

## 2. DIFFRACTION GEOMETRY AND ITS PRACTICAL REALIZATION

Table 2.3.5.1. X-ray tube maximum ratings

Sealed-off (3 kW)*				Rotating anode (18 kW)†			
Anode	Focus (mm)	Power (kW)	Brightness (W mm <sup>-2</sup> )	Anode	Focus (mm)	Power (kW)	Brightness (W mm <sup>-2</sup> )
Mo	0.4 × 12	3.0	625	Mo, Cu	0.5 × 10	18.0	3600
	1 × 10	2.4	240		0.3 × 3	5.4	6000
	2 × 12	2.7	112		0.1 × 1	1.2	12000
Cu	0.4 × 12	2.2	460	Ag	0.5 × 10	12.0	2400
	1 × 10	2.0	200		0.3 × 3	5.4	6000
	2 × 12	2.7	112		0.1 × 1	1.2	12000
Cr	0.4 × 12	1.9	400	Cr	0.5 × 10	10.0	2000
	1 × 10	1.9	180		0.3 × 3	4.5	5000
	2 × 12	2.7	112		0.1 × 1	1.0	10000

\* Philips. † Rigaku.

operate at lower total power. X-ray tubes normally have a life of several thousand hours. It varies with power, anode-cooling efficiency, on-off cycles, and similar factors.

Most X-ray generators are now designed for constant-potential operation using solid-state rectifiers and capacitors in the high-voltage transformer tank. They produce higher intensity at the same voltage than self-rectified or full-wave-rectified operation because the characteristic line spectrum is produced only in the portion of the cycle in which the voltage exceeds the critical excitation voltage of the target element. The gain thus increases with decreasing wavelength. The operation of modern X-ray generators is very simple and requires little attention. Safety interlocks provide electrical protection, and window-shutter interlocks aid in radiation safety. Large ray-proof plastic enclosures are available to surround the X-ray tube tower and diffraction instrumentation and are recommended for safety. Some legal requirements are outlined in Part 10.

Air-cooled tubes can operate at only a fraction of the power of water-cooled tubes and are used for special applications where low intensities can be tolerated. Small portable air-cooled X-ray tubes have recently become available in a variety of forms (see, for example, Kevex Corporation, 1990). The tube, high-voltage generator and control electronics are packaged in compact units with approximate dimensions 27 by 10 cm weighing about 3 kg. They have a single 0.13 mm Be window, a focal-spot size 0.25 to 0.50 mm, and are available with a number of target elements. They can be AC or battery operated. Some tubes are rated at 70 kV, 7W, and others at 30 kV, 200W, depending on the model.

#### 2.3.5.1.1. Stability

Modern X-ray generators have a high degree of electrical stability, of the order of 0.1 to 0.005%, which is sufficient for most applications. The current is continually monitored in the generator and used in feedback circuits to regulate the output. The high voltage is also monitored in some generators. Maximum long-time stability is obtained if the generator and X-ray tube are run continuously over long periods of time so that they reach stable operating conditions. Experienced technicians often advise that the X-ray tube life is shortened by frequent on-off use because the filament receives maximum stress when turned on. The tube may be left operating at low

power, 20 kV, 5–10 mA, when not being used. It is inadvisable to operate at voltages below about 20 kV for long periods of time because space charge builds up, causing excessive heating of the filament and shorter life. The stability can be determined by measuring the intensity of a diffraction peak or fluorescence as a function of time. This is not an easy experiment to perform because the stability of the detector system must first be determined with a radioactive source and a sufficient number of counts recorded for the required statistical accuracy.

Alternatively, a monitor method can be used to correct for drifts and instabilities. The monitor is another detector with a separate set of electronics. It can be used in several ways: (1) as a dosimeter to control the count time at each step; (2) to measure the counts at each step and use the data to make corrections, *i.e.* counts from specimen divided by monitor counts. (It is usually advisable to average the monitor counts over a number of steps to obtain better statistical accuracy.) A thin Be foil or Mylar film inclined to the beam is ideal because they have little absorption and strong scattering. The monitor detector can be mounted out of the beam path and must be able to handle very high count rates and have an extended linear range to avoid introducing errors. In synchrotron-radiation EXAFS experiments, the beam passes through an ionization chamber placed in the beam to monitor the incident intensity.

Spikes in the data may arise from transients in the electrical supply and filtering at the source is required, although modern diffractometer control systems have provision for removing aberrant data.

#### 2.3.5.1.2. Spectral purity

Spectral contamination from metals inside the tube may occur and increase with tube use. This reduces the intensity by coating the anode and windows and may not be noticed when using an incident-beam or diffracted-beam monochromator. It can be measured by removing the monochromator or  $\beta$  filter, operating the tube at high kV, and recording the diffraction pattern of a simple powder (*e.g.* Si or W), a rolled metal foil, or a single-crystal plate (Ladell & Parrish, 1959). The contaminating elements can be identified from the extra peaks. It is advisable to check the spectral purity when the tube is new and periodically thereafter.