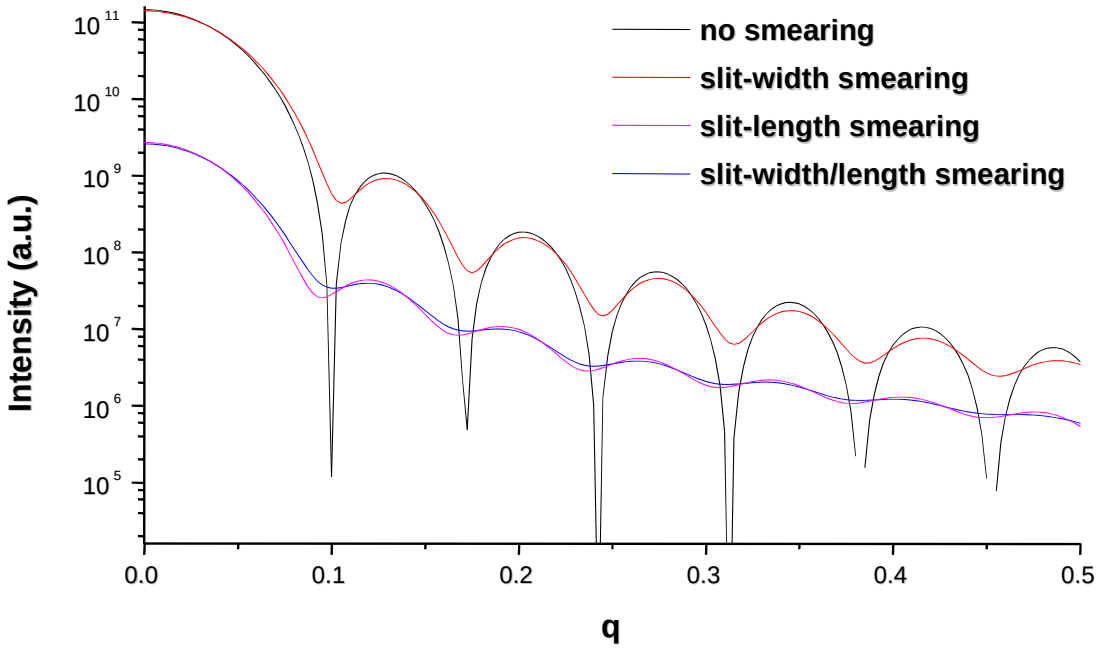


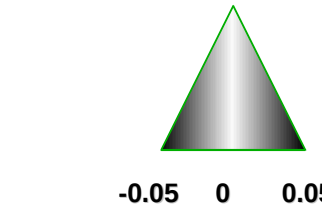
Beam-width profile



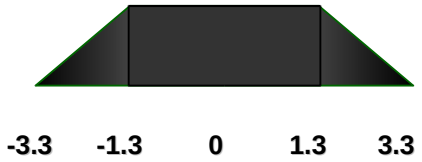
Beam-length profile

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



