INTERNATIONAL ATOMIC ENERGY AGENCY
UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION
INTERNATIONAL CENTRE FOR THEORETICAL PHYSICS
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H4.SMR/676-19

SECOND SCHOOL ON THE USE OF SYNCHROTRON RADIATION IN SCIENCE AND TECHNOLOGY: *"JOHN FUGGLE MEMORIAL"*

25 October - 19 November 1993

Miramare - Trieste, Italy

Instrumentation for X-Ray Spectrometry

OPTICS

H. Tolentino
National Laboratory for Synchrotron Radiation
(LNLS)
Campinas, Brazil

instrumentation for X-ray spectrometry

optics

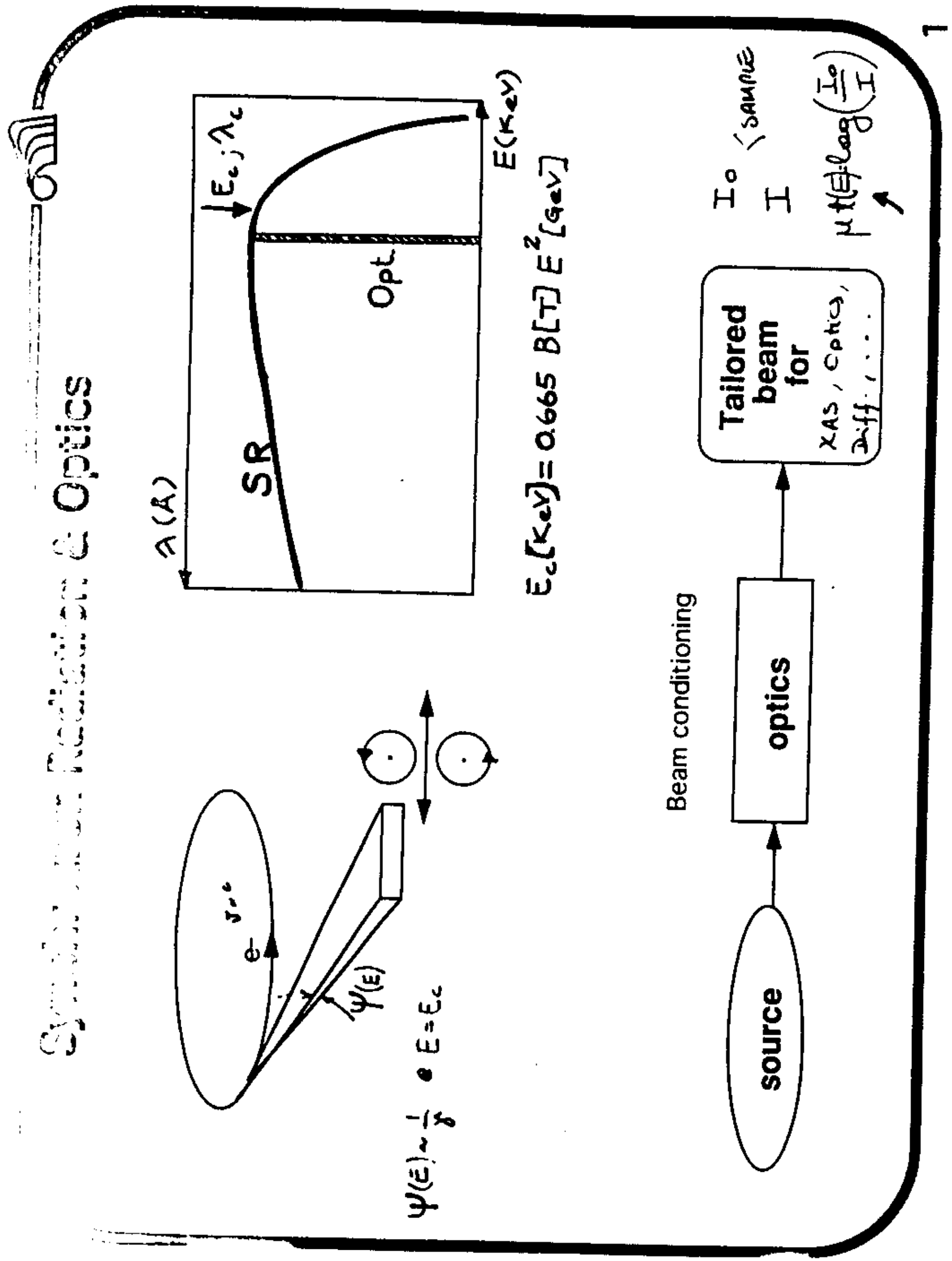
needs : x-ray source
optics
detectors
specific needs

optics

beam shapers
mirrors
monochromators

new trends

high brilliance sources
state of polarization
high resolution

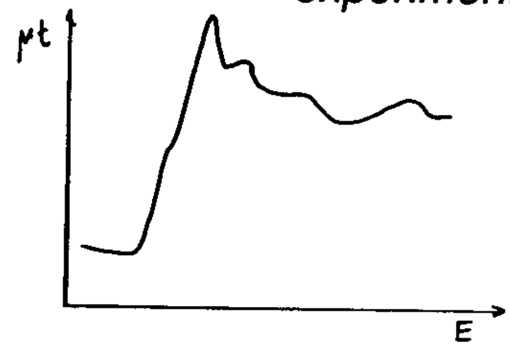


x-ray optical system

beam characteristics
sample conditionning
detection scheme

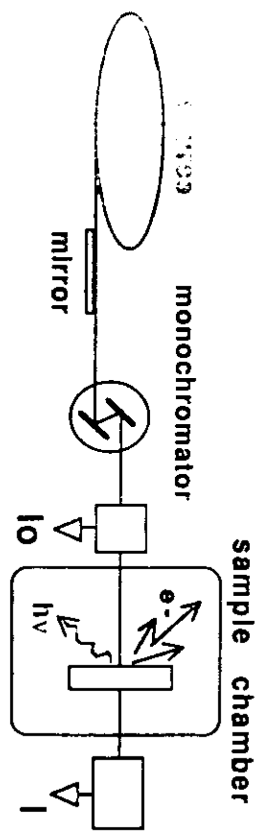
to transform the beam to match it to the experiment

XAS requirements



- o energy range
- o energy tunability
- o energy resolution
- o harmonic purity
- o polarization state
- o intensity / stability
- o signal / noise
- o focusing possibilities
- o high mechanical precision, stability and reproducibility

XAS workstation



Total reflection

$$n = 1 - \delta - i\beta$$

$$\delta = (r_0/2\pi)\, N f(0)\, \lambda^2$$

$\delta \sim 10^{-5} > 0$

$n < 1$

Snell's law

$\cos\theta_i = n \cos\theta_r$

$\cos\theta_c = (1-\delta)$

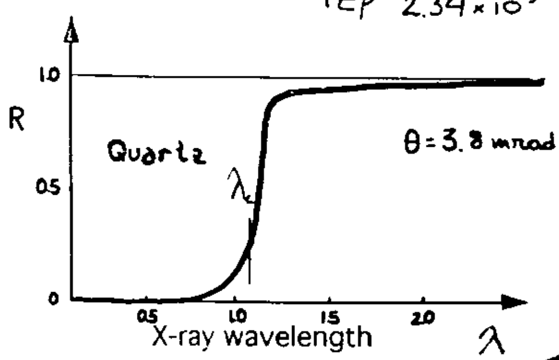
$1 - \frac{\theta_c^2}{2} = 1 - \delta$

$$\Theta_c = 2.34 \times 10^{-3} \sqrt{\frac{Z\rho}{A}}\ \lambda$$

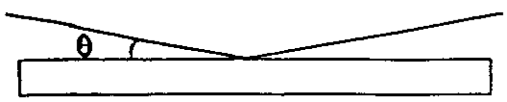
$\theta_c = \sqrt{2\delta} \ \alpha\ \lambda$

$\delta \sim 10^{-5}$ $\theta_c \sim 1$ mrad

$$\lambda_c = \sqrt{\frac{A}{Z\rho}}\ \frac{\Theta}{2.34 \times 10^{-3}}$$

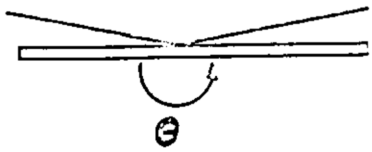


High-pass wavelength filter

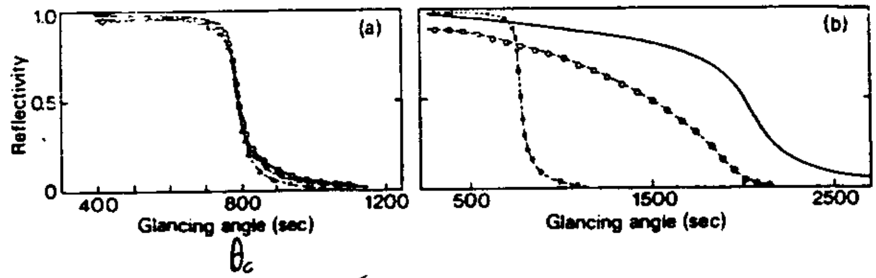


Low-pass energy filter

$\lambda = 1.54 Å$

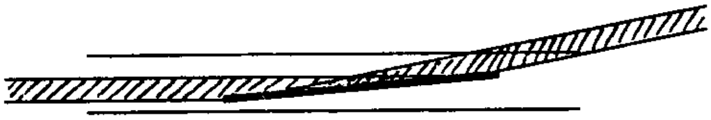


$z \uparrow$: $\beta \uparrow$



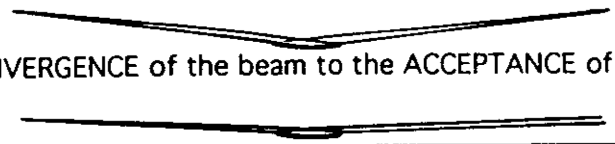
Low-pass energy filter : HARMONICS n > 1

Slit



Thermal load : the POWER is spread over a large area

Focusing element :
focus
match the DIVERGENCE of the beam to the ACCEPTANCE of the crystal



focusing mirrors

efficiency is determined by aberrations
and imperfections

o focusing geometry

meridional

sagittal

o accuracy and roughness

multilayers

layered synthetic microstructures

o flexibility

o Bragg's law

o layers of high and low Z

stability

interdiffusion

interface roughness

layer thickness

o reflectivity

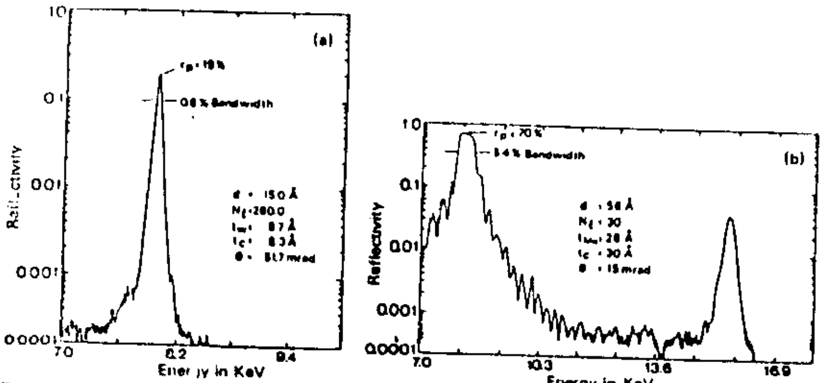
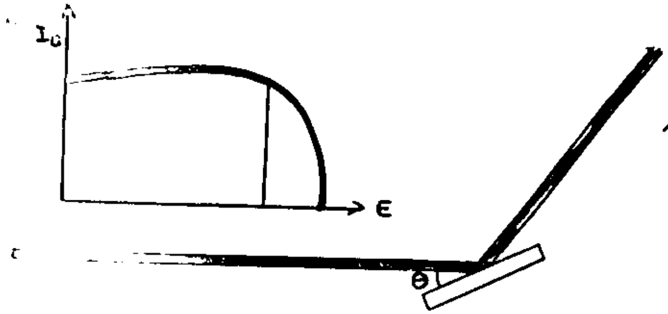


Fig. III.1.- Typical reflectivity profile obtained with multilayers (After Bilderback et al., 1983).

