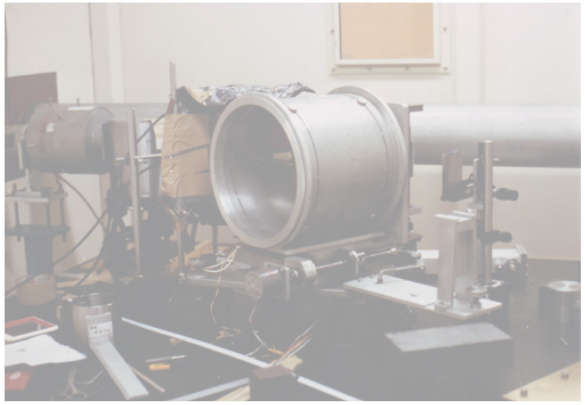
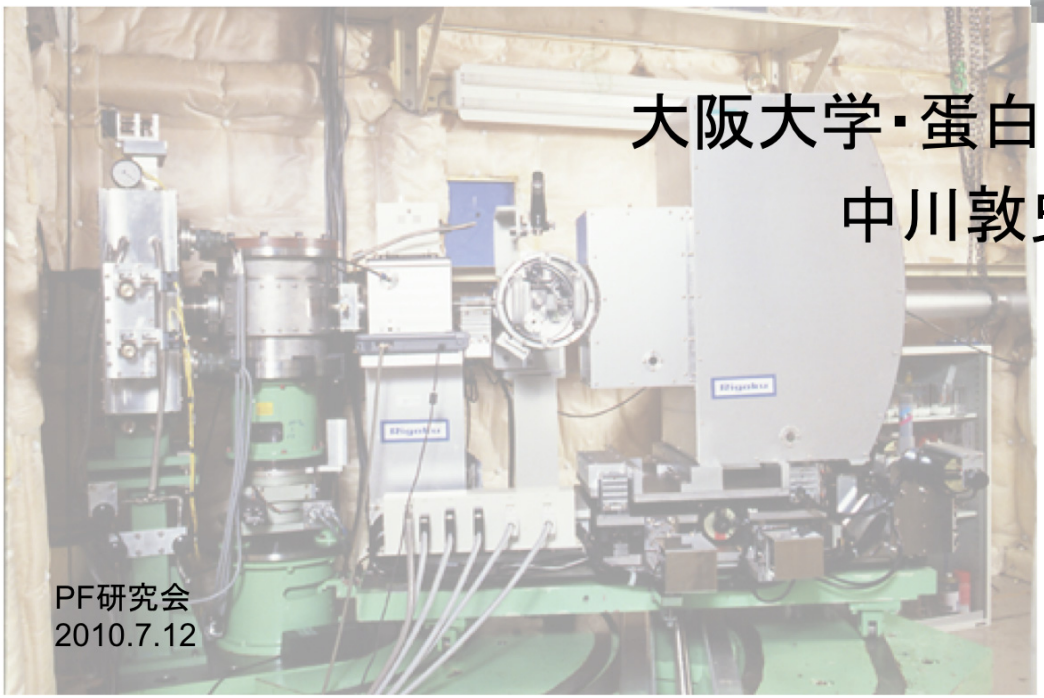


