X-ray Crystallography II

Scattering by X-rays

- Two types of scattering
  - Elastic - incident and scattered radiation is the same wavelength
  - Inelastic - incident and scattered radiation is not the same wavelength
    - Fluorescence occurs
- Intensity of scattered radiation is

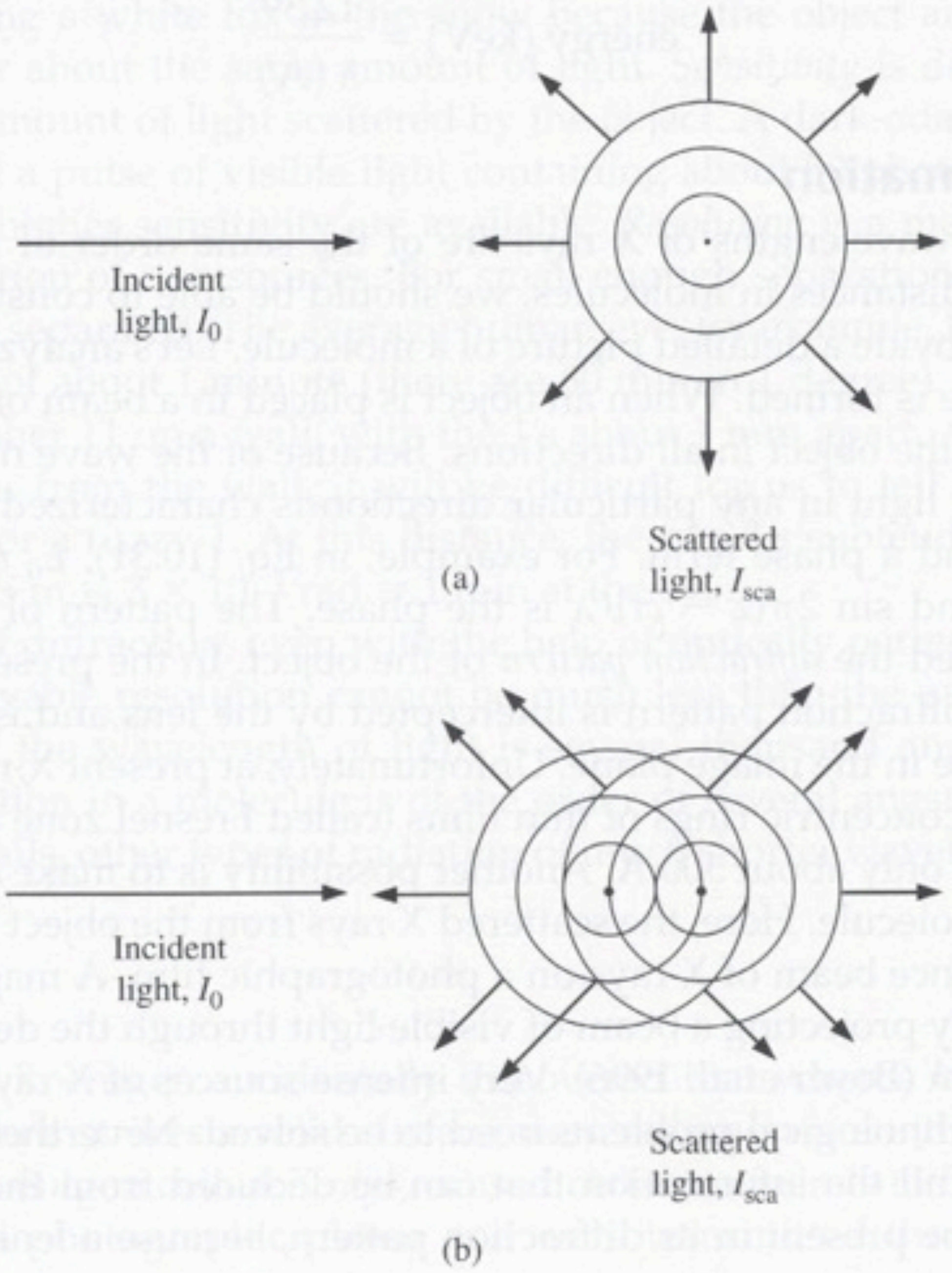
$I_{sca}=(CI_o)/r^2$ Where I_{sca} is scattered Intensity

I_o is incident intensity

r distance from scattering point to detector

C proportionality factor

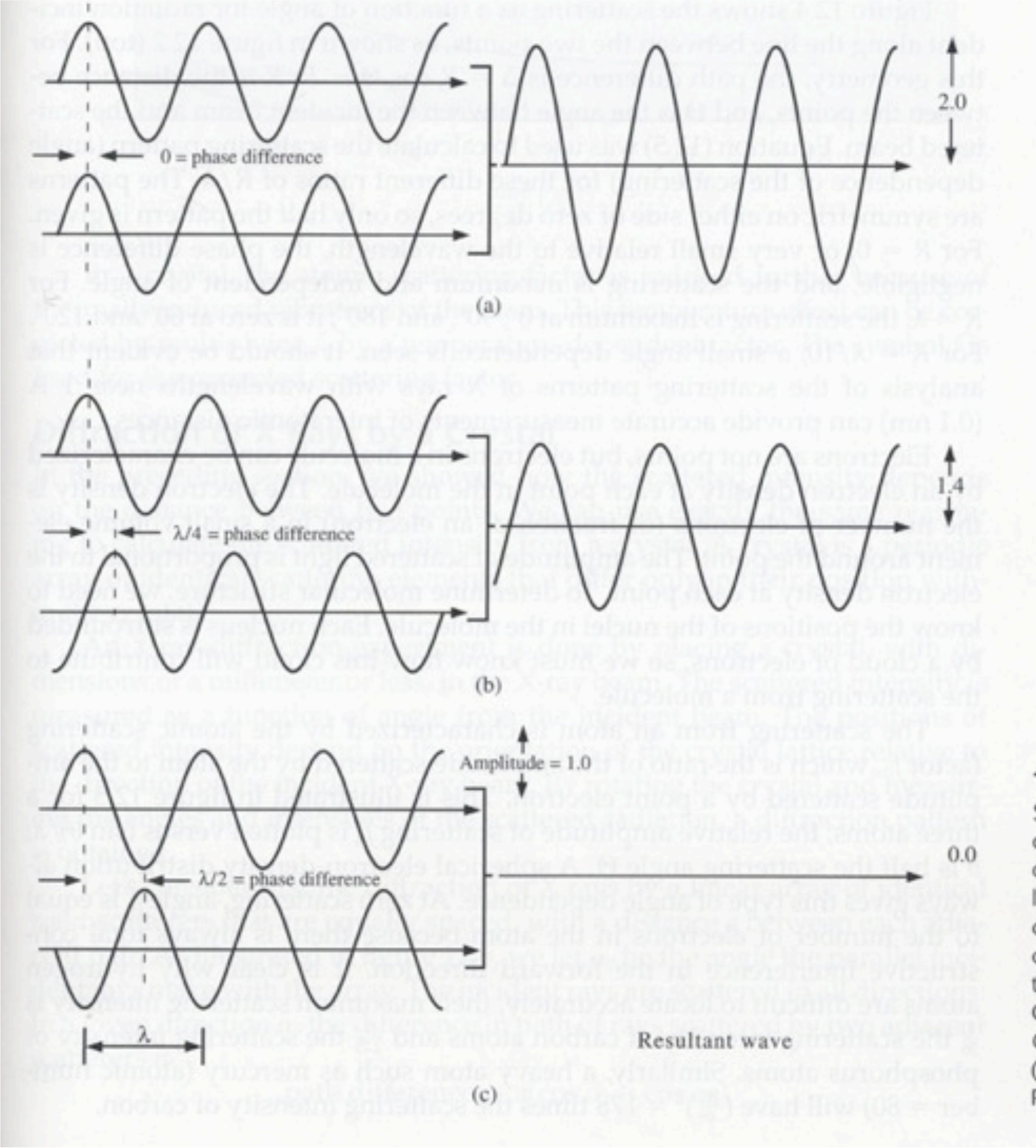
Scattering by Two Electrons



▶ **FIGURE 12.1**

(a) The scattering from a single electron for light polarized perpendicular to the scattering plane is independent of angle in the scattering plane. The scattering intensity decreases inversely proportional to the square of the distance from the scattering electron. (b) For scattering from two electrons, the angle dependence of the scattering amplitude depends on the interference between the two scattered waves.