BRAGG DIFFRACTION



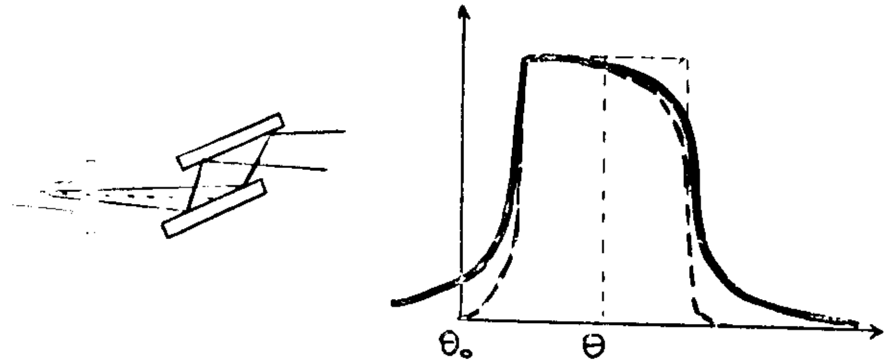
λ, Δλ

$$n\lambda = 2 d_{hkl} \sin\theta \left(1 - \frac{\delta(\lambda)}{\sin^2\theta}\right) \quad ; \quad n = 1 - \delta - i\beta$$

$$\frac{\Delta\lambda}{\lambda} = \cot\theta \, \Delta\theta \quad \leftarrow \alpha$$

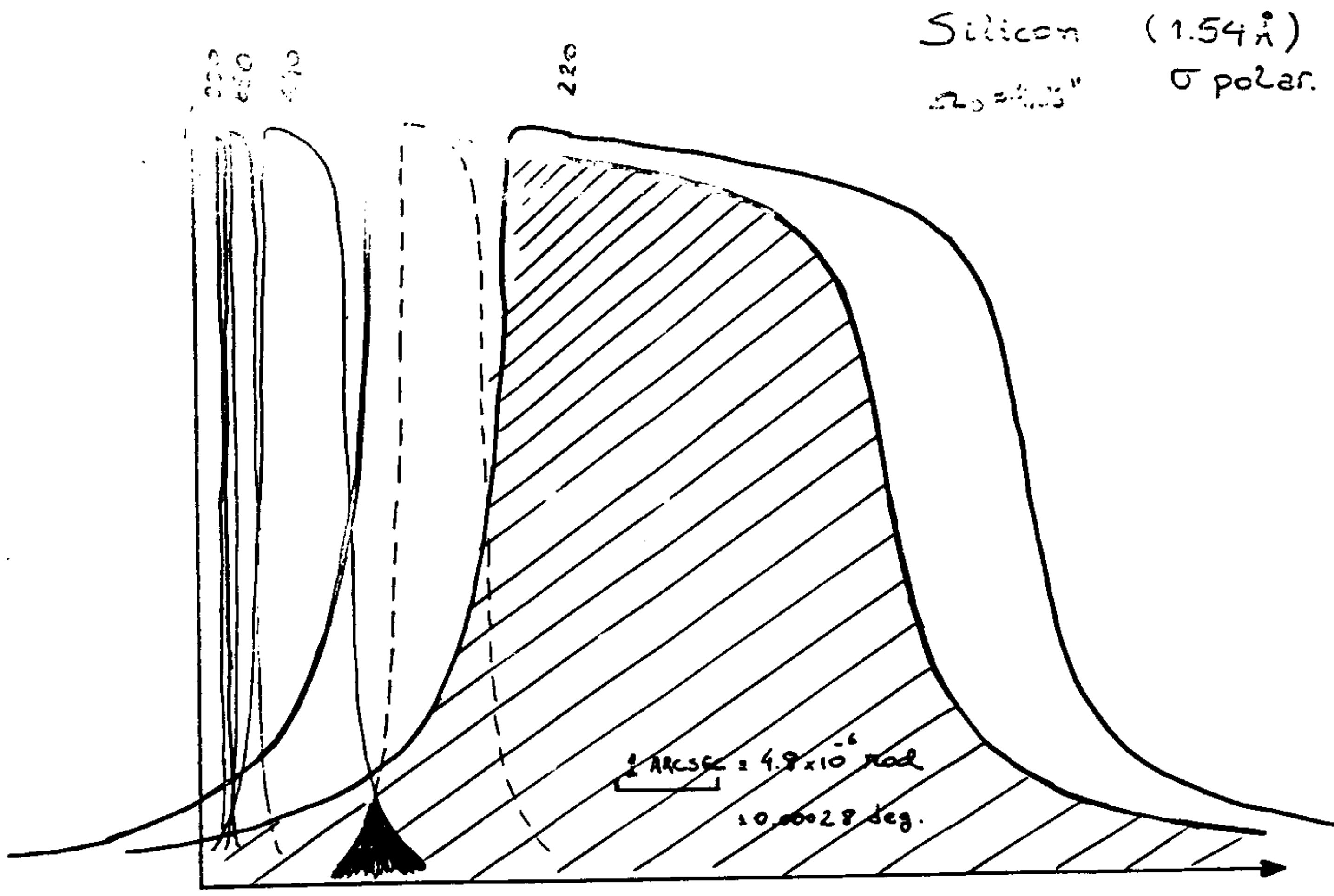
$$\leftarrow \Omega_D \propto \delta \sim \lambda^2 Z$$

$$\Omega_D \cot\theta \propto \frac{1}{h^2+k^2+l^2}$$

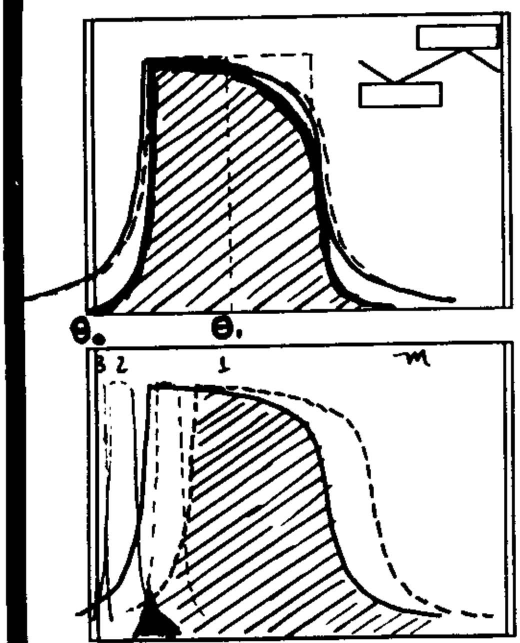


RESOLUTION & INTENSITY

Si(333) ↓ Ω_D ↑ Ge(111)



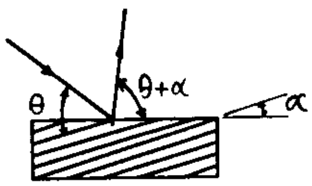
Dynamical theory of perfect crystals



- $m\lambda = 2d_{hkl}\sin\theta \; (1 - \delta / \sin^2\theta)$
- $n = 1 - \delta - i\beta$
- $\Omega_D \propto \delta \propto \lambda^2 Z$

  $\Omega_D \, \mathrm{ctg}\,\theta \propto \frac{1}{h^2 + k^2 + l^2}$ ‖ RESOLUTION
- $\Delta\theta_0 \propto \lambda^2 Z$
- HARMONICS $(m > 1)$ REJECTION → DETUNING / MIRROR
- ASYMMETRIC CRYSTAL

  $\Omega_A = \gamma_A \, \Omega_D \qquad \gamma_A = \sqrt{\frac{\sin(\theta-\alpha)}{\sin(\theta+\alpha)}}$



RESOLUTION ↔ ACCEPTANCE

Asymmetric crystal

$$\gamma_A = \sqrt{\frac{\sin(\theta - \alpha)}{\sin(\theta + \alpha)}}$$

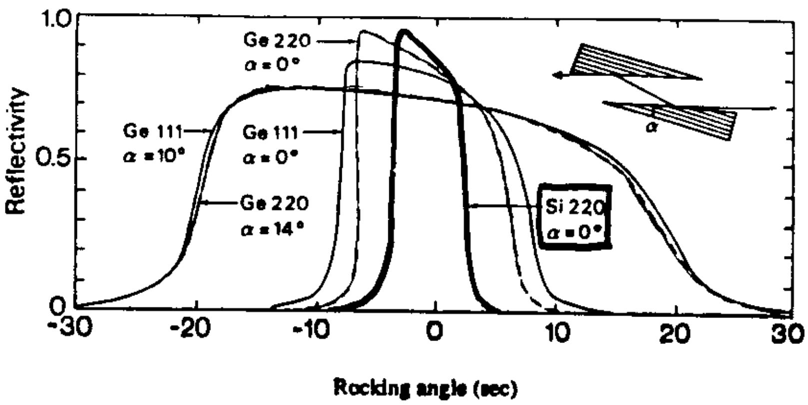


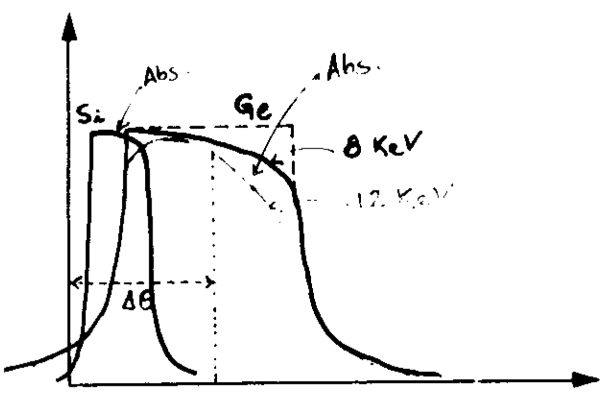
Fig. 6. Calculated double-reflection curves of grooved monochromators for symmetric and asymmetric reflections of silicon and germanium and for X-rays of 1.54 Å wavelength (After Kohra et al., 1978).

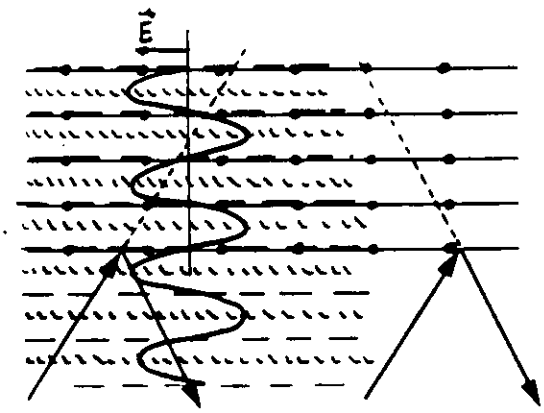
$$\Omega_A = \gamma_A \Omega_D$$

16

Absorption

$$n = 1 - \delta - i\beta$$





8 KeV	Si(111)	Ω = 7.4 arcsec	= 1.1 eV	Si : 1 s	1840 eV
	Ge(111)	Ω = 16.6 arcsec	= 2.6 eV	Ge : 1 s	11100 eV

13

Primary Extinction

WITHIN THE RANGE OF TOTAL REFLECTION ∴ Ω

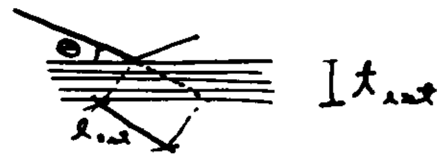
THE ENERGY OF THE PRIMARY BEAM IS WHOLLY DIVERTED INTO THE REFLECTED BEAM

* PRIMARY BEAM DIMINISHES RAPIDLY IN INTENSITY

~→ LARGE ABSORPTION COEFICIENT

PRIMARY EXTINCTION (DARWIN) : NO ORDINARY ABSORPTION

$$\ell_{ext} = \frac{1}{N|f(2\theta)|\, r_0\, \lambda\, C}$$



$$t_{ext} = \frac{1}{N|f(2\theta)|\, r_0\, 2d\, C}$$

$$t_{abs} = \frac{\sin\theta}{2\mu} \qquad t_{ext} \ll t_{abs}$$

	t_{ext}	t_{abs} (μm)
e.g.: Si (111)	1.53	8.7
Ge (111)	0.68	2.94

RESOLUTION

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

$$\frac{\Delta\lambda}{\lambda} = \frac{\Delta E}{E} = \left[\left(\frac{\Delta d}{d}\right)^2 + \left(\Delta\theta\cdot\cot g\,\theta\right)^2\right]^{1/2}$$

$\Delta\theta$: ANGULAR DISTRIBUTION ⊗ MOSAIC STRUCTURE

$\frac{\Delta d}{d}$: DIFFRACTION PATTERN P.C. ⊗ STRAINS AND VARIATIONS d

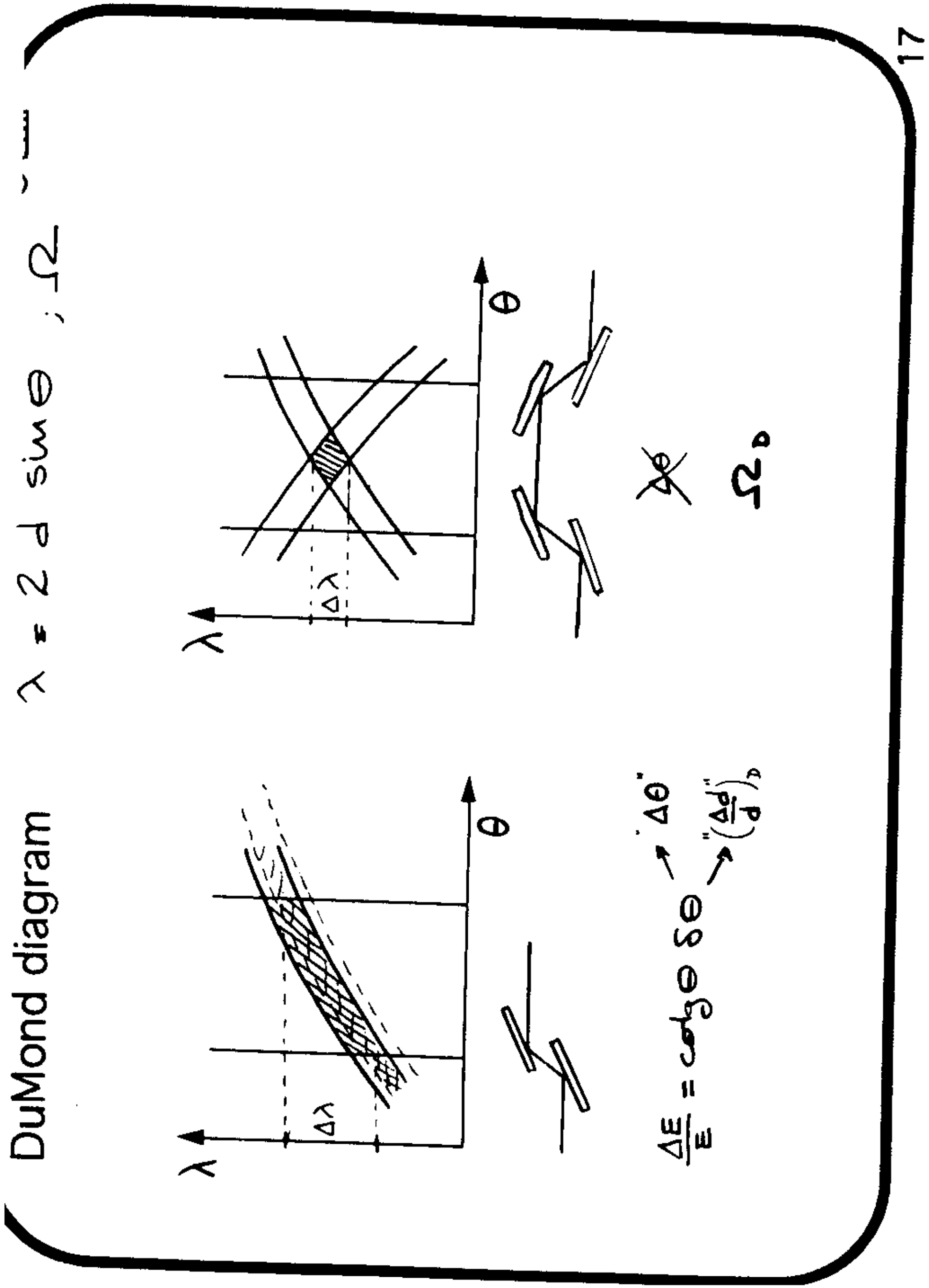
↓

Ω_D

$$\Omega_D = \left(\frac{\Delta d}{d}\right)_D \tan\theta \qquad \left(\frac{\Delta d}{d}\right)_D = \frac{\Delta\lambda}{\lambda} = \frac{4d^2}{\pi} N|f(2\theta)|\, r_0\, C = \frac{2d}{\pi\, t_{ext}}$$

$$\left(\frac{\Delta d}{d}\right)_D \simeq 0.64/N_p \qquad N_p = \frac{t_{ext}}{d}$$

