ELECTRONIC AREA DETECTOR DATA REDUCTION SYSTEM AT THE SRS

J. Campbell, D. Croft, J. Helliwell, P. Machin, M. Papiz, A. Thompson

To cite this version:

HAL Id: jpa-00225831
https://hal.archives-ouvertes.fr/jpa-00225831
Submitted on 1 Jan 1986

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
ELECTRONIC AREA DETECTOR DATA REDUCTION SYSTEM AT THE SRS

J.W. CAMPBELL, D. CROFT, J.R. HELLIWELL(1), P. MACHIN, M.Z. PAPIZ and A.W. THOMPSON

SERC Daresbury Laboratory, GB-Warrington WA4 4AD, Great-Britain

Abstract - With the current detector and computer technology, data collection at synchrotron x-radiation sources poses special problems. Fast data collection rates are essential for kinetic experiments at room temperatures. If maximum reflection measuring rates of $10^2$ to $10^3$ reflections per second for monochromatic and $10^5$ reflections per second with white beam are to become feasible, some simplifications at data collection time must be made, at the expense of possible complications during data reduction at a later stage.

The main benefit of EAD's over film is that a 3D profile scan of each reflection can maximise the peak-to-background ratio. The benefits are even more marked on synchrotron sources since the intrinsic mosaicity of protein crystals can be small. With optimum collimation the angular spread of a reflection can be reduced 5-fold over a conventional source, thus giving further improvement in signal-to-noise.

Brief details are given of the EAD system (hardware and software) for protein crystallography at the SRS wiggler beam line, based on a TV system for $0.5 \leq \lambda \leq 1.5$ Å.

I - INTRODUCTION

For many experiments in protein crystallography at a synchrotron x-radiation source, film is still the most efficient and reliable data recording medium. Where photon count rates are low, for instance in an anomalous dispersion experiment, weak scattering samples or monochromatic time-resolved crystallography, the sensitivity of electronic area detectors (EAD) offers more efficient and accurate methods of data collection. The main benefits of EAD over film are improved counting statistics and an on-line control of the experiment.

Computational and detector hardware problems are encountered when high x-ray fluxes at the sample are combined with the kind of sensitivity available in an EAD. Monochromatic data collection rates at a synchrotron facility using an EAD can be in the range 100-600 reflections per second which corresponds to a dataset within 10 min. In practice the limitations of encoding the signal, reading out the digitised image, predicting reflection positions on the detector image and writing the integrated intensities to a storage medium can increase the data collection times considerably. When hardware is a limiting factor to the rates of data collection, surplus x-ray flux can be sacrificed to improve protein crystal lifetimes, spectral resolution and to stabilise the x-ray beam by reducing heat loading on optical elements.

A number of EAD are being commissioned at the Daresbury SRS which could be used for the purpose of protein crystallography. Two multi-wire proportional chambers, suitable for the 7.2 experimental station on the bending magnet, where they would be

(1) Present address: Department of Physics. University of York, Heslington, GB-York YO1 5DD. Great-Britain
used at wavelengths above $\lambda = 1.5 \, \text{Å}$, and a TV detector system based on the design of Arndt and Gilmore [1,2] as manufactured by Enraf-Nonius. The TV detector system has been installed on the wigglner beam line (station 9.6) and will be used in the wavelength range $0.5 < \lambda < 1.5 \, \text{Å}$. The discussion here will be limited to the TV detector system, with an assessment of the problems and usefulness of running such a system at a synchrotron facility.

