The Swiss-Norwegian Beam Lines at ESRF

Дифракционный эксперимент на пучке синхротронного излучения

Д. Ю. Чернышов
SNBL at ESRF, Grenoble, France
Кафедра кристаллографии, СПбГУ, Санкт-Петербург

Фонд Династия Зимина

Династия

План лекции

1. Введение. Как подают предложение на эксперимент и получают время в Европейском Центре Синхротронного Излучения
2. Типовая схема линии для структурных исследований.
3. Образец, детектор, дифрактометр, давление и температура на образце.
4. Разрешение кристаллографическое и приборное. Разрешение дифрактометра с плоским позиционно-чувствительным детектором.
5. Стратегия сбора данных монокристального эксперимента.
6. Заключение. На что обратить внимание при выборе прибора для структурного эксперимента.

Как подают предложение на эксперимент и получают время в ESRF

All 30 public beamlines at ESRF are now in operation, and provide some 500 shifts of beam time each year for user experiments, after deducting machine-dedicated runs and maintenance days. In addition, several externally funded, Collaborating Research Group (CRG) beamlines, make available 1/3 of their beam time to ESRF users. Beamlines at ESRF operate 24 hours a day in three shifts, each of 8 hours.

Two proposal review rounds are held each year, with deadlines for submission of applications on **1st March** and **1st September** for the scheduling periods **August to February**, and **March to July** respectively.

Requests for beam time are based on shifts of 8 hours. Allocations of beam time will usually be made in multiples of 3 shifts.

Check your proposal with ESRF Beamline staff before submission

www.esrf.eu

REVIEW COMMITTEES and AREAS OF EXPERTISE

Chemistry-related Studies: CH

Environmental and Cultural Heritage Matters: EC

Disordered systems and Liquids: HD

Electronic and Magnetic Properties: HE

Crystals and Ordered Systems, Structures: HS

Applied Materials and Engineering: MA

Medicine: MD

Methods and Instrumentation: MI

Macromolecular Crystallography: MX

Soft Condensed Matter and Biological Materials: SC

Surfaces and Interfaces: SI

Before submitting a proposal for ESRF Beam Time, we strongly recommend that you first consult (and keep) the **Proposal Guidelines** **specific to the category of your new proposal(s)** very carefully, as these contain **detailed instructions** regarding the electronic submission of your proposal(s).