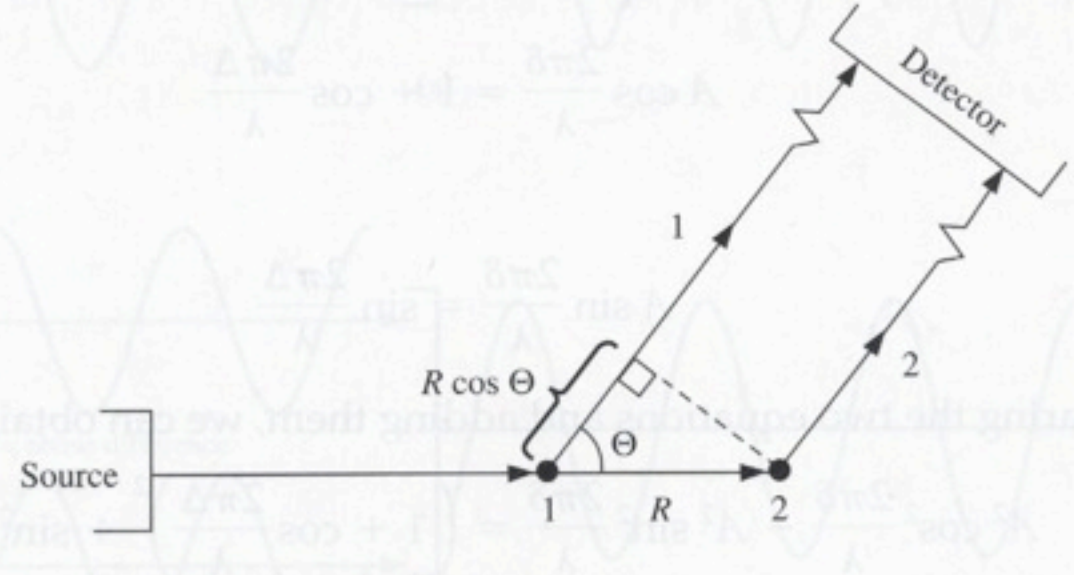
Summing of Waves



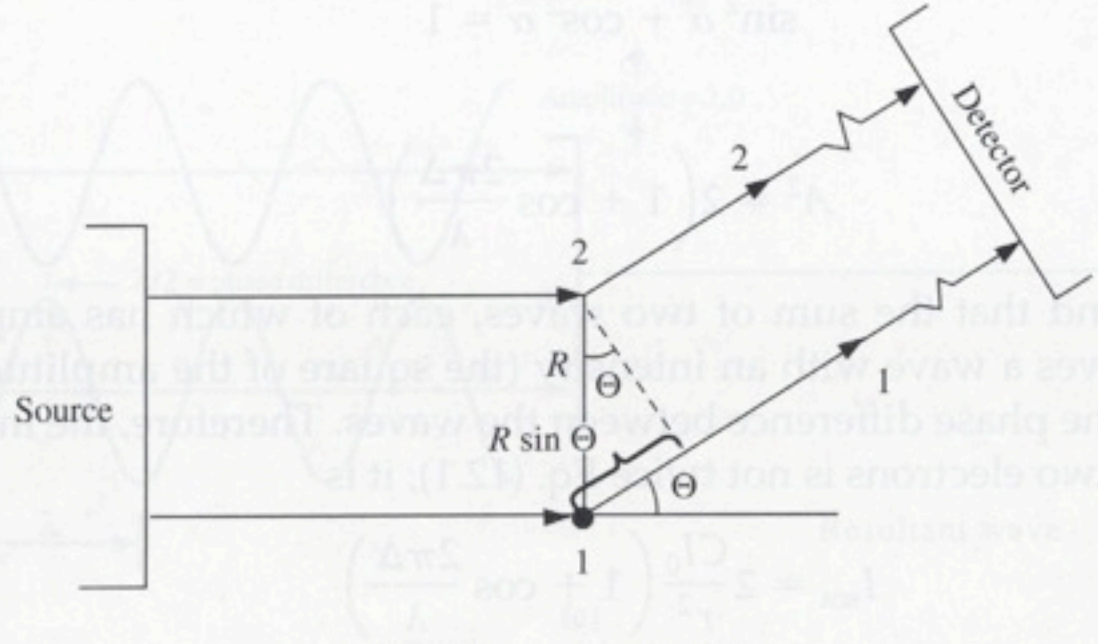
◀ **FIGURE 12.3**

Summing of two waves: (a) waves completely in phase; (b) waves out of phase by one-quarter wavelength (partial reinforcement occurs); (c) waves out of phase by one-half wavelength (total destructive interference occurs). [From J. P. Glusker and K. N. Trueblood, 1972, *Crystal Structure Analysis: A Primer* (New York: Oxford University Press), p. 19, fig. 5.]

Path Differences



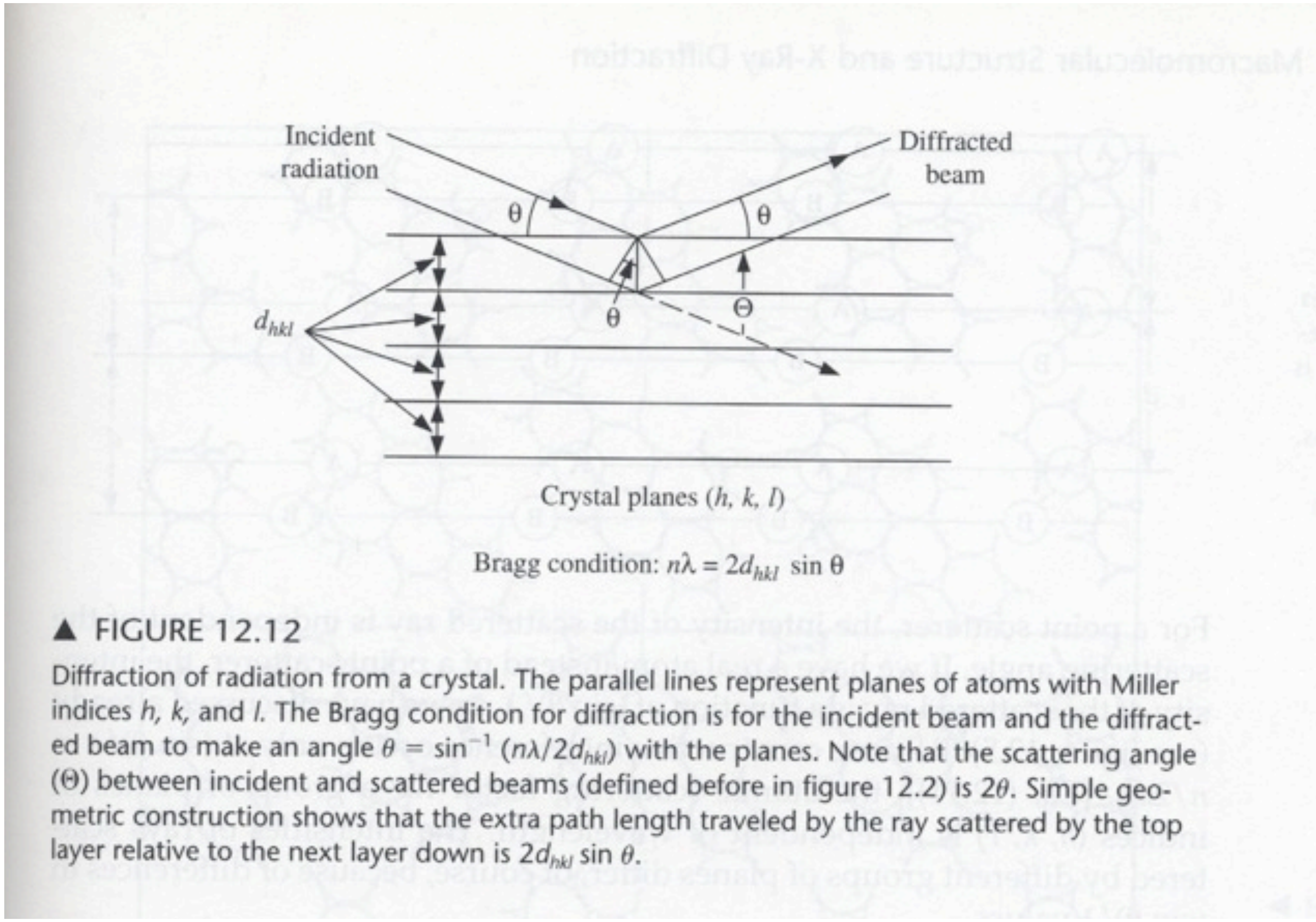
Path difference $\Delta = R\cos\Theta - R$



Path difference $\Delta = R\sin\Theta$

◀ FIGURE 12.2
Path difference for two beams scattered from two electrons separated by distance *R*. The angle between the incident beam and the scattered beam is Θ. At the top, the two scatterers are on a line with the incident beam; at the bottom, the scatterers are perpendicular to the incident beam direction. Trigonometry shows that the path difference Δ is related to the scattering angle Θ and the distance between scattering points *R*.