$\Omega_D \cot g\,\theta$: RELATIVE ENERGY RESOLUTION WITH PERFECT CRYSTAL WITH PARALLEL "WHITE" BEAM



single crystal monochromators for XAS

1 crystal :

- focusing
- high flux
- dispersive

2 crystals :

- several applications
- flux & resolution
- focusing
- cooling

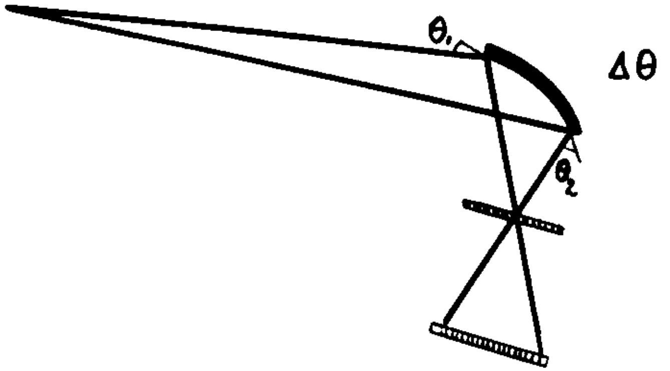
4 crystals :

- high resolution
- high collimation

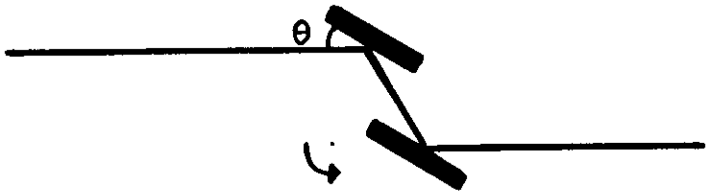
backdiffraction :

- high resolution
- large angular acceptance

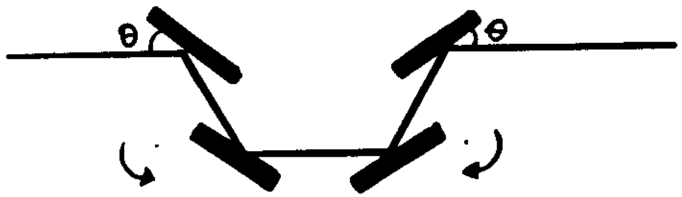
high flux



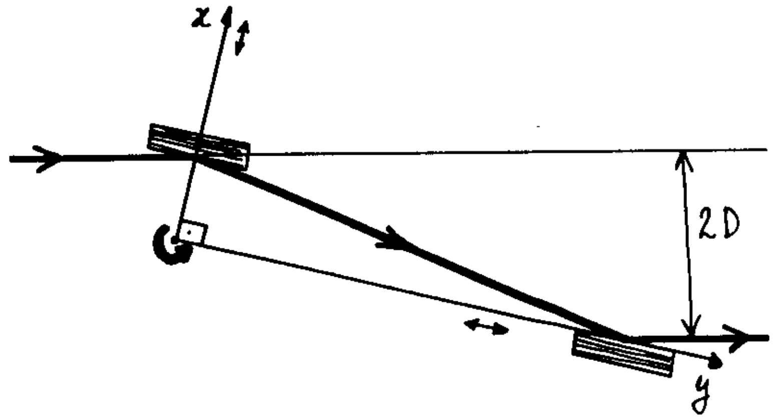
1C

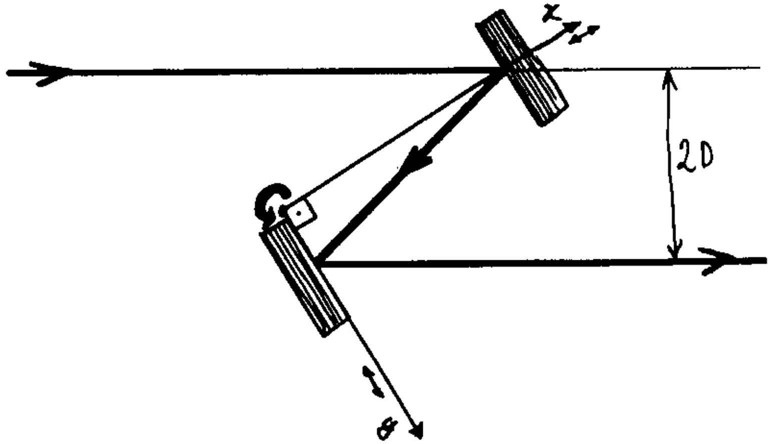


2C



4C



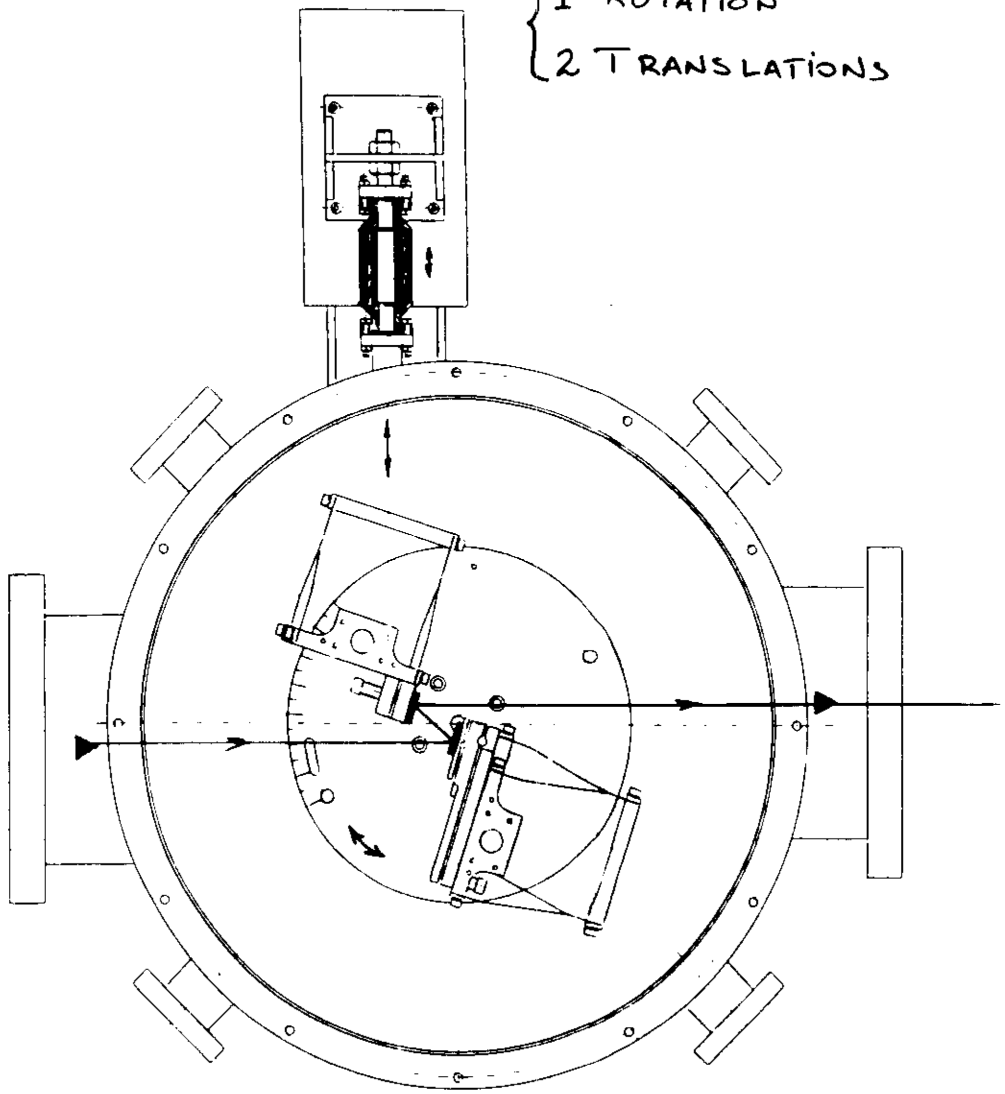


$$x = \frac{D}{\cos\theta}$$

$$y = \frac{D}{\sin\theta}$$

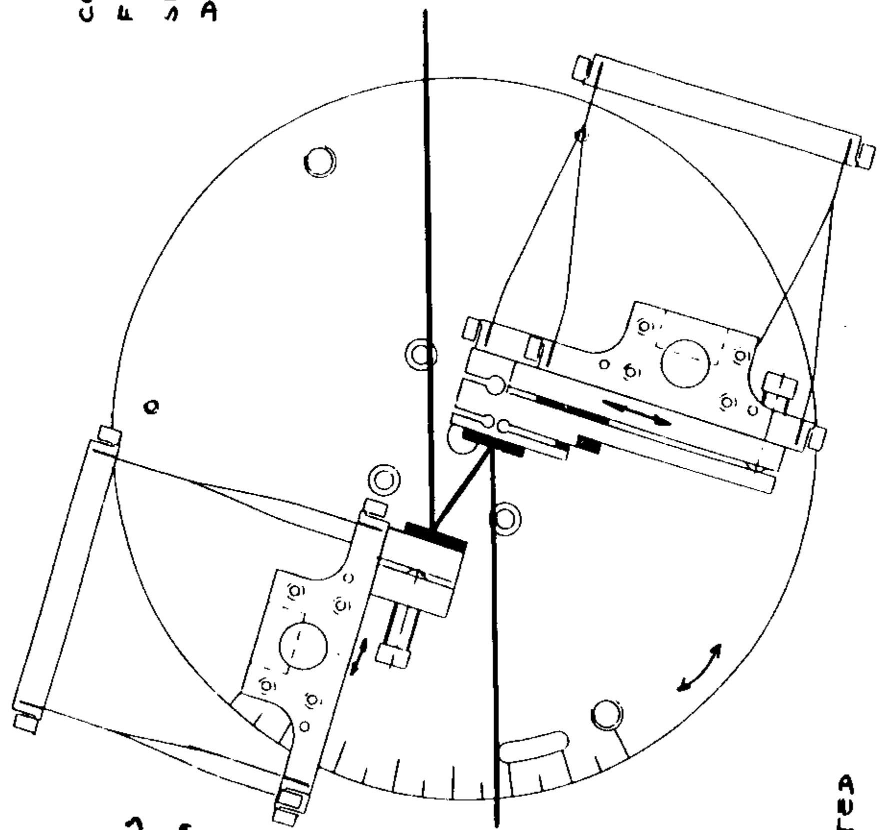
Two Crystal Monochromator
using Elastic Translators