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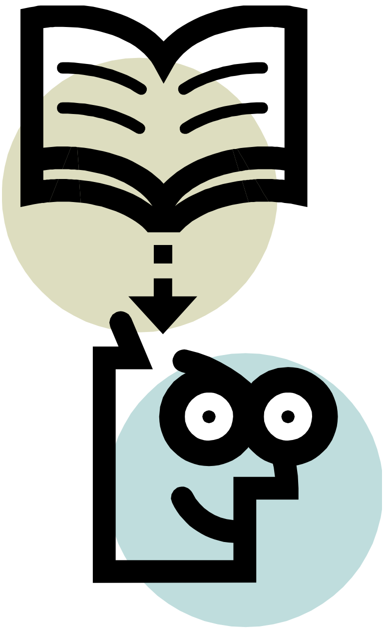
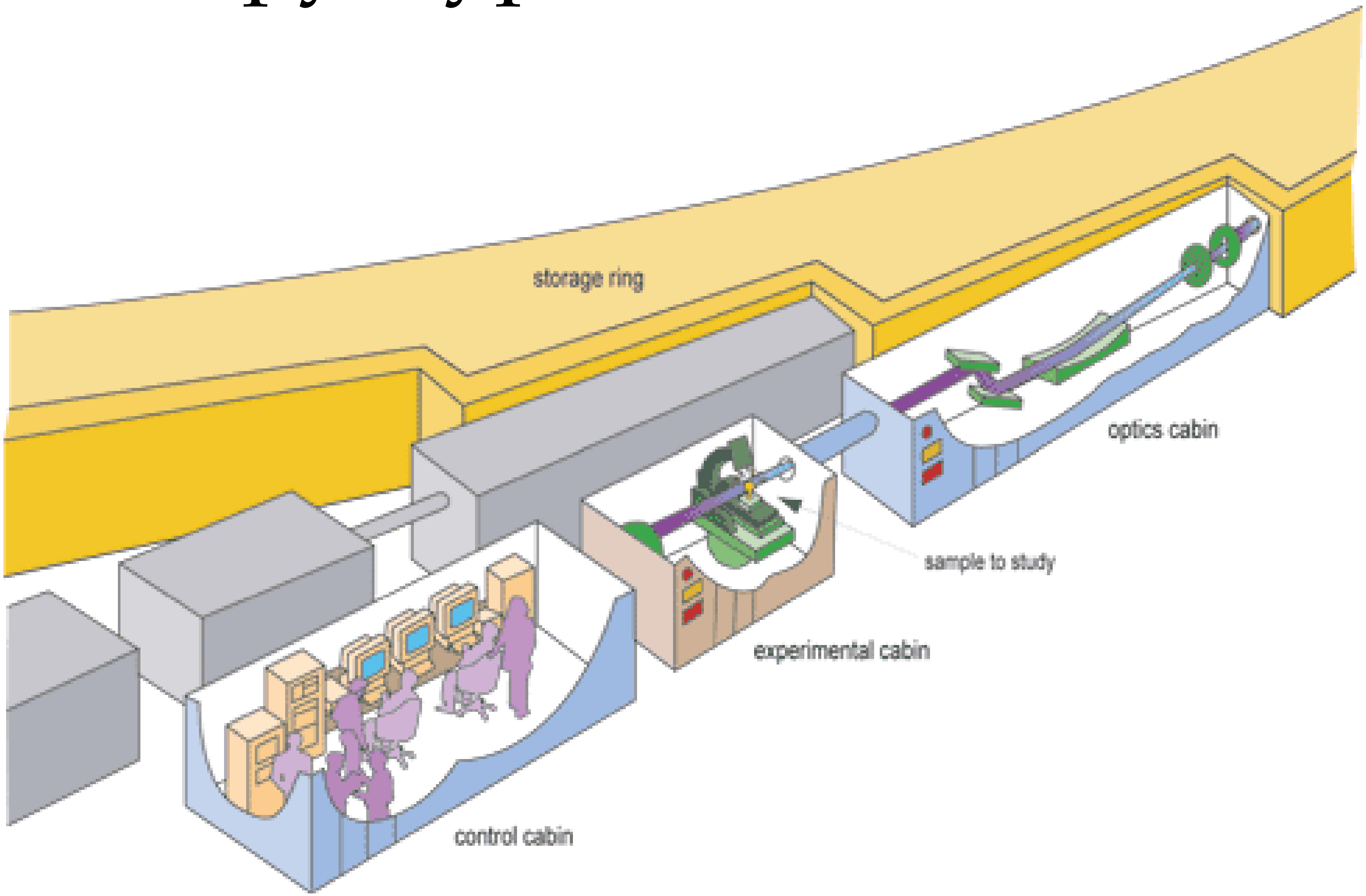
ASSESSMENT & BEAM TIME ALLOCATION