Bragg's Law

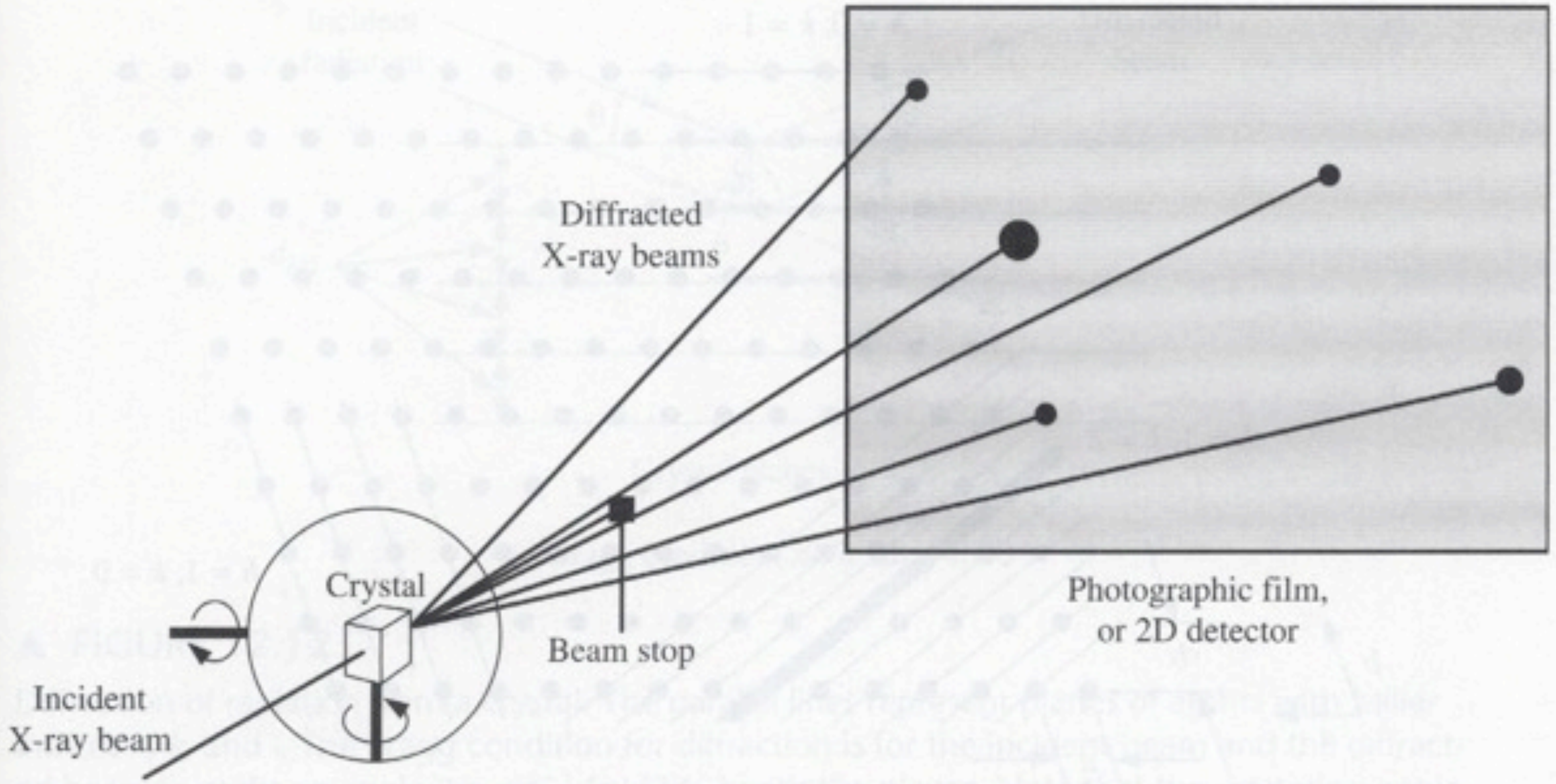


Bragg condition: $n\lambda = 2d_{hkl} \sin\theta$

▲ FIGURE 12.12

Diffraction of radiation from a crystal. The parallel lines represent planes of atoms with Miller indices *h, k,* and *l*. The Bragg condition for diffraction is for the incident beam and the diffracted beam to make an angle $\theta = \sin^{-1}(n\lambda/2d_{hkl})$ with the planes. Note that the scattering angle (Θ) between incident and scattered beams (defined before in figure 12.2) is 2θ. Simple geometric construction shows that the extra path length traveled by the ray scattered by the top layer relative to the next layer down is $2d_{hkl} \sin\theta$.