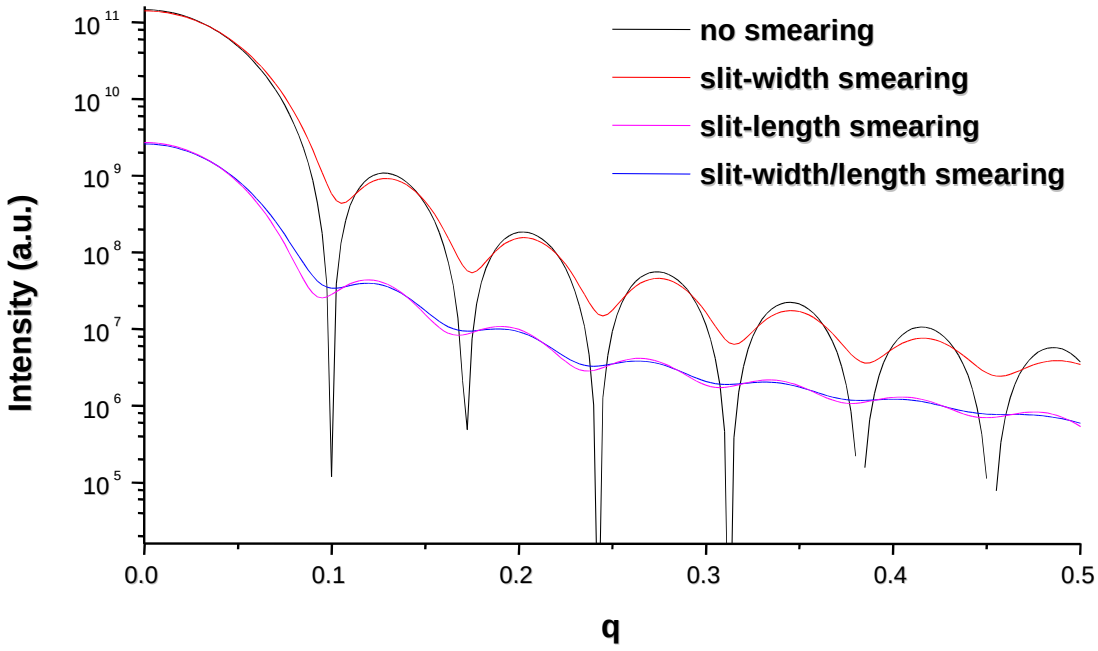


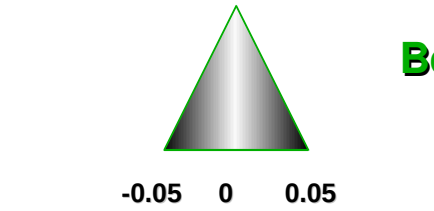
Beam-width profile



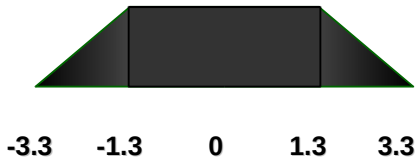
Beam-length profile

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





Beam-width profile