{ 1 ROTATION
2 TRANSLATIONS



TWO-CRYSTAL MONOCROMATOR
WITH ELASTIC TRANSLATORS

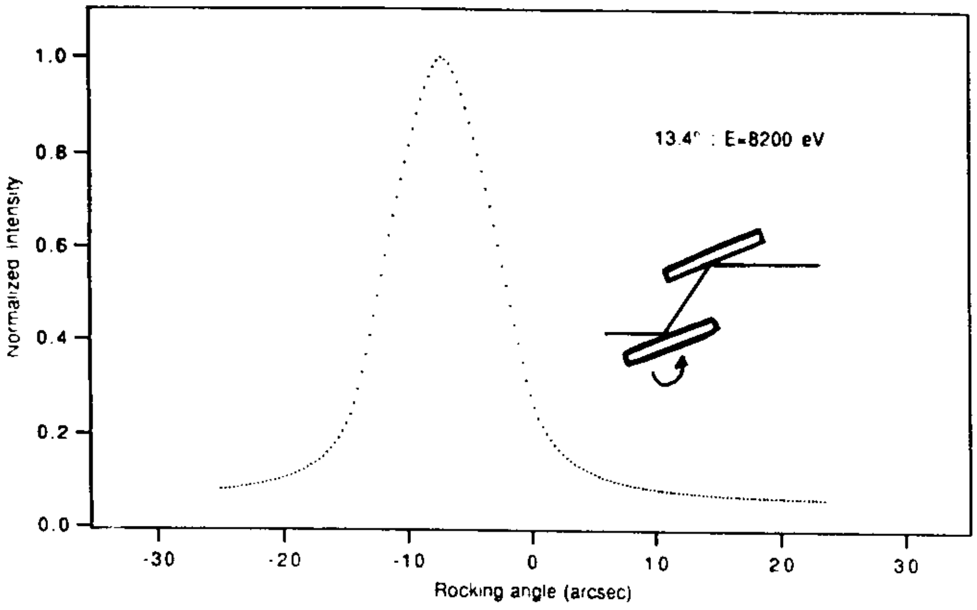
CONSTANT OFF-SET
FIXED BEAM POSITION
SMALL DEAD TIME
DETUNNING

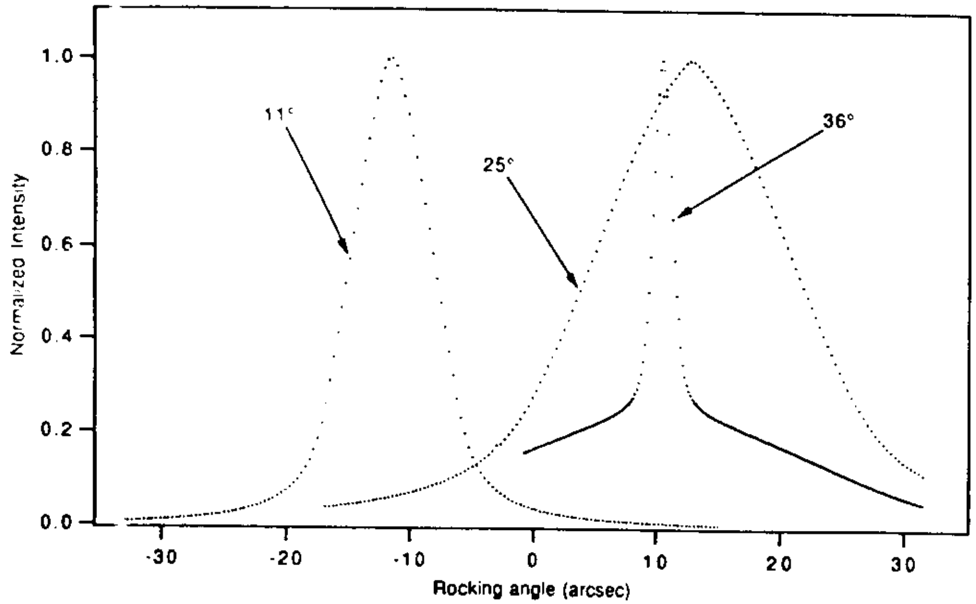


...NG ROTATION
...WO TRANSLATIONS
...LASTIC DETUNNING

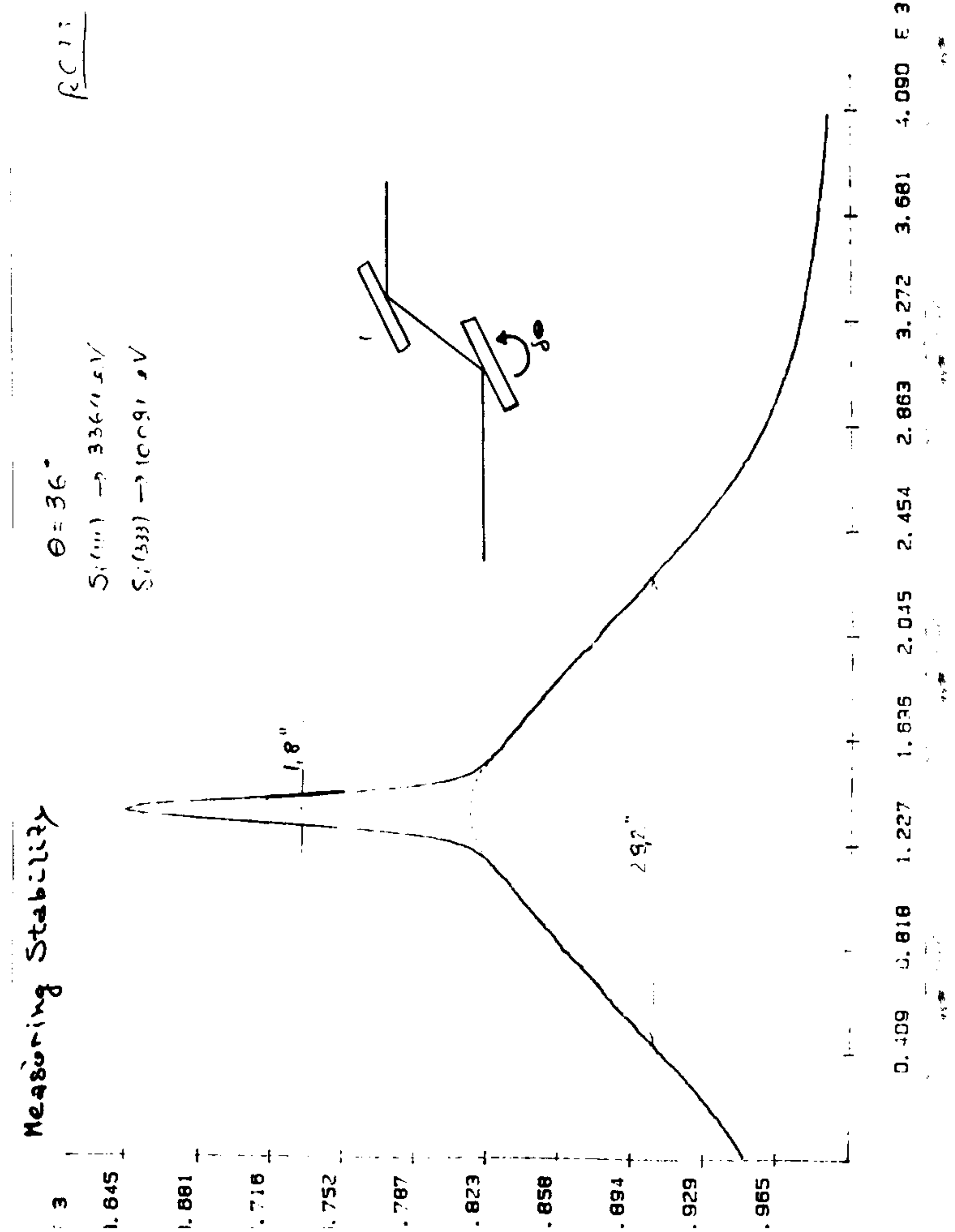
TRANSLATORS
• SMALL ERROR
• REPETITIVE
• DYNAMICALLY CORRECTED

CUSATIS & GILES,
II WORKSHOP SYNC. LIGHT



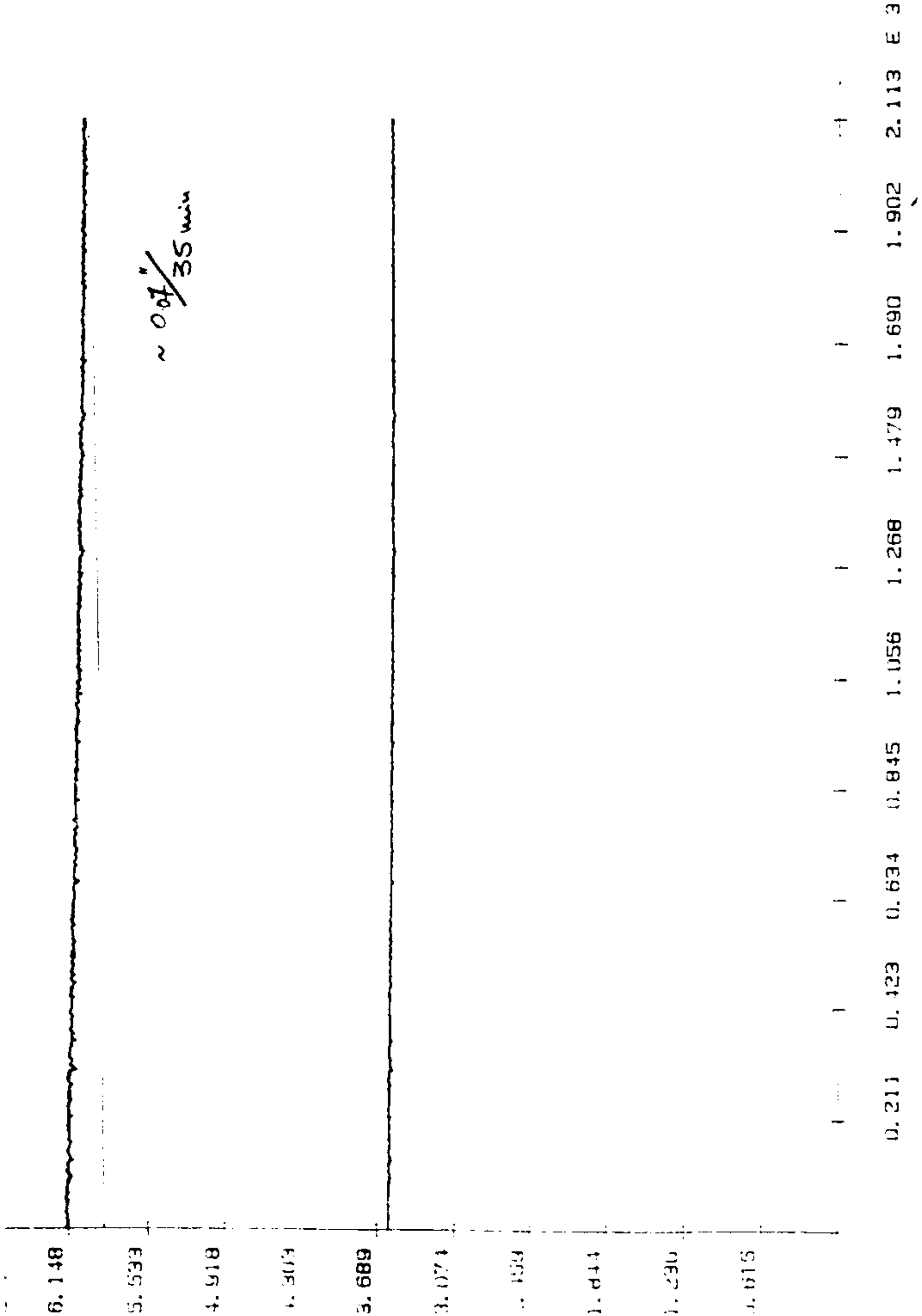


rcdac : rc64, 65, 103



rcdac : rc 51

SRN Vol 4 n°1 (JAN/FEB. 1991)



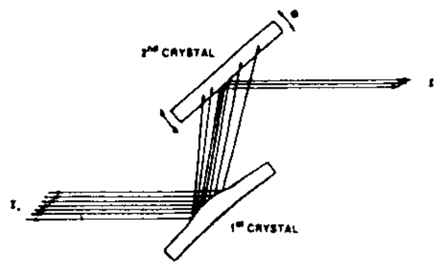


Figure 2 Schematic of the hot optics problem. The thermal bump on the first crystal's surface produced by the powerfulincident beam spreads out the diffracted rays so that they do not simultaneously diffract from the 'cold' second crystal, entailing a loss of throughput

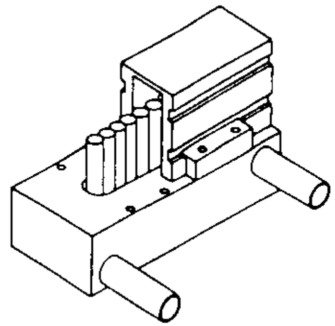


Figure 3 Geometric view of the monochromator and jet cooling manifold

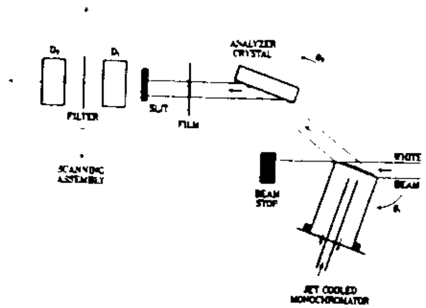


Figure 4 Double crystal experimental arrangement used at X25 tostudy thermal strains in the water cooled crystals Thedouble-crystal diffracted intensity for the fundamental reflection is measured with the ionization chamber D_1, and the first harmonic is measured with D_2

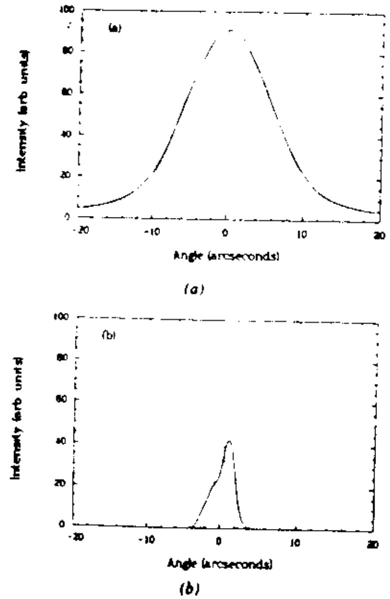


Figure 5 Rocking curves obtained at 175 mA beam current, for (a) the (111) reflection at 6.17 keV and (b) the simultaneous(333) reflection at 18.51 keV. The solid (———) curves are experimental, and the dashed (----) ones were derived from theory