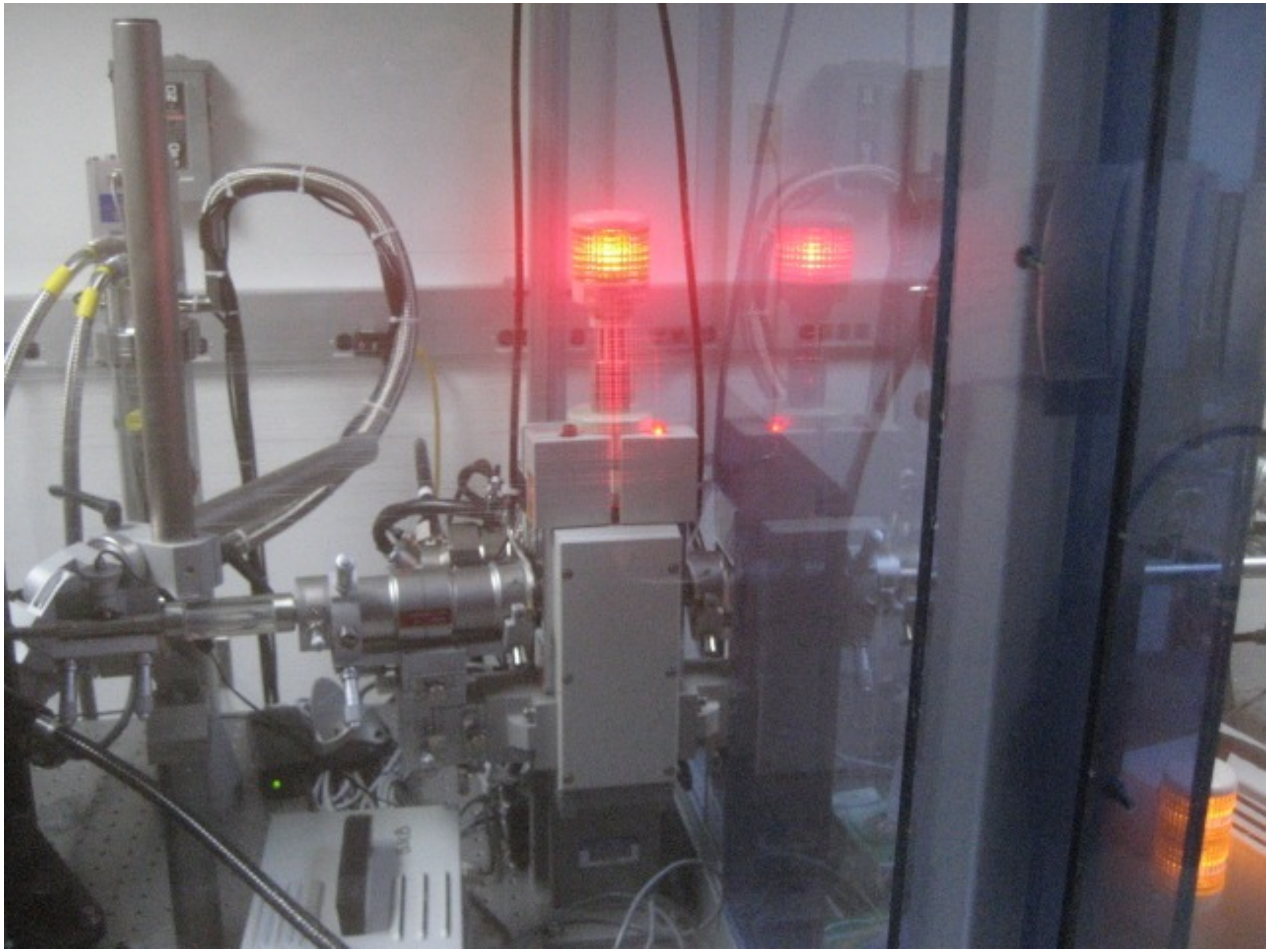
Experimental Setup

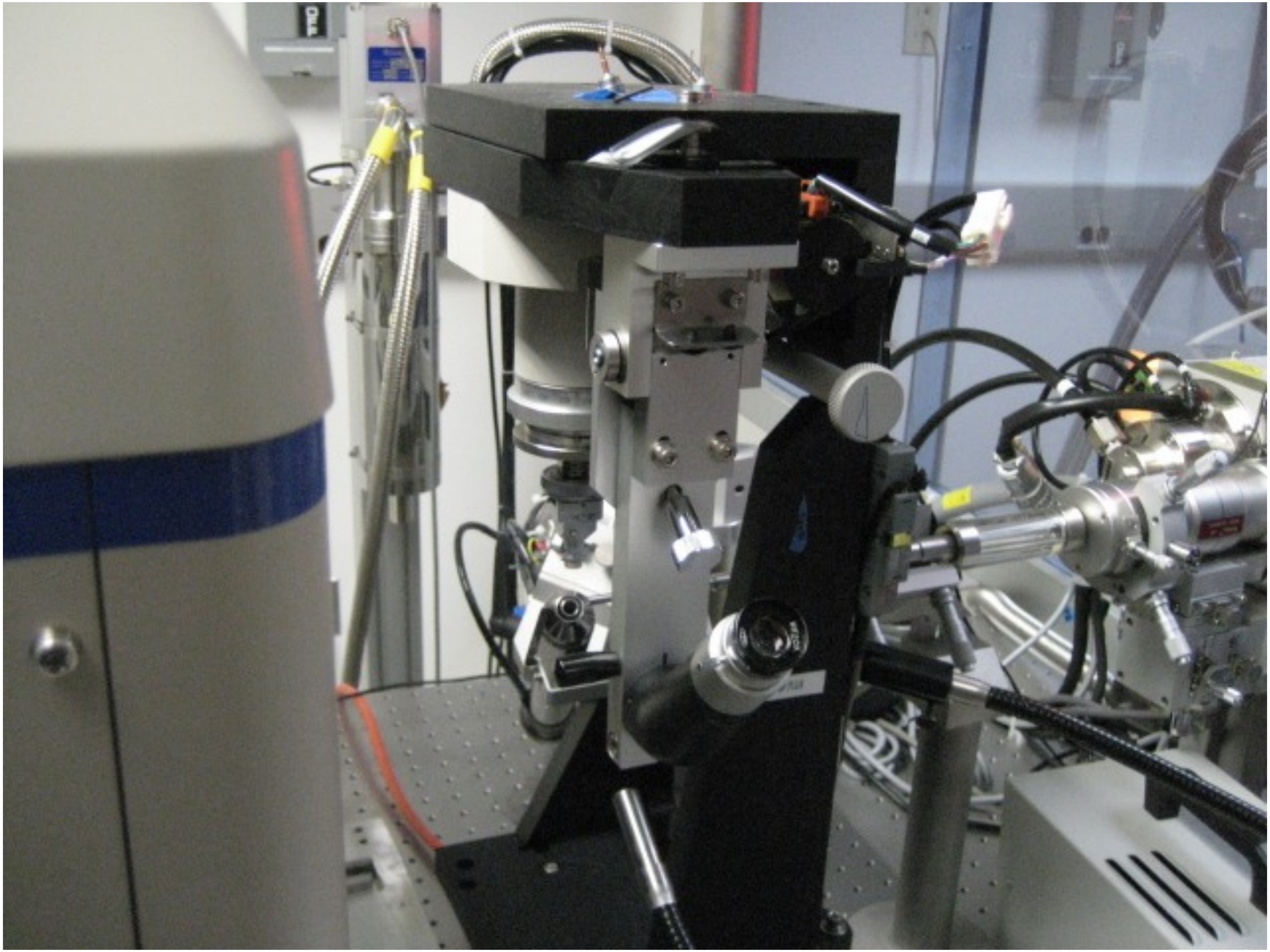


▲ FIGURE 12.10

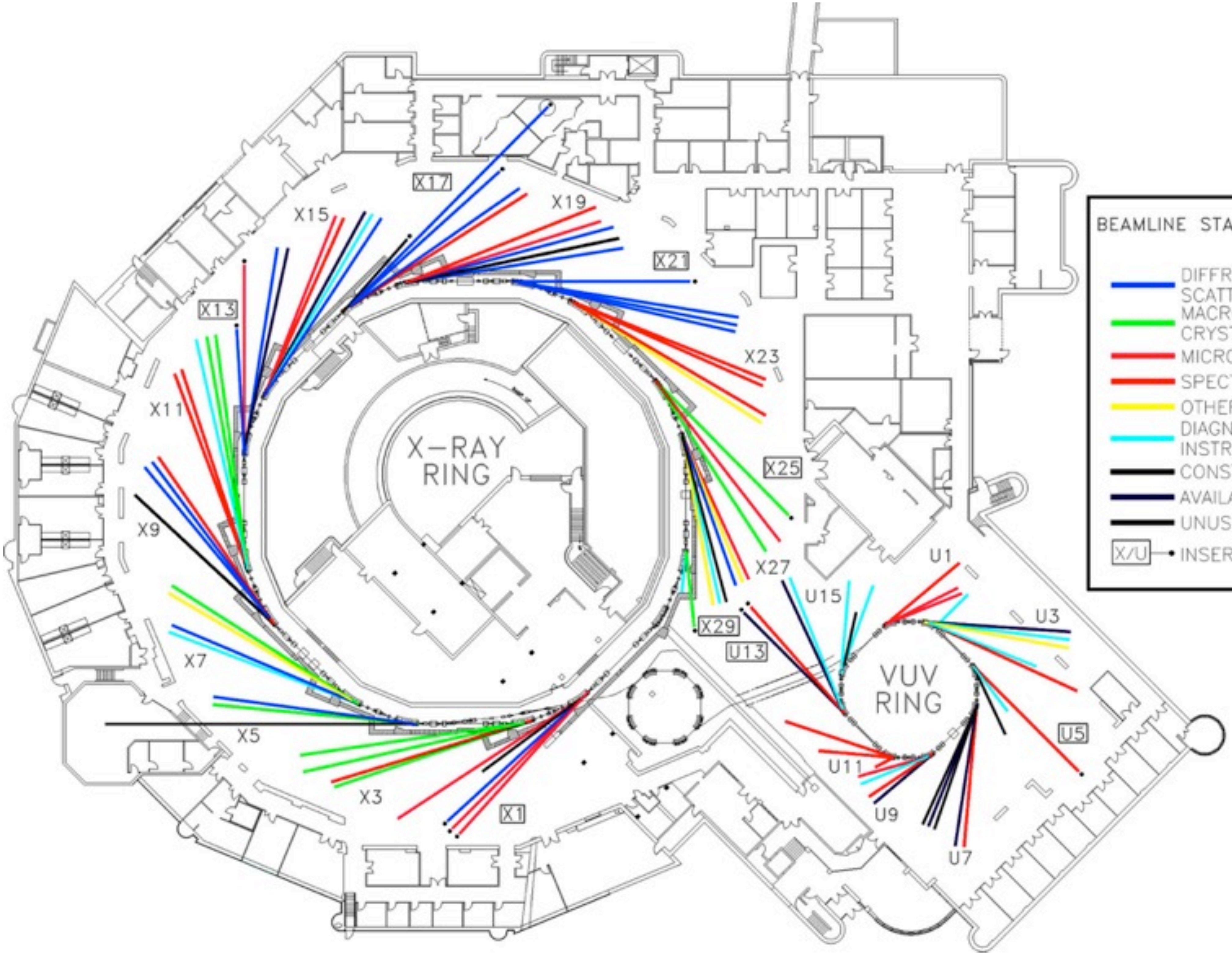
Experimental arrangement for measuring the X-ray scattering from a crystal. A beam of X rays is incident on a crystal that is mounted so that it can be rotated around perpendicular axes. The intensities of the diffracted X-ray beams are measured with a photographic film or an equivalent imaging plate. A beam stop is used to attenuate the incident X-ray beam.