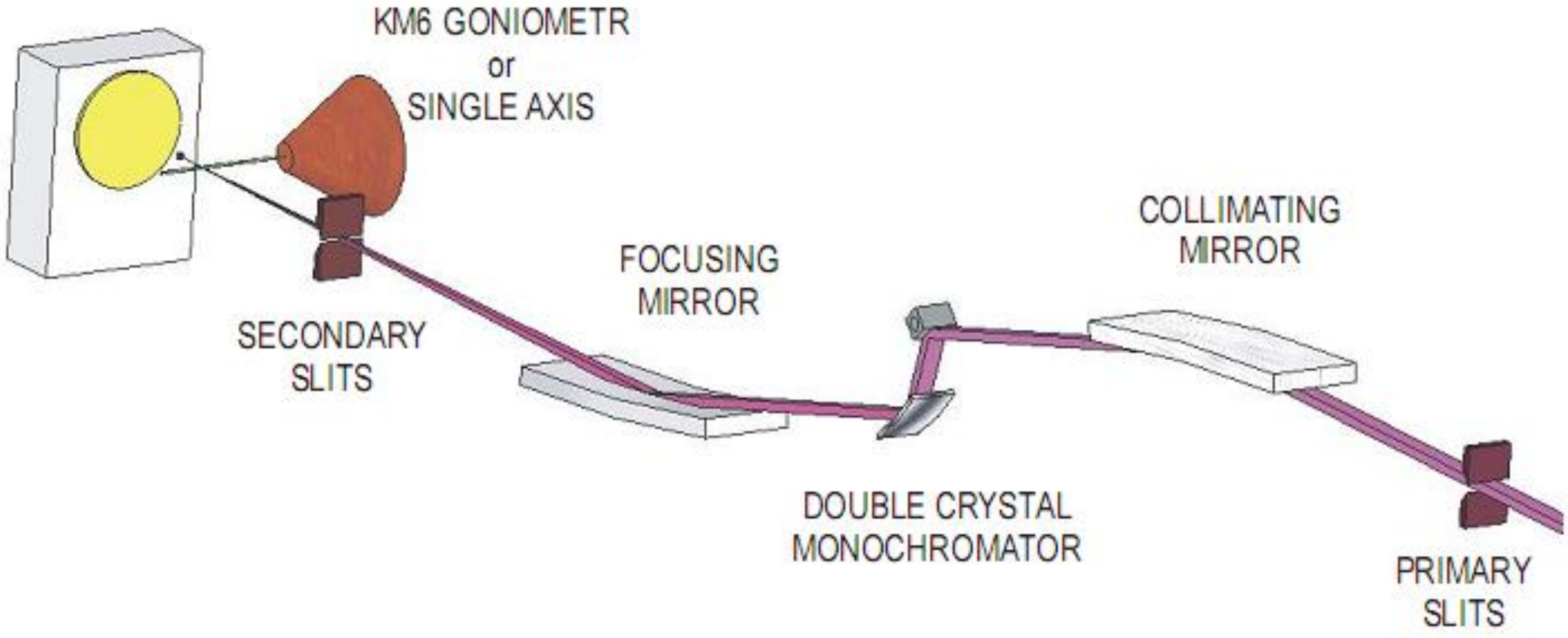


The Review Committees, assisted by external referees where appropriate, consider the proposals: they first assign each proposal a grade between 0 and 5 on the basis of scientific merit, 5 being the top score. At their meetings, the committees discuss the projects, and arrive at a final grade for each proposal; they also rank the proposals in order of priority for each beamline. This round, we have introduced the notion of "quartiles", to give proposers a more precise indication of the relative ranking of their projects. The scores for each beamline have therefore been divided into sections, the first section with the best scores being allocated beamtime. Thus the second section represents good rankings, below the cut-off for allocation, and the fourth, the lowest set of rankings.

When beam-time is allocated, invitations for experiments, together with detailed instructions, are then communicated to the Proposers concerned by the ESRF User Office several weeks ahead of the scheduled experiment.

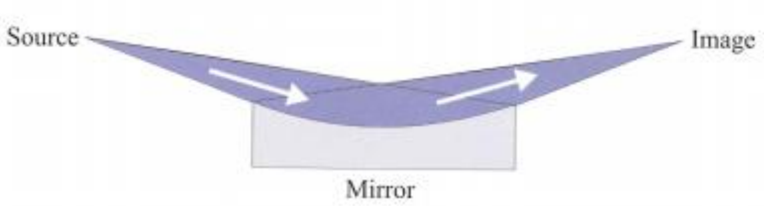
Типовая схема линии для структурных исследований.

