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On the correction of reflection intensities recorded on imaging plates for incomplete absorption in the phosphor layer

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Abstract

When an X-ray beam is incompletely absorbed in the phosphor layer of an imaging plate (IP), a correction for oblique incidence of the beam on the plate becomes imperative. The perpendicular transmission through the phosphor of Fuji imaging plates has been measured as 30% (HR-IIIN plate, conventional source) for 0.707 Å radiation, and 50 and 85% (BASIII plate, synchrotron radiation) for 0.642 and 0.394 Å radiation, respectively. For a given wavelength, the correction factor is a function of the angle between the diffracted beam and the normal to the surface of the plate. For a flat IP, mounted perpendicularly to the incident beam, the correction increases with the 2θ angle of the reflections, but for other orientations of the IP the dependence is more complex.

1. Introduction

Area detectors, reintroduced in crystallography a decade ago after extensive use of scintillation counters, are rapidly becoming the detectors of choice. They offer an increase in the data-collection rate by one to two orders of magnitude, allowing efficient use even for the most demanding tasks. In particular, data collection for charge-density analysis requires accurate extended data-sets collected at short wavelengths to minimize absorption and extinction, and to allow collection of data at very high values of $\sin\theta/\lambda$. The time required to obtain such data with point detectors is typically of the order of one month, while with area detectors it may be reduced to a few days or less. Testing of the accuracy of area detectors is therefore of prime importance.

Two of the most commonly used area detectors are imaging plates (IP) and CCDs (charge-coupled devices). IPs were developed initially for medical radiography. They have been employed extensively in macromolecular crystallography and for this purpose are used mostly with wavelengths above 1 Å. However, for small-molecule structure analysis, Mo $K\alpha$ or shorter-wavelength radiation is preferable. We report here on a systematic bias, due to nonzero transmission of the diffracted beam through the IP phosphor, which affects the accuracy of short-wavelength data collected with imaging plates. Similar effects may occur with CCD detectors.

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2. Experimental

Three sets of measurements were made on a small test crystal of chromium hexamine hexacyanochromium, $Cr(NH_3)_6$-$Cr(CN)_6$ (Bolotovsky *et al.*, 1995). The first two were performed at the X3A1 beamline at the National Synchrotron Light Source at Brookhaven National Laboratory with 0.642 and 0.394 Å radiation, respectively, using 20 × 40 cm Fuji BASIII IPs mounted in a flat holder attached to the 2θ arm. The third data-set was measured at a rotating-anode source with Mo $K\alpha$ radiation, a cylindrical IP holder and 20 × 25 cm

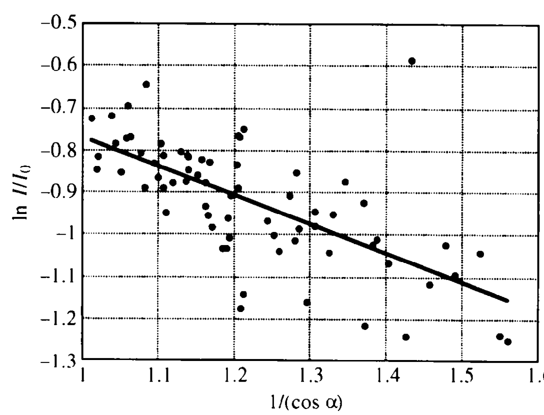

Fig. 1. Transmission of a BASIII imaging plate as a function of the angle of incidence α; λ = 0.642 Å.

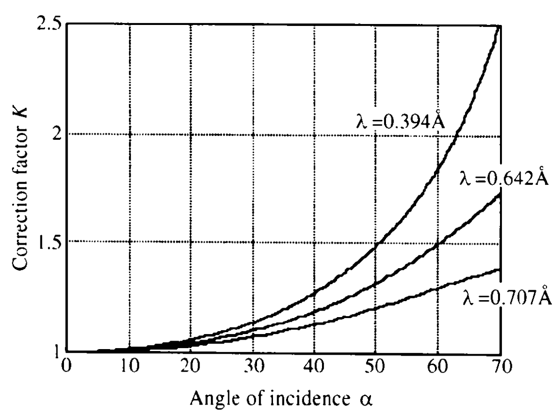

Fig. 2. Correction factor K for three different wavelengths, such that $I_{corr} = I_{obs}/K$.

HR-IIIN Fuji imaging plates. The BASIII plates have a coverage of 15–20 g per 0.3048 m^2 of phosphor material, while the coverage for the HR-IIIN plates is 40–50 g per 0.3048 m^2. All data were read with Fuji BAS2000 scanners and integrated with the *DENZO* package (Otwinowsky & Minor, 1997).