放射光を用いた異常分散の利用と生体超分子複合体の構造解析

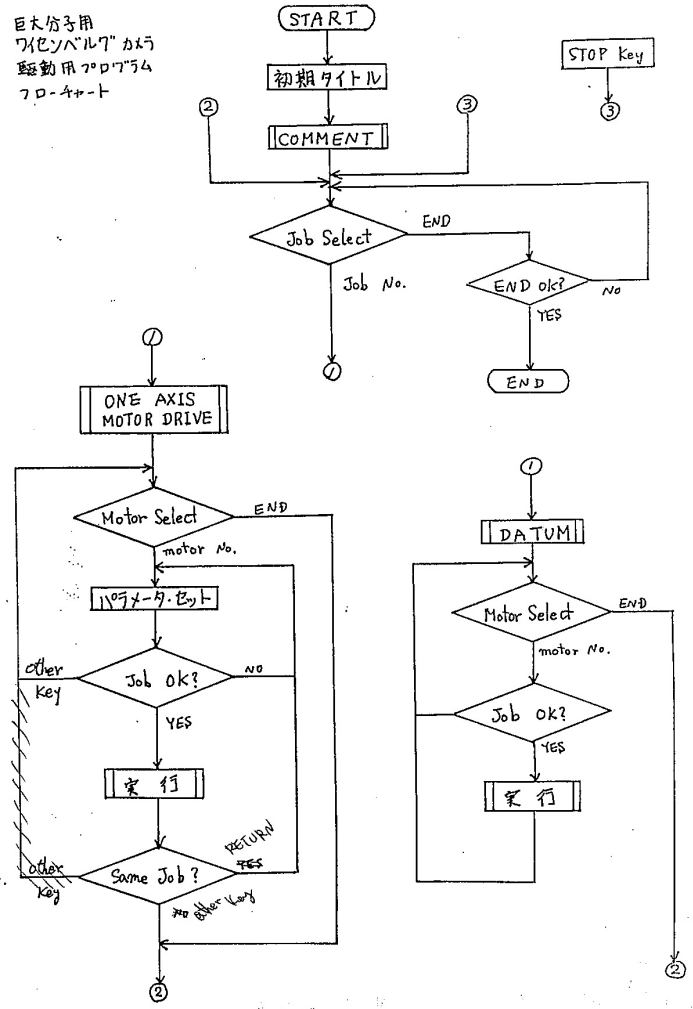
大阪大学・蛋白質研究所

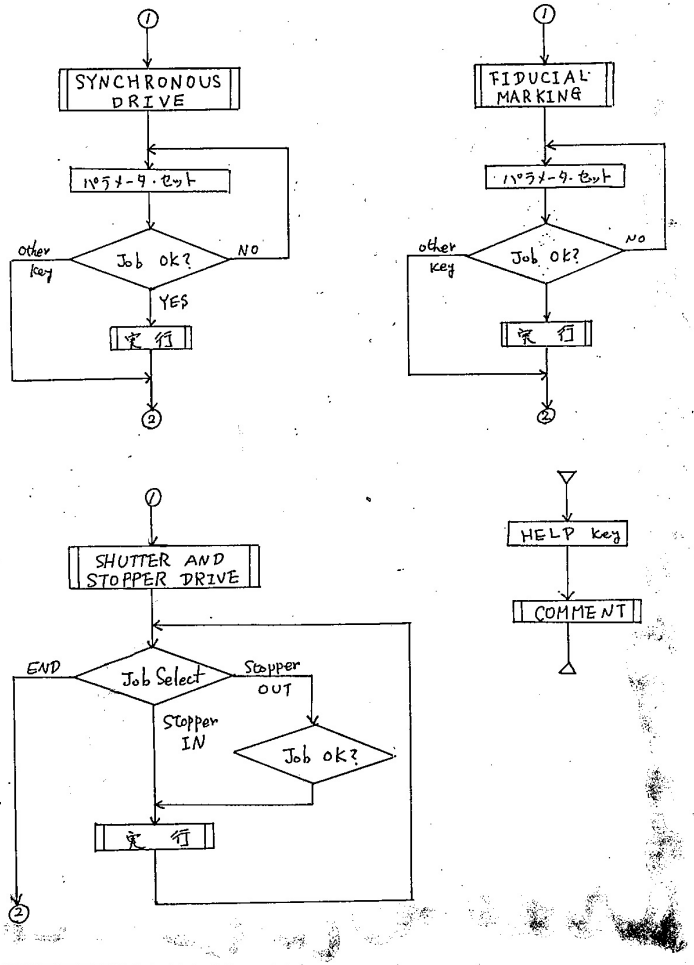
中川敦史



PF研究会
2010.7.12

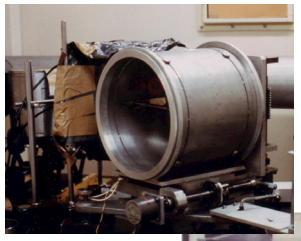
巨大分子用ワイセンベルグカメラ(2号機)駆動用プログラムのフローチャート

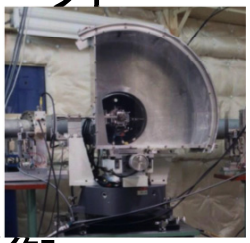


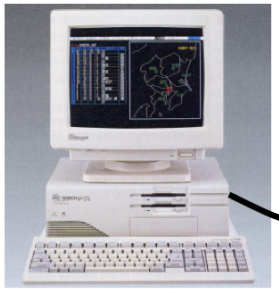
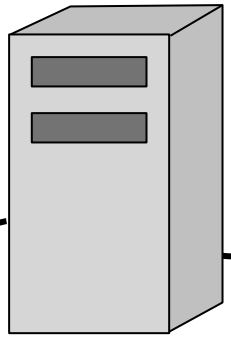


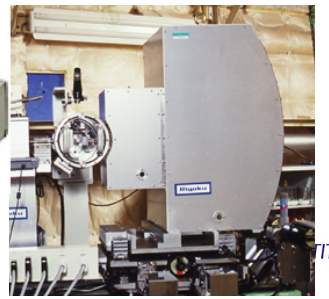
巨大分子用ワイセンベルグカメラの制御

- 1号機
  - 専用のパルスモータコントローラ
- 2号機
  - PCからのTTL信号でパルスモータドライバーを駆動
- 3号機　以降
  - 専用のコントローラをPCから制御

