Journal of
Synchrotron
Radiation

ISSN 0909-0495

Received 4 June 2009
Accepted 16 June 2009

Response to Krumrey's *Comments on Determination of X-ray flux using silicon pin diodes by R. L. Owen et al. (2009). J. Synchrotron Rad. 16, 143–153*

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The manuscript referred to by M. Krumrey aimed to provide a basis for macromolecular crystallographers to determine the photon flux incident on, and hence dose absorbed by, a crystal during an experiment. Radiation damage is a pressing concern in macromolecular crystallography (MX), and it is important that a tool be provided to obtain the incident flux, to allow the dose metric to be used in ongoing studies. For this tool to be widely useful, an accuracy of $\sim$5–10% is required over an energy range 6–20 keV using commercially available devices.

(i) We appreciate the significant contribution made by Krumrey and colleagues over the years to the now vast literature on calibrating photodiodes and other detectors; Krumrey & Tegeler (1992) was overlooked in the literature search during the preparation of the manuscript and we apologize for omitting it. As mentioned in the introduction of Krumrey & Tegeler (1992), the idea of tilting a detector to determine its thickness is an old one and we saw no reason to provide a specific reference for this. Also, the energy range used in their work (0.15–2.5 keV) meant that it was not obvious that it was immediately applicable to MX. The simple expression we used [equation (5), Owen *et al.* (2009)] is derived directly from the geometry of the diode, and the primary aim of the paper was to determine whether the behaviour of devices used could be explained considering only primary absorption. More sophisticated models including both charge-carrier recombination and diffusion (Gullikson *et al.*, 1995; Lutz, 1999) were considered but found not to be well suited to the devices used.

(ii) The neglect of Compton scattering is explicitly addressed in §1.4 of our paper, where we agree with Krumrey that photoelectric cross-section data alone can be used to calculate absorbed energy between 6 and 20 keV. As no attempt was made to calibrate the diodes at energies greater than 20 keV, we felt it was unnecessary to include Compton scattering, since the effect of omitting it contributed negligibly to the error in our flux calculations.

(iii) We regret that we did not make Fig. 5 of our paper and its legend clear enough. The origin of the error bars is detailed in the legend of Fig. 5, and the size of these was determined from the r.m.s. scatter of ten experiments from the mutual mean; a pessimistic approach was taken and this scatter was not divided by $\sqrt{10}$ as is customary. The key point of interest is that the data and theory are superposed, *not* fitted to one another. We found it striking and satisfying that the average value (centre of each error bar) agreed very well with theoretical expectation (blue line).

It is true that a scintillation device does not faithfully detect every incident photon, but the absorption of air and the front window, and the finite thickness of the scintillation crystal were all taken into account in this work, and the photon pile-up effects were both accounted for and minimized by keeping the count rate low. We did not take such factors as escape peaks into account, but they are (theoretically) not significant for this device in this energy range. This is particularly true when using a single-channel analyzer with no energy discrimination.

The linearity of the OSI S100VL device that we calibrated was demonstrated across the pA to mA range by three methods: of these the most convincing was the method illustrated in the results shown in Fig. 3 of our paper (comparison with the dead-time-corrected count rate of a scintillator). We also compared the diode with an ionization chamber and additionally verified that the observed current on the diode in an adsorbtively attenuated beam was consistent with the product of the transmittances of a group of six foils (inserted 10 m upstream), much as is described in the hypothetical discussion in §1.2 of our paper. However, since the counter-comparison data seemed sufficiently convincing, we left out the redundant ion chamber and transmittance-product linearity checks.

We were careful not to report the value of 3.37 MΩ as a shunt resistance because it was not a shunt resistance. We reported this value as an 'input impedance' because it is the terminal-to-terminal opposition of the device to small DC currents. Obviously, the *V–I* curve for any diode is not linear, but for very small voltages and currents such as those with which we were concerned it is a very good approximation to treat it as such. Although related to the shunt resistance of the device, the input impedance and the shunt resistance are not the same thing. Our best determination of the actual shunt resistance of this device is that it is about 100 MΩ (consistent with the manufacturer's specification).

We measured the temperature dependence of the input resistance of the S100VL diode in question and found it to be within 4% of 3.37 MΩ over a 10 K range centred at 296 K. The temperature at both of the beamlines described in the paper is regulated to within 1 K; we therefore saw no need to mention thermal fluctuations as a source of error in our measurements.

(iv) In obtaining the fluxes shown in Table 2 and Fig. 6 of our paper, we used the manufacturers' specifications and any deviation from these feeds directly through to the calculated flux. We did not

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attempt to alter these specifications to obtain better agreement between the devices, because our goal was to demonstrate the precision to which photon flux can be measured using our simple diode model with an 'off the shelf' component and the product literature available with it. We found it noteworthy that the largest disagreement between the four diodes was only 28%, considering that the device thicknesses varied by a factor of 50 and no corrections other than equations (2)–(4) detailed in our paper were employed. The differences no doubt arise from the approximations made in obtaining/using equation (4) as pointed out by Krumrey and throughout our manuscript, and we agree that one may always obtain better accuracy by taking more corrections into account. However, this work endeavoured to find the simplest procedure for obtaining reasonably accurate flux measurements at MX beamlines (energy range 6−18 keV).

(v) None of the statements made by Krumrey in this remark were challenged in our report and we hoped that the specific mention of the PTB at BESSY at the end of §1.3 would guide the interested reader to the correct literature. We saw no need to list commercial calibration services nor to reiterate product literature in our paper. We aimed to report on a simple method to determine photon flux to an accuracy of 5–10% at MX beamlines using commercially available devices. In fact, it is quite unnecessary to measure MX beamline flux to better than 5% accuracy as dose calculations are already limited to this uncertainty by other sources of error. Scattering losses, fluorescent X-ray losses, and the geometric complexity of integrating these effects over the beam profile, the crystal and the material surrounding it easily introduce a 5% error in the calculated absorbed dose (Paithankar *et al.*, 2009).

References

Gullikson, E., Gramsch, E. & Szawlowski, M. (1995). *Appl. Opt.* **34**, 4662–4668.

Krumrey, M. & Tegeler, E. (1992). *Rev. Sci. Instrum.* **63**, 797–801.

Lutz, G. (1999). *Seminconductor Radiation Detectors.* Berlin/Heidelberg/New York: Springer.

Owen, R. L., Holton, J. M., Schulze-Briese, C. & Garman, E. F. (2009). *J. Synchrotron Rad.* **16**, 143–151.

Paithankar, K. S., Owen, R. L. & Garman, E. F. (2009). *J. Synchrotron Rad.* **16**, 152–162.