

2.1. LABORATORY X-RAY SCATTERING

Table 2.1.6

Important detector properties at 8 keV as reported by various vendors

Only typical values are given to allow approximate comparisons. Detector properties strongly depend on individual detector designs and are subject to high development rates.

	Scintillation	Gas ionization (Xe/CO ₂ gas filling)		
		Wire based (0D)	Wire based (1D/2D)	Micro-gap (1D/2D)
DQE	~95%	~95%	~80%	~80%
Dynamic range	>6 × 10 ⁶	>10 ⁶	>10 ⁴ (1D) >10 ⁶ (2D)	>8 × 10 ⁷ (1D) >10 ⁹ (2D)
Maximum global count rate	>2 × 10 ⁶ c.p.s.	>7.5 × 10 ⁵	>10 ⁵ (1D) >4 × 10 ⁴ c.p.s. (2D)	>8 × 10 ⁵ (1D) >1.6 × 10 ⁶ c.p.s. (2D)
Maximum local count rate	n/a	n/a	>10 ⁴ (1D) >10 ⁴ c.p.s. mm ⁻² (2D)	>9 × 10 ⁵ c.p.s. mm ⁻² (1D, 2D)
Noise	~0.3 c.p.s.	~1 c.p.s.	~1 c.p.s. (1D) <5 × 10 ⁻⁴ c.p.s. mm ⁻² (2D)	<0.01 c.p.s. (1D) <5 × 10 ⁻⁴ c.p.s. mm ⁻² (2D)
Energy resolution	~3500 eV (~45%)	~1600 eV (~20%)	~1600 eV (~20%)	~1600 eV (~20%)
Detection mode	Photon counting	Photon counting	Photon counting	Photon counting

	Semiconductor				
	Si(Li)	Strip	Pixel	CCD	CMOS
DQE	>98%	>98%	>98%	~20–60%	~75%
Dynamic range	>10 ⁶	>7 × 10 ⁶ per strip	>10 ⁹	>5 × 10 ⁴	>1.6 × 10 ⁴
Maximum global count rate	>10 ⁵ c.p.s.	>10 ⁸ c.p.s.	>10 ⁷ c.p.s. mm ⁻²	n/a	n/a
Maximum local count rate	n/a	>7 × 10 ⁵ c.p.s. per strip	>10 ⁴ per pixel	n/a	n/a
Noise	~0.1 c.p.s.	~0.1 c.p.s. per strip	~2.5 × 10 ⁻³ c.p.s. mm ⁻²	<0.1 c.p.s. per pixel	<0.05 c.p.s. per pixel
Energy resolution	~200 eV (~4%)	~1600 eV (~20%)†	>1000 eV (~12.5%)	n/a‡	n/a
Detection mode	Photon counting	Photon counting	Photon counting	Integrating§	Integrating

† ~380 eV/~5%; Wiacek *et al.* (2015). ‡ >300 eV/>6% in photon-counting mode, see text. § Photon-counting mode possible, see text.

as the detector of choice. An important disadvantage these days is the limitation to 0D detection.

2.1.7.2.2. Gas-ionization detectors

The gas-ionization detectors in current use are proportional counters and can be of the 0D, 1D or 2D detection type. Common to all proportional counters is a gas-filled chamber permeated by a non-uniform electric field between positive and negative electrodes, held at a constant potential difference relative to each other. Typically the noble gases Ar or Xe are used as gas fill, mixed with a small amount of quenching gas such as CH₄ or CO₂ to limit discharges. When an X-ray photon travels through the gas-filled volume, it may be absorbed by a noble-gas atom, resulting in the ejection of an electron (photoelectric and Compton recoil). This electron, accelerated by the electric field towards the anode, will cause an avalanche by subsequent ionization along its path (gas amplification), generating an electric pulse which can be registered. The height of the generated pulse is proportional to the energy of the incoming X-ray photon and permits the use of pulse-height selection to achieve moderate energy resolution.

2.1.7.2.2.1. Wire-based proportional counters

In a point proportional detector (0D detection), the pulses generated are measured at one end of a wire (or a knife edge). Position-sensitive (1D and 2D detection) proportional detectors have the added capability of detecting the location of an X-ray photon absorption event. In a 1D proportional detector, pulses

are detected at both ends of the wire. Thus the time difference between the measurements of a given pulse can be used to determine the location of the discharge. 2D proportional counters consist of three arrays of wires (multiwire proportional counter, MWPC; Sauli, 1977; Charpak *et al.*, 1968), where one array forming the anode plane is placed between two cathode arrays with their wires oriented parallel and orthogonal to the anode-plane wires, respectively.

Low count rates and low-to-moderate detector noise result in low-to-moderate dynamic ranges. Wire-based proportional counters are not competitive with micro-gap and semiconductor detectors, as can be seen in Table 2.1.6, and are therefore being driven out of the market.

2.1.7.2.2.2. Micro-gap detectors

The maximum count rates in 'classical' metal-wire-based proportional counters are severely limited by the long ion-drift times in the chamber (which typically have a cathode to anode spacing of ~10 mm). This issue has been successfully addressed by so-called micro-gap technology using parallel-plate avalanche chambers with a readout electrode separated from a resistive anode. The key feature is the resistive anode, which allows a very small amplification gap (1–2 mm cathode to anode spacing) at an increased average electric field intensity, while preventing discharges (Durst *et al.*, 2003; Khazins *et al.*, 2004). As a result, micro-gap detectors can achieve count rates several orders of magnitude higher than classical proportional counters at higher position sensitivity. Micro-gap detectors of the 1D and 2D detection type are available. Moderate count rates and very small

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noise levels result in very high dynamic ranges. Notably, in contrast to wire detectors, micro-gap detectors are not likely to be damaged by accidental exposure to a high-intensity direct beam, as a patterned anode plane is used rather than wires.