3. Results and discussion

We find that at wavelengths below 1 Å the diffracted beams are only partially absorbed by the IP phosphor and that the absorption in the IP therefore is a function of α, the angle between the diffracted beam and the normal to the IP surface. As the path length of the beam in the phosphor layer is equal to $d/\cos\alpha$, where d is the thickness of the layer, the transmission, defined as $T=I/I_0$, equals $\exp(-\mu d/\cos\alpha)$. A correction factor for oblique incidence is required, which, relative to the reflections recorded at normal incidence, will be equal to

$$\begin{aligned} K &= [1-\exp(-\mu d/\cos\alpha)]/[1-\exp(-\mu d)] \\ &= [1-\exp(\ln T_{\perp})/(\cos\alpha)]/(1-T_{\perp}) \qquad (1) \end{aligned}$$

with $I_{corr}=I_{obs}/K$, and $T_{\perp}$ equal to the transmission of the phosphor layer at the normal incidence.

To apply the correction, the transmission through the IP phosphor for each of the wavelengths was determined as follows. For intensity measurement with the oscillation method, two IPs were mounted with the phosphor layers facing each other, such that the beams pass the poly(ethylene terephthalate) (PET) backing of the first plate before being absorbed in the two phosphor layers. With this arrangement, the ratio of intensities of corresponding reflections on the two plates gives the transmission through the first phosphor layer.† The transmission was plotted against $1/\cos\alpha$ and fitted by the least-squares method to give $T_{\perp}$. The plot for 0.642 Å is shown in Fig. 1. Although considerable scatter occurs for individual reflections, the overall trend is evident. The values of $T_{\perp}$ for 0.394, 0.642 and 0.707 Å were found to be 85, 50 and 30%, respectively.‡

Values of $1/K$ derived with expression (1) are shown as a function of α in Fig. 2. For a flat-plate geometry with the IP mounted perpendicularly to the incident beam, the high-order reflections are strongly affected. We note that at significant nonzero transmission, the intensities may also be biased by nonuniformity of the chemical/physical composition and variation of the thickness of the phosphor layer. This effect would be more severe for shorter wavelengths.

To test the validity of the correction, seven oscillation photographs, with the same φ oscillation range, were taken with the IP holder on the 2θ arm offset by −10, 0, 10, 20, 30, 40 and 50°. In this way, identical reflections are measured with different values of the incidence angle α. Relative scale factors between the plates were obtained from the mean ratio of equivalent reflections recorded on each set of two plates. Figs.

† The absorption in the very thin protective layer on the face of the imaging plate is negligible.

‡ Our direct measurement of the transmission using a scintillation counter placed behind the plates gives values of 65, 30 and 10% for 0.394, 0.642 and 0.707 Å, respectively. As the absorption in the backing is estimated to be not more than 2%, the difference with the 'face to face' method may indicate a dependence of the readout efficiency on the depth of the color centers.

Table 1. *R_{merge} factor for the 0.394 Å synchrotron data-set on $Cr(NH_3)_6Cr(CN)_6$ before and after correction for oblique incidence*

$N_{rejected}$: reflections rejected because of a large deviation from the mean. The numbers in the top line correspond to a less stringent test than those in the second line of each entry. $R=\sum(I-I_{mean})/\sum I$; $R_w=[w(I-I_{mean})^2/\sum I^2]^{1/2}$.

	$N_{reflections}$	$N_{rejected}$	R	R_w
Before correction	13 202	978	9.54	13.80
		2258	4.71	7.83
After correction	13 202	774	4.25	6.76
		1536	3.50	4.87