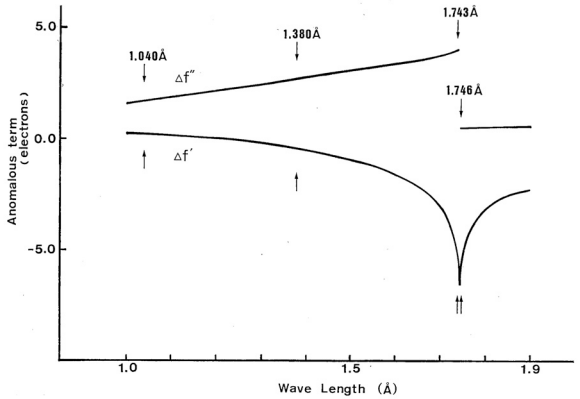


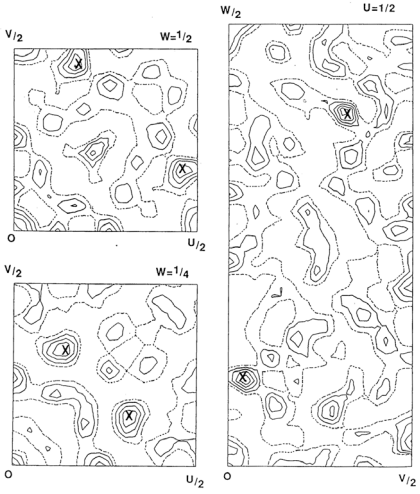



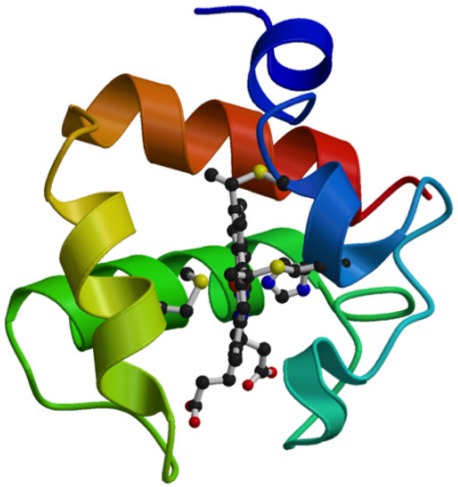


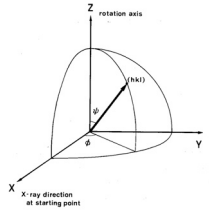
MAD法による硫酸還元菌チトクロムc-553の構造解析

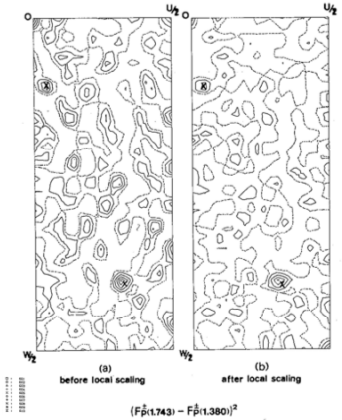


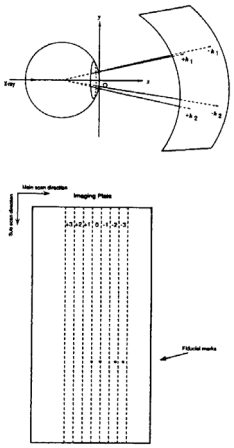


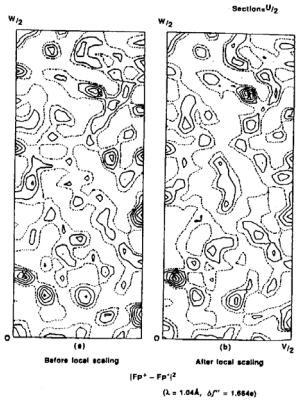
Bijvoet Patterson (1.04 Å)