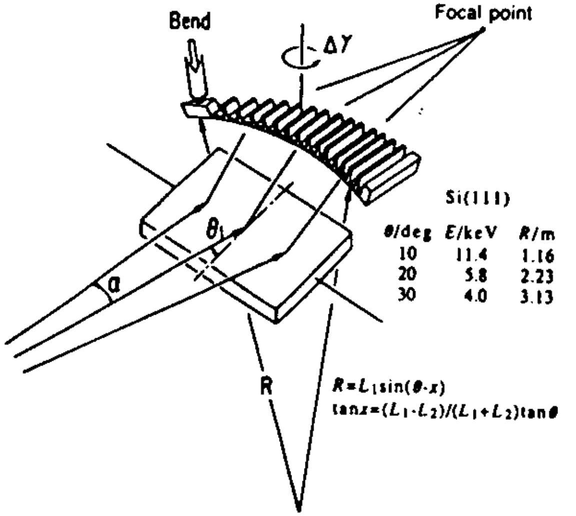
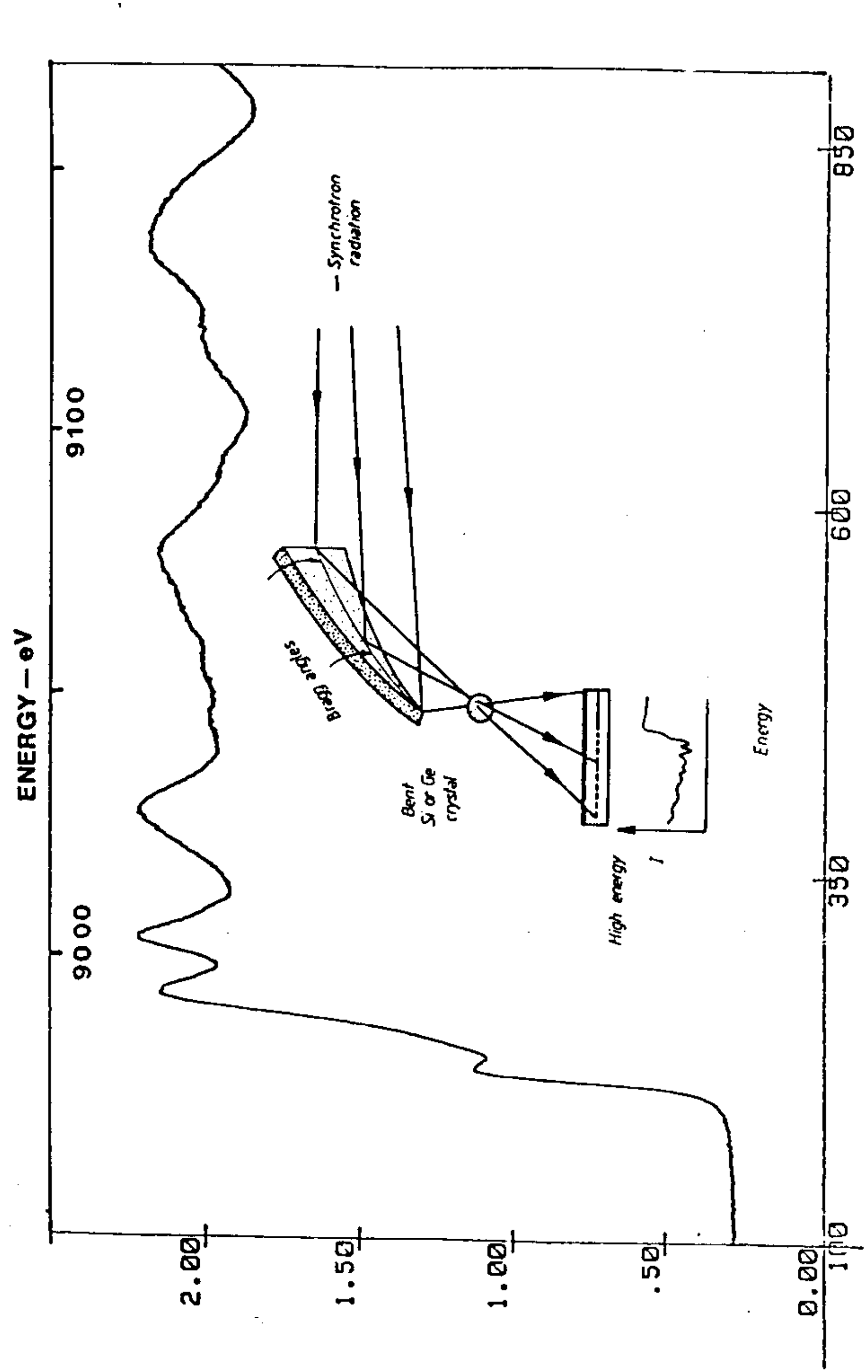
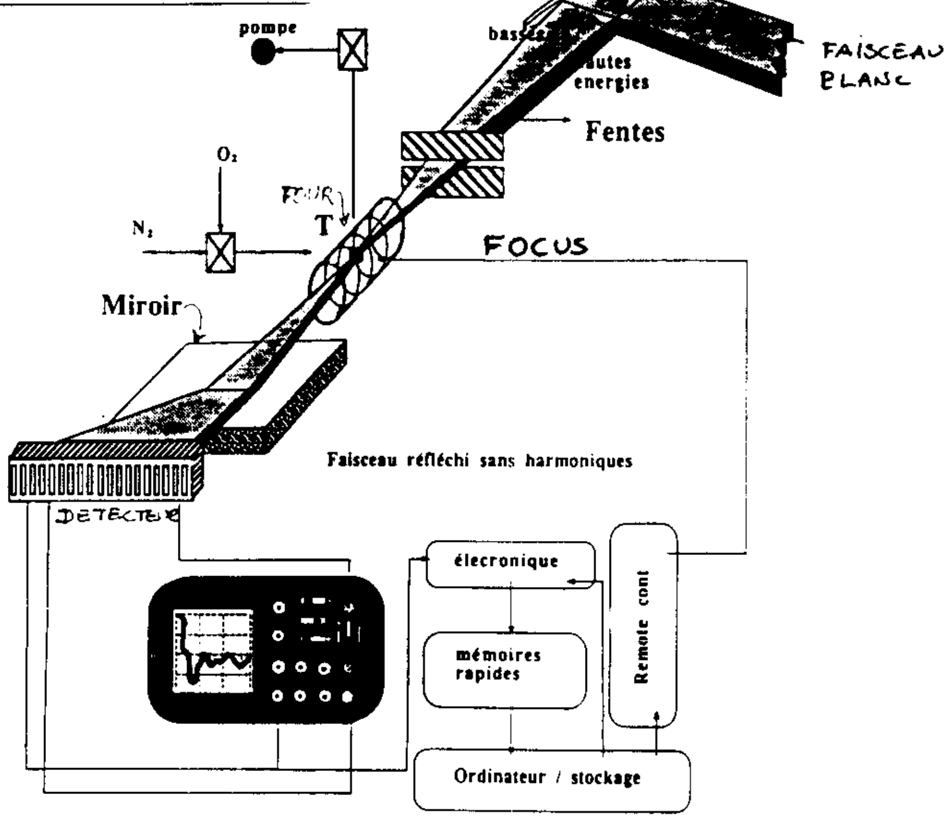


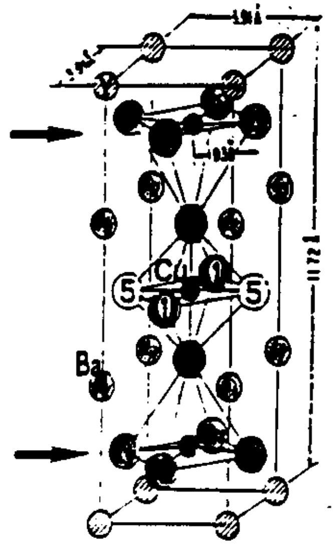
FIG. 2. The sagittally focusing double-crystal monochromator. The second crystal is shaped like an isosceles triangle with some ribs in order to prevent anticlastic bending. The basis of the triangle is secured to a crystal holder and the free end can be displayed by a screw in order to realize optimum sagittal radius.



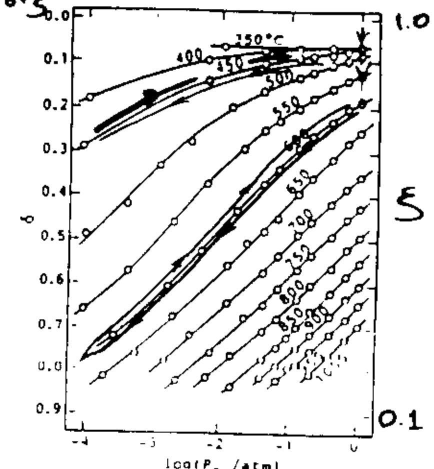
ETUDE In situ de prise d'oxygène

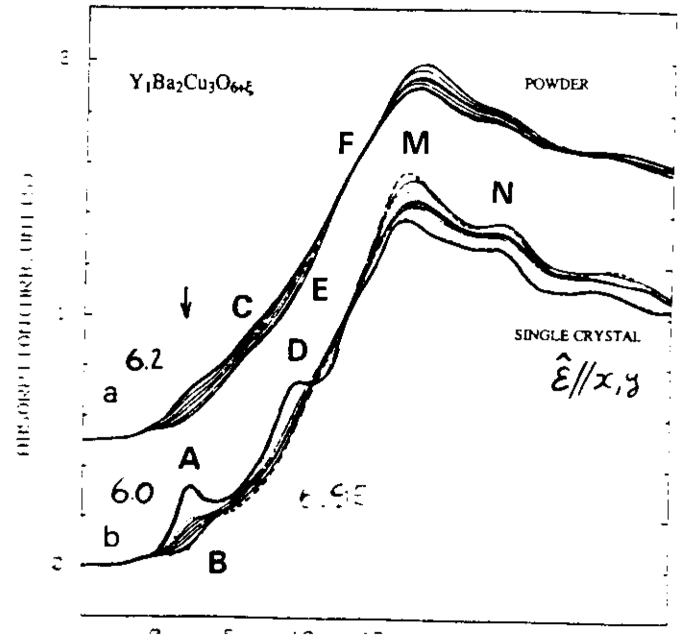


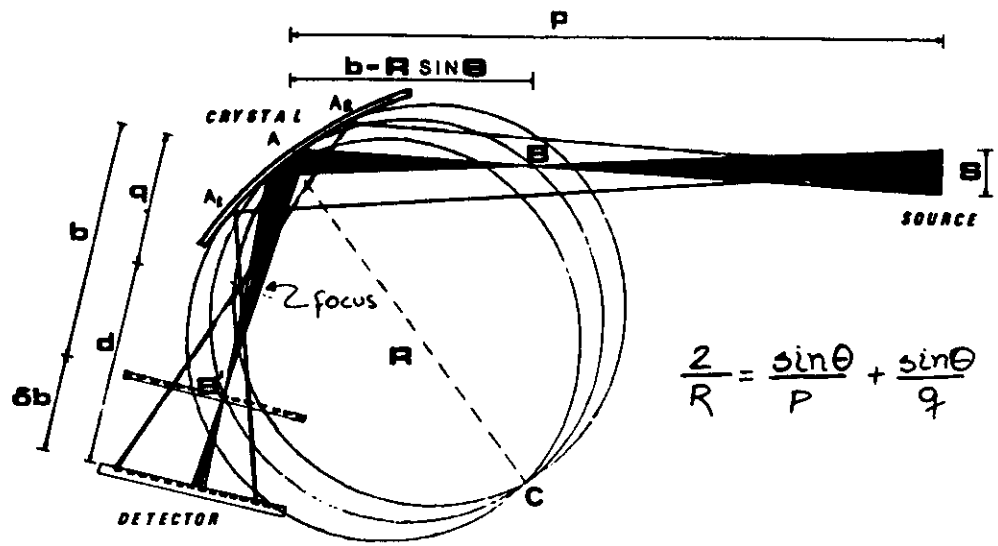
Description schématique
du spectromètre d'Exafs Dispersif



$YBa_2Cu_3O_{6+\xi}$ T , pO_2







$$\frac{2}{R} = \frac{\sin\theta}{p} + \frac{\sin\theta}{q}$$

Resolution

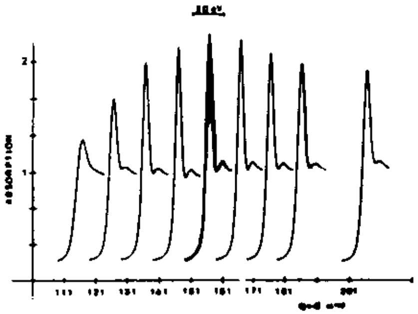
$\delta E = E \cot\Theta \, \delta\Theta$

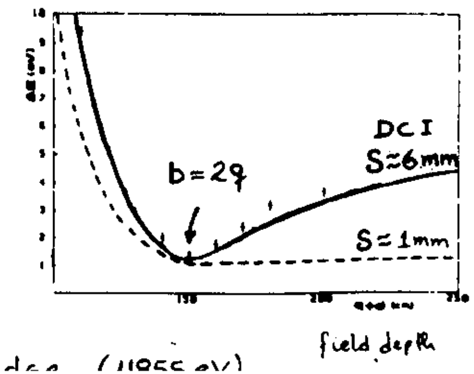
- detection — pixel, distance
- optics — Ω
- source — Rowland circle

$p \gg q \Rightarrow b \approx 2q$

H.Tolentino et al. JAC (1987)

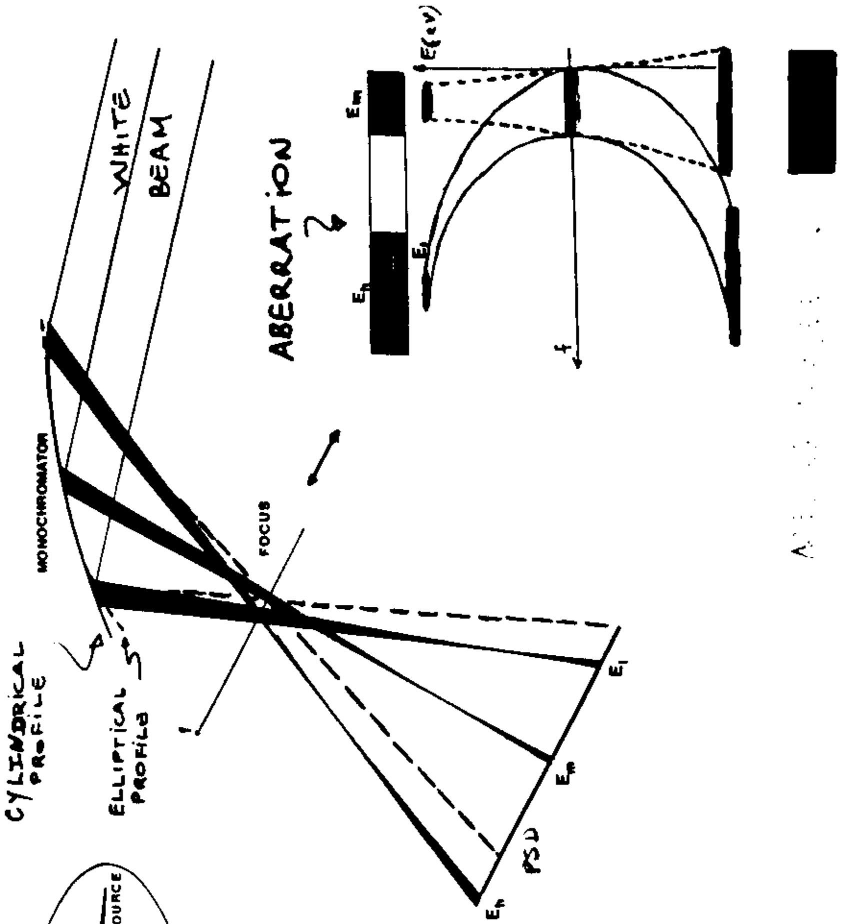
Resolution x detector position

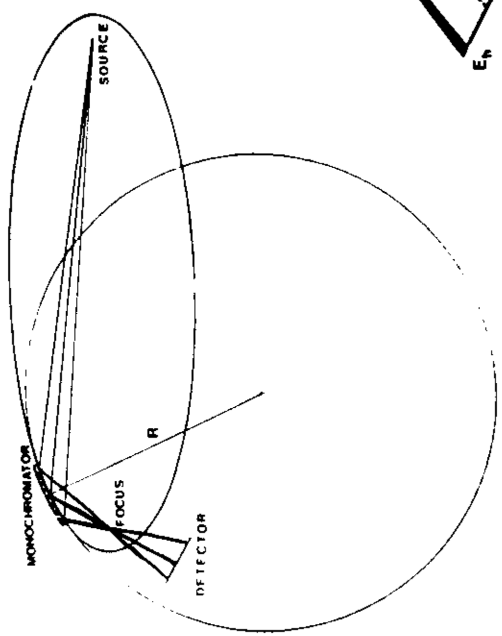




Si 311 + As K edge (11855 eV)