II - INSTRUMENTATION

The protein crystallography station on the wigglner beam line has a number of optical elements (figure 1) which provide a wide range of experimental conditions at the sample [3]. A 0.5 mm vertical x-ray source size is produced at the sample with a 75 cm, cylindrically bent, Pt coated, fused quartz focussing mirror. A monochromator vessel containing a 200 mm triangular bent horizontally dispersing monochromator is used in experiments where high fluxes are required; for instance a Ge(111) monochromator is used under conditions of 2 GeV machine energy, 5 T wigglner magnetic field and 200 mA electron beam current to produce a flux of $7 \times 10^{11}$ photons s$^{-1}$ mm$^{-2}$ at 0.1% bandwidth. Single-crystal Si(111) monochromators, of various oblique cuts, are available for anomalous dispersion experiments with a $\delta \lambda/\lambda > 3 \times 10^{-4}$. For rapid tunability a Si(220) channel cut monochromator follows the first monochromator vessel, with the mirror set to reflect a parallel beam a minimum $\delta \lambda/\lambda$ of $8 \times 10^{-5}$ can be achieved. Because the Pt mirror has been set to a 3 mrad tilt to reject $\lambda < 0.5 \, \text{Å}$, for the case of a Si(111) or Ge(111) monochromator, a vessel containing a flat gold-coated mirror has been installed after the monochromator vessel to reject harmonics of fundamentals greater than 1.5 Å.

![Fig.1 - Scheme of beam optics. Mono1 is a 20 cm triangular Ge(111) or Si(111) monochromator—Mono2 is a channel cut Si(220) monochromator.](image)

The Enraf-Nonius TV diffractometer is mounted on a motorised experimental carriage to facilitate alignment to the x-ray beam. The 28 arm, on which the detector is mounted, swings in the vertical plane. The protein crystal is mounted on a Kappa goniostat with a horizontal $\omega$ axis. The detector has an active area of $64 \times 48 \, \text{mm}^2$. A 6-bit ADC register is read into a 512 x 512 16-bit deep mass store every 40 ms. The gain of the image intensifier plus camera can be varied through a range of 1:250. A MICROVAX-I computer runs the crystallographic software. At the present time the primary x-ray beam decay is monitored from a V/F converter and PDP-11/23 computer via CAMAC. In the future CAMAC will be connected directly to the MICROVAX-I computer. Data are transferred over ETHERNET or on magnetic tape to a VAX-11/750 where the raw data are reduced and merged.

III - SOFTWARE

The data collection software package MADNES (J. Pflugrath and A. Messerschmidt, this meeting) was used. This program suite measures small rotation images of data, reduces them to indexed three-dimensional (3D) reflection profiles and optionally integrates the intensities within the profiles. The 3D profiles were modified by correcting each $\phi$ rotation slot within the profile for primary beam decay.
Fig. 2 - Intensity versus time measurements from the ion chamber. (a) Full time scale of the experiment. (b) Expanded view of the first 550 s.
The integrated intensities were merged and \( R_{\text{sym}} \)'s calculated by a modified version of the rotation film merging program AGROVATA written by Phil Evans (MRC, Cambridge).

IV - EXPERIMENTAL

The detector hardware and software were evaluated by collecting data of pea lectin during 'single bunch' running of the SRS. The machine running conditions were: 2 GeV energy, wiggler magnetic field of 5 T and a beam current at injection of 20 mA, decaying to 7 mA by the end of the experiment. The primary x-ray beam was monitored with an air gap ion chamber placed after the collimator. The readings from the chamber integrated at 1 s intervals, were written through CAMAC and an LSI-11/23 onto a Winchester disk. These intensity readings were used to correct the crystallographic data for beam intensity fluctuations. Monochromatic radiation was produced from a Si(111) monochromator of oblique cut, 6.75°. The wavelength of radiation was 0.884 Å and the correlated spectral dispersion \( \left( \delta \lambda / \lambda \right)_{\text{COR}} = -2 \times 10^{-3} \). The divergences of the beam in the horizontal and the vertical directions were 0.15° and 0.013°, respectively. The protein crystal used to collect data was spg. \( P2_12_12_1 \), cell dimensions \( a = 50.8 \) Å, \( b = 61.4 \) Å and \( c = 136.4 \) Å. The rotation axis was \( b \) and the \( \phi = 0° \) position was defined by the \( c \) axis antiparallel to the x-ray beam. Images were collected at 0.1° intervals with integration times of 30 s. An electronic background image was measured at the start of data collection and was subtracted from all subsequent data images. Corrections for non-uniform detector response were made during the experiment from a pixel look-up table. The look-up table was generated by illuminating the detector with a uniform plane wave of x-radiation from an uncollimated Cu Kα sealed x-ray source. A total of 3.3° of data were collected within a time of 80 min.