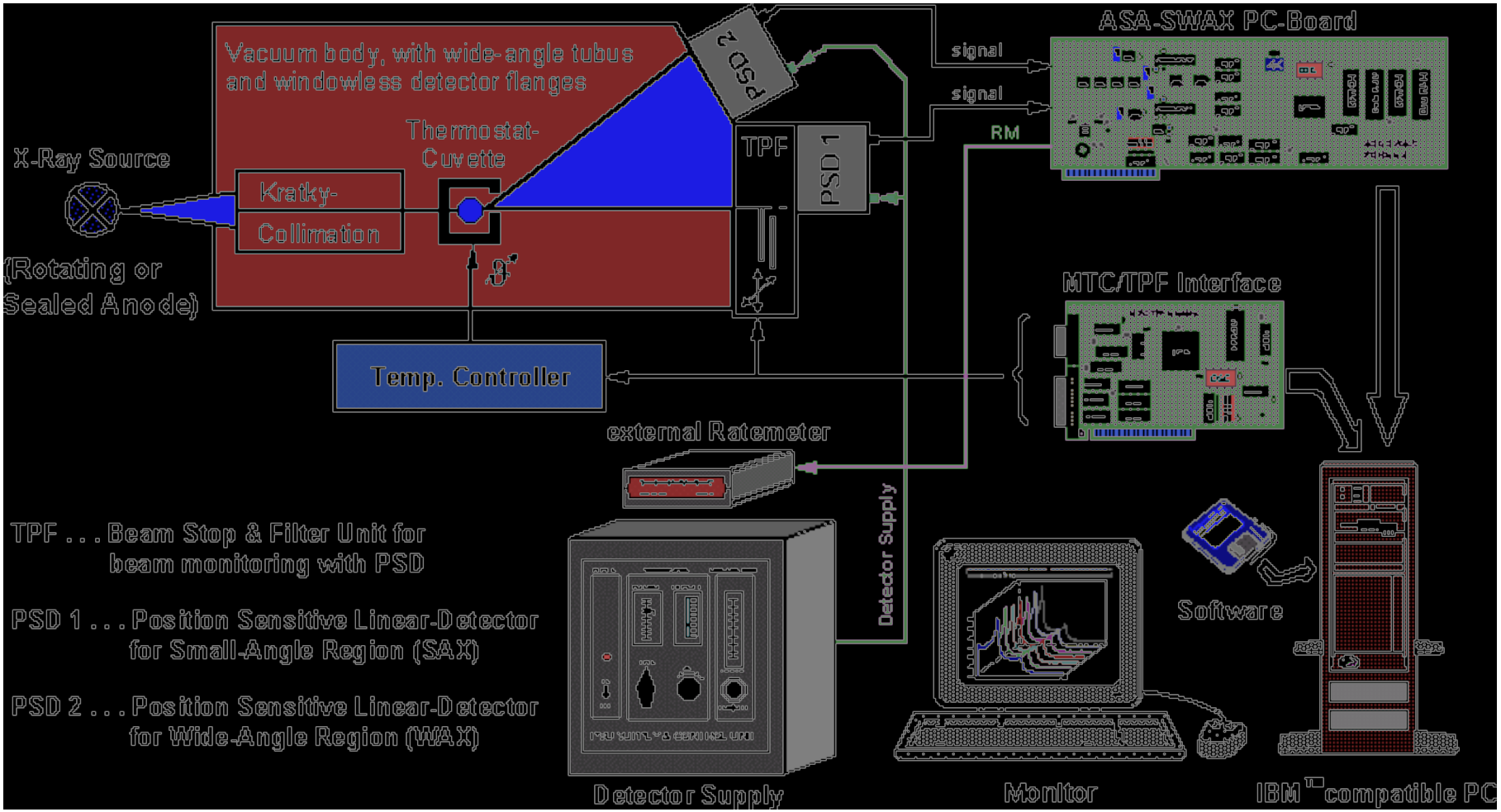
Beam-length profile

Further smearing effects by wavelength distribution and polydispersity.

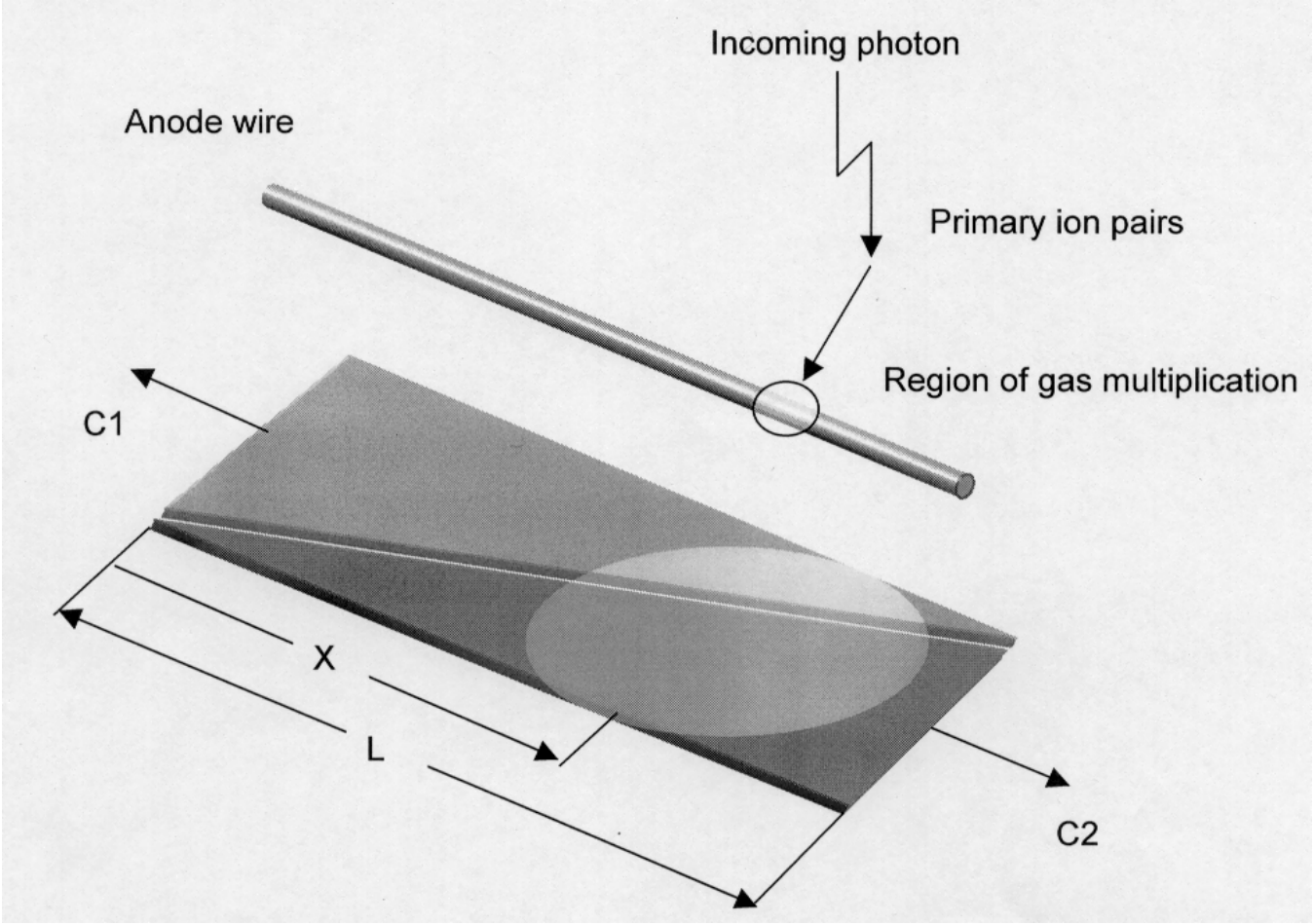
Flexible Sample Environment

- 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:

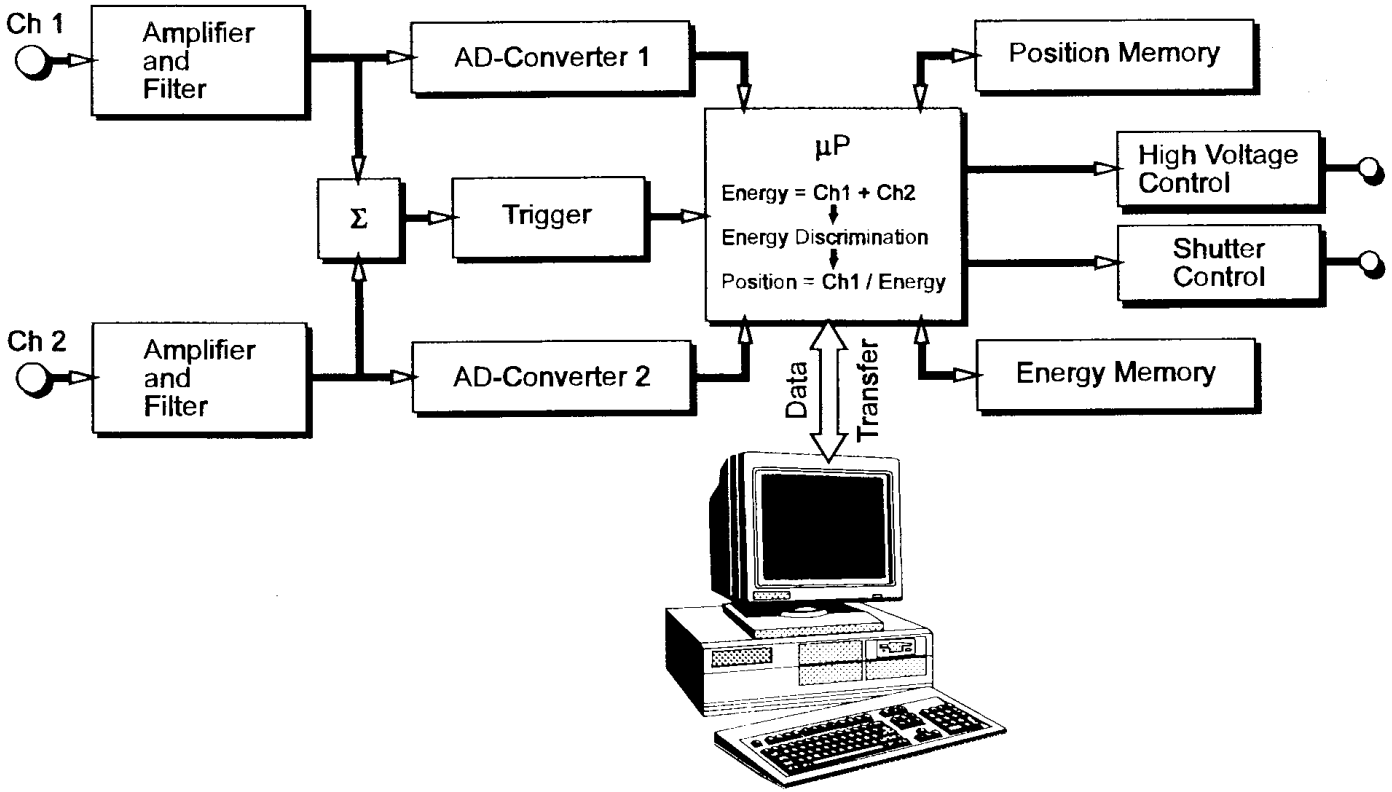
$X = L*C2/(C1+C2)$

Energie:

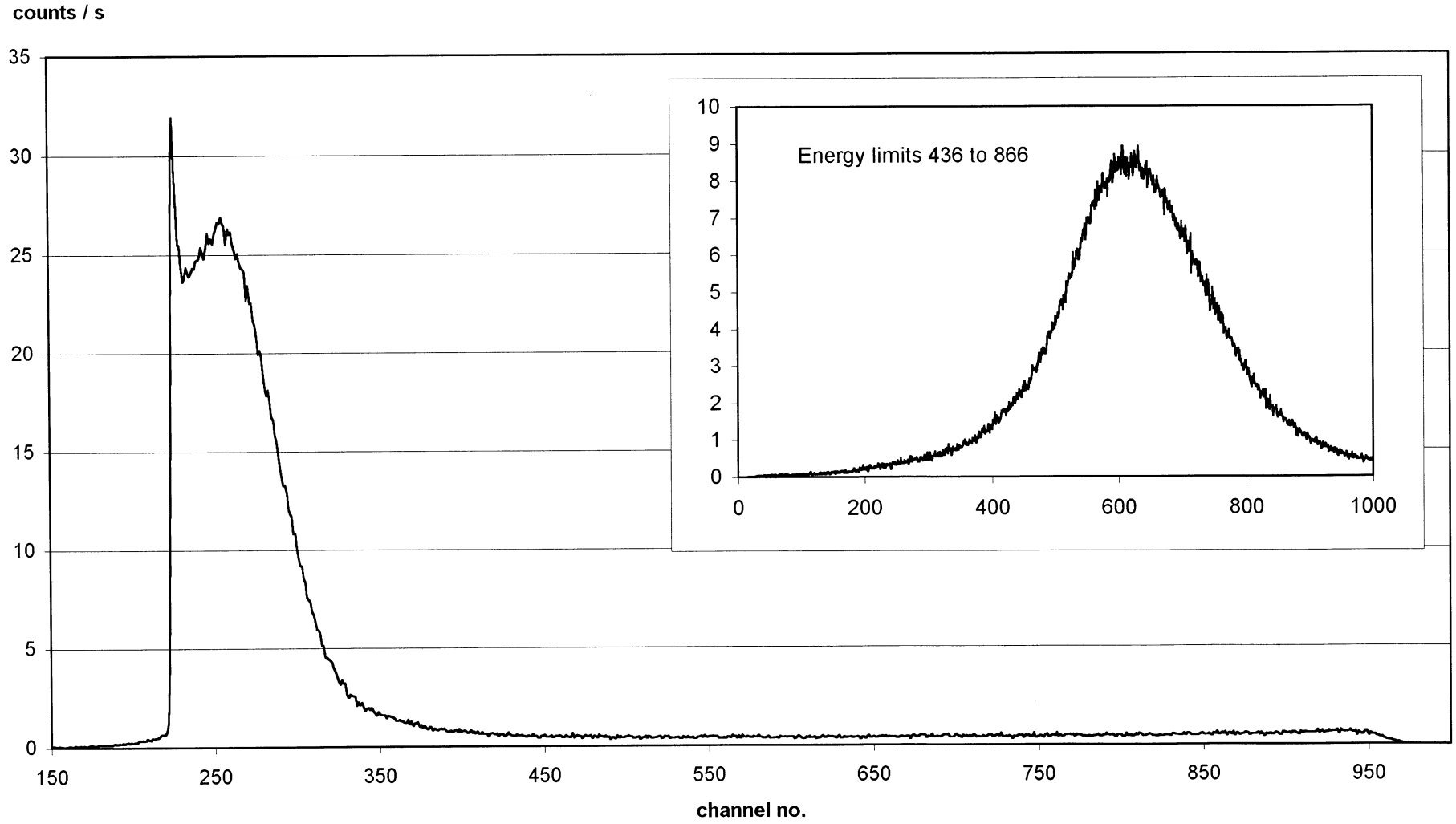
$E = C1+C2$

C1,C2	integraded cathode signals
L	actve length of the detector
X	position of incoming photon

Amplitude Spectra Analyser (ASA)