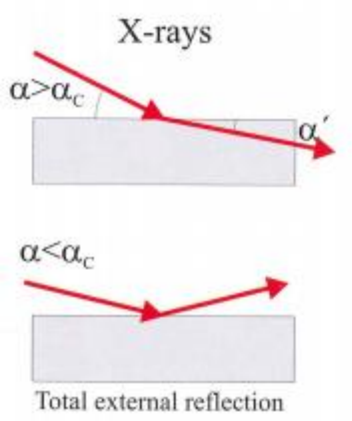




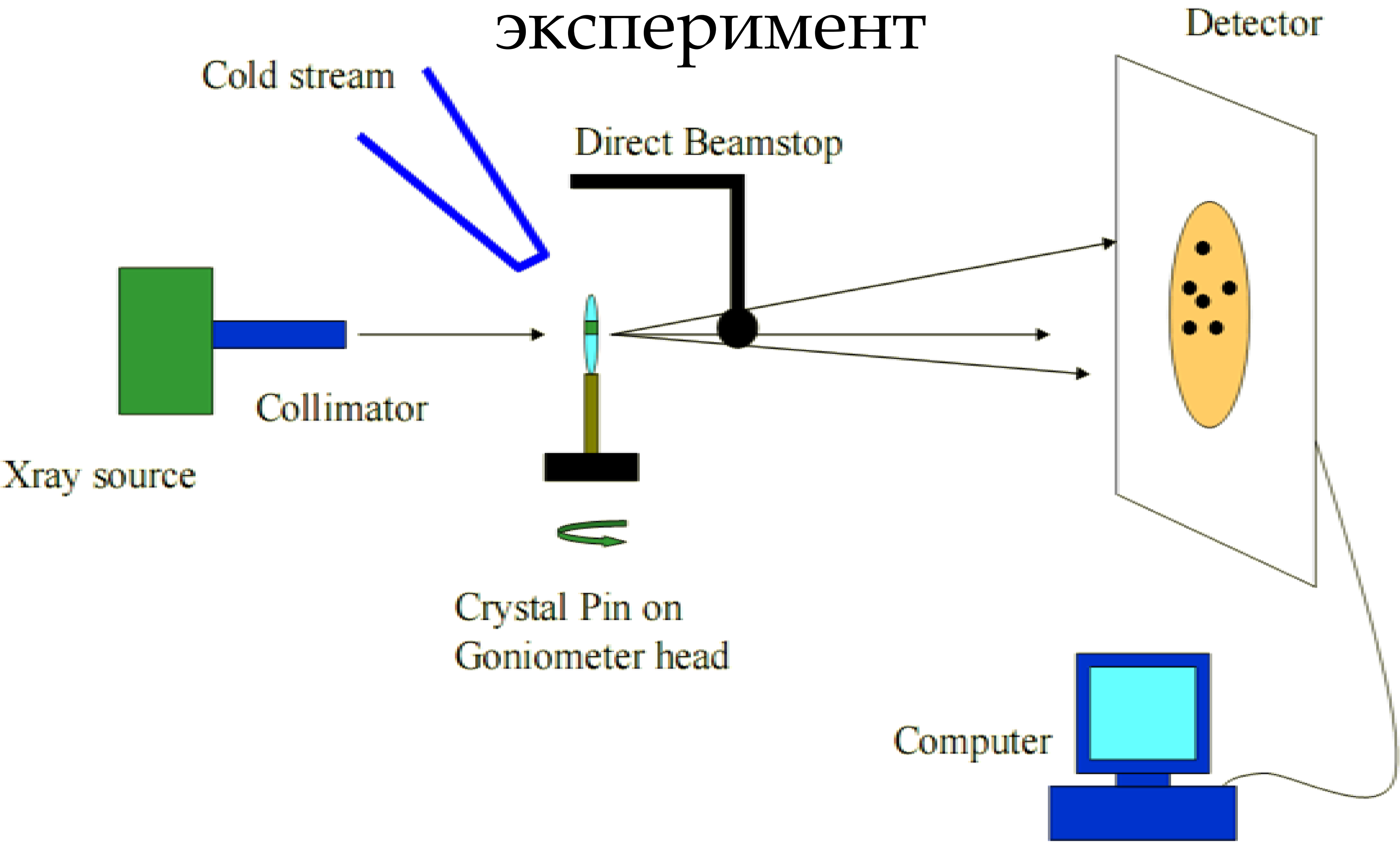
Оптические компоненты дифракционной линии