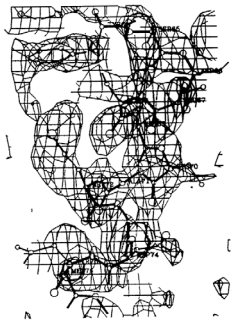


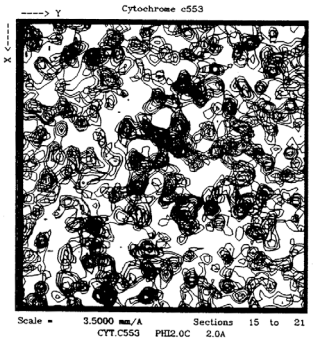


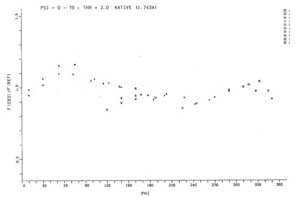












INSTITUTE for PROTEIN RESEARCH

1995年頃までに
MADによる構造解析が報告されたビームライン

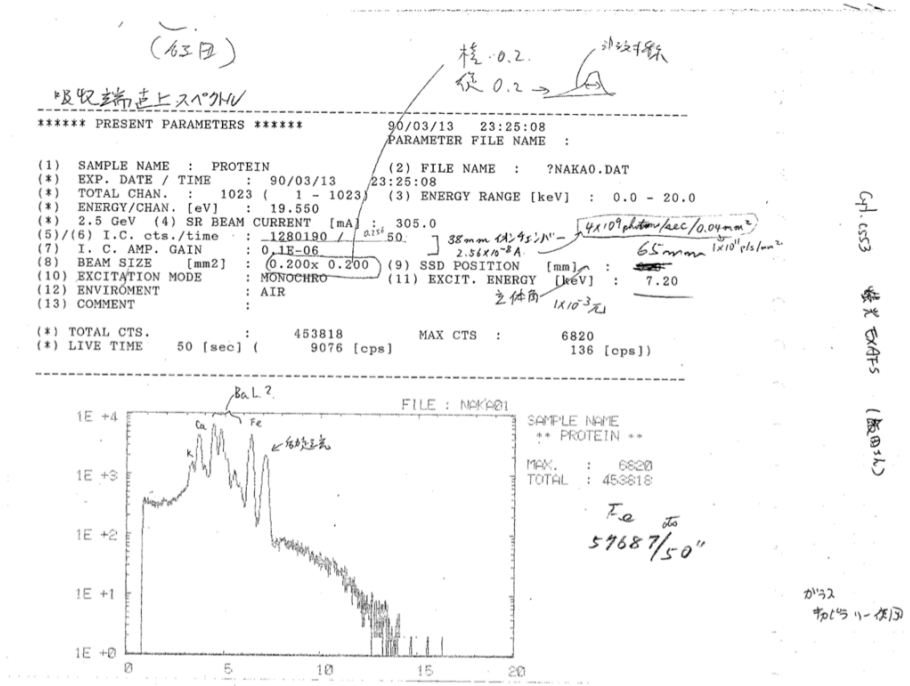
TABLE II
PARAMETERS FOR BEAM LINES THAT HAVE REPORTED MAD STRUCTURES

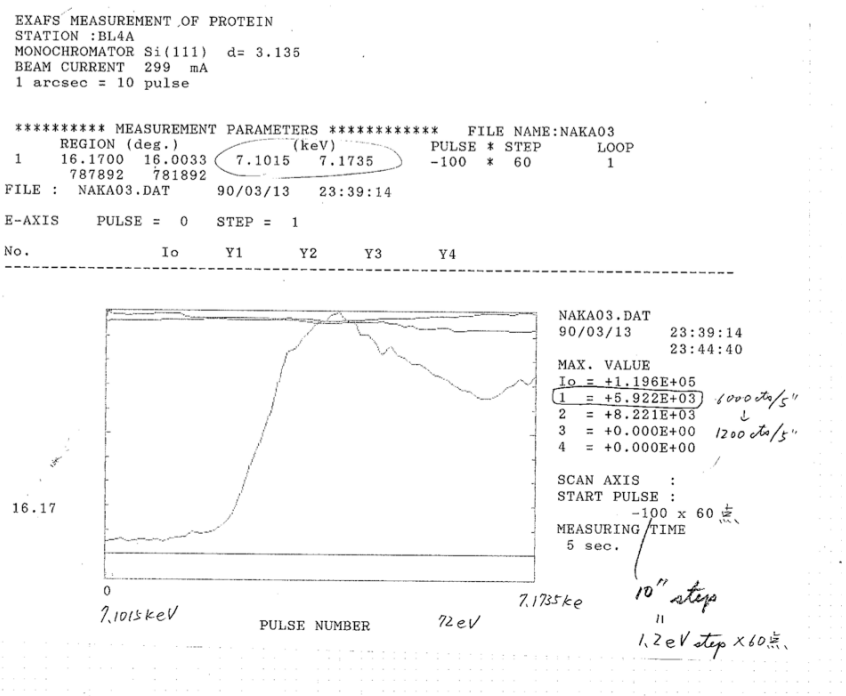
Source (energy)	Station	Magnet	λc (Å)	Mono	Mirror	Accessible λ range (Å)	Δλ/λ	Detector	Ref.
SSRL (3.0–3.5 GeV)	1-5 ES2	Bending	2.6	Double crystal	Yes	0.86–2.25	2×10^{-4}	BAS2000 MWPC	35, 49, 50 51–54
NSLS (2.584 GeV)	X4A	Bending	2.48	Double crystal	Yes	0.35–3.5	10^{-4}–10^{-3}	BAS2000	55–69
	X12C	Bending	2.48	Double crystal	Yes	0.89–1.61	10^{-4}–10^{-3}	MAR FAST	33, 53 70, 71
	X25	Hybrid wiggler	2.54	Double crystal	Yes	0.41–4.1	10^{-4}–10^{-3}	MAR BAS2000	72
CHESS (5.2–5.6 GeV)	F1	Wiggler	0.53	Bent triangle (channel cut)	Yes	0.9	1×10^{-3}	BAS2000 Kodak	73
	F2	Wiggler	0.53	Double crystal	Yes	0.3–1.8	1.5×10^{-4}	CCD BAS2000	47, 74, 32 75
PF (2.5 GeV)	BL14A	Vertical wiggler	0.6	Double crystal	Yes	0.15–2.5	2–8×10^{-4}	BAS2000 Scint.	20, 76, 77 78, 51, 79
	$BL6A_2$	Bending	3.1	Bent triangle	Yes	0.5–2.5	1×10^{-3}	Weissenberg BA100 BAS2000 IPR4080	25
SRS (2.0 GeV)	9.5	Wiggler	0.9	Channel cut	Yes	0.2–2.2	1.2×10^{-4}	MAR	80
LURE (1.72 GeV)	D23	Bending	3.4	Double crystal	None	0.95–2.2	–	MWPC	17
ESRF (6.0 GeV)	Troika	Undulator						MAR	61
DESY	BW-6	Wiggler		Double crystal	Yes				81

A single, bent-triangle monochromator will have less energy resolution than a double-crystal monochromator because of the horizontal source size, vertical length of the source in the case of an insertion device, and any deviations from ideality in the bending of the crystal.
Despite of these restrictions, Nakagawa *et al.* were able to solve the structure of cytochrome c_{553} on BL6A2 at the Photon Factory. (Hendrickson and Ogata, Meth. Enzymol. vol. 276 (1997))

