A Neutron Biological Diffractometer in J-PARC

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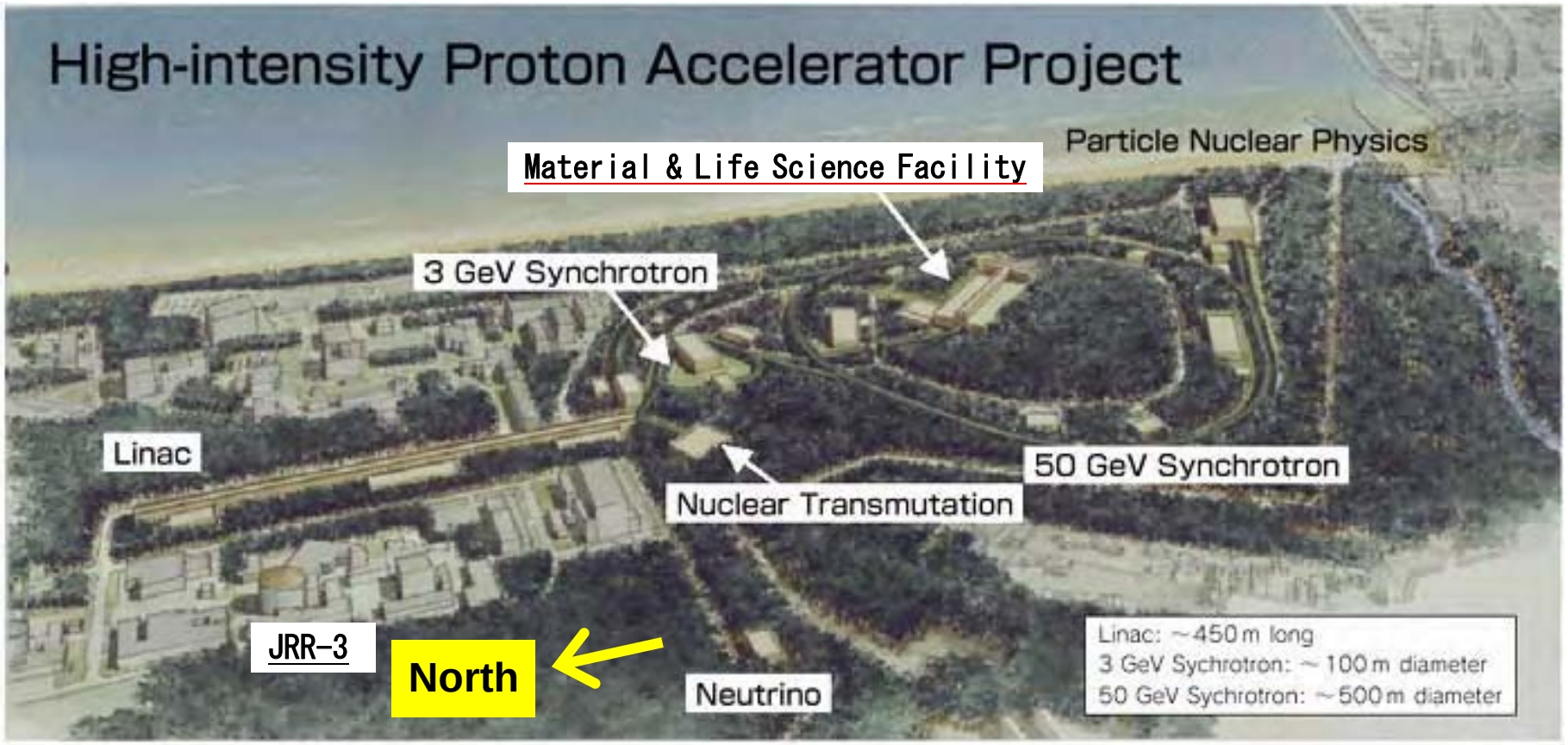
Abstract

At J-PARC in JAERI, Japan, Ibaraki Prefectural Government decided to build a neutron diffractometer for biological macromolecules for industrial use. The construction will finish in 2009. This diffractometer aims to make clear hydrogen-bond and hydration structures in biological macromolecules concerned enzyme activity mechanisms and to stimulate the industrial application such as pharmaceutics. The diffractometer is designed to cover the sample crystals which have their cell edges up to 135 Å. It is expected to measure 100 samples per year if they have 2mm^3 in crystal volume. The efficiency is more than 50 times larger than the present high performance diffractometers, BIX-3 and BIX-4 in JRR-3 reactor, in JAERI. To realize this performance, two important items should be developed; one is an area detector which must have a spatial resolution less than 1mm with time resolution of micro sec orders etc., and the other is a software which de-convolutes overlapped Bragg spots and presents an accurate integrated intensity of each Bragg reflection. This diffractometer will be installed at a coupled moderator, which has wider pulse shape but more intense peak and integrated intensities than a decoupled moderator, because it is the most important point to collect Bragg reflections from biological macromolecule crystals. The current status of these developments in J-PARC will be reported with the latest parameters of this diffractometer.

J-PARC

(Japan Proton Accelerator Research Complex)



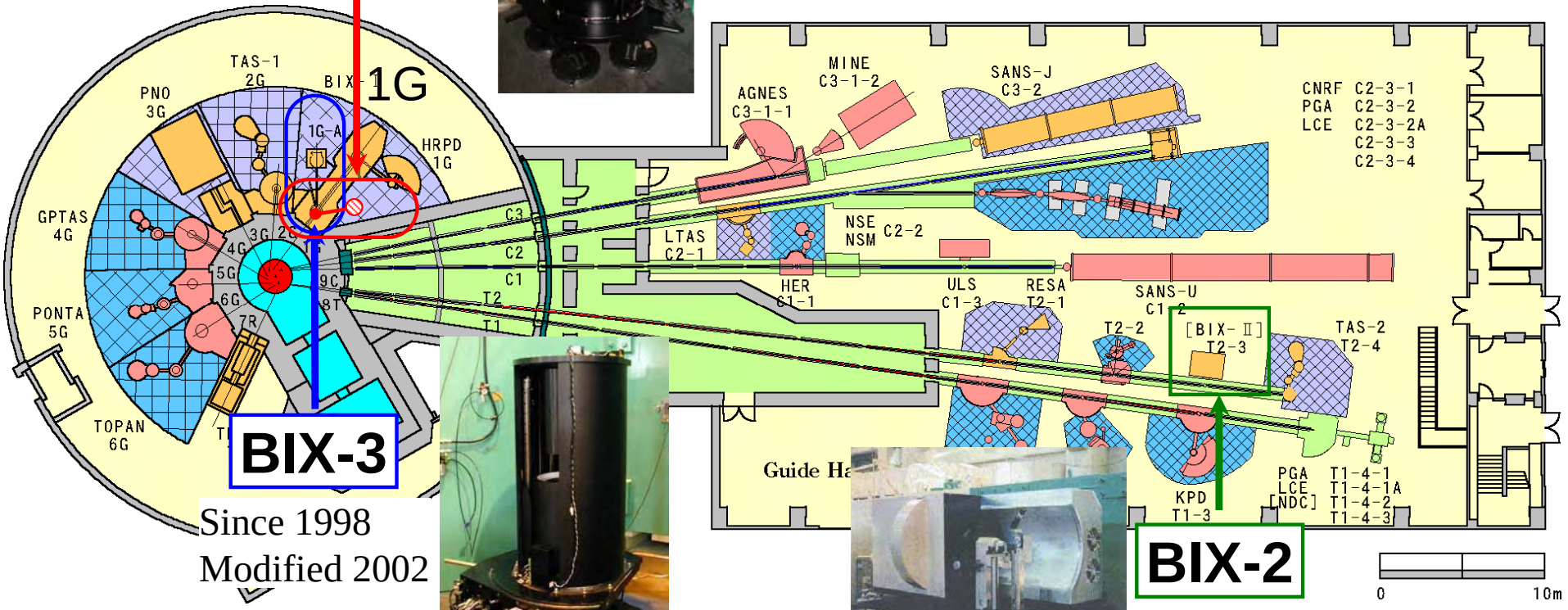
— The first neutron beam will come in 2007.