- Коллимирующие щели
- Фокусирующие зеркала / линзы
- Монохроматор
- Гониостат
- детектор

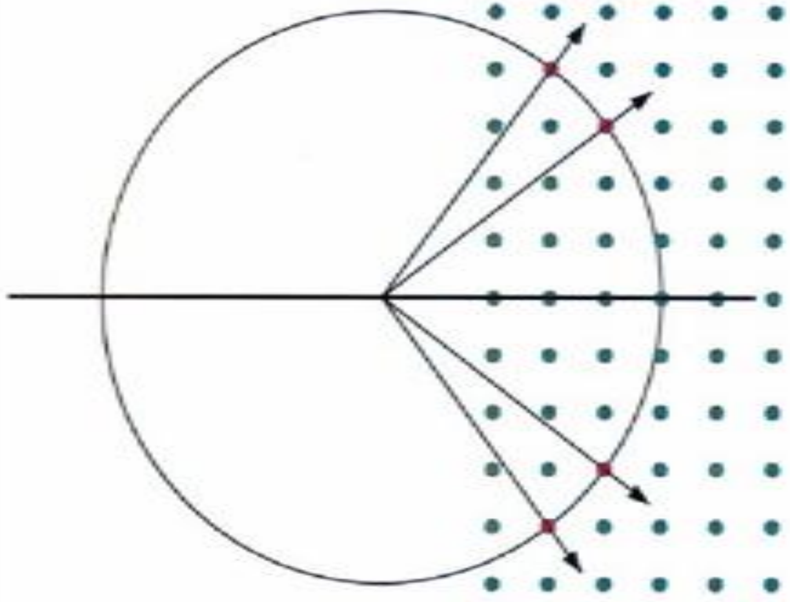


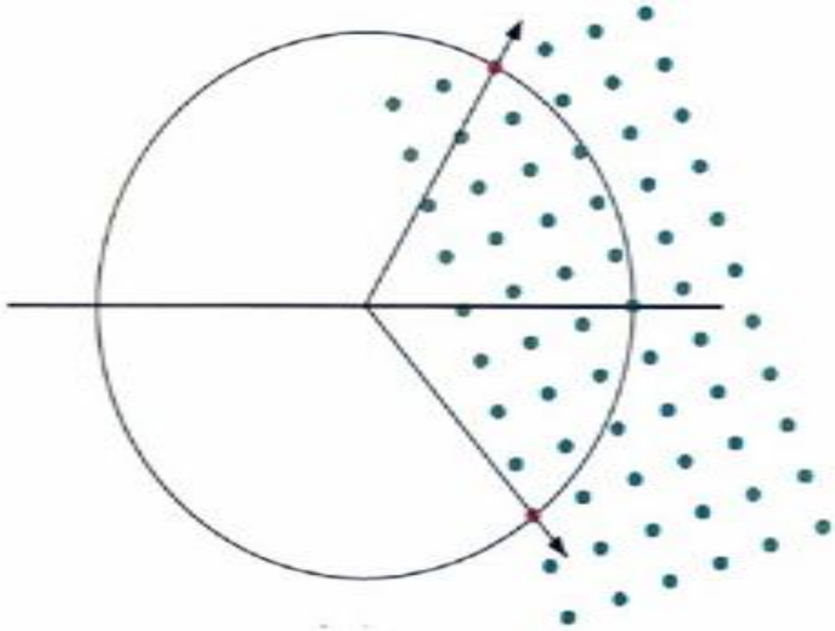