INSTITUTE for PROTEIN RESEARCH

蛋白質結晶（チトクロムc_{553}）からの蛍光スペクトルの測定

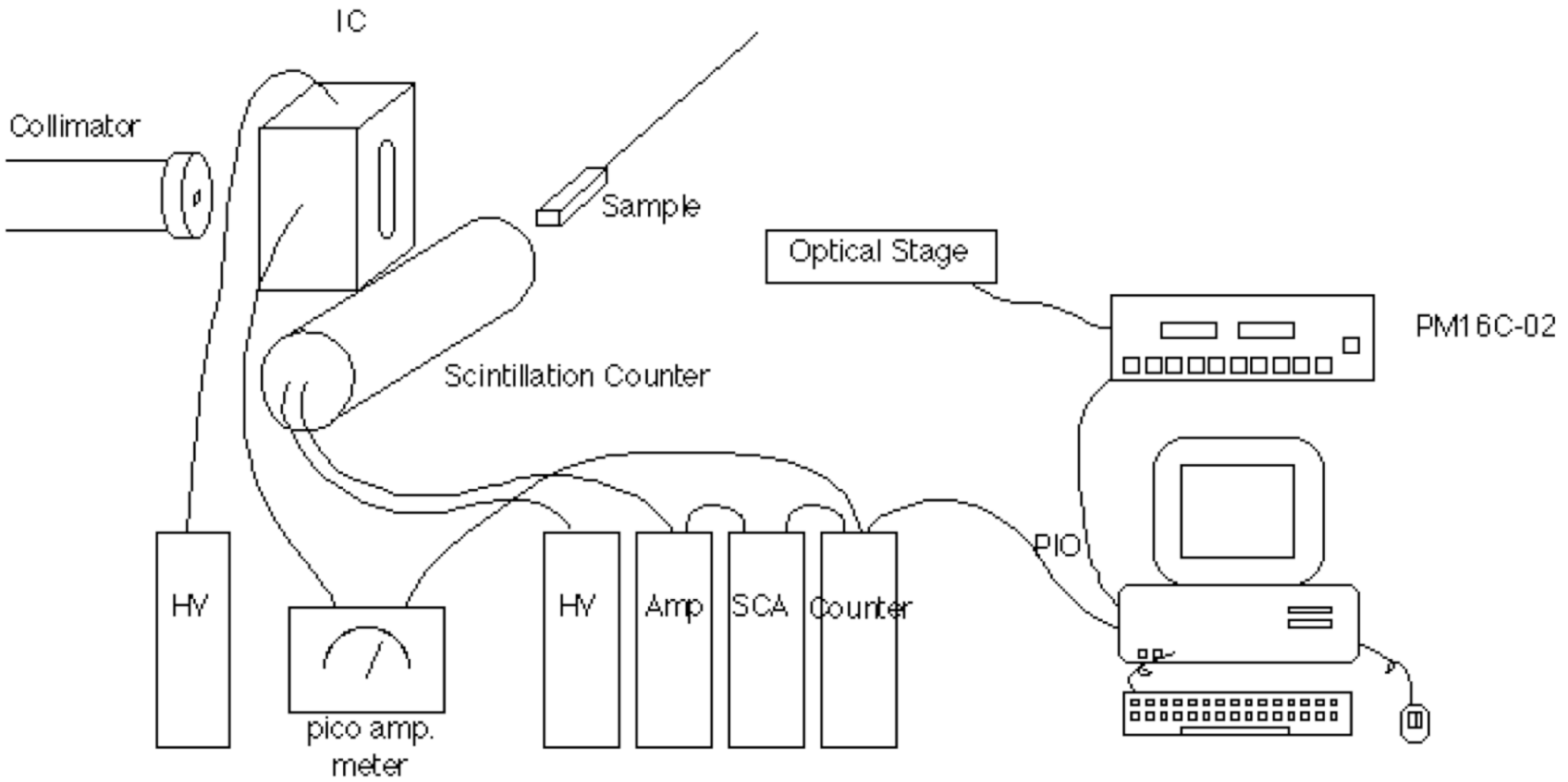




taken at 1990.3.13
at BL4A
by 飯田厚夫さん

INSTITUTE for PROTEIN RESEARCH

蛍光測定システム


IC
Collimator
Sample
Optical Stage
PM116C-02
Scintillation Counter
PIO
HV
pico amp.
meter
HV
Amp
SCA
Counter

光学系の評価（波長分解能）

KEK NATIONAL LABORATORY FOR HIGH ENERGY PHYSICS
1–1 OHO, TSUKUBA-SHI, IBARAKI-KEN, 305, JAPAN

TELEPHONE : 0298 (64) 1171
TELEX : 3652-534
CABLE : KEK OHO

8 October 1992

Prof. Wayne Hendrickson
Department of Biochemistry and
Molecular Biophysics
Columbia University
New York, NY 10032
U.S.A.

Dear Prof. Hendrickson

I have tried to measure XAFS data of $HoCl_3$ solution today. I am sending herewith some result of the experiments.

I used 60mM $HoCl_3$ solution to check energy resolution of our beamline. The data were collected with slit open and slit closing as I_0=1/2, 1/5, 1/10. The last one is the data from 30mM $HoCl_3$ solution, which is simular concentration of your crystal.
I also have data of $Yb(NO_3)_3$ solution. If you need, I will send them, too.

Would you please check these data? I will be very happy if you will give me some comments of the result.