BIX-Type Diffractometers at JRR-3, JAERI

BIX-4
(BIX-3M)
Since 2002

Reactor hall

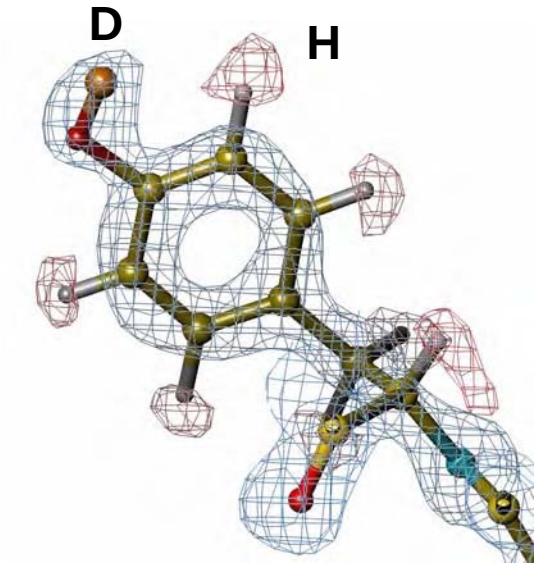
Guide hall

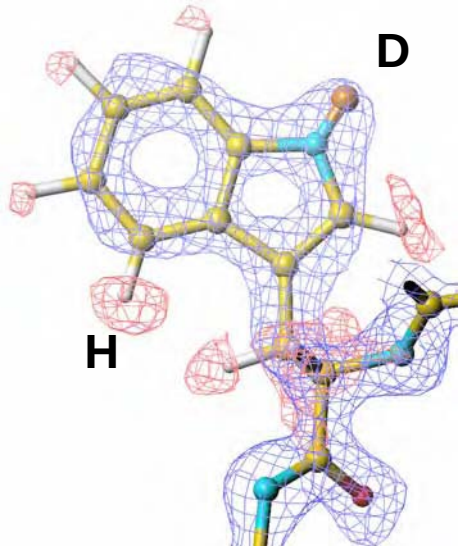


BIX-3
Since 1998
Modified 2002

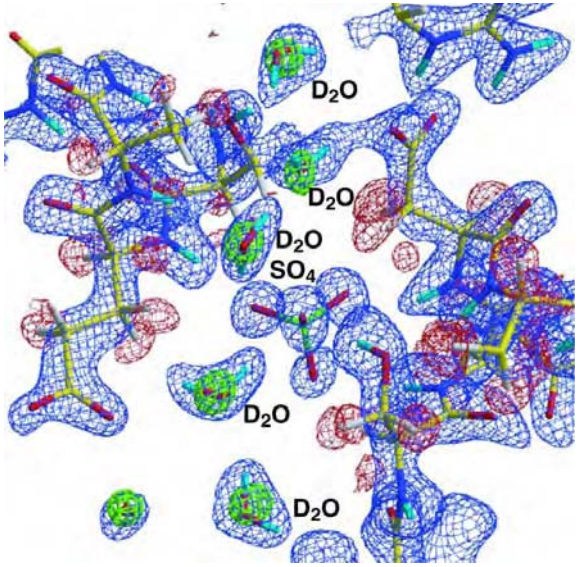
BIX-2
Since 1996

Typical Results of Neutron Protein Crystallography





Tyr10 (left), Trp3 (right)
in Rubredoxin (WT)



Various shape waters
in Met-myoglobin
(green contours are X-rays')

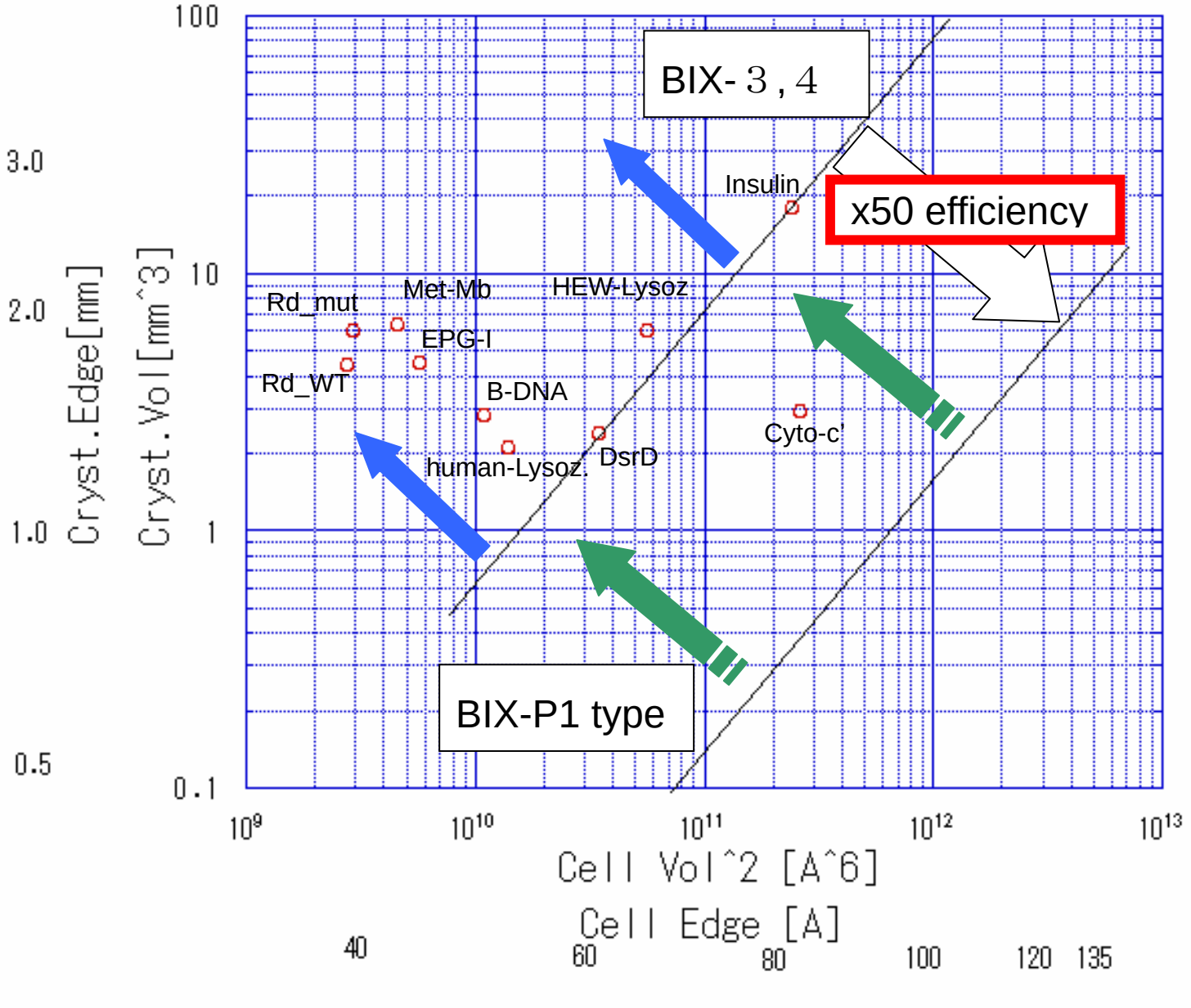
Both data were analyzed at 1.5 Å resolution, taken at BIX-3, JRR-3

