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The Correction of Reflection Intensities for Incomplete Absorption of High Energy X-rays in the CCD Phosphor

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Beamlines: X3A1

Introduction: CCD detectors have been widely used for macromolecular and chemical crystallography studies. For protein structure studies, X-ray beams with wavelengths above 1 Å are commonly applied. However, for small-molecule structure analysis, and in particularly in charge-density studies, Mo-K α or shorter-wavelength X-rays, available at synchrotron sources are preferable. Because the phosphor layer of the CCD or image plate cannot completely absorb the short-wavelength X-ray, an oblique-incidence dependent correction is imperative [1].

Experimental: Two sets of measurements were made on a crystal of diaquobis(hydrogen phthalate)Cu(II). The data were collected at the X3A1 beamline at NSLS at Brookhaven National Laboratory with 0.394 Å. A Bruker SMART-6000 CCD detector was used. In the first data set, the reflections were collected by locating the CCD at 2 θ =40, 30, 20, 10, 0 and -10 degree, respectively. At each 2 θ position, 110 frames with 0.3deg oscillation of phi angle were collected. In the second data set, a full data of 1350 frames were collected, with the CCD located at 2 θ =30°. The perpendicular transmission of SMART-6000 CCD phosphor was determined as 56.08% for 0.394 Å radiation.

Results: The first data set provides a convincing proof of the oblique-incidence dependence of the intensities through analysis of the intensities of identical reflections measured at different values of the incident angle α [1] (Fig. 1). Furthermore, application of the oblique correction $I_{\text{corr}} = I_{\text{obs}} [1 - T_{\perp}] / [1 - \exp(\ln(T_{\perp}) / \cos(\alpha))]$ (here T_{\perp} is the perpendicular transmission of the phosphor layer) leads to a significant improvement in both the R_{merge} factor and the final R of the structure refinement (Table 1), showing that the correction cannot be ignored in high-accuracy short-wavelength studies.

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Reference:

[1] Zaleski J, et al, J. Appl. Cryst. (1998). 31, 302-304

[2] Bartl H., et al, Z. Kristallogr. (1980), 152(3-4), 161-7.

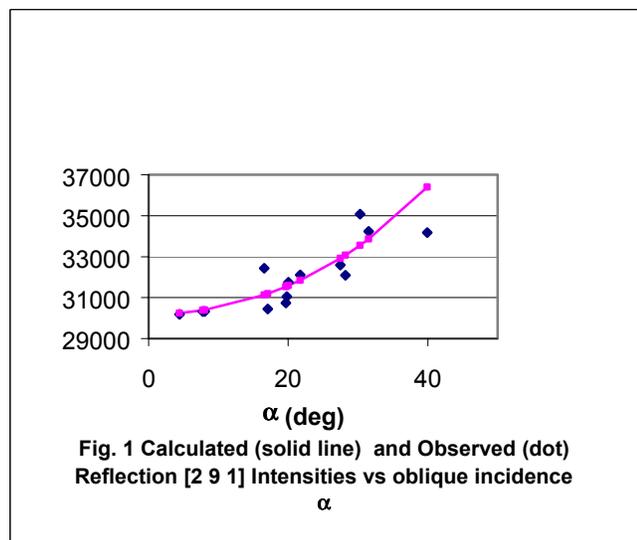


Table 1. The result of R_{merge} and structure refinement for the 0.394 Å synchrotron data with and without oblique correction*

	without correction	with correction	Ref. No.
R_{merge}	5.85%	4.30%	49163
$R_{\text{refinement}}$	3.46%	2.78%	14383*

*unique reflections with $I > 2\sigma(I)$