OPTICAL ABERRATION





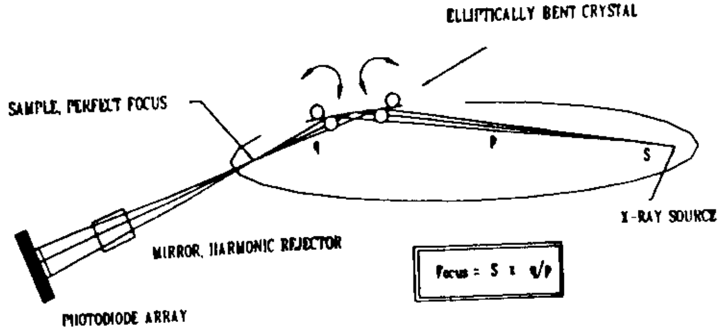
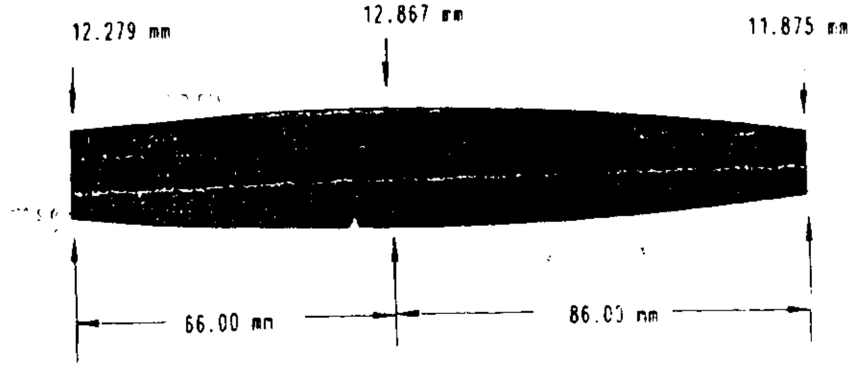
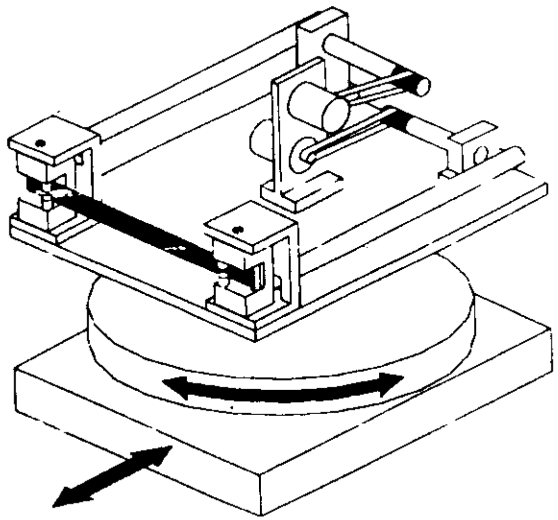
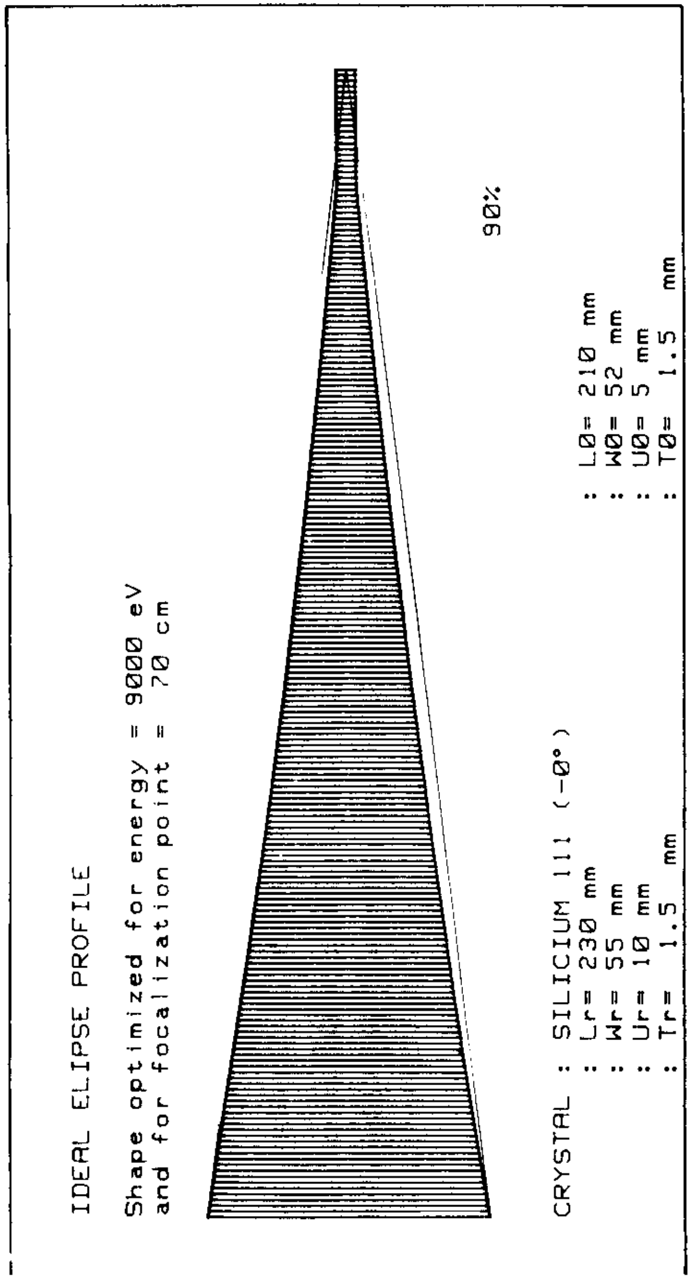


Figure 1. Schematic of dispersive XAFS geometry and components. The ideal optics requires that the source and focus be located at the foci of an ellipse. The crystal is bent to match an arc of the ellipse by a four-point bender as indicated.







$$\omega(x) = \frac{W}{2}\left(1-\frac{2x}{L}\right)\Phi(x) \simeq \frac{W}{2}\left(1-\frac{2x}{L}\right)\left(\frac{1-Kx}{1+KL/2}\right)$$

linear correction

FOUR - CRYSTAL MONOCHROMATOR

TOLENTINO & RODRIGUES
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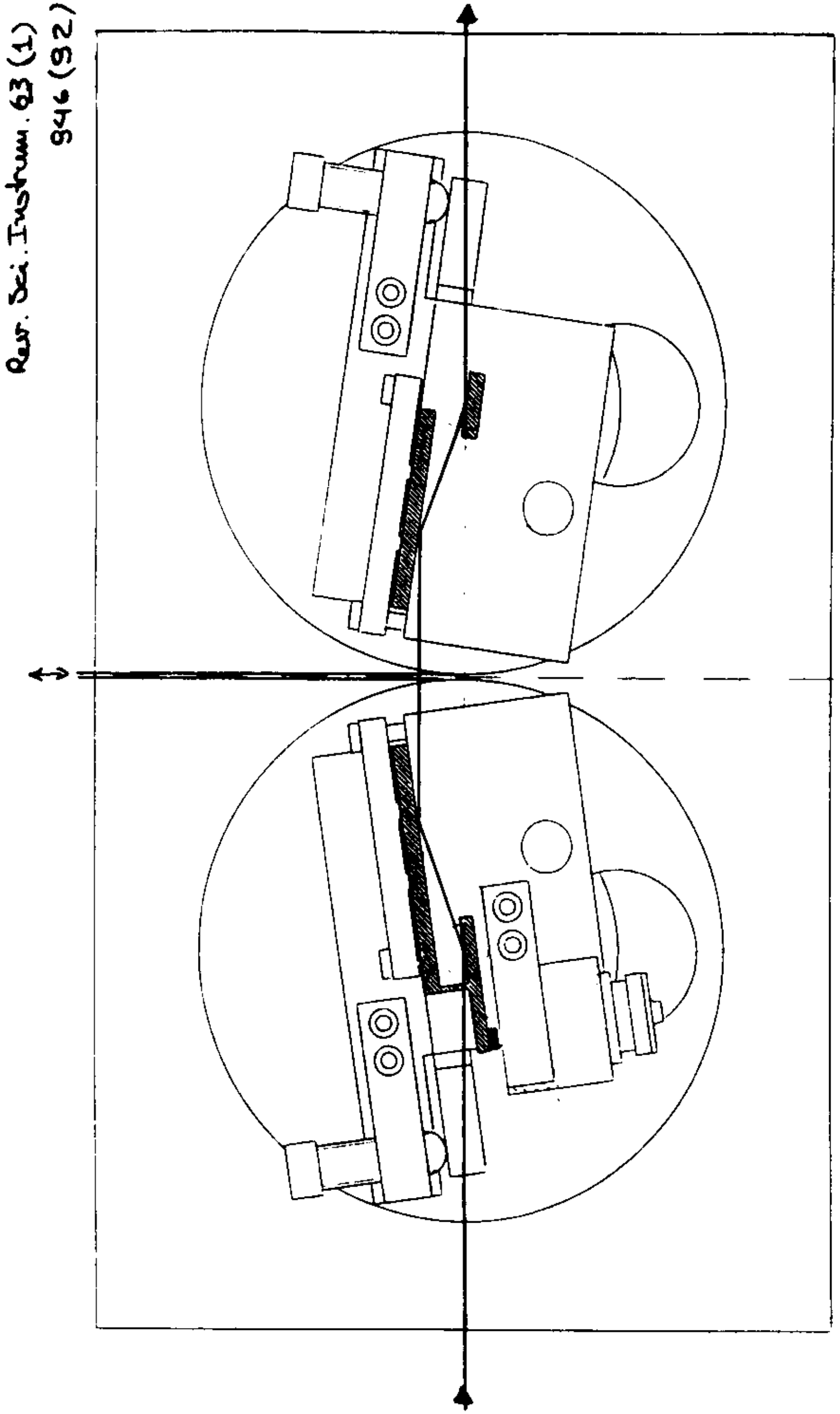
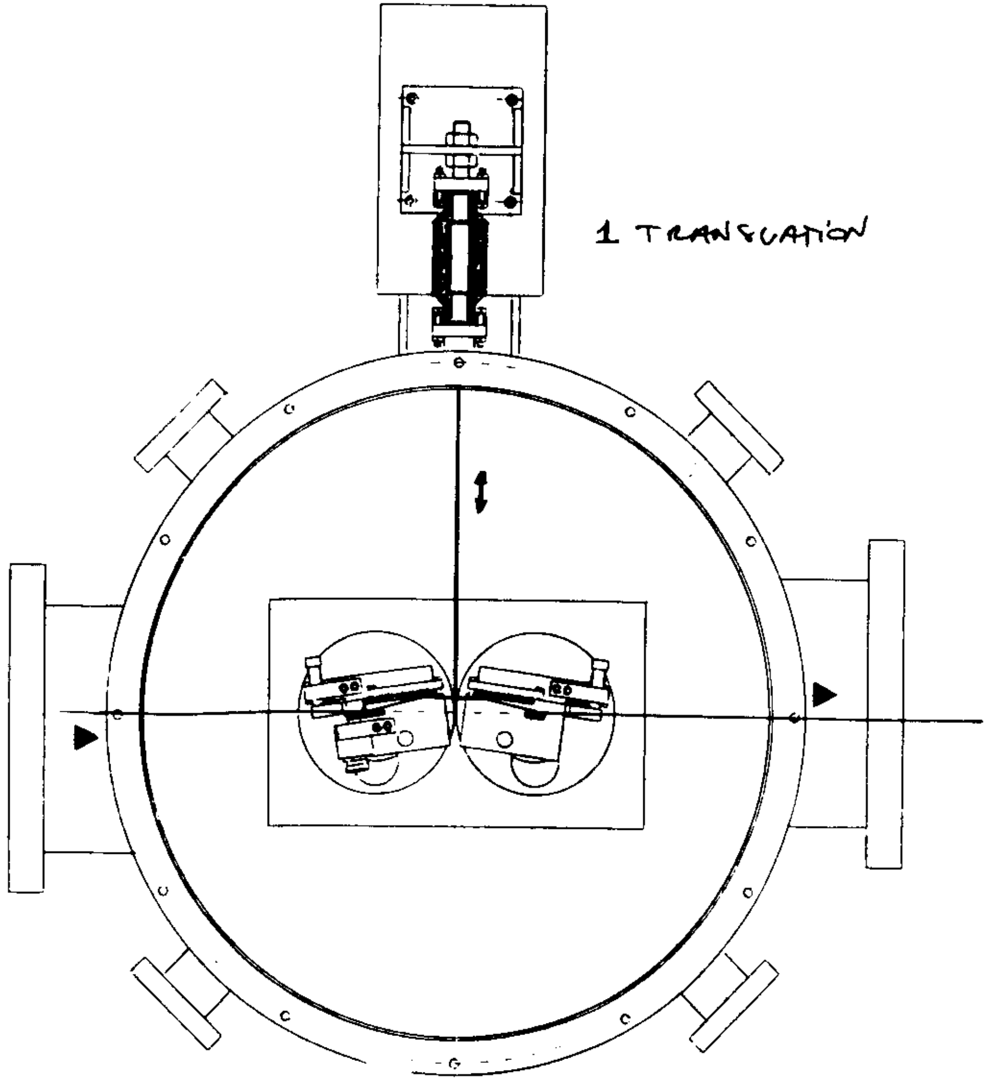
FOUR CRYSTAL