Design Criteria
For Neutron Biological Diffractometer in J-PARC

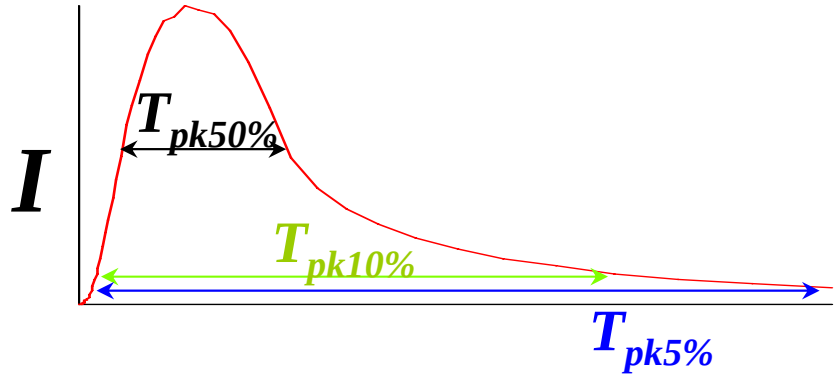
- Maximum unit cell dimension 135 Å as sample crystals
- Minimum d-spacings 1.2 Å in biomacromolecules and 0.7 Å in organic compounds
- Determination of more than 100 structures of biological macromolecules with crystal volume 2mm^3 per year

x50-100 times efficiency

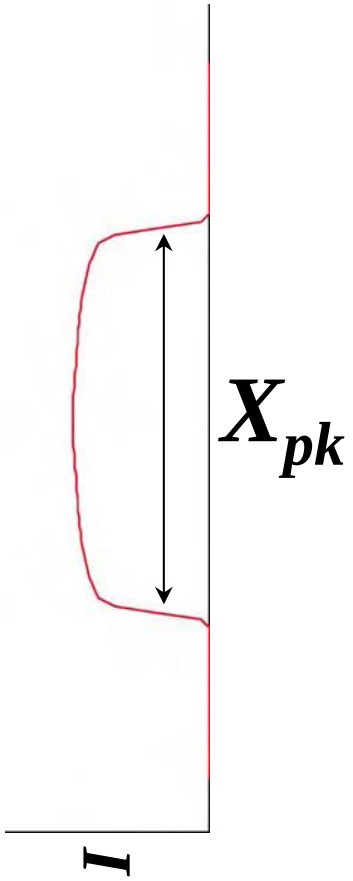
The relationship between cell and crystal volume



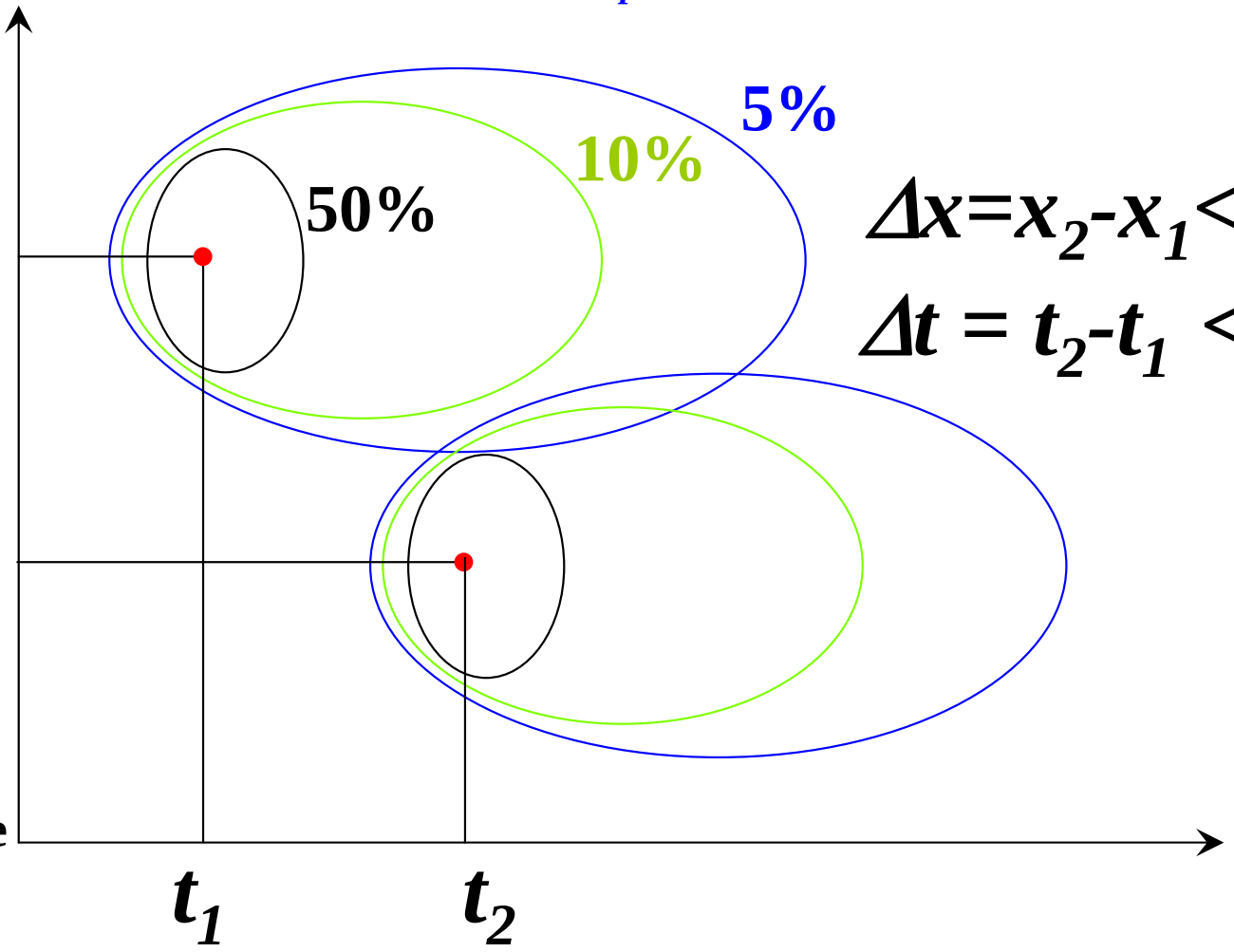
Judgment of spot-overlapping



Broadening along Time-axis from Pulse Shapes



Spatial Broadening from Beam Divergence



$$\Delta x = x_2 - x_1 < X_{pk}$$

$$\Delta t = t_2 - t_1 < T_{pkn\%}$$

Simulation condition

Moderator	**para-H_2 Coupled**
Crystal Lattice	**Cubic**
	***a*=135 Å**
h_{min},h_{max},k_{min},k_{max},l_{min} & l_{max}	**–112, 112 –112, 112, –112, 112**
***L1*(m)**	**40**
***L2*(mm)**	**300**
Detector Size (mm^2)	**130 ×130**
λ_{min} & λ_{max} (Å)	**0.7 3.88**
d_{min}(Å)	**1.200**
Sample Size(mm)	**1.00**
Detector No.	
1~6(2θ =30,60,90,120,150,180°)	
Divergence(degree)	**0.15 0.20 0.25 0.30**
Peak height(%)	**5 50**

Results

Peak height 5% ($\Delta t = < T_{pk5\%}$)

Div(deg)	0.15	0.20	0.25	0.30
Overlap	37.1%	51.5%	67.0%	77.5%

Peak height 50% ($\Delta t = < T_{pk50\%}$)

Div(deg)	0.15	0.20	0.25	0.30
Overlap	1.4%	4.3%	9.8%	14.7%

Research and Development Items

- ***Guide Tube Design :***

  ⇨ ・20mm-square straight tube and higher Qc super mirror seems better at present.

