01.X-4 IMAGE PLATE PROTEIN DATA: AN EXAMPLE OF EXPERIMENT WITH A WEISSENBERG CAMERA USING SR IN THE PHOTON FACTORY. By N. Sakabe, National Laboratory for High Energy Physics, Oho, Tsukuba, Ibaraki 305, Japan.

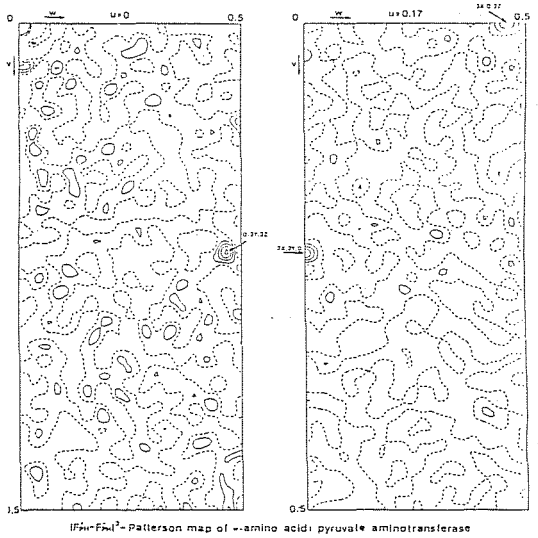
Fuji Imaging Plate(IP) is an area detector based on a phosphostimulable phosphor($BaFBr:Eu^{2+}$) screen to store a two-dimensional image produced by the irradiation of X-rays. The characterization of IP with Fuji computed radiography(FCR) system has been reported by J. Miyahara (J.Miyahara, K.Takahashi, Y.Amemiya, N.Kamiya & Y.Satow, Nucl. Instr. & Meth. Phy. Res. 1986, A246 572-578).

Mersalyl derivative of ω-amino acid:pyruvate aminotransferase crystals were used in this experiment. This enzyme is a tetrameric protein of 172,000 daltons and crystallized in space group I222 with a=124.67,b=137.90 and c=61.45A. The data was collected with a Weissenberg camera (N. Sakabe, J.Appl.Cryst.1983,16 542) with 143.5 mm radius of a cylindlical cassette in combination of IP (HR), when PF was operated at 2.5GeV and 150mA. The intensity data was collected at λ= 1.004A. The oscillation axis was the c-axis (4°/mm), and the oscillation angle was 18°. The exposure was stopped after 12 times oscillation, and exposure time was about 4 min. The total range of 162°were recorded at ten shots with overlapped area 2°. A complete data set was collected with a single

Method/ Scan step	Imaging plate/100μm			Photographic film/25μm		
Miller index	(7 8 1)	(4 5 1)	(5 6 1)	(7 8 1)	(4 5 1)	(5 6 1)
Integrated intensity	135	342	2933	1716	4386	14142
I/I(7 8 2)	1.00	2.54	21.8	1.00	2.56	8.24
Heighest peak value	23	44	440	53	95	206
Background level	4	3	4	29	28	29
S/N ratio	5.75	14.7	110.0	1.83	3.39	7.21
Half width(RxSmm)	0.3x0.25	0.3x0.25	0.3x0.25	0.23x0.2	0.28x0.15	0.30x0.25
Peak size(RxSmm)	1.0x0.6	0.9x0.7	1.3x1.2	0.33x0.3	0.45x0.3	0.53x0.35
Half width area (pixels)	5	6	4	49	50	55
Peak area (pexels)	37	44	100	113	165	256

crystal up to 2.3A resolution and the merge $R(F^2)$ was 0.056. The comparison of diffraction spots on IP and a film is in the Table. In order to know the feasibility, $(F^+ - F^-)^2$ Patterson maps were calculated and heavy atom vectors are clearly appeared even if the occupancy of the Hg is about a half. The absorption correction (C.Katayama, N.Sakabe & K.Sakabe, Acta Cryst. 1972, A28 293-294) is effective since after the correction, R value was reduced from 0.052 to 0.038. We conclude that the combination of the Weissenberg camera and IP is one of the best system for the data collection of protein crystals using SR.

We are grateful to Mr. J. Miyahara of Fuji Photo Film Co., Ltd. for reading out IP with FCR.



$(F^+ - F^-)^2$-Patterson map of ω-amino acid: pyruvate aminotransferase

01.X-5 SYNCHROTRON RADIATION LAUE DATA COLLECTION FOR KINETIC STUDIES. By [2]Donald Bilderback, [1]Keith Moffat, [1]Wilfried Schildkamp, [1]Doletha Szebenyi, [3]Brenda Smith Temple, and [1]Karl Volz.
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X-ray diffraction data from single crystals of typical proteins are very weak, numerous, and subject to systematic errors arising from radiation damage. Even with intense synchrotron x-ray sources, exposure times for monochromatic oscillation photographs lie in the range of seconds to minutes. This precludes time-resolved studies on those biochemical systems where lifetimes of structural intermediates are seconds or less, as is almost always the case. If the x-ray monochromator is dispensed with, then a polychromatic x-ray beam falling on a stationary crystal will yield a Laue diffraction pattern (Moffat et al., Science 223, 1423 (1984); NIM 222, 245 (1984); NIM A246, 627 (1986); Helliwell, J. Mol. Struct. 130, 63 (1985)). Such Laue patterns require extremely short exposure times for strongly scattering protein crystals, in the ms to s time range; yield integrated intensities even with a stationary crystal; may be quantitated with very good precision; may contain a substantial fraction of the entire unique data in one pattern; and do not suffer greatly from overlapping orders (Cruickshank et al., Acta Cryst. A, in press; unpublished results of the authors, and of D.W.J. Cruickshank, J.R. Helliwell and colleagues). These properties fit the Laue technique well both for static, and even more so for dynamic, data collection; that is, for time-resolved crystallography on a biochemical time scale.
The principles of Laue diffraction from macromolecular crystals, and preliminary application to time-resolved studies, will be discussed.

01.1-1 GREATLY REDUCED RADIATION DAMAGE IN RIBONUCLEASE CRYSTALS MOUNTED ON GLASS FIBERS.
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We have been engaged in a program of X-ray data collection, primarily from bovine pancreatic ribonuclease-A (RNase-A) crystals, at temperatures ranging from 320K to 98K as part of a study of protein structural dynamics. During the course of investigations at 220K, 180K, 160K, 130K, and 98K we have mounted RNase-A crystals on glass fibers and have noticed that these crystals exhibit almost no radiation damage whereas an RNase-A crystal at 160K that was mounted in a quartz capillary tube showed significant deterioration. Another data set, collected at 103K from a crystal of sperm-whale metmyoglobin mounted on a glass fiber, also exhibited no radiation damage.

RNase-A crystals in 50% MPD:H_2O and maintained at ~273K with an ice bath (MPD = 2-methyl-2,4-pentanediol), can be picked up on a glass fiber that has a ball of uncured epoxy cement near the tip. The glass fiber itself is glued to a brass pin which is mounted on a standard goniometer head. The crystal is pressed into the uncured epoxy and, once adhering to the fiber, the whole assembly is quickly transferred to the diffractometer where the crystal is prevented from drying out by virtue of the nitrogen-gas cold-stream of the low-temperature device. The crystal is not necessarily covered completely by the epoxy.

Data collection from RNase-A at 160K, with the crystal mounted in a quartz capillary tube, showed a 21% decay of the intensity standards after 14.6 days of X-ray exposure. RNase-A crystals mounted on glass fibers, however, showed virtually no decay of the intensity standards even after extensive X-ray exposure. For