"Home" Source - Rotating Anode





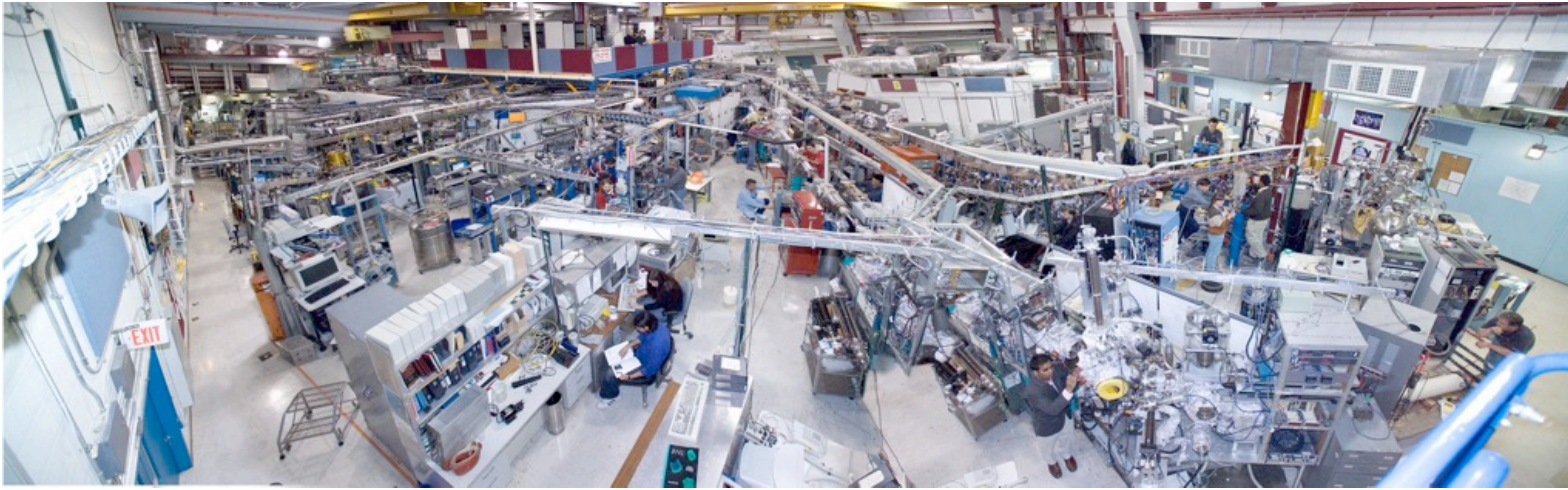
National Synchrotron Light Source



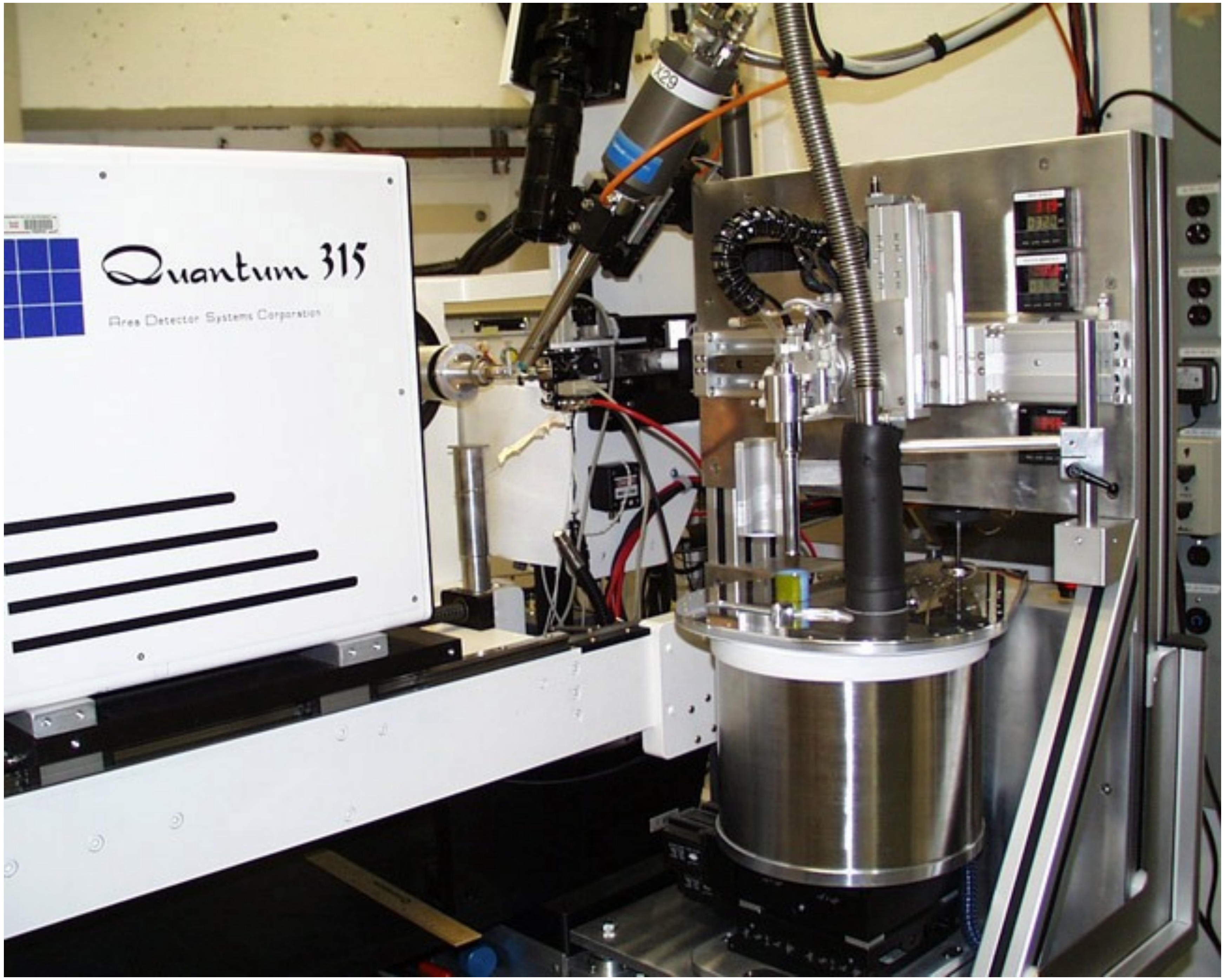
BEAMLINE STATUS AS OF DECEMBER, 2007

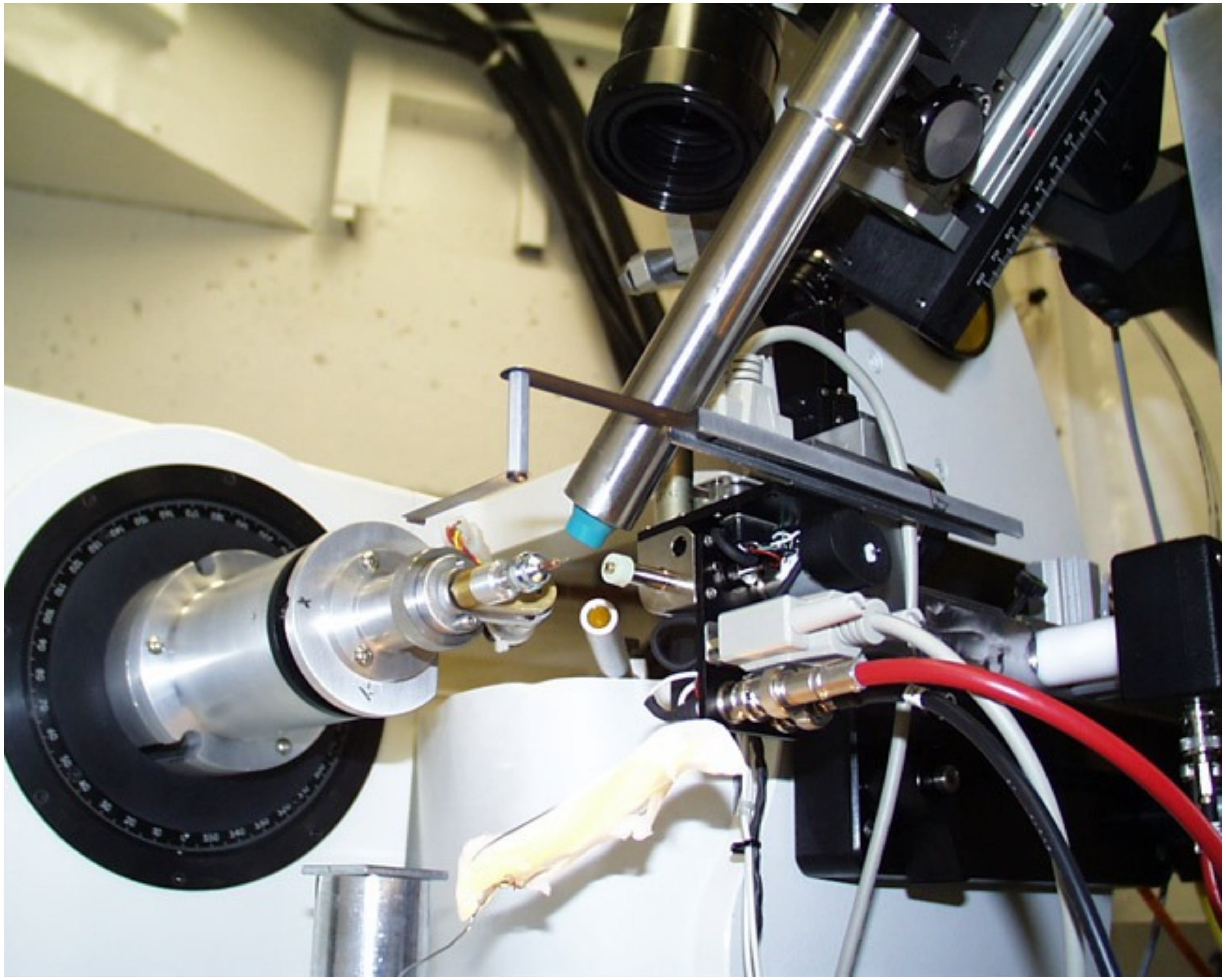
		VUV	X-RAY
🟦	DIFFRACTION AND SCATTERING	0	20
🟩	MACROMOLECULAR CRYSTALLOGRAPHY	0	10
🟥	MICROSPECTROSCOPY	3	8
🟥	SPECTROSCOPY	9	10
🟨	OTHER	1	4
🟦	DIAGNOSTIC AND INSTRUMENTATION	8	5
⬛	CONSTRUCTION	0	2
⬛	AVAILABLE	6	2
⬛	UNUSED	4	4
X/U —•	INSERTION DEVICES		

Experimental Floor at NSLS

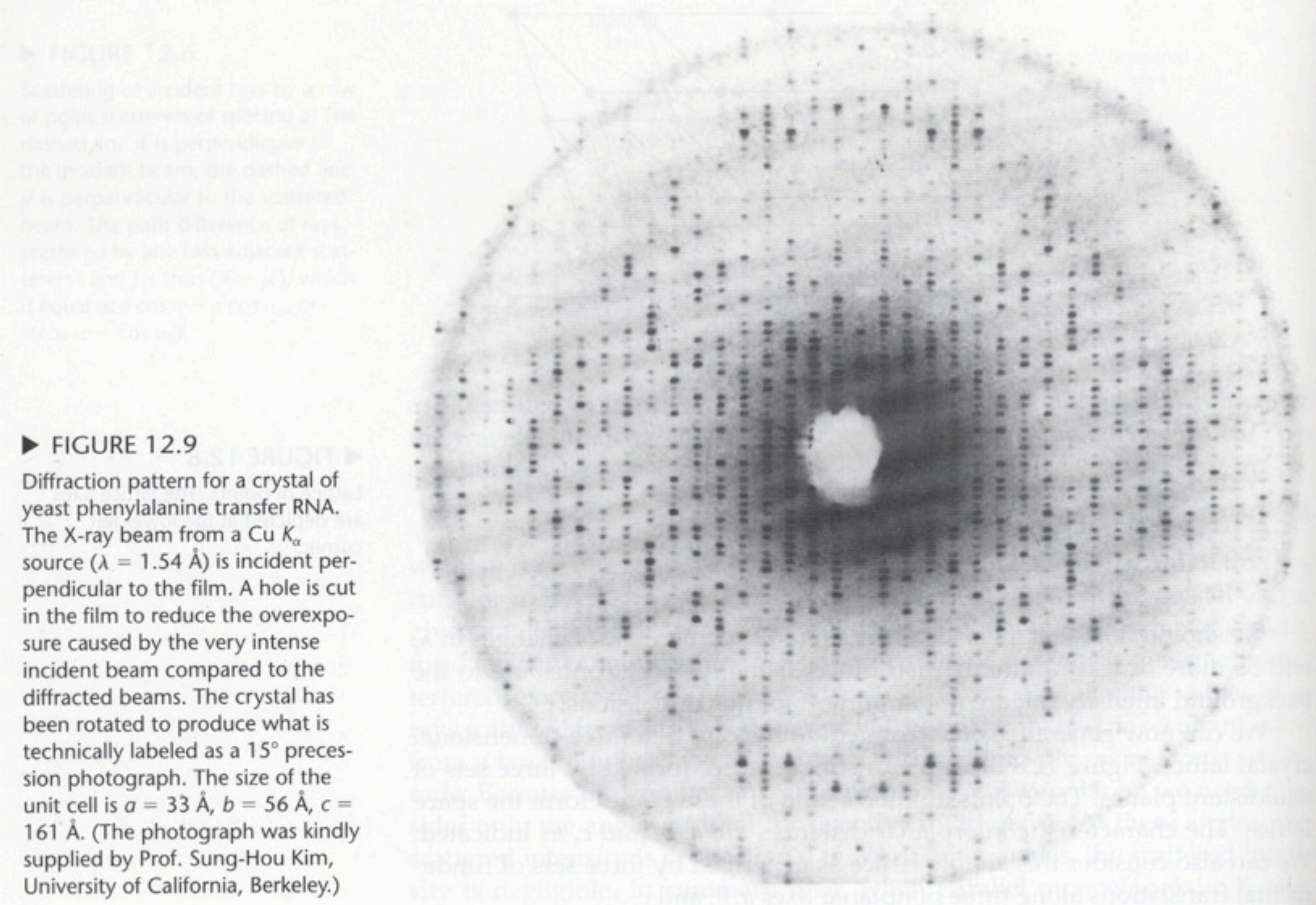


X29 Beamline NSLS





Diffraction Pattern