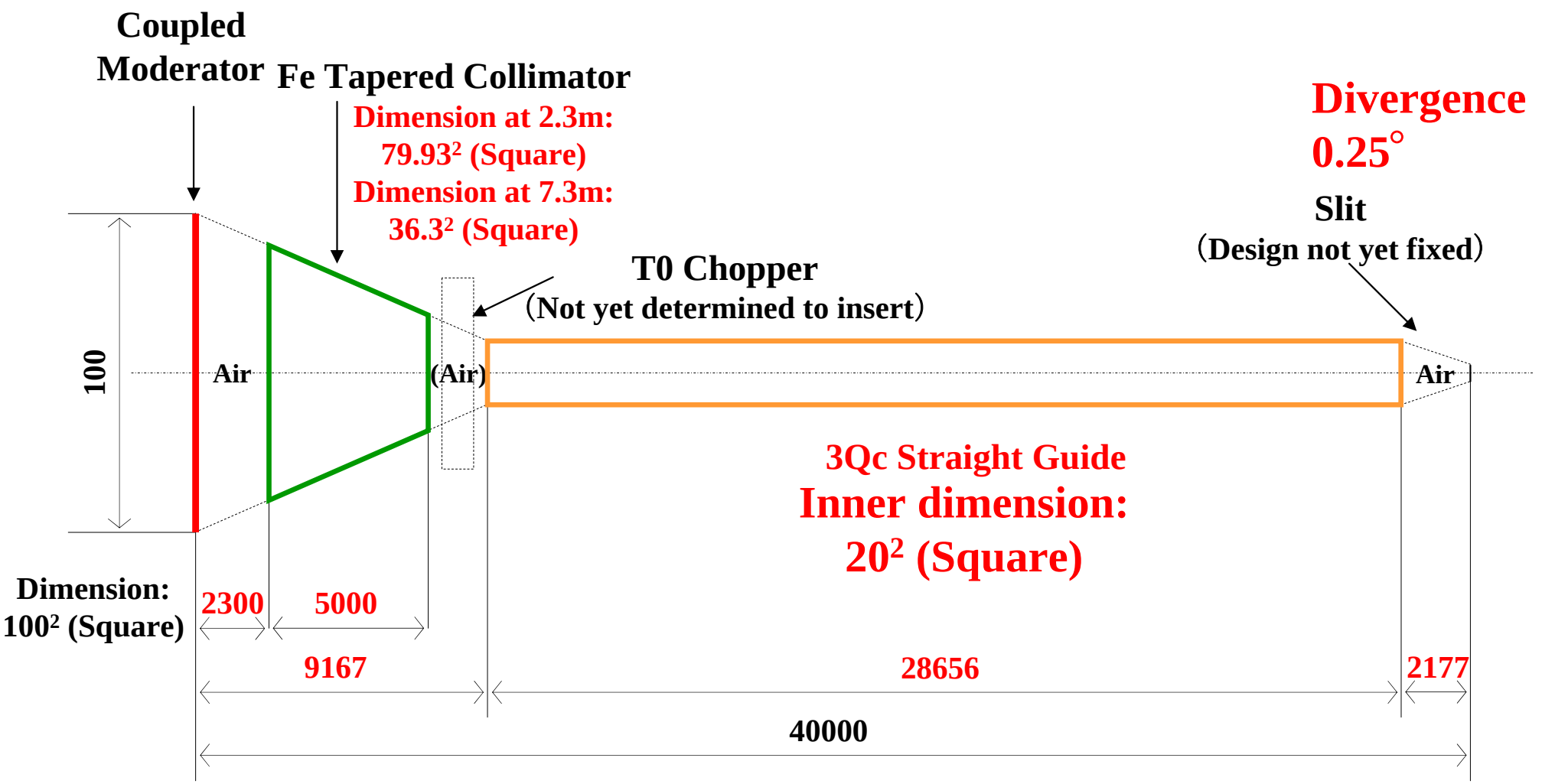
- ***Software :*** **Reliable indexing and deconvolution of overlapped spots**

  ⇨ ・After indexing, a proper software to deconvolute the partially overlapped spots in time by profile-fitting *etc* is necessary.

- ***Detector :*** **Less than 1mm spatial resolution with minimum gap**

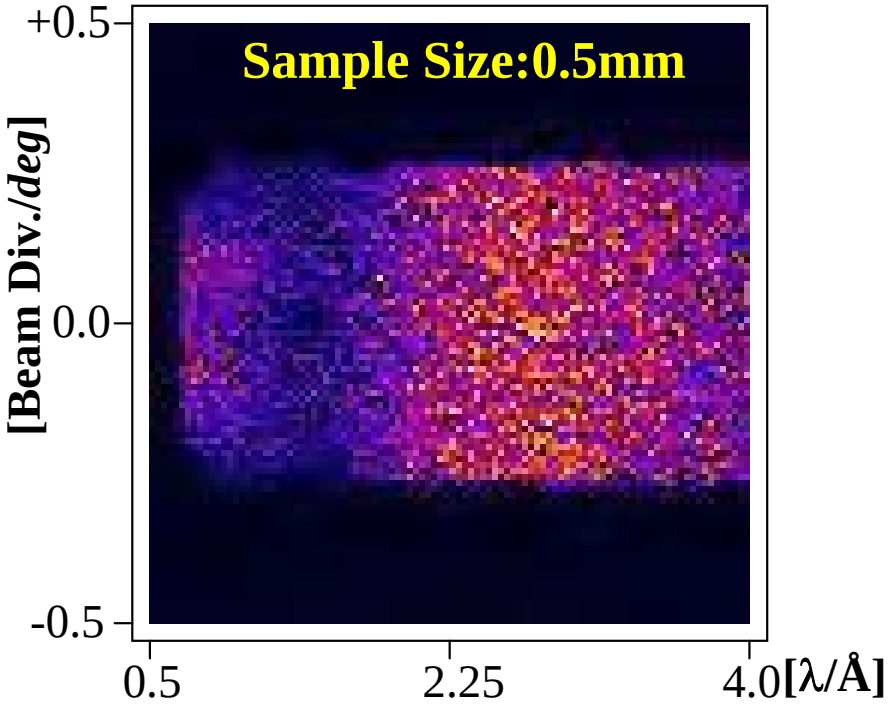
  ⇨ ・Necessary to simulate what kind of detector shape will maximize measurement efficiency in a realistic case, in order to develop a new detector.

