7.1. Detectors for X-rays

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7.1.1. Photographic film*

In 1962, when Volume III of *International Tables for X-ray Crystallography* was published, photographic film was the commonest detector for X-rays. Now it has been largely supplanted by the electronic devices described in other sections, but it is still much used in powder cameras and in preliminary investigation of specimens.

X-rays and other radiations cause blackening of silver halide emulsions, and their intensity can be measured accordingly. The blackening of the film is expressed in units of density:

$$D = \log_{10} (\mathscr{I}_{\text{incident}}/\mathscr{I}_{\text{transmitted}}), \qquad (7.1.1.1)$$

where $\mathscr{I}$ refers to the intensity of the ordinary light incident on the film. Measured densities must be corrected by subtracting the fog density D_F measured on a non-exposed part of the film.

Important features of the photographic process for strongly ionizing radiations such as X-rays and electrons are:

(i) For a given total exposure E the relationship between D and E is, to a close approximation, independent of the time variation of the intensity of the incident radiation. It does not matter whether the X-ray quanta arrive continuously or in short intense bursts (Mees, 1954).

(ii) The density D increases linearly with E up to $D \simeq 1$, then increases more slowly.

Photographic intensity measurements may be made either visually or by using a microdensitometer.

7.1.1.1. *Visual estimation*

Visual estimation consists of comparing the spot or line to be measured with a series of exposure-calibrated marks similar in shape to the object of measurement, and preferably made with the same specimen and incident beam. Lack of complete similarity and unfavourable background usually cause the error of such measurements to be larger than the optimum contrast threshold of the eye. For a spot area of 1 mm^2, the latter amounts to roughly 1% or 0.004 density units, a difference that can in fact be detected under favourable circumstances (low density and low background).

7.1.1.2. *Densitometry*

If the blackening is measured with a microdensitometer, an accuracy of 0.002 density units up to densities of at least 2 is easily attained. Higher precision is rarely required, as the limiting factors are graininess of the film and irregularities in the emulsion and processing. The grains in processed X-ray film are larger than those produced by visible light, and occur in clusters around each absorbed quantum. The resulting statistical fluctuations may be minimized by appropriate choice of densitometer slit dimensions and scanning speed. If the X-rays are not incident normally on double-coated film, it may be necessary to make corrections for obliquity (Whittaker, 1953; Hellner, 1954).

* Editorial condensation of the entry by P. M. de Wolff in Chapter 3.1 of Volume III.

7.1.2. Geiger counters†

Geiger-Müller counters (Geiger & Müller, 1928) are now obsolete for data collection, but are still used in portable monitors for X-rays. A cross section of a once-popular type is shown in Fig. 7.1.2.1(*a*). The cathode *C* is a cylinder made of a metal such as chrome-iron, about 2 cm in diameter and 10 cm long. The anode *A* is a tungsten wire about 0.7 mm in diameter mounted coaxially with *C* and terminated by a bead to prevent destructive electrical discharges from its tip. About 1400 V DC is applied between *C* and *A*. X-rays enter at a low-absorption end window *W*, made of mica about 0.013 mm thick or other suitable material; beryllium would now be used. The gas filling may be argon at a pressure of about 55 cm Hg or krypton at a lower pressure. A small amount of halogen (~0.4% of chlorine or bromine) helps to avoid destructive discharges. Separating the anode and the window is a dead space in which X-rays are absorbed but not detected.

The quantum-counting efficiency varies with wavelength; for Cu K and its neighbours, it is about 50% and, for Mo K, it is about 10%. For the longer wavelengths, it is limited by absorption in the window and the dead space, so it is important to keep these as thin as practicable. For the shorter wavelengths, it is limited by the transparency of the gas in the sensitive volume.

† Editorial condensation of the entry by W. Parrish in Chapter 3.1 of Volume III. Several papers on the relative advantages of various detectors are collected in Parrish (1962). Sections 7.1.2–7.1.4 have been slightly revised by J. I. Langford.

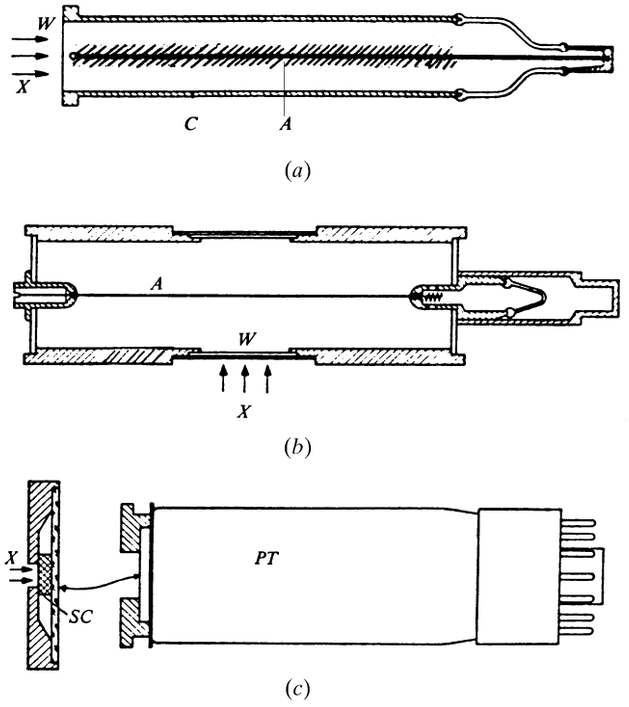

Fig. 7.1.2.1. Detectors used for diffractometry: (*a*) Geiger counter, (*b*) side-window proportional counter, (*c*) end-window scintillation counter. The arrows *X* show direction of incident X-ray beam, *W* thin window, *C* cathode, *A* anode, *SC* scintillation crystal, *PT* photomultiplier tube.

references