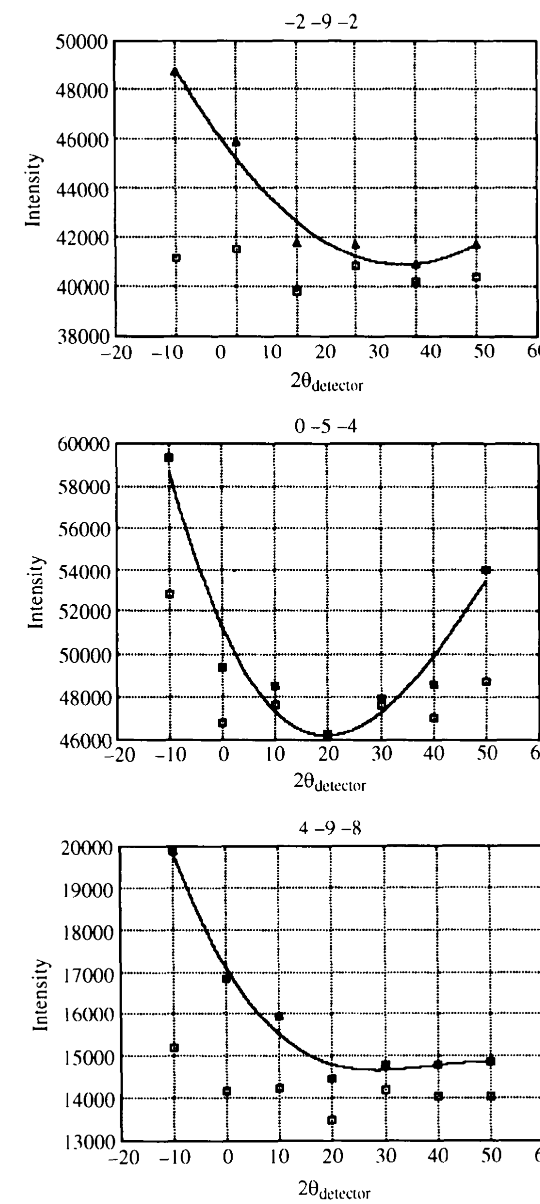

Fig. 3. Intensities of three reflections before (filled squares) and after (open squares) correction for oblique incidence; $\lambda = 0.642$ Å.

3 and 4 show a number of intensities at 0.642 and 0.394 Å, respectively, before and after correction using (1). Uncorrected intensities show a strong dependence on the position of the detector arm, while the corrected intensities are nearly uniform. A corresponding improvement is obtained in the $R_{\rm merge}$ factors upon correction, as illustrated for the 0.394 Å data-set in Table 1. It is noteworthy that the number of rejected reflections drops significantly upon application of the correction, although it is our general experience that outliers always occur in imaging-plate data, even at longer wavelengths.

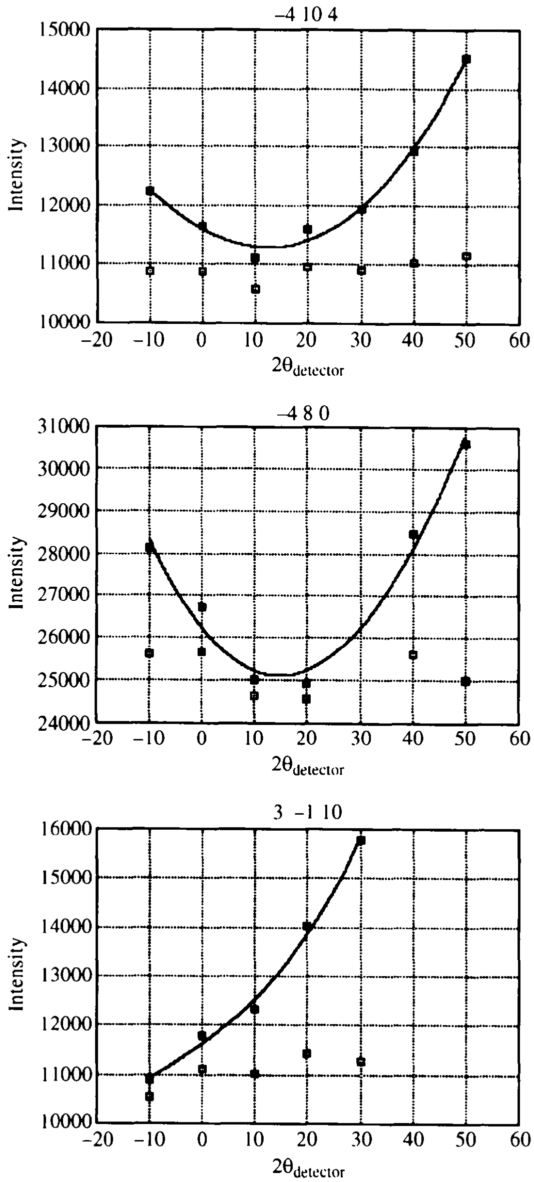

Fig. 4. Intensities of three reflections before (filled squares) and after (open squares) correction for oblique incidence; λ = 0.394 Å.

We have also found, in a recent study with Mo $K\alpha$ data, that a correction based on a transmission significantly larger than 30% may improve the data even further; thus it is advisable to explore carefully which correction is optimal. For larger transmissions, *i.e.* shorter wavelengths, the value of $dK/dT_{\perp}$ is less and so the correction is less sensitive to a variation of $T_{\perp}$.

4. Conclusions

We conclude that imaging-plate intensities collected at wavelengths shorter than about 1 Å must be corrected for the finite thickness of the phosphor layers. The omission of such a correction will lead to significant bias in the results, or even a failure of the refinement for very short wavelengths. In a spherical-atom refinement, the thermal parameters will be particularly affected, while in an aspherical-atom refinement the charge-density parameters are also likely to be biased. The correction for oblique incidence is a function of the wavelength and the crystal-to-plate distance, and must therefore be considered separately for each set of conditions. It is likely that a similar correction applies to CCD data collected at short wavelengths. Its magnitude will depend on the type and thickness of the phosphor used on a particular instrument.

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