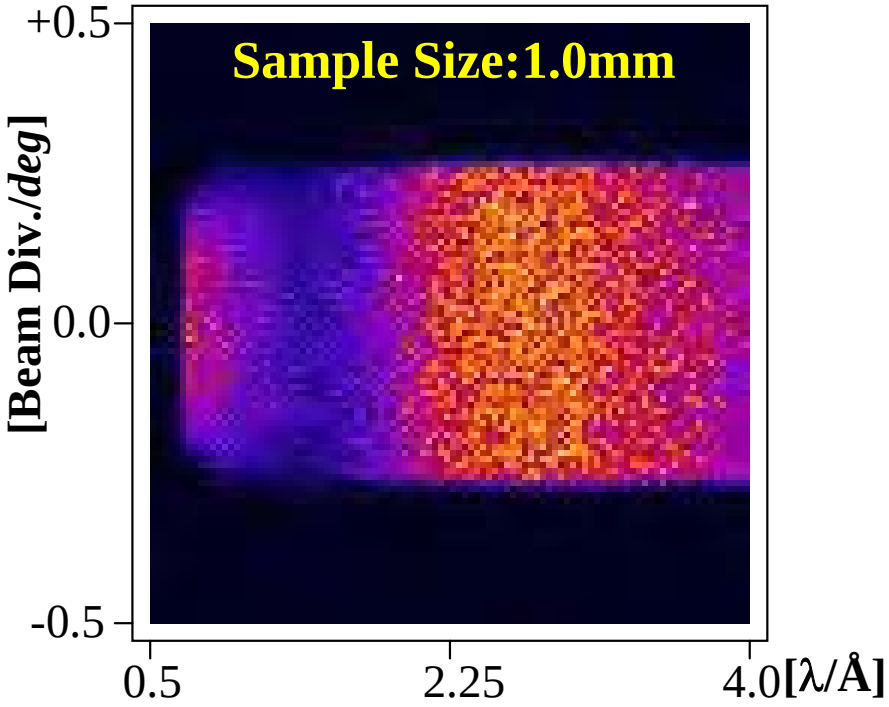
Optical Parameters of BIX-P1 Type Diffractometer

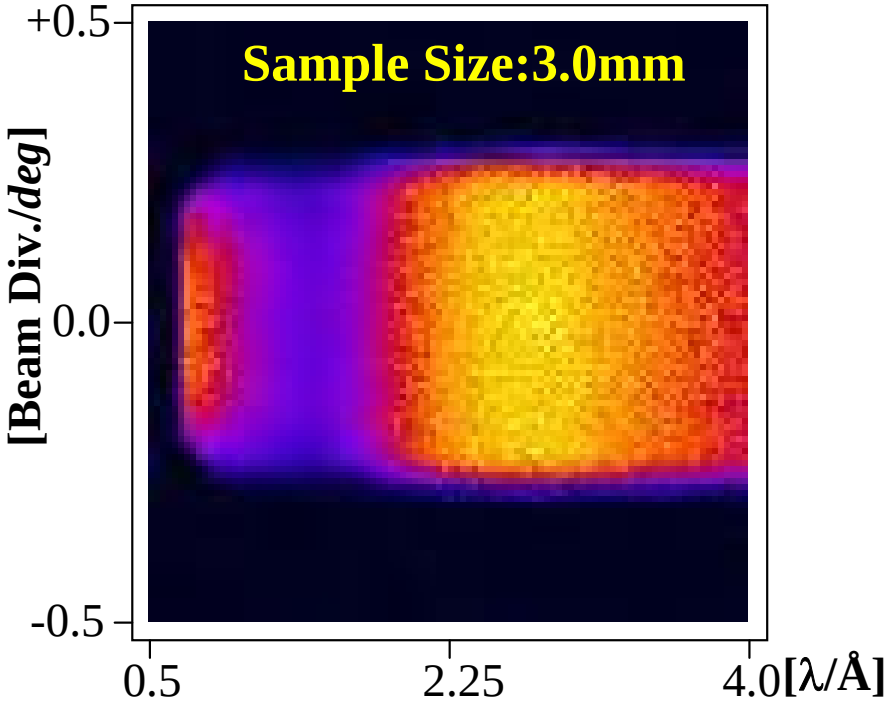


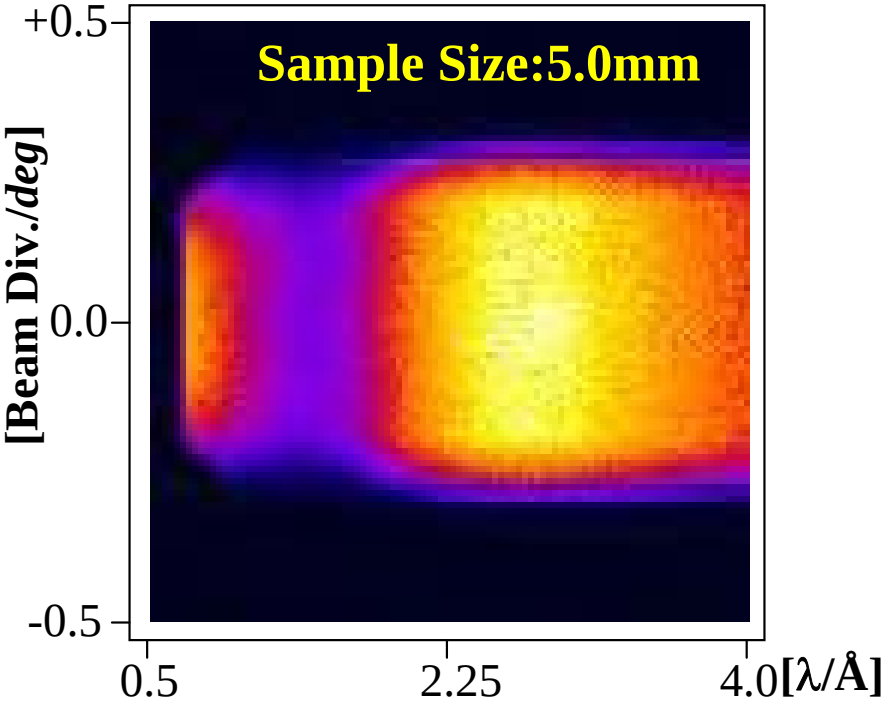
Unit: mm
Scale: 1/2 (Vertical)
1/200 (Horizontal)

Profile of Direct Beam Result No.1 (by MCSTAS)