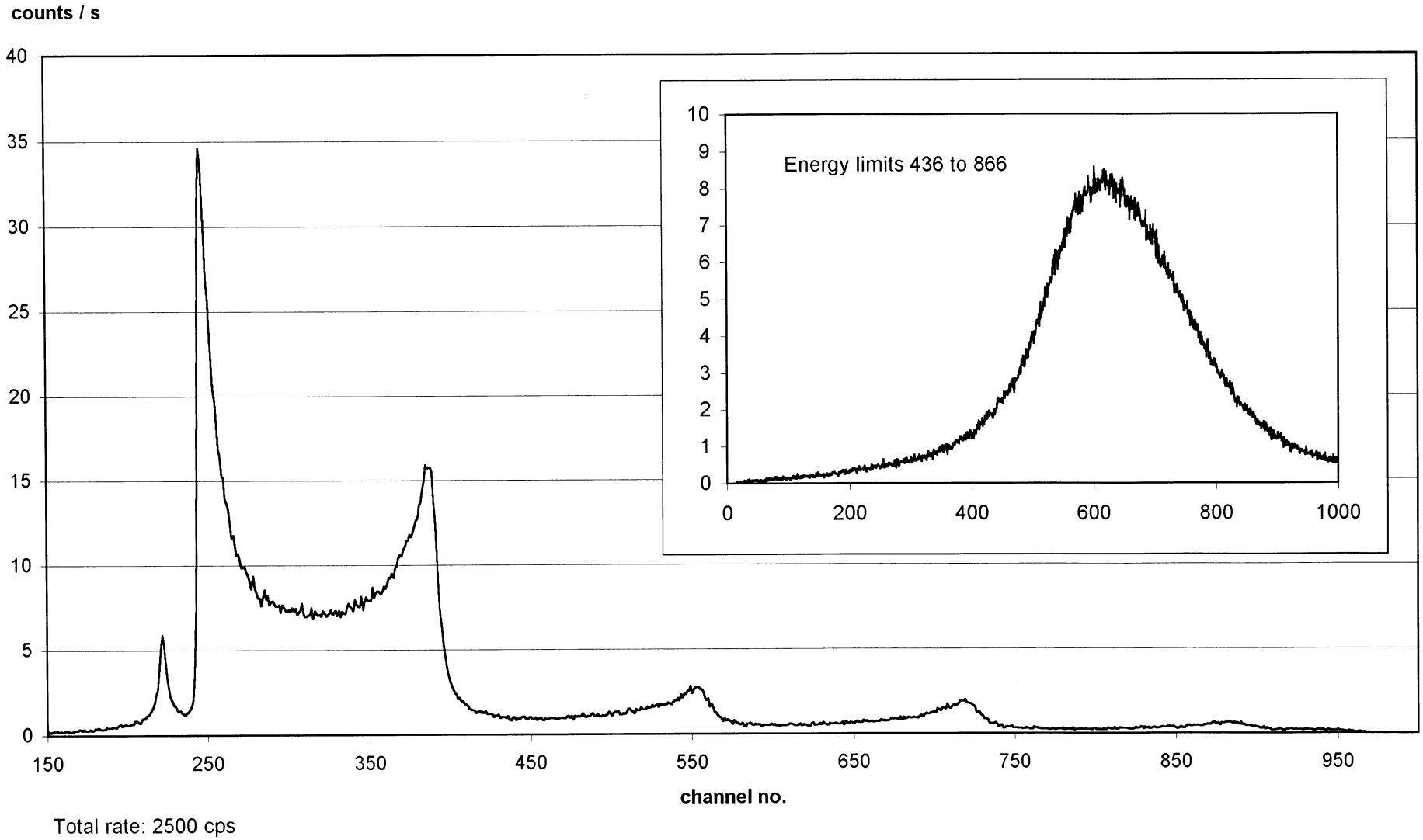


Lupolen



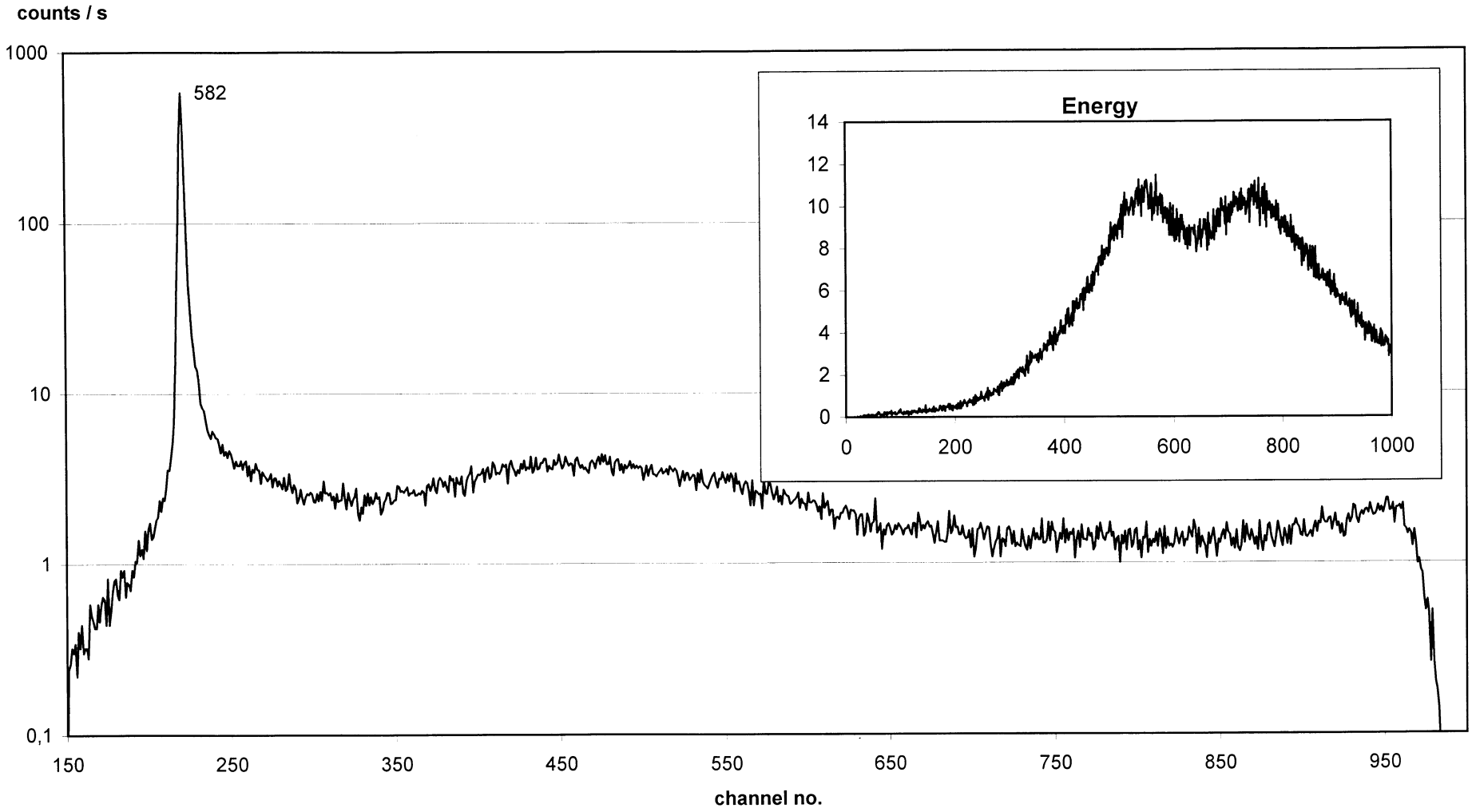
Total rate: 2500 cps

Silverstearate



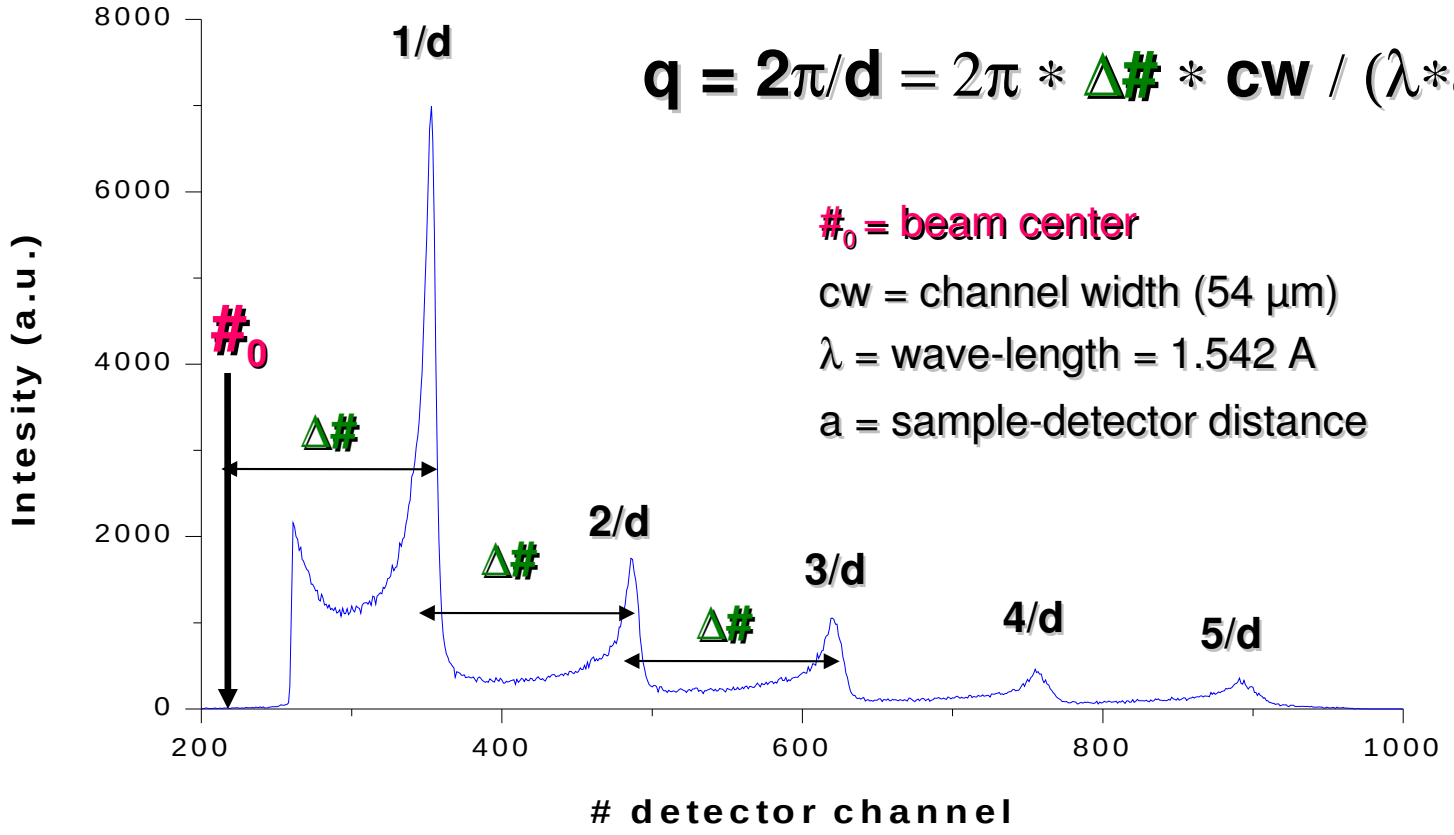
Total rate: 2500 cps

'burn in' effect on the wire



Total rate: 5200 cps

