

## 4.2. X-RAYS

$$\eta_c = bZE_0/2. \quad (4.2.1.9)$$

Crystallographers are more accustomed to thinking of the spectrum in terms of wavelength. Equation (4.2.1.7) can be transformed into

$$dN_\lambda = hcbZ(1/\lambda^2 - 1/\lambda\lambda_0) d\lambda, \quad (4.2.1.10)$$

which has a maximum at  $\lambda = 2\lambda_0$ . In practice, the *emerging* spectrum is modified by target absorption, which is greatest for the longer wavelengths and moves the maximum more nearly to  $1.5\lambda_0$ .

It is of interest to compare the X-ray flux in a narrow wavelength band selected by an appropriate monochromator with the flux in a characteristic spectral line, in order to examine the practicability of XAFS (X-ray absorption fine-structure spectroscopy) or optimized anomalous-dispersion diffractometry experiments. For these purposes, the maximum permissible wavelength band is about  $10^{-3}$  Å. From equation (4.2.1.10), we see that, for a tungsten-target X-ray tube operated at 80 kV,  $dN_\lambda$  is about  $1.1 \times 10^{-5}$  photons with the  $K\alpha$  energy  $\text{electron}^{-1} \text{steradian}^{-1} (10^{-3} \delta\lambda/\lambda)^{-1}$  for an X-ray wavelength in the neighbourhood of 1.5 Å. By comparison, from equation (4.2.1.2), a copper-target tube operated at 40 kV produces about  $5 \times 10^{-4}$   $K\alpha$  photons  $\text{electron}^{-1} \text{steradian}^{-1}$ . In spite of this shortcoming by a factor of about 45, laboratory XAFS experiments are sufficiently common to have merited at least one specialized conference (Stern, 1980; see also Tohji, Udagawa, Kawasaki & Masuda, 1983; Sakurai, 1993; Sakurai & Sakurai, 1994).

The use of continuous radiation for diffraction experiments is complicated by the fact that the radiation is polarized. The degree of polarization may be defined as

$$p = (I_{\parallel} - I_{\perp}) / (I_{\parallel} + I_{\perp}), \quad (4.2.1.11)$$

where  $I_{\parallel}$  and  $I_{\perp}$  are the intensities of radiation with the electric vector parallel and perpendicular to the plane containing the incident electrons and the direction of the emitted photons. For an angle of  $\pi/2$  between the electrons and the emitted beam,  $p$  varies smoothly through the spectrum; it is negative for the softest radiation, approximately zero at  $\nu/\nu_0 \sim 0.1$  and reaches values between +0.7 and +0.9 near the Duane–Hunt limit (Kirkpatrick & Wiedmann, 1945). Since practical use of white radiation is likely to be in the vicinity of  $\nu/\nu_0 \sim 0.1$ , the effect is not a large one.

It should also be noted that the spatial distribution of the white spectrum, even after correction for absorption in the target, is not isotropic. The intensity has a maximum at about  $50^\circ$  to the electron beam and non-zero minima at  $0$  and  $180^\circ$  to that beam (Stephenson, 1957).

## 4.2.1.3. X-ray tubes

The commonest source of X-rays is the high-vacuum, or Coolidge, X-ray tube, which may be either demountable and pumped continuously when in operation or permanently sealed after evacuation. The vacuum tube contains an electron gun that incorporates a thermionic cathode, which produces a well defined electron beam that is accelerated towards the anode or target, formerly often called the anticathode. In most X-ray tubes intended for crystallographic purposes, the anode is massive, *i.e.* its thickness is large compared with the range of the electrons; it is usually water-cooled and its surface is normal to the incident electron beam. Usually, it is desirable for the X-ray source to be small (between  $25 \mu\text{m}$  and  $1 \text{mm}$  square) and for the X-ray intensity from the tube to be the maximum possible for the amount of power that can be dissipated in the target. These objectives are best achieved by

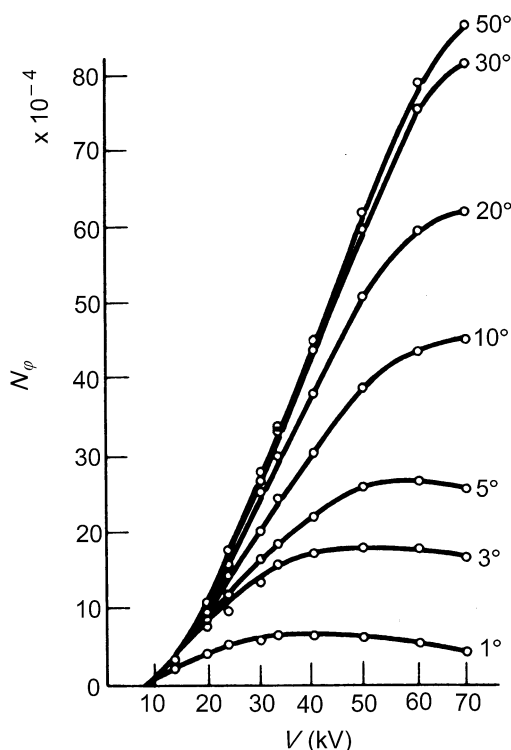


Fig. 4.2.1.2. Experimental measurements of  $N_\phi$  for Cu  $K\text{-}L_3$  as functions of the accelerating voltage for different take-off angles. From Metchnik & Tomlin (1963).

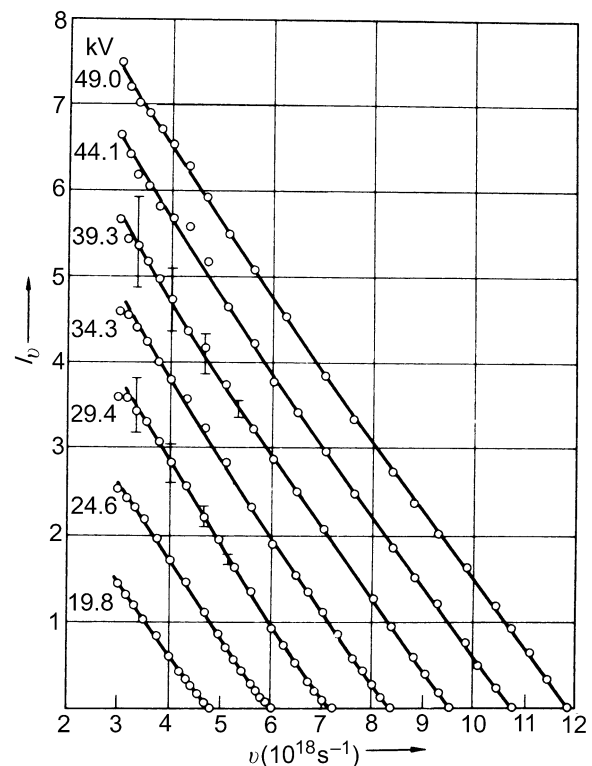


Fig. 4.2.1.3. Intensity per unit frequency interval *versus* frequency in the continuous spectrum from a thick target at different accelerating voltages. From Kuhlenskampff & Schmidt (1943).