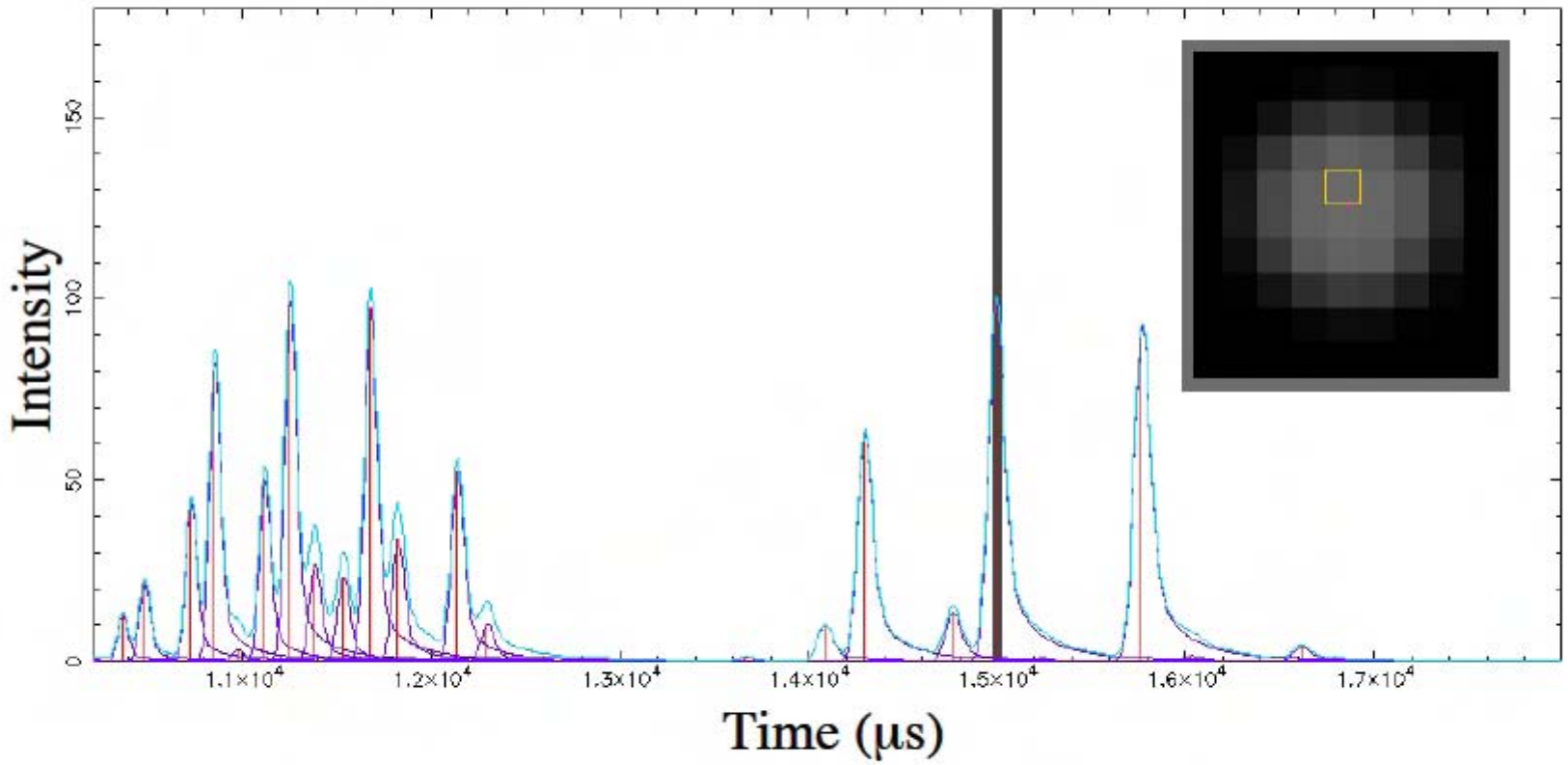






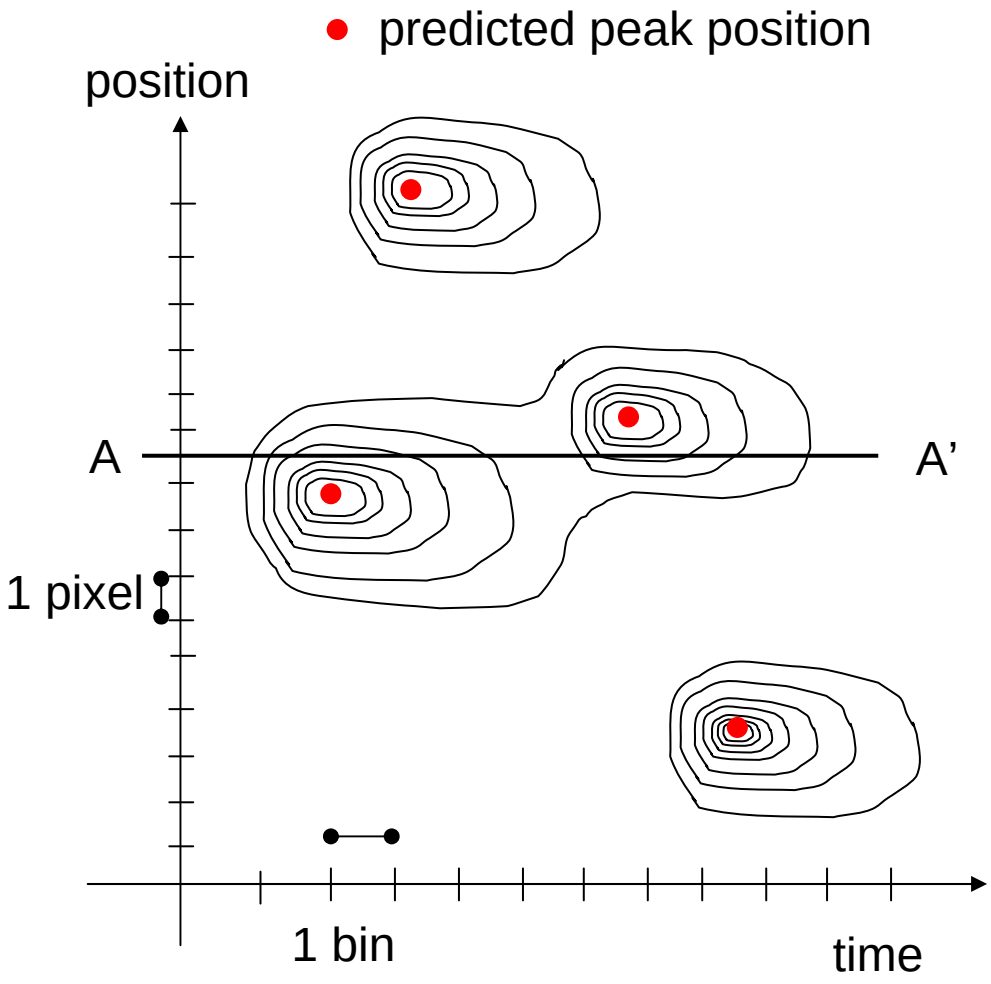


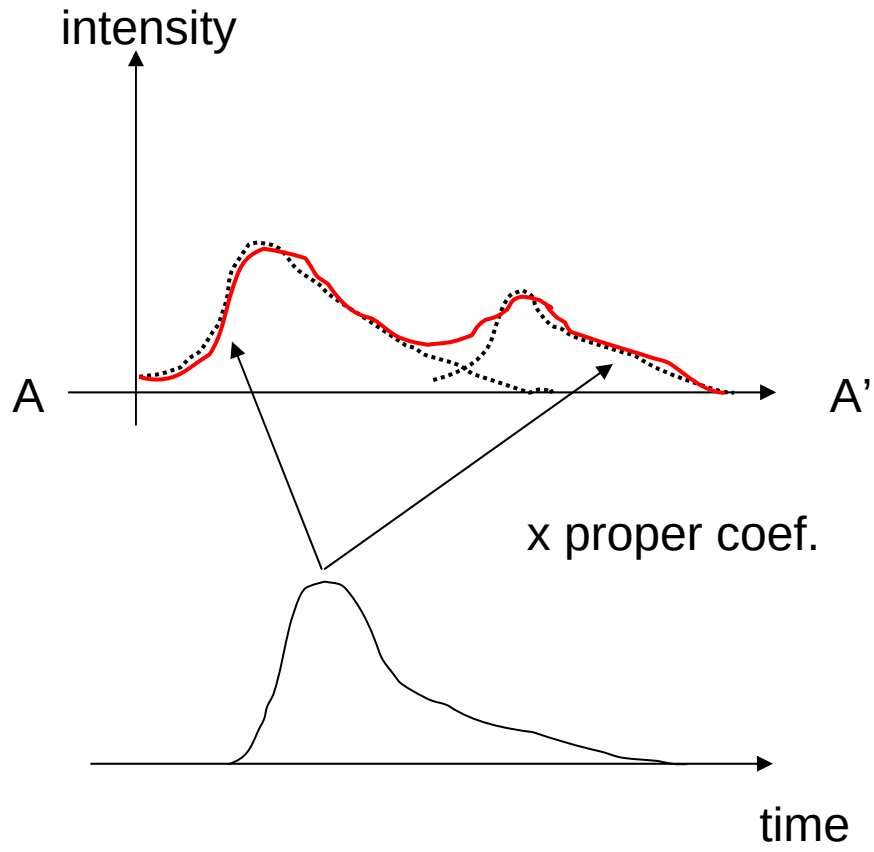
Peak profile



2θ=37.9°, 42 $\overline{15}$ 39 reflection, d=2.2787

Strategy of deconvoluting partially overlapped spots



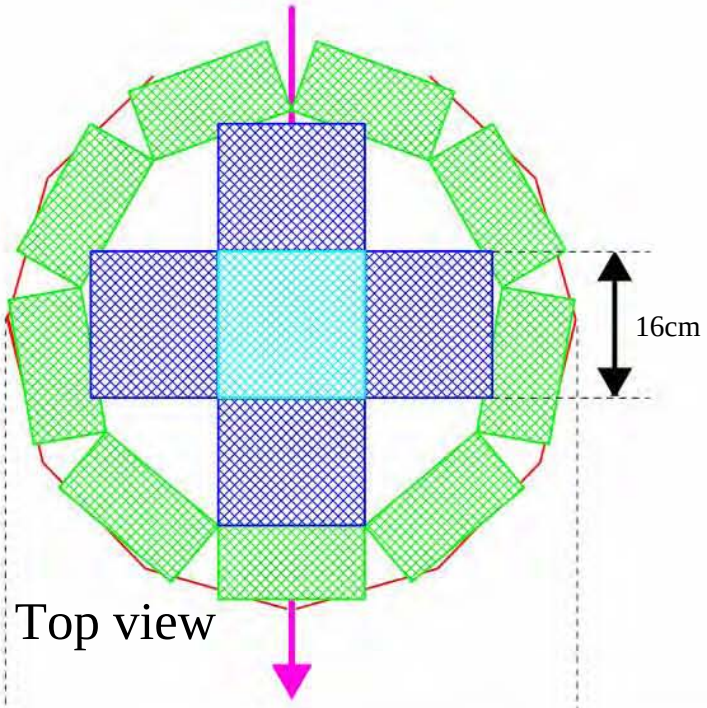


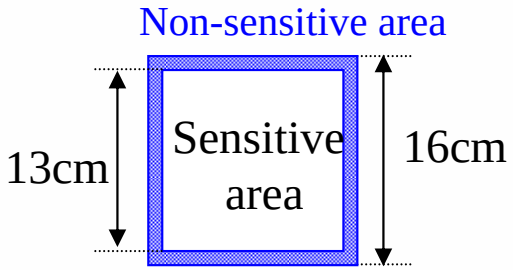
Original time structure of pulse(λ)