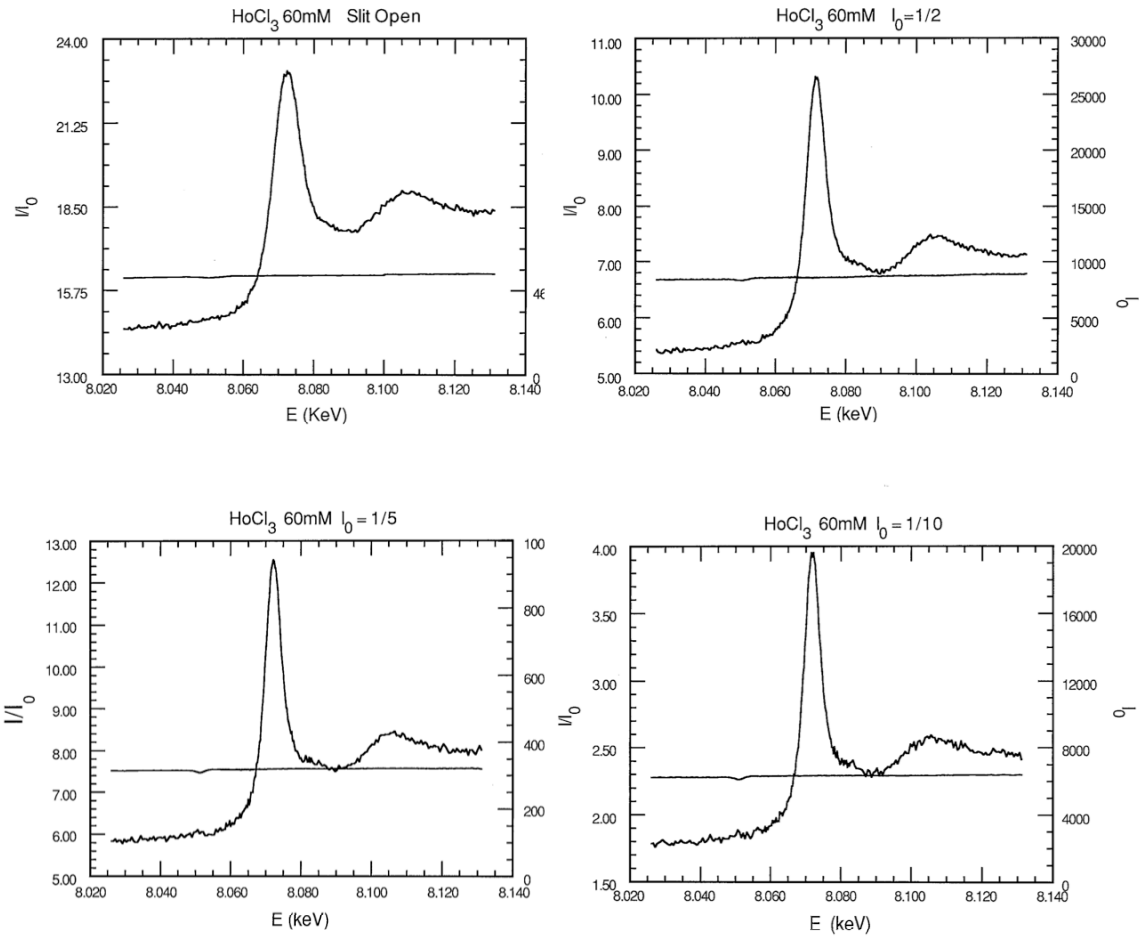
Sincerely yours

Atsushi Nakagawa

Atsushi Nakagawa
Photon Factory,
National Laboratory for High Energy Physics

Tel : 81-298-64-1171
Fax : 81-298-64-2801

e-mail : nakagawa@kekvax.kek.jp
nakagawa@jpnkekvx.bitnet



INSTITUTE for PROTEIN RESEARCH

ビームライン評価 by W. Hendrickson

HENDRICKSON_LAB TEL: 2123057379 Oct 13,92 0:35 No.001 P.01

Howard Hughes Medical Institute
Research Laboratories
Columbia University
Department of Biochemistry and Molecular Biophysics
630 West 168th Street
New York, New York 10032
Wayne A. Hendrickson
(212) 305-3456

FAX: (212) 305-7379

TO: ATSUSHI NAKAGAWA
FROM: WAYNE
DATE: 12 Oct '92
SUBJECT: Ho energy res'n
Number of page
MESSAGE:

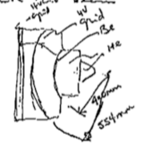
HENDRICKSON_LAB TEL: 2123057379 Oct 13,92 0:35 No.001 P.03

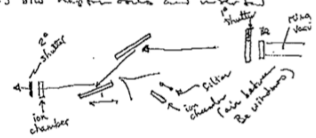
6 March 1990

Ho- MBP Experiment at LURE

18:20 Beam delivered 310 mA, 1.85 GeV

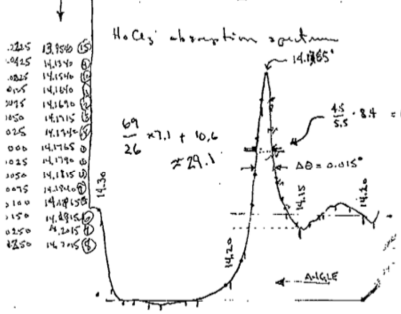
Filter prepared of HoCl3 pressed b/w Kapton foils and inserted [illegible]





Si 111 crystals
2nd crystal sagittally focused
No cooling of xtal. Mounted on copper slide.

HoCl3 absorption spectrum



$\lambda = 2d \sin\theta$

$\frac{d\lambda}{d\theta} = 2d \cos\theta$

$\frac{\Delta\lambda}{\lambda} = \cot\theta \, \Delta\theta = .0010$

$\Delta E_{K_\alpha} = \frac{\Delta\lambda}{\lambda} \cdot E_0 = (.0010)(8071) = 8.$

From Templeton Sm results

$\Delta\lambda/\lambda = 0.0015/1.845 = 0.0008$

$\Rightarrow \Delta E_{K_\alpha} \approx 6.6$ eV

Gd: $\Delta\lambda = 1.7107 - 1.7098$

$\Delta\lambda/\lambda = .0009/1.7104$

$\Delta E = 3.8$

$\Delta E(Ho) = 4.2$

7 March 1990

21:45 Bill's largest crystal in the beam for first 30 sec. 0.5° photo.

T = 18°C (16.5° on probe)
23:00 T = 16°C (14.5° ")
01:18 T = 14°C (12.5° ")
01:46 T = 12°C (10.5°

① θ = 14.1765° @ φ = 94.0 → 97.0° align = 94.2° x-rays along z
φ = 4.0 → 6.0° x-rays along z
0.1° frames, 40" per frame

02:46 T = 10°C (8.5°)

② θ = 14.1790° φ = 94.0 → 97.0°

03:32 T = 8°C (6.5°)

③ θ = 14.1815°

HENDRICKSON_LAB TEL: 2123057379 Oct 13,92 0:35 No.001 P.02

Howard Hughes Medical Institute
Research Laboratories
Columbia University
Department of Biochemistry and Molecular Biophysics
630 West 168th Street
New York, New York 10032
Wayne A. Hendrickson
(212) 305-3456

12 October 1992

Dr. Atsushi Nakagawa
Photon Factory
KEK, Tsukuba

Dear Atsushi:

I am very pleased by your experiments with holmium and encouraged by the results. I have made rough analyses of the spectra with the following results:

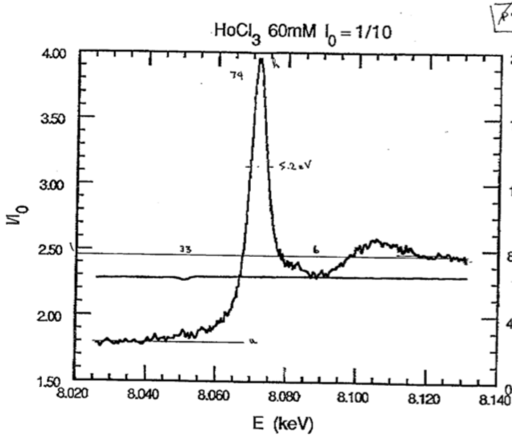
I/Imax	ΔE(FWHM)	f"(max)	Resolution
1.0	7.3 eV	19.0e	7.4 x 10^{-4}
0.5	6.0	24.0	5.5
0.2	5.1	25.7	3.7
0.1	5.2	26.6	4.0
Ho(LURE)	6.8	29.1	6.7
Gd(TTPH)	3.8	31.2	1.0

The LURE numbers are from our $HoCl_3$ specrum analyzed in the same way as for yours, and the Gd results are from Templeton et al. in Acta A38, 74-78 (1982). It seems that the intrinsic ΔE is about 4.1eV at Ho; while not everything is completely consistent, this implies energy resolutions by subtraction in quadrature as given above. Even at I/Imax = 0.5, this resolution would be excellent for a Cu experiment.

