NSRRC_BL17B2 蛋白質結晶學實驗站之 Q-315 CCD 面積偵測器性能測試報告

Performance Test Report of ADSC Quantum-315 CCD Area Detector Used at NSRRC_BL17B2 PX Station

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(一) 簡介

基因體醫學核心設施 SPXF(Synchrotron Radiation Protein Crystallography Facility)包含了兩座光束線及實驗站(NSRRC_BL13B 和 NSRRC_BL13C)，為了滿足結構基因體學的研究需求，High-Throughput Crystallography 是必要的實驗手段。為了達到 High-Throughput 目標，在硬體設備上要儘可能使用最快最大的面積偵測器。目前市場上口碑最佳，最快最大的面積偵測器是美國 ADSC 公司 Quantum 系列的 CCD(Charge Coupled Device)面積偵測器，根據預算共購買兩台 CCD 偵測器，其型號分別是 Q315 和 Q210。這兩台 CCD 偵測器於 2003 年 4 月運抵 NSRRC，由於 CCD 偵測器的性能在高光強度的光源下表現較佳，因此應廠商要求，於 2003 年 9 月申請 NSRRC_BL17B2 光束線時間進行此二座偵測器的安裝及性能測試工作。NSRRC_BL12B2 蛋白質結晶學實驗站原配備一台 30 公分直徑，雙片 IP 的面積偵測器(Rigaku/MSC RAXIS-IV++)，各項偵測參數性能良好，兩片 IP 交替至曝光位置的時間(以下稱 Dead Time，DT)需要 150 秒，若使用此新型 CCD 面積偵測器，Dead Time 只有 2 秒，理論上數據收集效率可提高 75 倍。本報告將利用一些標準樣本的量測，來檢驗這台新的面積偵測器性能是否正常。

(二) 繞射儀系統

Q-315 CCD 面積偵測器是裝置在 CrystalLogic 公司製造的垂直 Phi 軸繞射儀上。下面列出此特繞射儀的規格：

Specification for Goniometer Used in NSRRC_BL17B2

Beam Height:

500 mm above base

Omega Axis:

Orientation: horizontal
Range: free rotation
Resolution: 0.0005 degree
Accuracy: +- 0.005 degree
Reproducibility: 0.0005 degree
Error sphere: <10 um
Slew speed: 600 degrees/min
Scan speed: 0.01~60 degrees/min
Home position: +- 0.005 degree
Distance from base of goniometer head to beam: 54 mm
Manual translation adjustment for goniometer head: +-20 mm
Distance from top-face of omega ring to beam: 300 mm
Distance from exit aperture to centered crystal: 10~15 mm

Motorized Detector Mount: (before reconfiguration)

Range: 50~500 mm
Accuracy: 0.1 mm
Slew speed: 300 mm/min
Weight capacity: 200 kg

Vertical Motorized Detector Mount: (before reconfiguration)

Range: -5~300 mm
Accuracy: 0.1 mm
Slew speed: 120 mm/min

Weight capacity: 200 kg

Motorized Detector Mount: (after reconfiguration)

Range: 50~800 mm
Accuracy: 0.1 mm
Slew speed: 300 mm/min
Weight capacity: 200 kg

Two-Theta Axis: (after reconfiguration)

Orientation: horizontal
Range: 0~45 degrees
Accuracy: +- 0.015 degrees
Reproducibility: 0.002 degree
Slew speed: 30 degrees/min
Weight capacity: 200 kg

Collimator & Shutter Assembly:

Collimator size: 0.1, 0.15, 0.25, 0.3, 0.5, 1.0 mm (x1), 0.2 mm (x2)
Collimator mounted on XY alignment device
Shutter latency: <10 msecs
Shutter repeatability: <1 msec
Shutter material: >1 mm tungsten carbide
Shutter synchronization with motion: <1 msec
Exit aperture: 1 mm
Exit aperture material: SS or silver or gold
Exit aperture mounted on XY alignment device
Ion Chamber: one with complete electronics

Beam Stop:

Material: >2 mm tungsten
Diameter: 2 mm O.D., 1.4 mm I.D.
Mounted on XYZ alignment device

Crystal Viewing System:

Two high-sensitivity CCD camera with 7:1 zoom
Double crosshair generator

Motor Controller:

Compumotor 6K series, or VEXTA 5 phase stepping motor series if requested.

Total Goniometer Weight:

Less than 1000 kg, but the design should be strong enough to prevent any mechanical deformation. The influence of this mechanical deformation on the coincidence of beam reference with omega axis should less than 10 um.