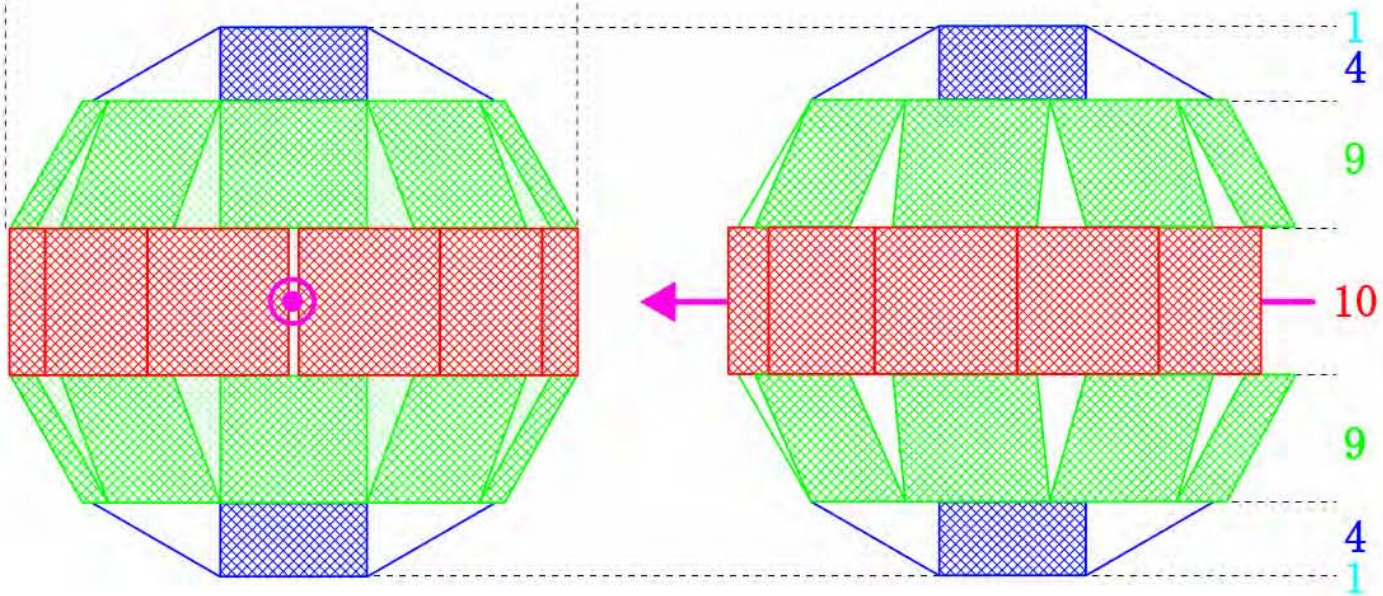
Detector Arrangement

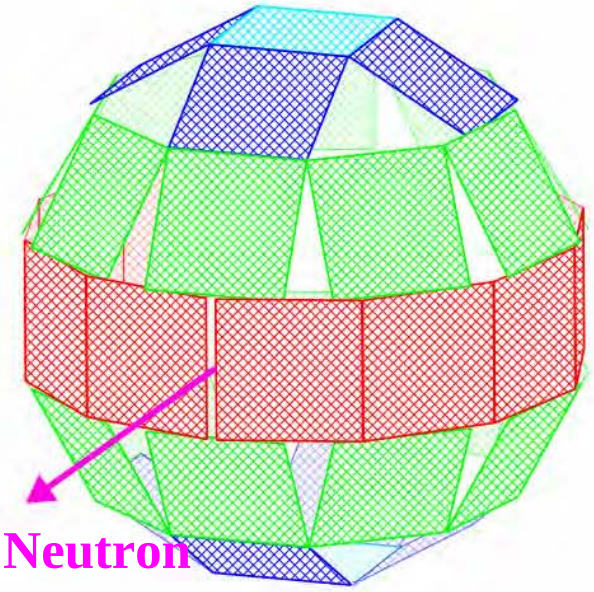
Supposed ZnS/^{6}LiF Scintillating Detector with Wavelength Shifting Fiber Read-Out (from M. Katagiri, JAERI)

L2	30 cm
Sensitive area	16×16 cm^2
Area with non-sensitive	13×13 cm^2
$2\theta_{min}$	3.66°
$2\theta_{max}$	161.0°
No. of Detectors	38
Total solid angle	54.26% (6.819str)