With an asymmetrically cut bent crystal, the actual energy resolution deteriorates as one moves away from the ideal asymmetric cut angle. For this reason it will be of interest to see the results from Yb, which has an LIII energy like that of the Cu K edge, and to know the cut angle and crystal material and face.

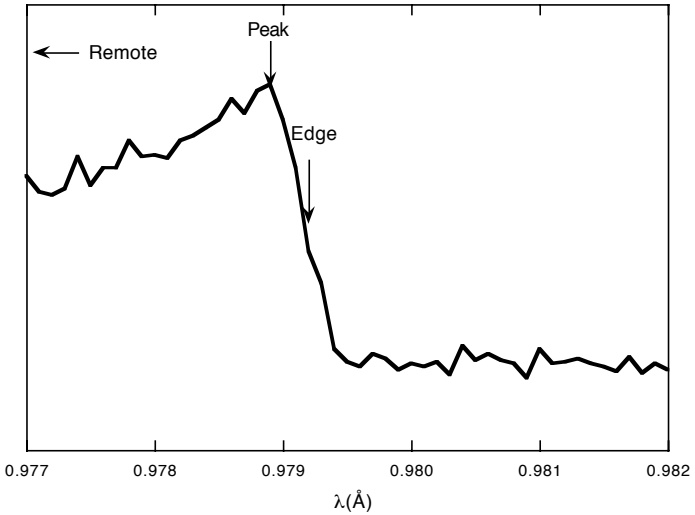
With best regards,

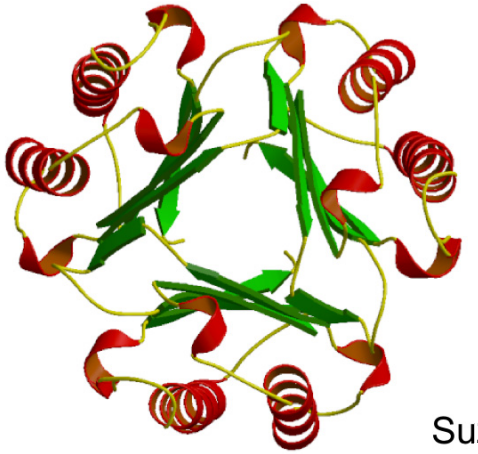
f'=19.9e p=26.6e

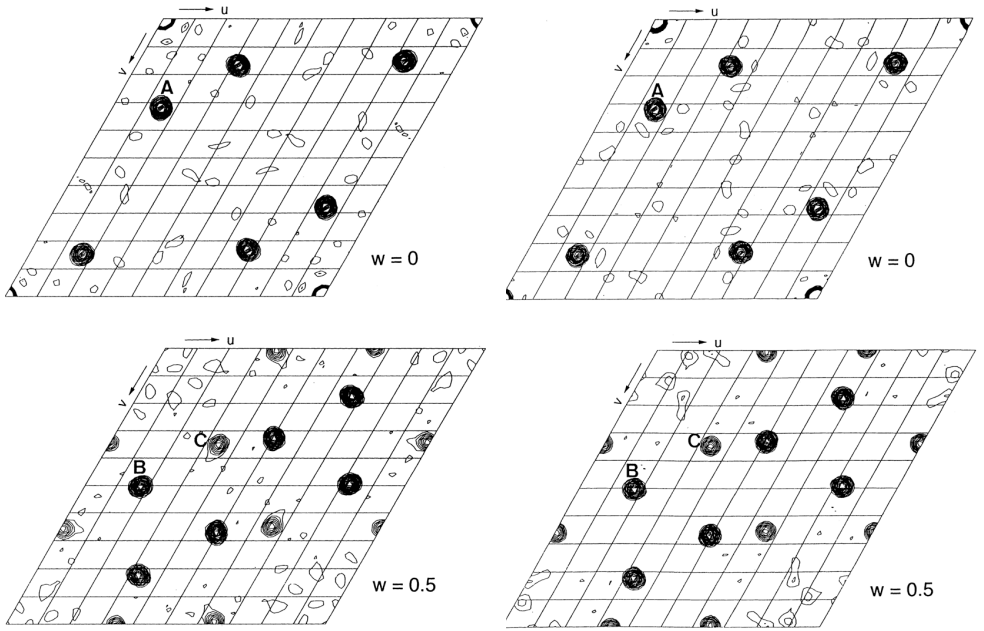


Macrophage Migration Inhibitory Factor (MIF) solved by Se-Met MAD

collaboration with Prof. I. Tanaka







Bijvoet difference Patterson

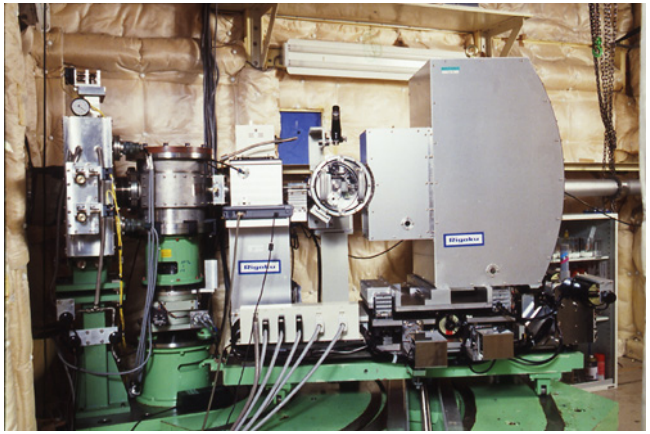
Dispersive difference Patterson

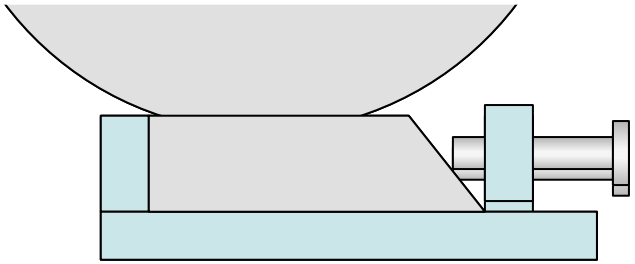
Suzuki *et al.* (1999) *Nature Struct. Biol.*, **3**, 259-266

INSTITUTE for PROTEIN RESEARCH

BL6Aでのトラブル

- ハッチのドア
- カセット取り





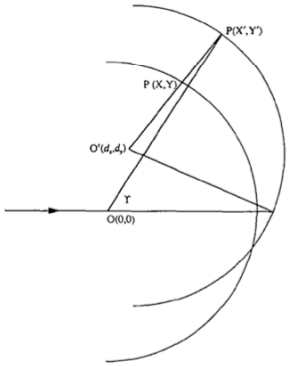


Fig. 1. Diffraction geometry. Deviation of cassette geometry from ideal is indicated by translation of the cylindrical camera axis from $O(0,0)$ to $O'(d_x,d_y)$. The crystal is at point O. Point $P(X',Y')$ is the position of a diffraction spot on the imaging plate when translation of the camera axis has occurred.

J. Appl. Cryst. (1992). **25**, 809–811

The Weissenberg method for the collection of X-ray diffraction data from macromolecular crystals: modifications to the data-processing program *WEIS*

BY BARRY A. FIELDS,[1] J. MITCHELL GUSS,[1]* MICHAEL C. LAWRENCE[2] AND A. NAKAGAWA[3]

[1] *Department of Inorganic Chemistry, University of Sydney, NSW* 2006, *Australia*

[2] *CSIRO Division of Biomolecular Engineering, Parkville, Victoria* 3052, *Australia*

[3] *Photon Factory, National Laboratory for High Energy Physics*, 1-1 *Oho, Tsukuba, Ibaraki* 305, *Japan*

(*Received* 5 *May* 1992; *accepted* 4 *August* 1992)

1993年の外国ユーザー

'93/4/8,10.11	Uppsala	
	* Janos Hajdu	