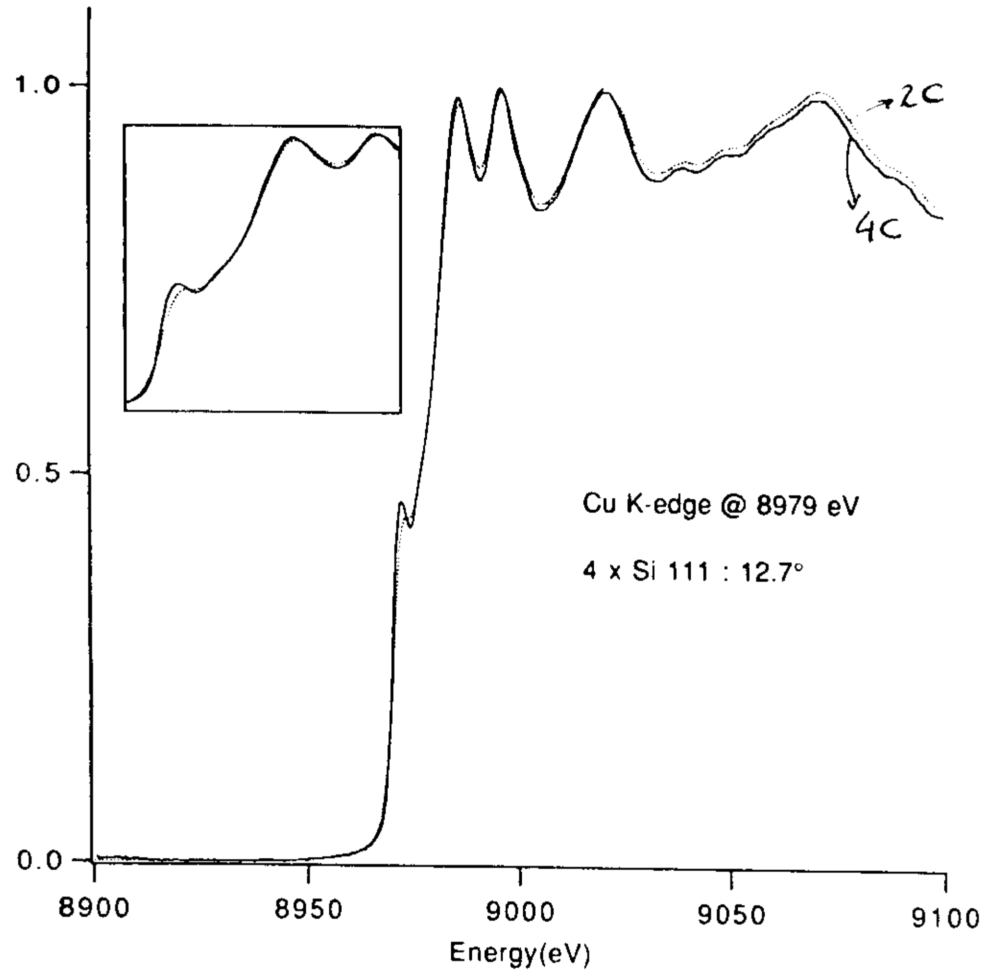
$\Delta\theta = \frac{\Delta X}{R}$

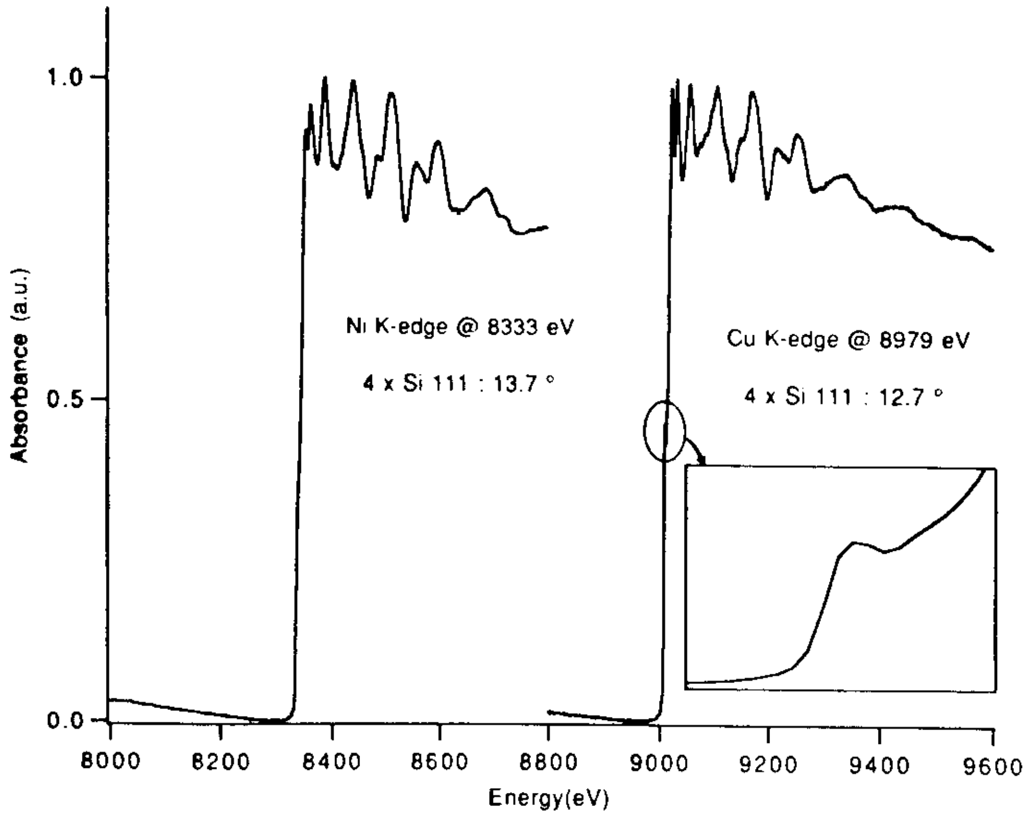




1

4-CRYSTAL MON.





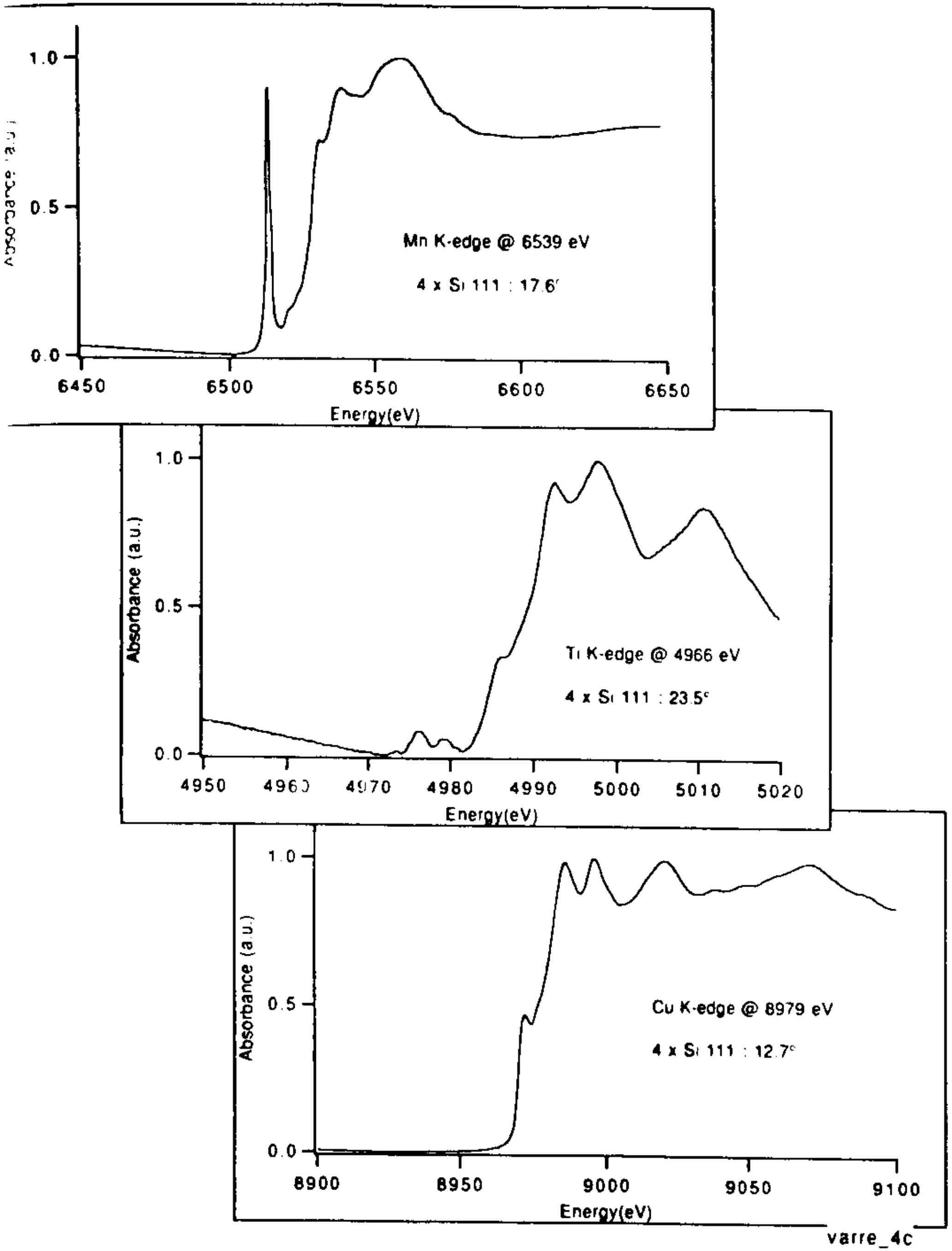
$\alpha \approx 1.6 \times 10^{-4}$

$\Omega \approx 6'' \approx 3 \times 10^{-5}$

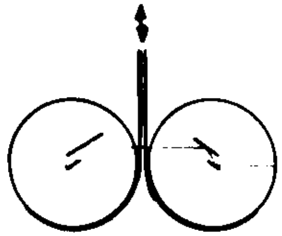
$\frac{\alpha}{\Omega} \approx 5$

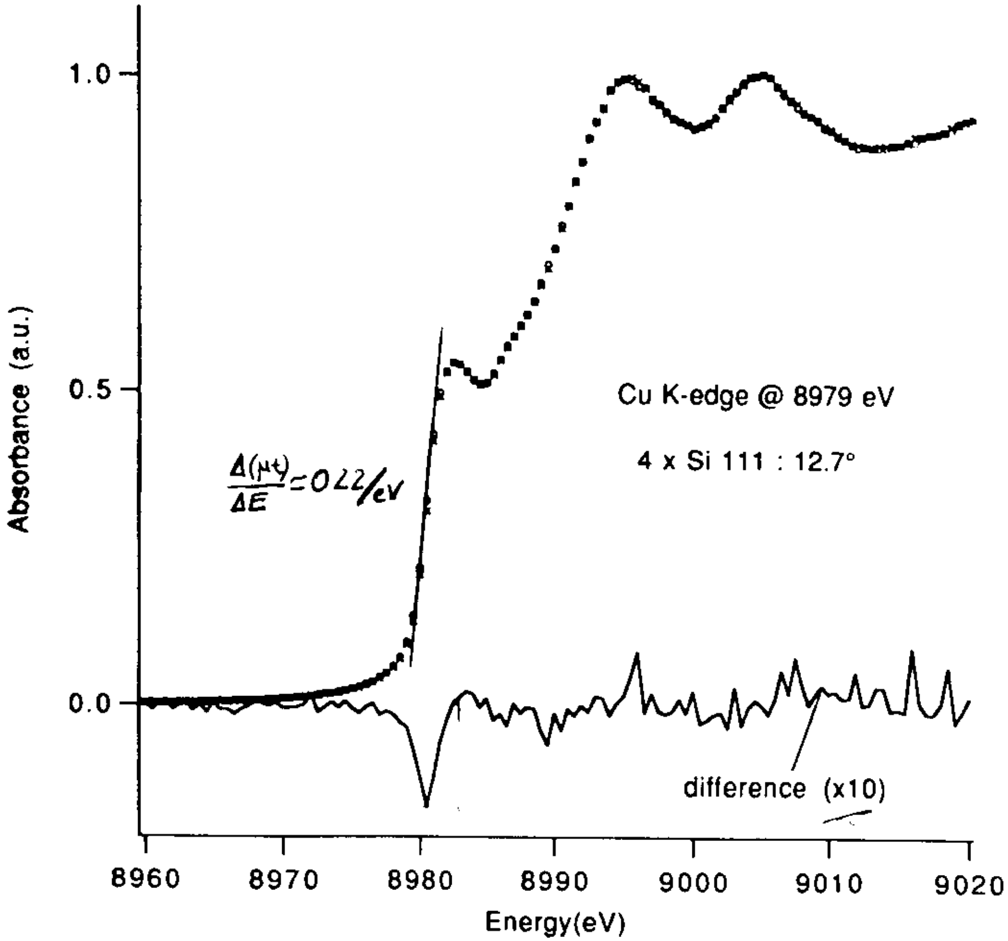
$\frac{2C}{4C} \sim 5 \rightarrow 6$

RESOLUTION



POSITION.