Calibration of the q-scale (SAXS) with Ag-behenate powder: Lamellar d-spacing: d = 58.38 A



$$q = 2\pi/d = 2\pi * \Delta\# * cw / (\lambda * a)$$

$\#_0$ = beam center

cw = channel width (54 µm)

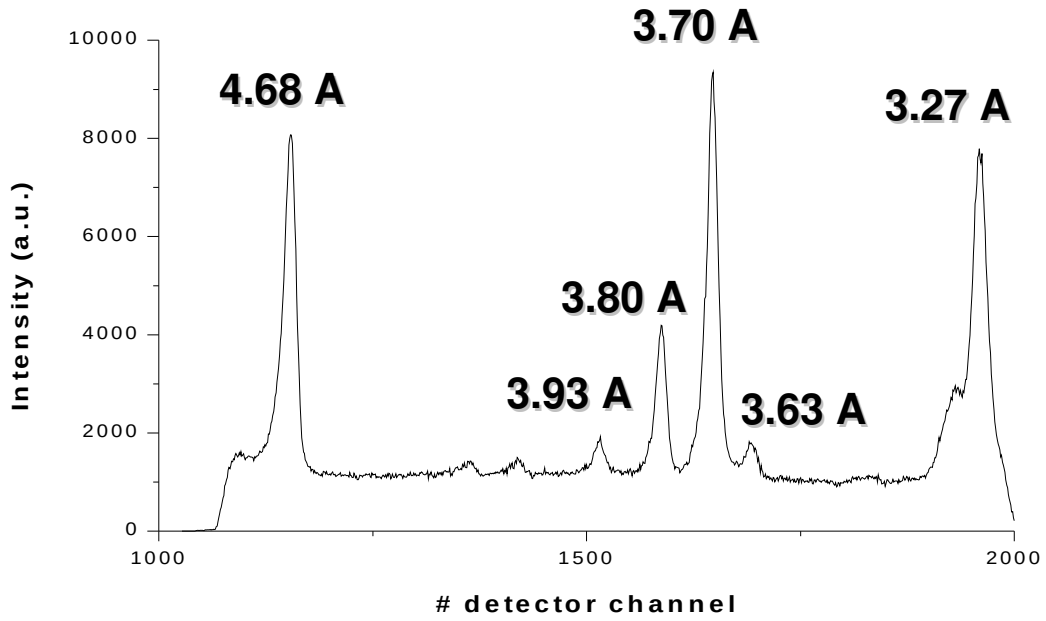
λ = wave-length = 1.542 A

a = sample-detector distance

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

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

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

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