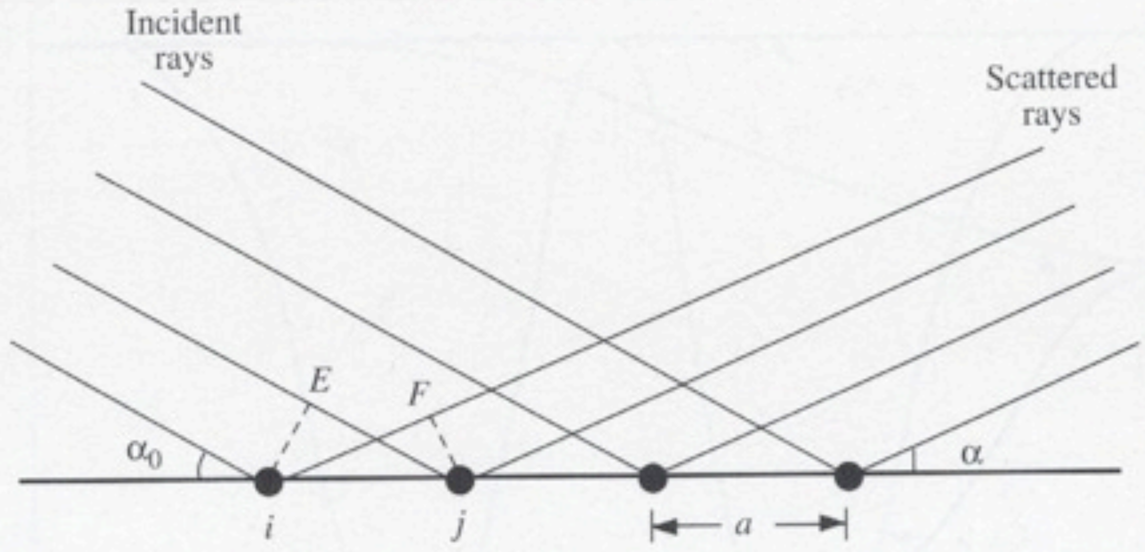


▶ FIGURE 12.9

Diffraction pattern for a crystal of yeast phenylalanine transfer RNA. The X-ray beam from a Cu K_α source ($\lambda = 1.54$ Å) is incident perpendicular to the film. A hole is cut in the film to reduce the overexposure caused by the very intense incident beam compared to the diffracted beams. The crystal has been rotated to produce what is technically labeled as a 15° precession photograph. The size of the unit cell is $a = 33$ Å, $b = 56$ Å, $c = 161$ Å. (The photograph was kindly supplied by Prof. Sung-Hou Kim, University of California, Berkeley.)