下圖為此繞射儀之照片：

(三) ADSC Q-315 CCD 面積偵測器系統

下表為ADSC Quantum CCD面積偵測器的一些特性參數：

Specification	Units	Quantum Q4R	Quantum Q210	Quantum Q315
Active Area	mm	188x188	210x210	315x315
# of Pixels	Quantity	2304 x 2304 5.31 Million	4096 x 4096 16.78 Million	6144 x 6144 37.75 Million
Pixel Size	Microns	82x82	51x51	51x51
Spatial Resolution FWHM	Microns	90	90	90
CCD Type		EEV 05-30 (1.1 K x 1.1K)	Thomson 7899 (2K x 2K)	Thomson 7899 (2K x 2K)
Dark Current	e/pixel/sec	0.030	0.015	0.015
Readout Times: Full Resolution 2x2 binned@430Khz	Seconds	9 and 3 1	1 330 ms @ 4 corner	1 330 ms @ 4 corner

下圖為此面積偵測器之照片：

(四) 性能測試結果(因專有名詞之故，此節將以英文撰寫)

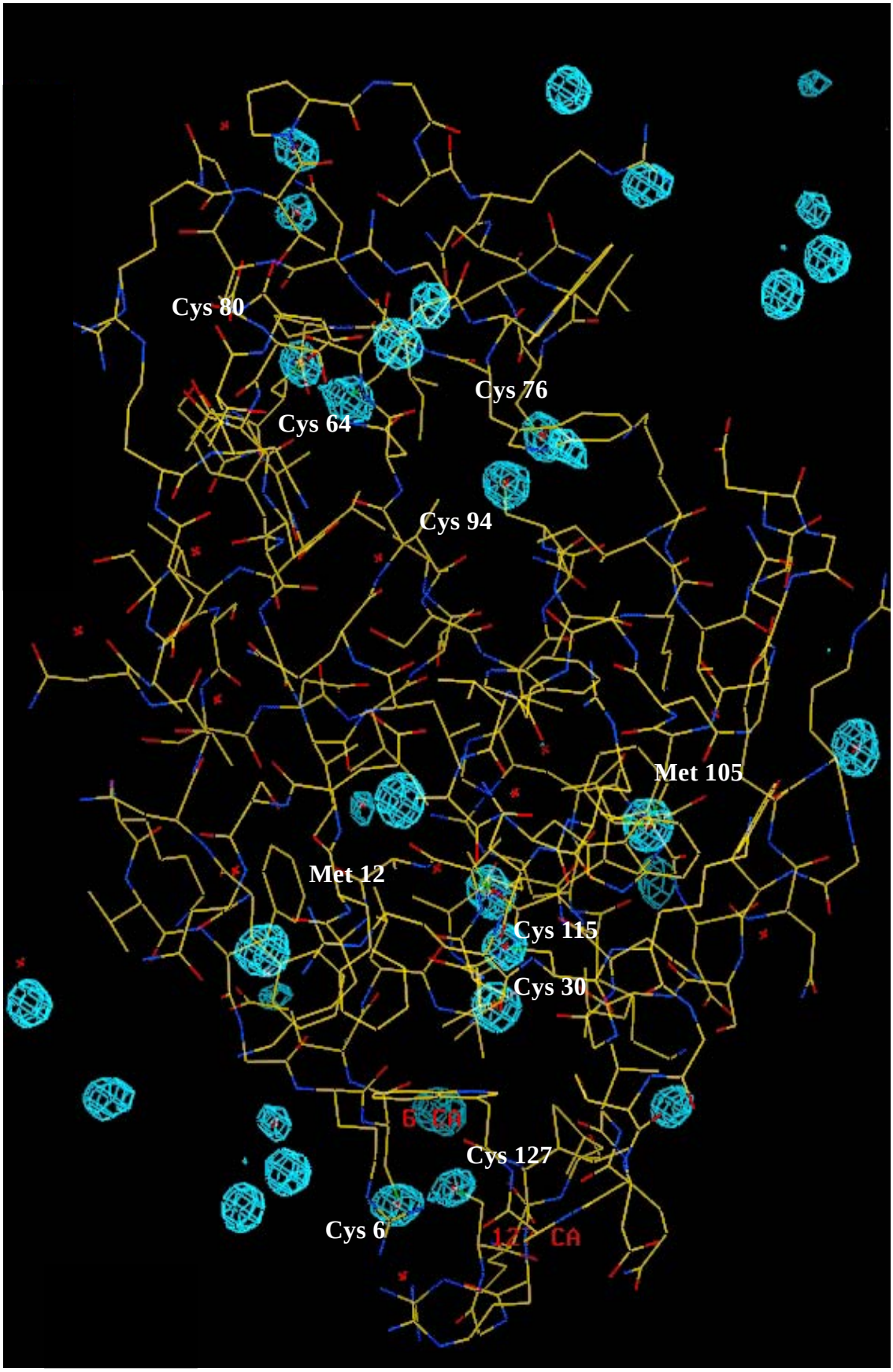
Lysozyme Crystal:

A well-accepted measure of the quality of data from a given detector is the measurement of the peak heights in the anomalous difference Patterson for P6 myoglobin. A strong set of Patterson vectors arising from the Fe atoms in the heme group has been the metric for high quality data. Recently, it has been argued that the test is not sensitive enough because the δ f" term for Fe at CuKα is 3.204 electrons.

Z. Dauter, et al. have reported a method for solving structures of strongly scattering macromolecules using the anomalous scattering signal of sulfur. A major prerequisite for using this method is data of extremely high quality because the δ f" term for sulfur at CuKα is only 0.557 electrons. This method may become more widely used as a tool for checking the quality of data produced by modern area detectors since the signal is much weaker, and, as a result is a more demanding experiment.

A total of 200 images of 0.5° oscillation angle and 20 second exposure time were collected at 10 keV and processed with HKL2000. The overall R_{sym} is 5.0% out to a maximum resolution of 1.40 Å with an average redundancy of 11.9, <I/σ(I)> of 28.6, completeness of 99.3% and χ^2=1.049. These data were imported into CNS and rigid body refinement using only the protein atoms of the lysozyme model from PDB. A difference Fourier map with the coefficients ΔFanom,ϕcalc-90° was calculated and the peak heights extracted. Below show the data statistics from HKL2000 and the results of the anomalous difference Fourier calculated for lysozyme. The phase angles were calculated using PDB entry 1LZ8. The position of each sulfur and chlorine is clearly marked by a significant peak. Features such as this are not possible unless the data is of the highest quality.

Shell Lower limit	Upper Angstrom	Average I	Average error	Average stat.	Norm. Chi**2	Linear R-fac	Square R-fac	Average Redundancy	Average I/error
30.00	3.02	83895.9	1724.6	363.0	0.936	0.031	0.035	12.6	
3.02	2.39	31465.0	716.9	129.3	0.995	0.053	0.060	14.5	
2.39	2.09	20937.0	488.8	173.3	1.081	0.056	0.062	14.1	
2.09	1.90	12442.9	323.5	149.7	1.162	0.065	0.065	13.3	
1.90	1.76	5934.2	152.5	116.7	1.129	0.083	0.081	15.1	
1.76	1.66	3658.9	136.7	125.3	1.047	0.115	0.108	14.9	
1.66	1.58	2550.3	153.8	147.2	1.061	0.158	0.143	12.5	
1.58	1.51	1575.1	214.5	211.1	0.946	0.207	0.181	7.3	
1.51	1.45	1178.3	353.8	351.4	0.943	0.220	0.196	3.1	
1.45	1.40	1006.3	436.2	434.6	0.954	0.242	0.204	2.0	
All reflections		18428.3	481.3	199.1	1.049	0.050	0.043	11.9	28.6



Anomalous difference Fourier of lysozyme using the coefficients ΔFanom,ϕcalc-90°

Atom	Peak Heights	
	Spring-8 BL 45XU	NSRRC BL17B2
CYS 30	5.60	12.55
CYS 64	4.97	10.77
CYS 80	4.96	10.35
MET 105	4.64	14.22
CYS 76	4.22	10.76
CYS 94	4.05	11.73
CYS 115	3.81	12.07
CYS 6	3.54	10.15
CYS 127	< 3.00	8.34
CL 201 (1)	5.05	8.09
CL 202	3.21	7.75
CL 203	3.43	7.60
CL 204	3.30	11.24
CL 205	3.72	7.69
CL 206	3.00	7.41

Peak heights, I/s, for the lysozyme anomalous difference Fourier

（五）結論

由上列標準樣本的數據分析顯示，這台 ADSC Q-315 CCD 面積偵測器可收集到極微弱的異常散射信號，且為高品質繞射數據，性能優良可放心使用。