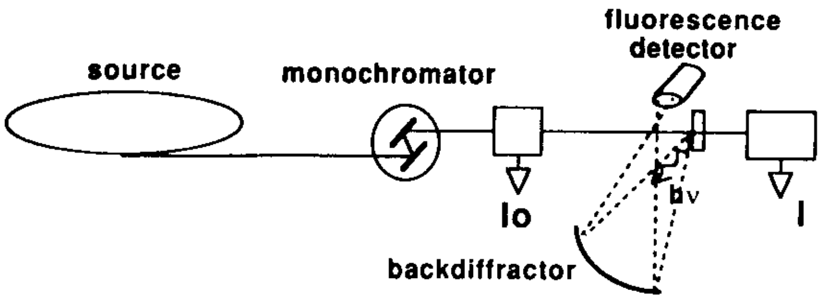


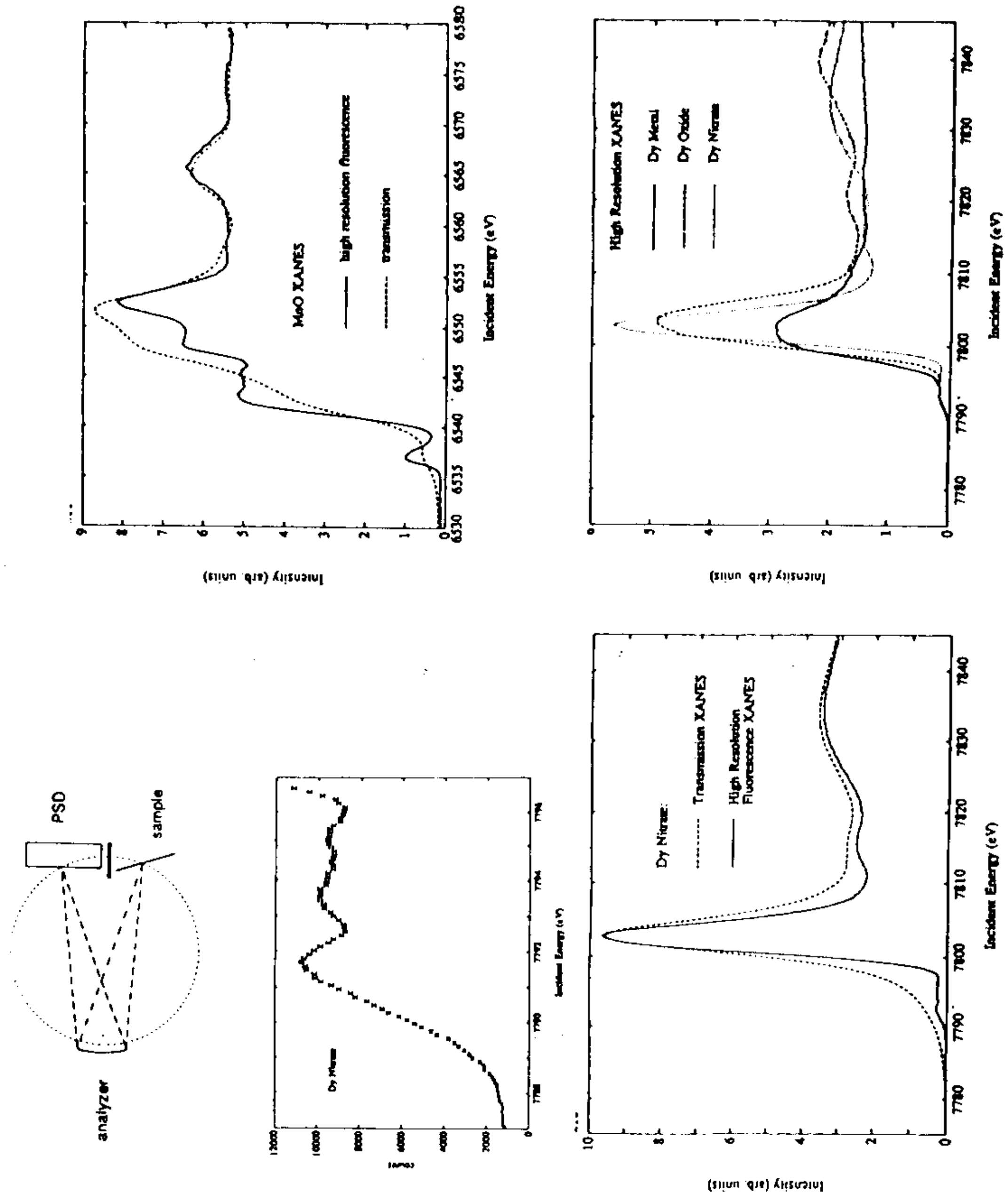
$\Rightarrow \delta E = 0.074\ eV$

$\frac{\Delta E}{E} = 8 \times 10^{-6}$; $\Delta\theta = 2 \times 10^{-6}$

XAS set up for

High Resolution Fluorescence Detection





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Section III. Beamline instrumentation: (g) Miscellaneous

PHASE-PLATE PERFORMANCE FOR THE PRODUCTION OF CIRCULARLY POLARIZED X-RAYS

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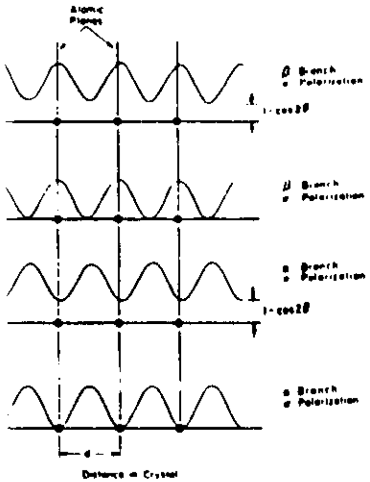
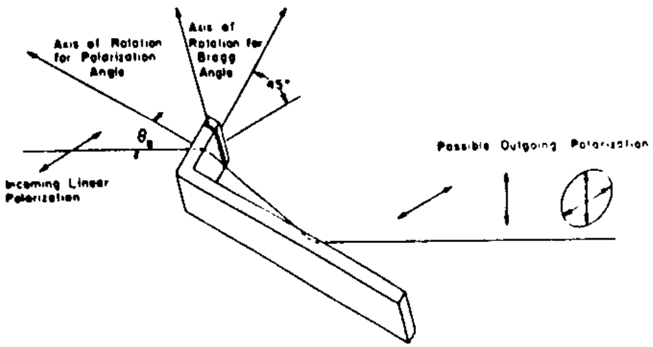
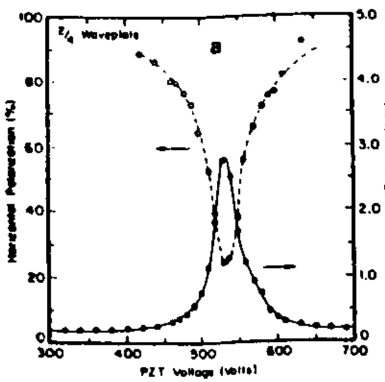
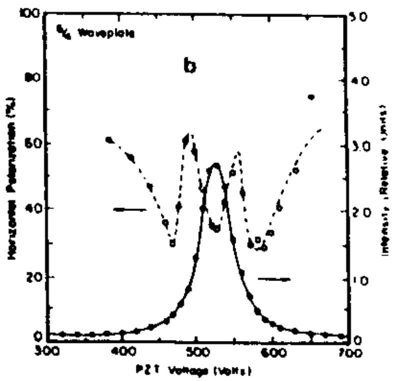








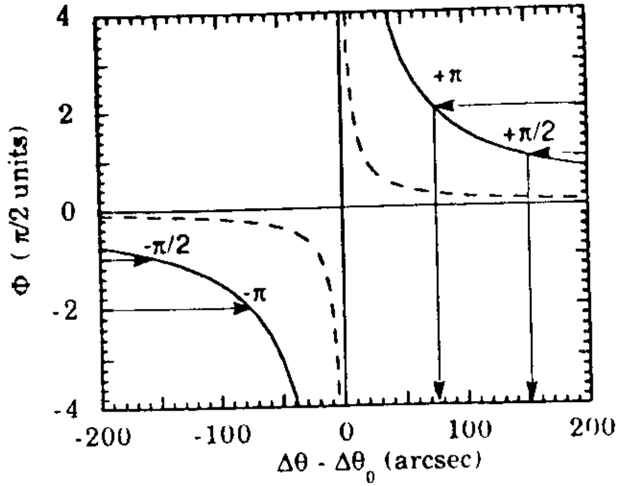


Figure 2. Calculated phase difference Φ between the σ and π components of the transmitted wave as a function of the angular offset defined in text. Calculations were done with λ = 1.712 Å. Full line (111) diamond crystal of thickness t_0 = 930 μm, (220) reflection and effective beam path t = 1680 μm. Dotted line (110) silicon crystal of thickness t_0 = 62 μm, (220) reflection and effective beam path t = 155 μm

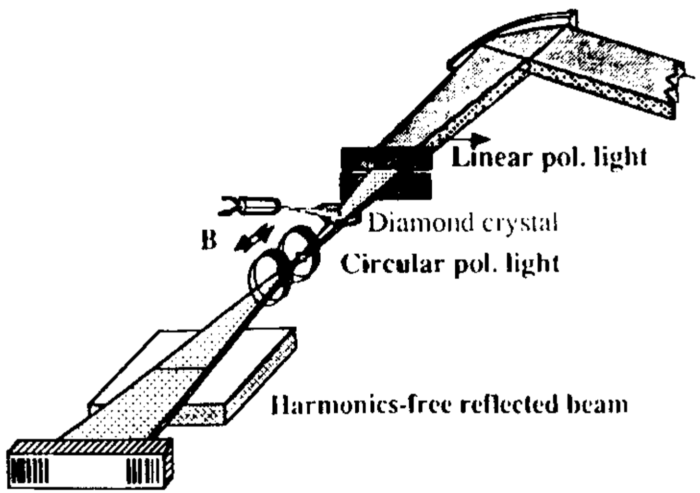
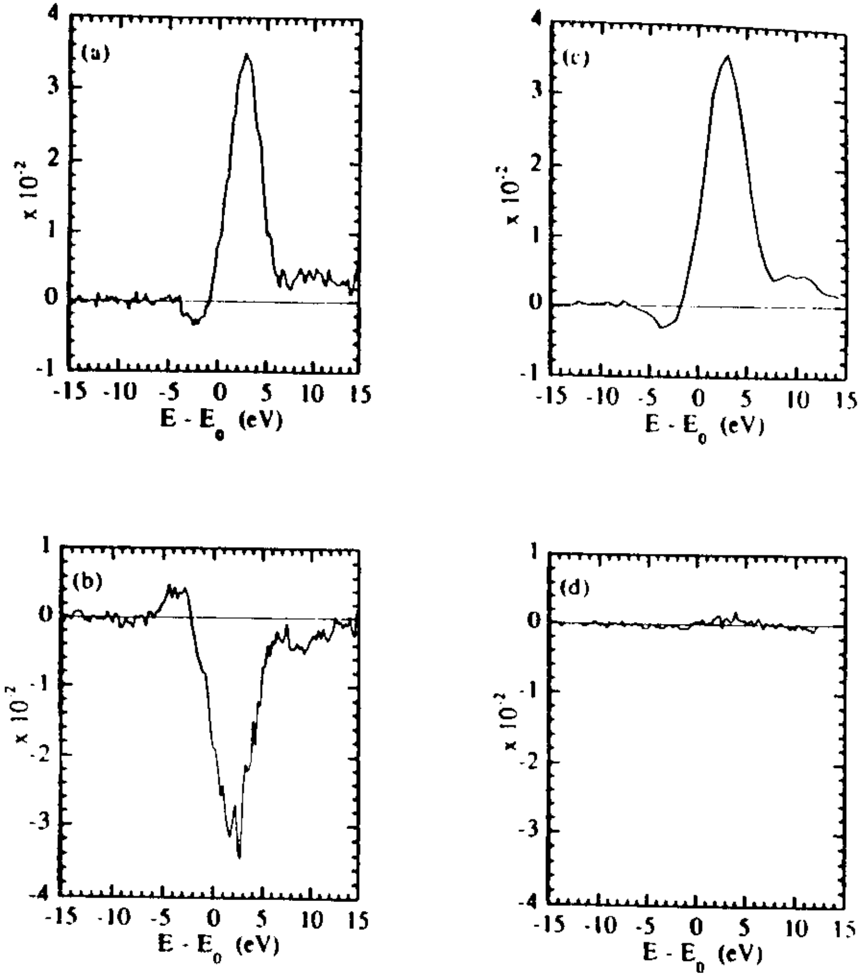
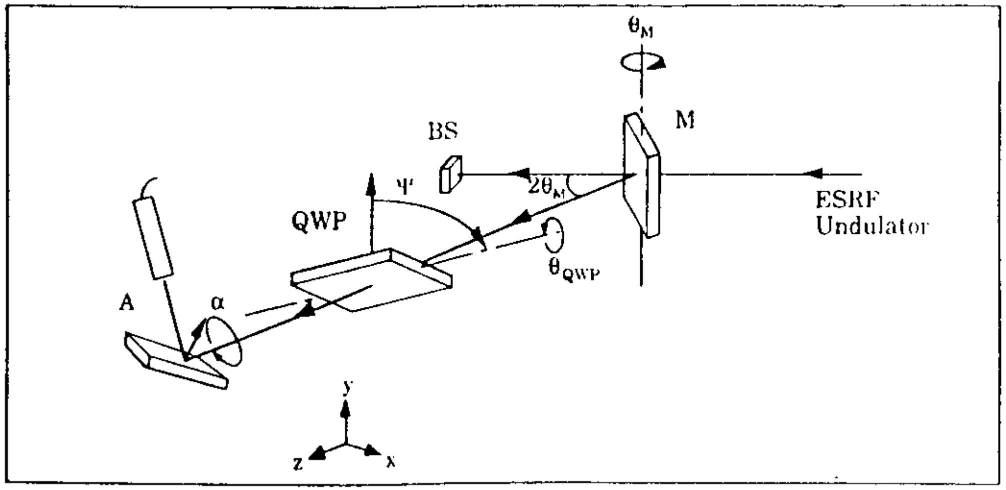


Fig. 3 Sketch of the dispersive x-ray absorption spectrometer at DCI (LURE). A diamond quarter wave plate with a diffraction plane rotated by 45° with respect to the horizontal plane produces circular polarization.





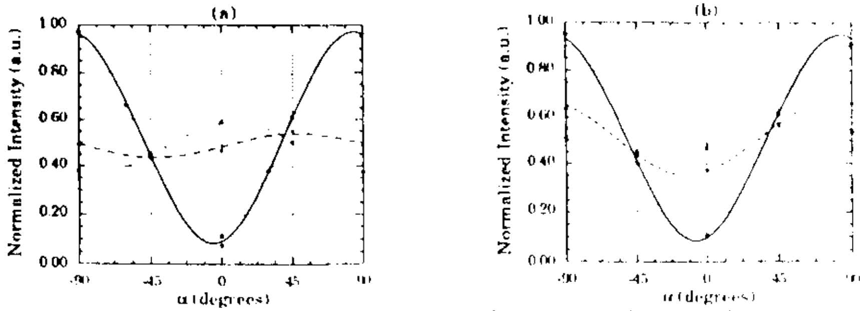


Fig. 8 Normalized integrated intensities from an Al 311 powder diffraction line as a function of the azimuthal angle α for three different positive and negative values of the angular offset from the center of the reflection profile. a) plain line: offset=-105 arcsec, P1= 0.83 ± 0.01; dashed line: offset= 170 [illegible], P1=-0.01 ± 0.03, dotted line: offset=-195 arcsec, P1=0.22 ± 0.01, b) plain line: offset 90 arcsec, P1 [illegible] 0.05; dashed line: offset=160 arcsec, P1=-0.28 ± 0.03; dotted line: offset=180 arcsec, P1=-0.01 [illegible]