- The scattering intensity is a function of angle from the incident beam. Brighter reflections have a lower angel of scatter
- The position of the reflection is dependent on orientation of the lattice to the incident beam

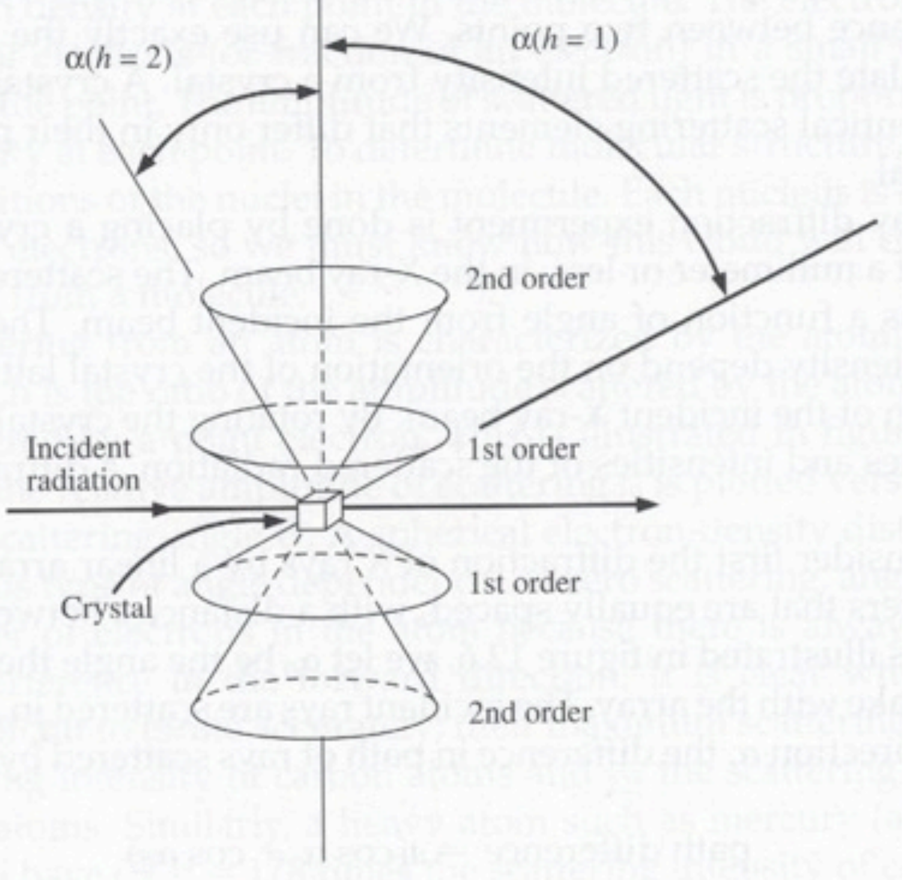
Diffraction One-Dimensional Lattice



▶ FIGURE 12.6

Scattering of incident rays by a row of point scatterers of spacing a. The dashed line iE is perpendicular to the incident beam; the dashed line jF is perpendicular to the scattered beam. The path difference of rays scattered by any two adjacent scatterers i and j is thus $(iF - jE)$, which is equal to $a \cos \alpha - a \cos \alpha_0$, or $a(\cos \alpha - \cos \alpha_0)$.

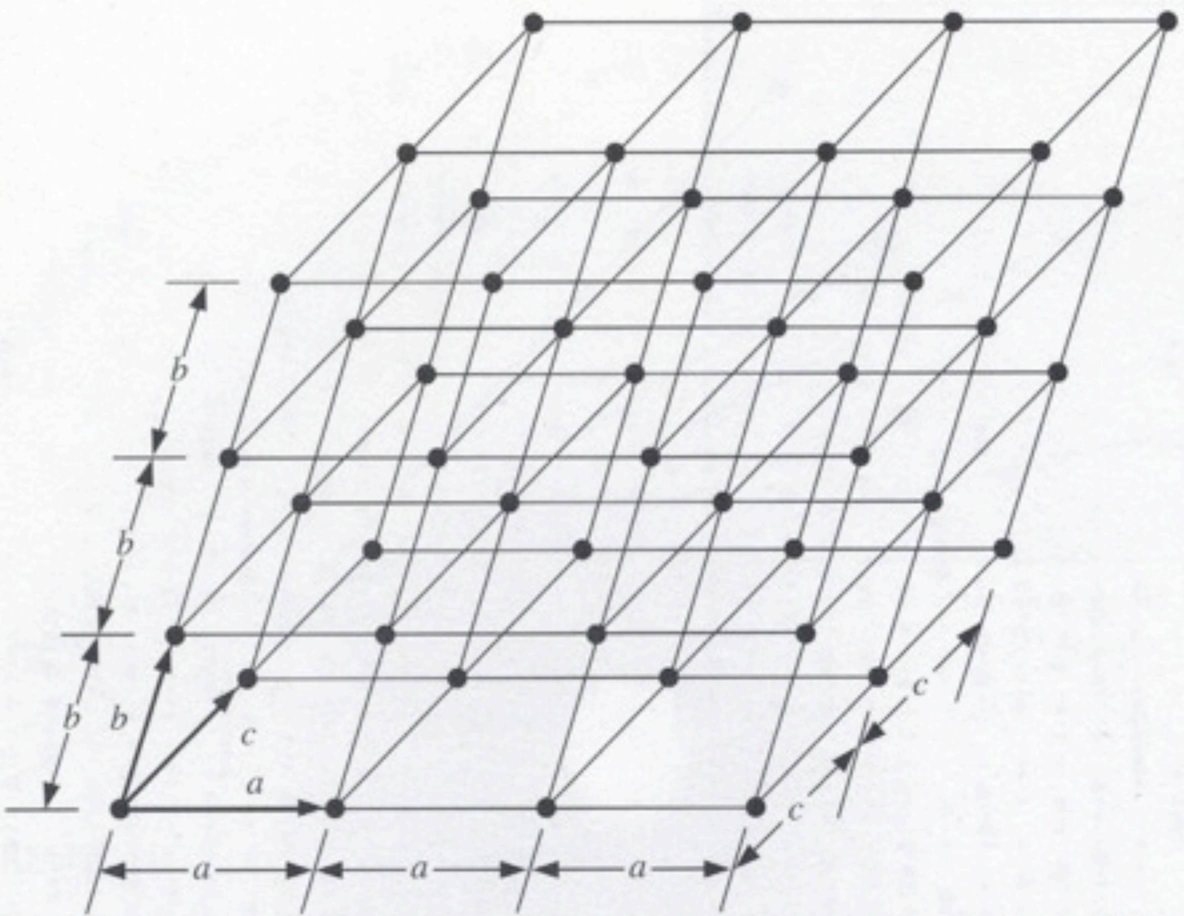
- Only get constructive reinforcement if $a(\cos\alpha - \cos\alpha_o) = h\lambda$



▶ FIGURE 12.7

Equidistant point scatterers arranged in the vertical direction at the apices of the cones. With an incident angle $\alpha_0 = 90°$, Eq. (12.6) reduces to $a \cos \alpha = h\lambda$, or $\alpha = \cos^{-1}(h\lambda/a)$. Cones with $h = 1$ (first order) and $h = 2$ (second order) are shown. Note that $\cos \alpha = \cos(-\alpha)$; thus, in this particular case, for each order there are two cones 180° apart. The zero-order diffraction ($h = 0$) is not shown.

Diffraction Three-Dimensional Lattice



◀ FIGURE 12.8
Lattice of points. The lattice axes are depicted at the lower left corner.