V - DISCUSSION

The results of data collection are shown in Tables 1 and 2. The deviation of intensities on symmetry related reflections is 5.8%. This is of the same order of magnitude as the deviations in the ion chamber readings (figure 2) used to correct the

| Table 1 - \( R_{\text{sym}} \) by resolution bins. |
|-----------------|------|-------|------|
| \( d_{\text{min}}(\AA)(a) \) | \( R_{\text{sym}} \)(b) | \( \langle I \rangle \)(c) | \( N \)(d) |
| 5.15 | 0.036 | 398 | 48 |
| 4.58 | 0.034 | 566 | 60 |
| 4.16 | 0.034 | 527 | 48 |
| 3.84 | 0.039 | 572 | 62 |
| 3.59 | 0.063 | 388 | 80 |
| 3.38 | 0.065 | 452 | 70 |
| 3.20 | 0.088 | 432 | 86 |
| 3.05 | 0.084 | 375 | 72 |
| 2.92 | 0.114 | 410 | 16 |

| Table 2 - \( R_{\text{sym}} \) in intensity ranges(a). |
|-----------------|-------|------|
| \( \langle I \rangle \) | \( R_{\text{sym}} \) | \( N \) |
| 72 | 0.177 | 44 |
| 147 | 0.098 | 98 |
| 258 | 0.065 | 94 |
| 353 | 0.079 | 68 |
| 459 | 0.063 | 66 |
| 542 | 0.054 | 44 |
| 650 | 0.064 | 28 |
| 760 | 0.043 | 18 |
| 860 | 0.045 | 20 |
| 948 | 0.086 | 14 |
| 1050 | 0.017 | 10 |
| 1172 | 0.026 | 8 |
| 1523 | 0.038 | 30 |

- Total 456 0.058 542

(a) \( d_{\text{min}}(\AA) \) minimum Bragg plane of the range.
(b) \( R_{\text{sym}} = \frac{\sum_{h \in I} \left| \langle I(h) \rangle - I_1(h) \right|}{\sum_{h \in I} I_1(h)} \)
(c) \( \langle I \rangle \) mean intensity.
(d) \( N \) number of reflections.

where \( I_1(h) \) are the set of equivalent reflections of indices \( h \) and mean intensity \( \langle I(h) \rangle \).

(a) average number of reflections greater than 3\( \sigma \) is 98%.

\[ \frac{\langle \sigma \rangle}{\langle I \rangle} = 0.083 \]

\[ \langle I \rangle = 456. \]

\[ \langle \sigma \rangle = 38. \]
data for fluctuations in the primary beam. The air gap ion chamber used in this experiment is inefficient and is influenced by the fluctuations in the environment. A better way of measuring the primary beam intensity would be with a sealed Xe/A gas filled ion chamber. A serious problem in this experiment was the beam movement observed over a period of 2500 s. Although this was corrected for, with a primary beam monitor, it was an undesirable feature of the experiment which it would be better to eliminate in the future. It is believed that the problem lies in the beam line optics and a possible solution would be to move away from single-crystal/mirror focusing optics to a system of a large vertical size parallel beam incident on an Si(220) channel cut monochromator.

The results suggest that useful data can be collected on the TV detector at the SRS. However, ultimate accuracy of data may not be achieved without a stable x-ray beam and an effective means of monitoring and controlling it.

REFERENCES