Монокристальный дифракционный эксперимент

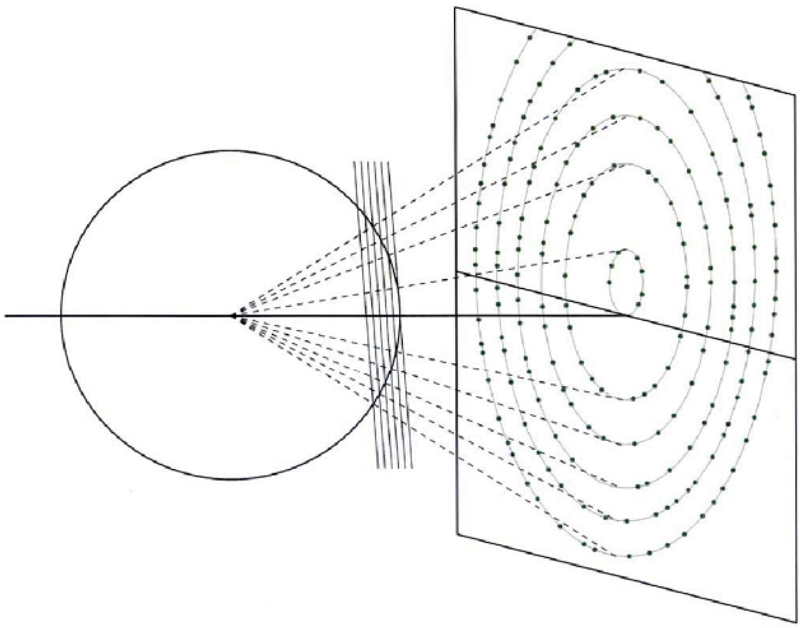


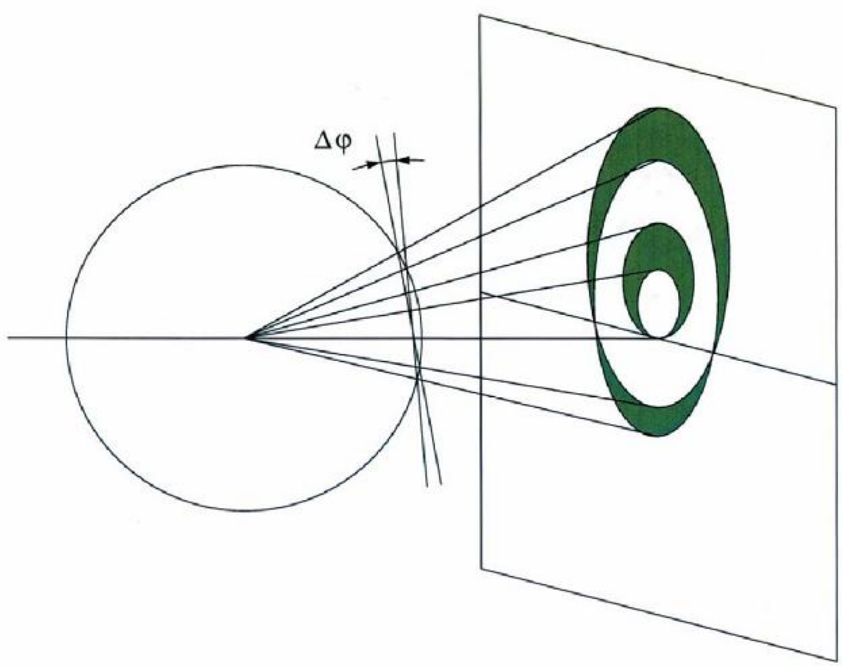
Монокристальный дифракционный эксперимент