- Only get constructive reinforcement if

$a(\cos\alpha-\cos\alpha_o)=h\lambda$

$b(\cos\alpha-\cos\alpha_o)=k\lambda$

$c(\cos\alpha-\cos\alpha_o)=l\lambda$

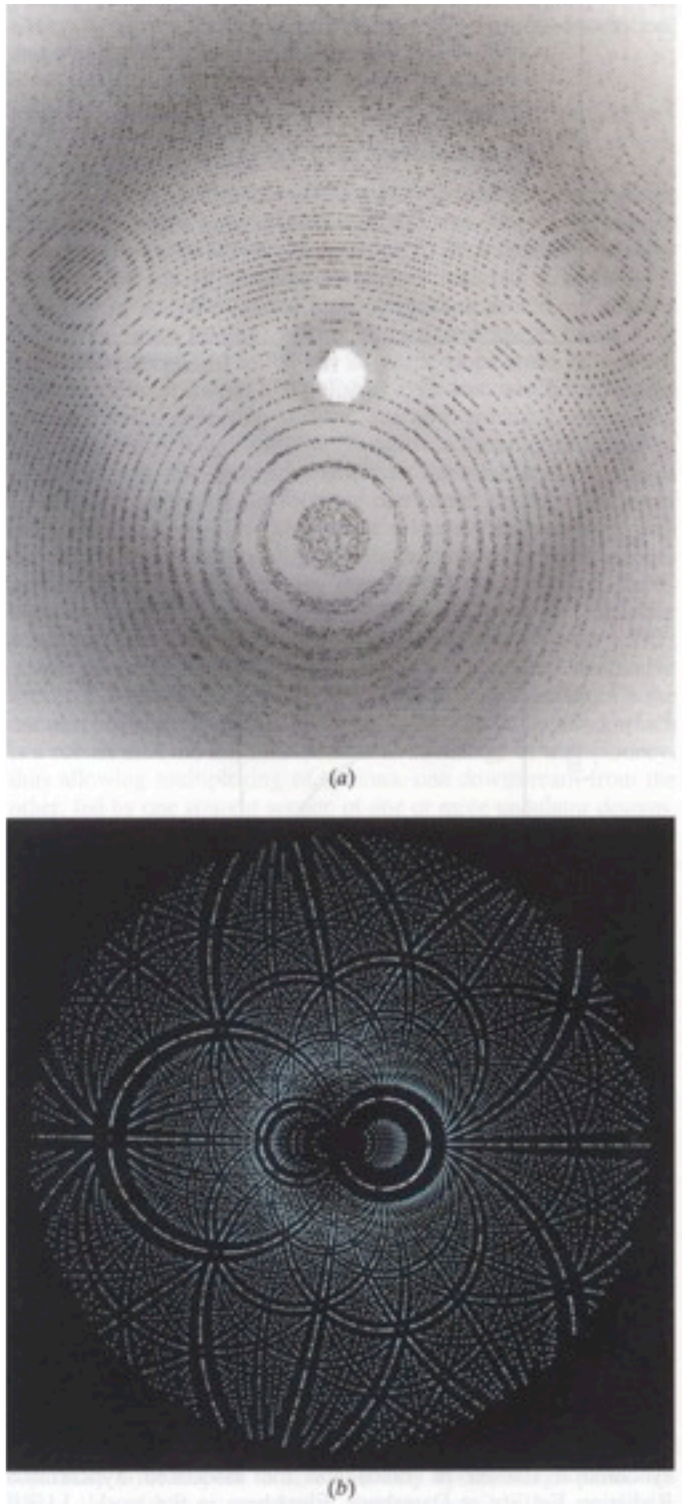
von Laue’s equations

hkl are called Miller indices

Each reflection is given a unique set of Miller indices

Reciprical Lattice

Monochromatic vs Polychromatic X-rays

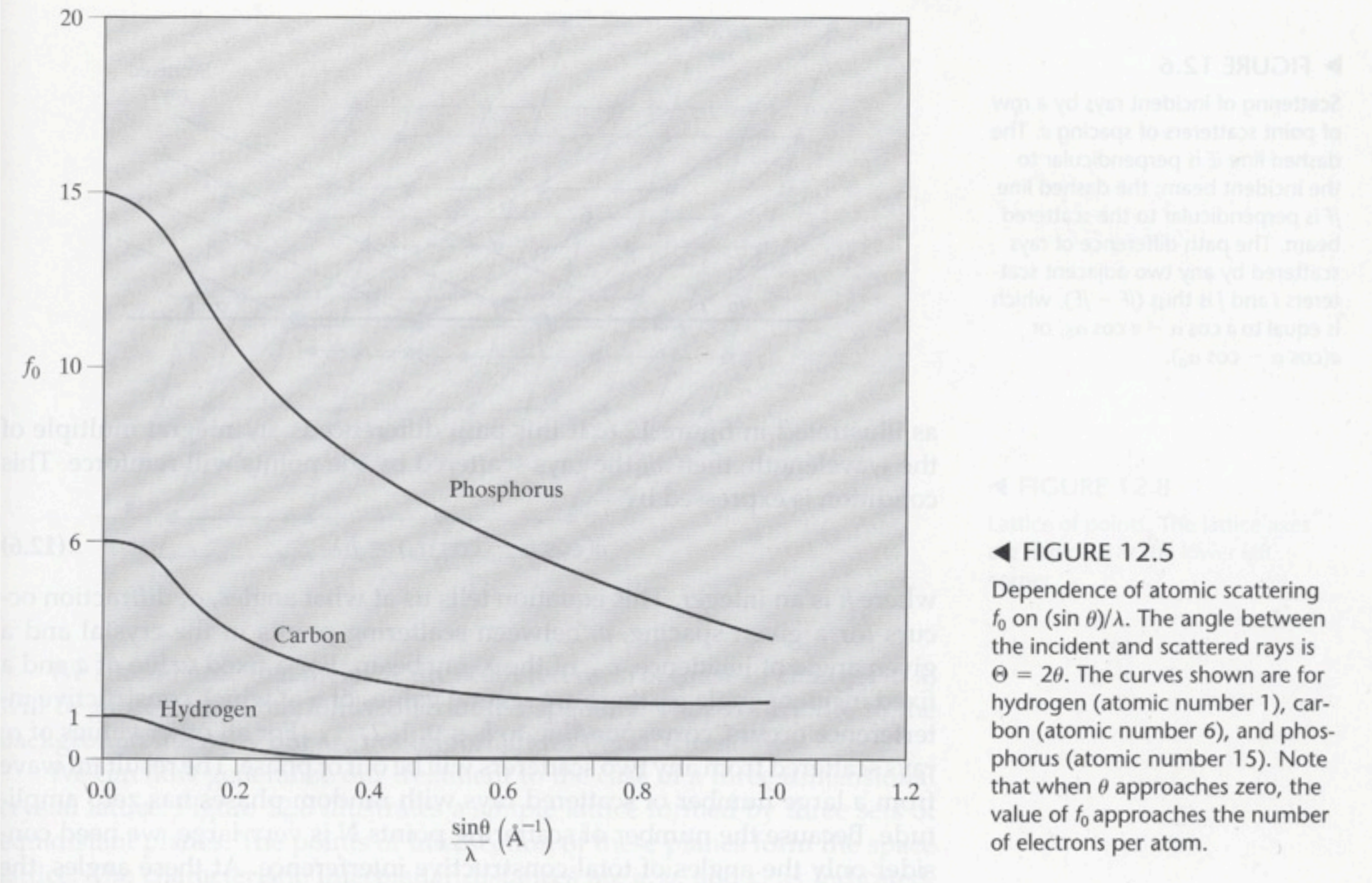


Monochromatic

Polychromatic

Fig. 8.1.4.2. Single-crystal SR diffraction patterns. (*a*) Rhinovirus monochromatic oscillation photograph recorded at CHESS (Arnold *et al.* 1987; see also Rossmann & Erickson, 1983). Copyright (1987) International Union of Crystallography. (*b*) Prediction of a protein crystal Laue diffraction pattern (for an illuminating bandpass, without monochromator, $\sim 0.4 < \lambda < 2.6$ Å). The colour coding is according to the multiplicity of each spot: turquoise for singlet reflections, yellow for doublets, orange for triplets and blue for quartet or higher-multiplicity Laue spots. Reproduced with permission from Cruickshank *et al.* (1991). Copyright (1991) International Union of Crystallography.

Scattering Factors



◀ **FIGURE 12.5**

Dependence of atomic scattering f_0 on $(\sin\theta)/\lambda$. The angle between the incident and scattered rays is $\Theta = 2\theta$. The curves shown are for hydrogen (atomic number 1), carbon (atomic number 6), and phosphorus (atomic number 15). Note that when θ approaches zero, the value of f_0 approaches the number of electrons per atom.

- Scattering factor (f_o) ratio of the amplitude scattered by an atom to the amplitude scattered by a point electron