2.1.7.2.3. Semiconductor detectors

Semiconductor (or solid-state) detectors are solid-state ionization devices in which electron–hole pairs instead of electron–ion pairs are generated by incoming photons, and they are sensitive to the entire electromagnetic spectrum from visible light to X-rays. The energy required for production of an electron–hole pair is very low compared to the energy required for production of paired ions in a noble-gas-filled detector. As a consequence, a larger number of charge pairs with a smaller statistical variation are generated in semiconductor detectors, resulting in intrinsically higher energy-resolution capabilities. The efficiency of semiconductor detectors is very high due to the high absorption of the semiconductor materials, usually reaching 100%, but may decline at higher photon energies if the photons are not fully absorbed in the semiconductor *e.g.* because of insufficient thickness.

2.1.7.2.3.1. The Si(Li) detector

The Si(Li) detector sensor consists of a lithium-drifted silicon crystal which must be cooled to prevent lithium diffusion and to reduce dark noise. An important advantage of this detector is its excellent energy resolution of even better than 200 eV (4%) at 8 keV (Cu radiation), allowing very effective filtering of $K\beta$ and fluorescence radiation and thus operation without a metal filter or a diffracted-beam monochromator. As Peltier cooling is sufficient, the Si(Li) detector type has found wide interest for applications benefitting from high energy resolution, unlike energy-dispersive detectors requiring operation under cryogenic conditions [*e.g.* Ge(Li)]. In particular, the Si(Li) detector significantly extends the application range of today's X-ray diffractometers by allowing energy-dispersive X-ray powder diffraction (EDXRD) as well as – to some extent – XRF (see Section 2.1.4.3).

An important disadvantage of Si(Li) detectors is their large dead time, which prohibits the handling of higher count rates. Moderate noise levels result in low-to-moderate dynamic ranges. An additional important disadvantage is the limitation to 0D detection.

2.1.7.2.3.2. Silicon micro-strip and silicon pixel detectors

Silicon micro-strip and silicon pixel detectors employ silicon sensors, which are one- or two-dimensional arrays of p–n diodes in the form of strips or pixels, respectively, individually connected to an array of readout channels. The development of this type of detector technology has obviously been driven by the idea of massive parallelism: each strip or pixel actually represents an individual detector. Accordingly, the silicon micro-strip and silicon pixel detectors are therefore of the 1D and 2D detection type, respectively.

Count rates recorded by silicon micro-strip and silicon pixel detectors are very high with very low noise levels, resulting in very large dynamic ranges. The energy resolution of most silicon micro-strip and silicon pixel detectors is of the order of 1600 eV (20%) at 8 keV (Cu radiation). Recently, a silicon micro-strip detector with an energy resolution of better than 380 eV at 8 keV has been introduced (Wiacek *et al.*, 2015). At

such high energy resolution Cu $K\beta$ is filtered out to below the detection limit while Mn, Fe and Co fluorescence is filtered completely, allowing this detector to be operated without a metal filter or a diffracted-beam monochromator for most applications.

2.1.7.2.3.3. CCD and CMOS detectors

Charge-coupled device (CCD) detectors are represented by one- or two-dimensional arrays of square or rectangular pixels consisting of metal–oxide–semiconductor (MOS) capacitors, and can detect X-ray photons directly or indirectly. The pixel size may be less than 10 μm . The majority of detectors use indirect detection, where the incoming X-ray photons are first converted to visible-light photons by a phosphor layer. CCD detectors employ the 'bucket brigade' readout method, in which charge is shifted one pixel at a time by phasing the bias on the gate electrodes that overlay each pixel until it reaches the output, resulting in relatively large readout times ranging from a few tenths of a second up to several seconds per frame. Cooling (Peltier-type) is required to reduce the dark-current noise representing the dominant noise source for long exposures. In some detector designs fibre-optic demagnification is used to increase the effective active detector area, resulting in an imaging area larger than the active area of the CCD chip at the cost of detector sensitivity and spatial resolution.

CCD detectors are usually operated as integrating detectors. As such, they have no dead time and therefore provide excellent linearity over a moderate dynamic range, but cannot have energy resolution. CCD detectors are the detectors of choice for single-crystal diffraction and imaging, but are not favourable for applications with weak diffraction signals, such as powder X-ray diffraction, owing to the relatively large dark-current noise.

CCD detectors may also function as counting detectors by making the exposure time sufficiently short. In single-event mode the energy of each photon can be determined, providing an energy resolution down to about 300 eV at 8 keV (Cu radiation) and allowing a spectrum at each pixel of the CCD array to be built up by a series of consecutive measurements. Such a detector can record energy-dispersive X-ray powder diffraction (EDXRD) as well as – to some extent – XRF (see Section 2.1.4.3); however, owing to the readout time, count rates are extremely low with high statistical noise.

Unlike the bucket-brigade readout of a CCD, the complementary metal–oxide–semiconductor (CMOS) active-pixel sensor (He *et al.*, 2011) uses a completely different architecture in which each pixel incorporates a readout preamplifier and is then read out through a bus, as in random-access memory (He *et al.*, 2011). Cooling is not required. CMOS detectors are immune to the blooming effect (in which a light source overloads the sensitivity of the sensor, causing the signal to bleed vertically into surrounding pixels forming vertical streaks). Additionally, they offer the very significant advantage of shutter-free operation, that is dead-time-free continuous scans which improve the efficiency of data collection and also improve data quality by eliminating shutter-timing jitter.

As a consequence of these characteristics, CMOS-detector active-pixel sensors are now replacing CCD chips in a number of high-end applications (*e.g.* professional digital photography and high-definition television), and have reached a level of performance where they are also starting to displace CCD chips in the most demanding scientific applications.