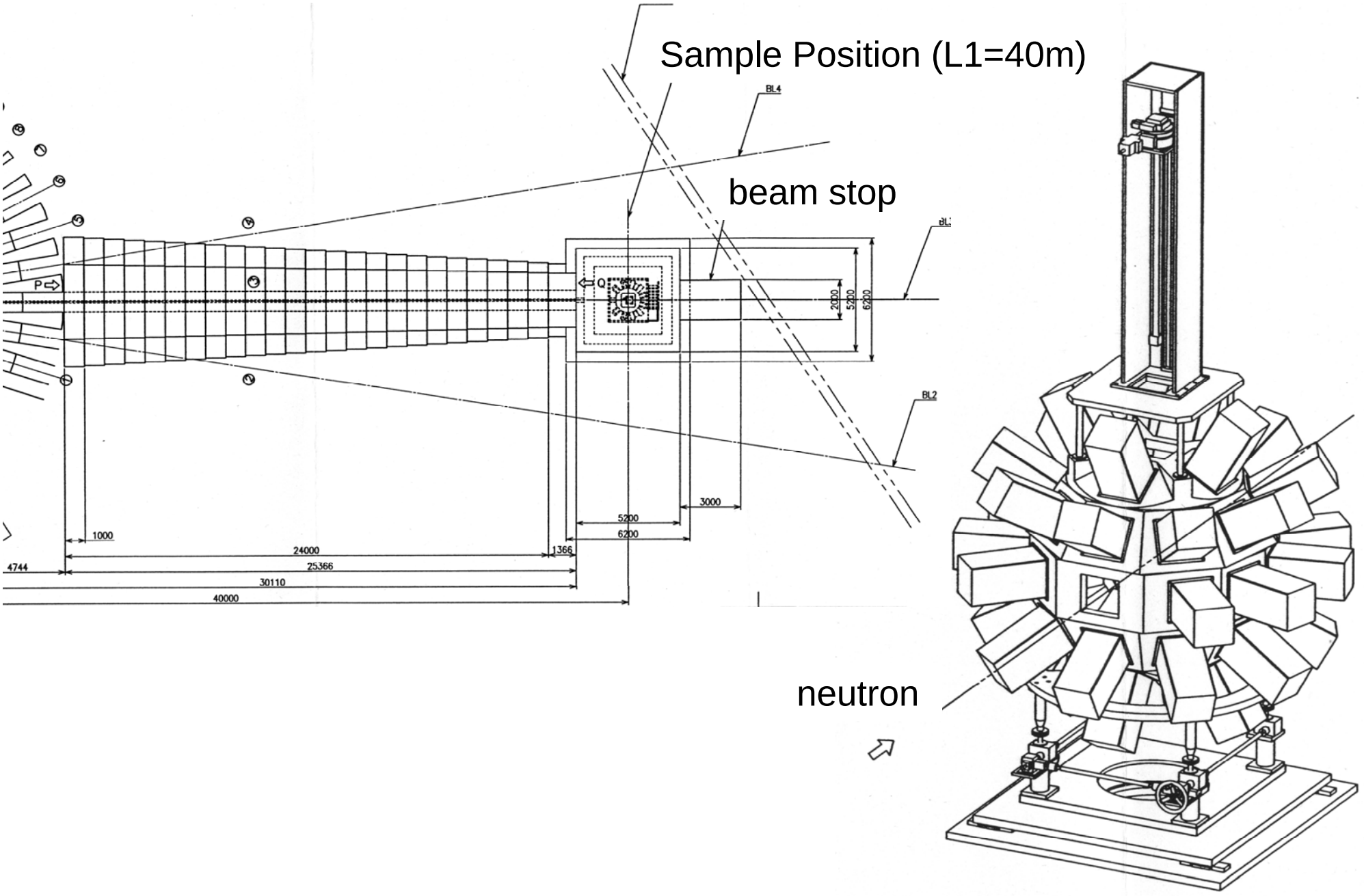




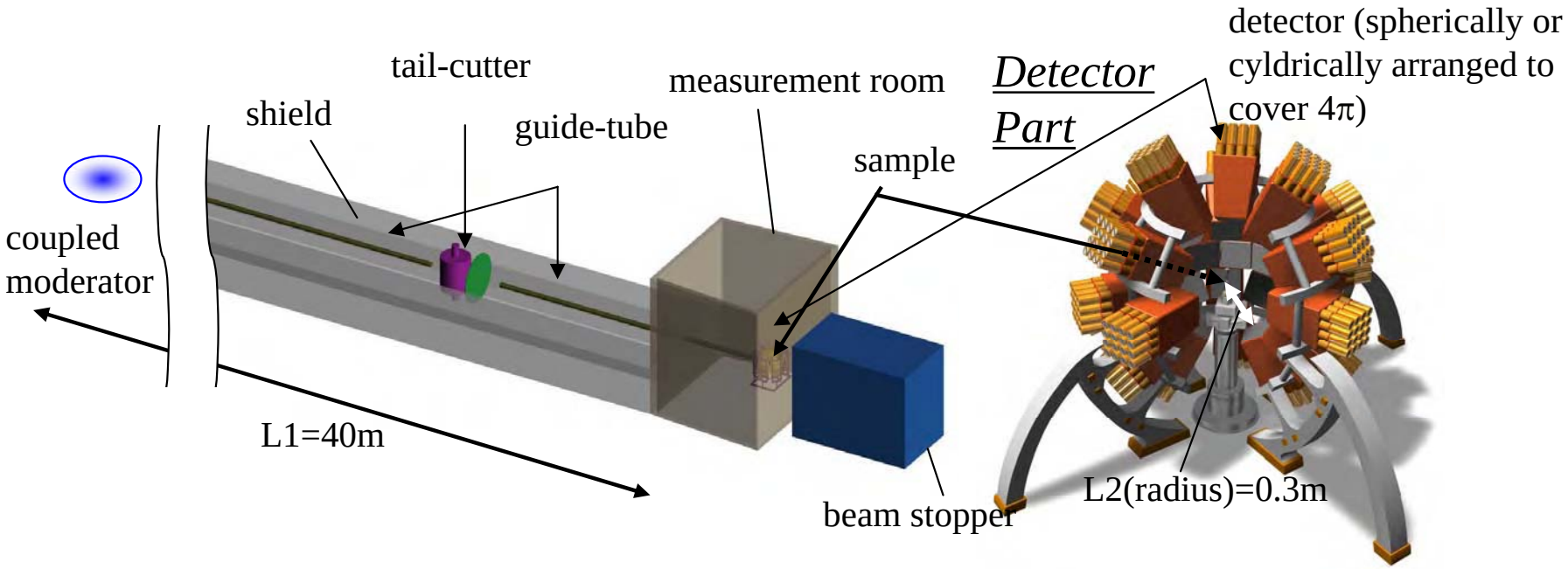




Engineering Design for the 1st Stage



Artistic Design



Current Design Parameters

Moderator and its view area	**Coupled H_2 (para);100 x 100 mm^2**
L1(m)	**40**
L2(m)	**0.3 (*)**
Guide Tube	**3Qc (20mmSq. & straight, 9.17<L1<37.83m)**
Beamline occupation angle	**4.3 deg**
Minimum d-spacings	**Less than 0.7 Å**
Maximum cell dimension	**135 Å**
Sample size (a x b mm^2)	**0.5 x 0.5 (standard size)**
Wavelength	**0.7 - 3.85 Å**
Detector spatial resolution	**Less than 1 x 1 mm^2 (*)**
Detector counting rate ($n/\mu sec/pulse/cm^2$)	**1mm^3 organic compound crystal : $2.7x10^{-3}$** **0.1mm^3 biomacromolecular crsytals : $9.5x10^{-7}$**

(*) L2 may become larger when the state-of-the-art detector spatial resolution is more than 1mm.

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- *Detector Development and Discussion*

  Dr. M. Katagiri(JAERI), J-PARC Detector Group Members(All Japan)

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