	Soichi Wakatsuki	
	Steven Edwards	
	Ian Clifton	
4/14,16	Univ. of Oxford	
	* Inger Anderson	
	Morell Mattnen	
	Thomas Taylor	
5/10,12	Univ. of .Oregon	
	* (Brian Matthews)	
	Larry Weaver	
	Michael Blaber	
	Robert Dubose	
	Raymond Jacobson	
6/30 7/2,3	Seoul National Univ.	
	* Se Won Suh	
	Sangsoo Kim	
	Kyeong Kyu Kim	
	Kwang Yeon Hwang	
	Dong Hae Shin	
	Changsoo Chang	
	Hyun Kyu Song	
	Kyeongsik Min	
10/13,15,17	Univ. of. California, Berkeley	
	* (Sung-Hou Kim)	
	Li-Wei Hung	
	XiangWei Weng	
	Alex Bohm	
10/26, 28, 30	Northwestern Univ.	
	* Alfonso Mondragon	
	Christpher Lima	
	Amit Sharma	
	Neal Lue	
10/27,29,31	Inst. of Food Research	
	* Richard Pickersgill	
	Gillian Harris	
	John Jenkins	
11/19	Univ. of Sydney	
	* Mitchell Guss	
	Ian Harvey	
11/21,23	CSIRO	
	* Jpseph Varghese	
	Albert Donkelaar	
	Michael Lawrenc	
11/22,24,26	Univ. of Toronto	
	* Emil Pai	
	Vincent Stoll	
	Bryan Eger	
11/27,28 12.1	Univ. of Alberta	
	* Randy Read	
	Norma Duke	
	Bart Hazes	
12/1,2,3	Univ. of Alberta	
	* Michael James	
	Stanley Moore	
	Ernst Bergmann	
12/5,7	Univ. of Alberta	
	* William Bridger	
	William Wolodko	
	Marie Fraser	
	Natalia Strynadka	
12/8,10,12	The Rockefeller Univ.	
	* Seth Darst	
	Joseph Kim	
	Ferre-d' Amare	
12/9,11,13	Harrington Cancer Center	
	* (Allen Edumundson)	
	Luke Guddat	
'94/2/24,26, 3/2	Birkbeck College	
	* (Tom Blundell)	
	Venugopai Dhanaraj	
	Matthew Groves	
3/8,10,12	Univ. of Oxford	
	* David Stuart	
	Edith Jones	
	Robert Esnouf	
	Jingshan Ren	
	David Stammens	
3/11,13,15	Daresbury Laboratory	
	* Samar Hasnain	
	Fraser Dodd	
	Richard Strauge	
	Ian Harvey	
3/19,21	Univ. of Sydney	
	* Mitchell Guss	
	Paul Curmi	
	Samantha Ashby	
	Sarah Tilley	
3/20	Biomolecular Research Inst.	
	* Joseph Varghese	
	Jennifer Martin	
	Thomas Garrett	
3/29,31	MRC	
	* Nobutoshi Ito	
	Chris Oubridge	

延べ２１グループ（実際は１９グループ）、延べ人数は７１人（実際は６９人）

INSTITUTE for PROTEIN RESEARCH