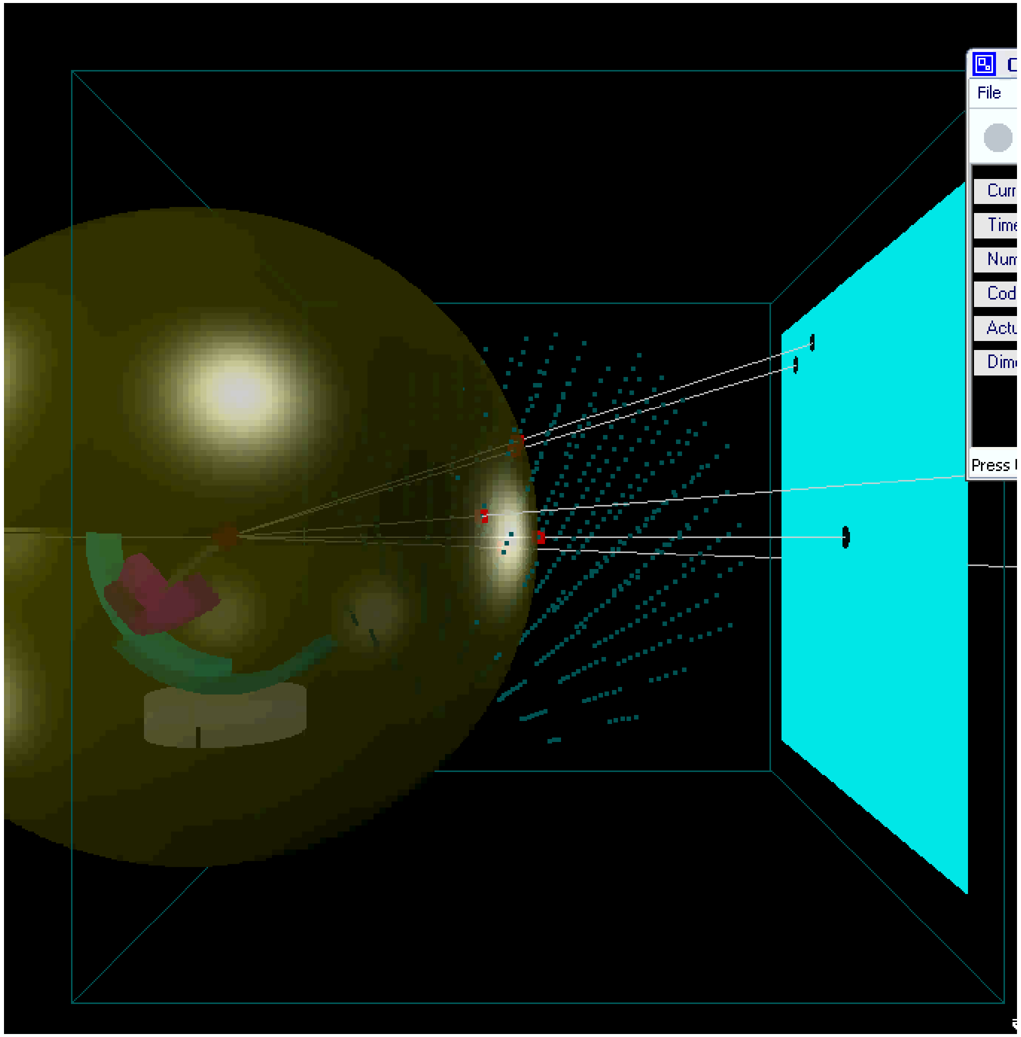
Монокристальный дифракционный эксперимент





Acta Crystallographica Section D (1999). D55: 1703-17

Монокристальный дифракционный эксперимент

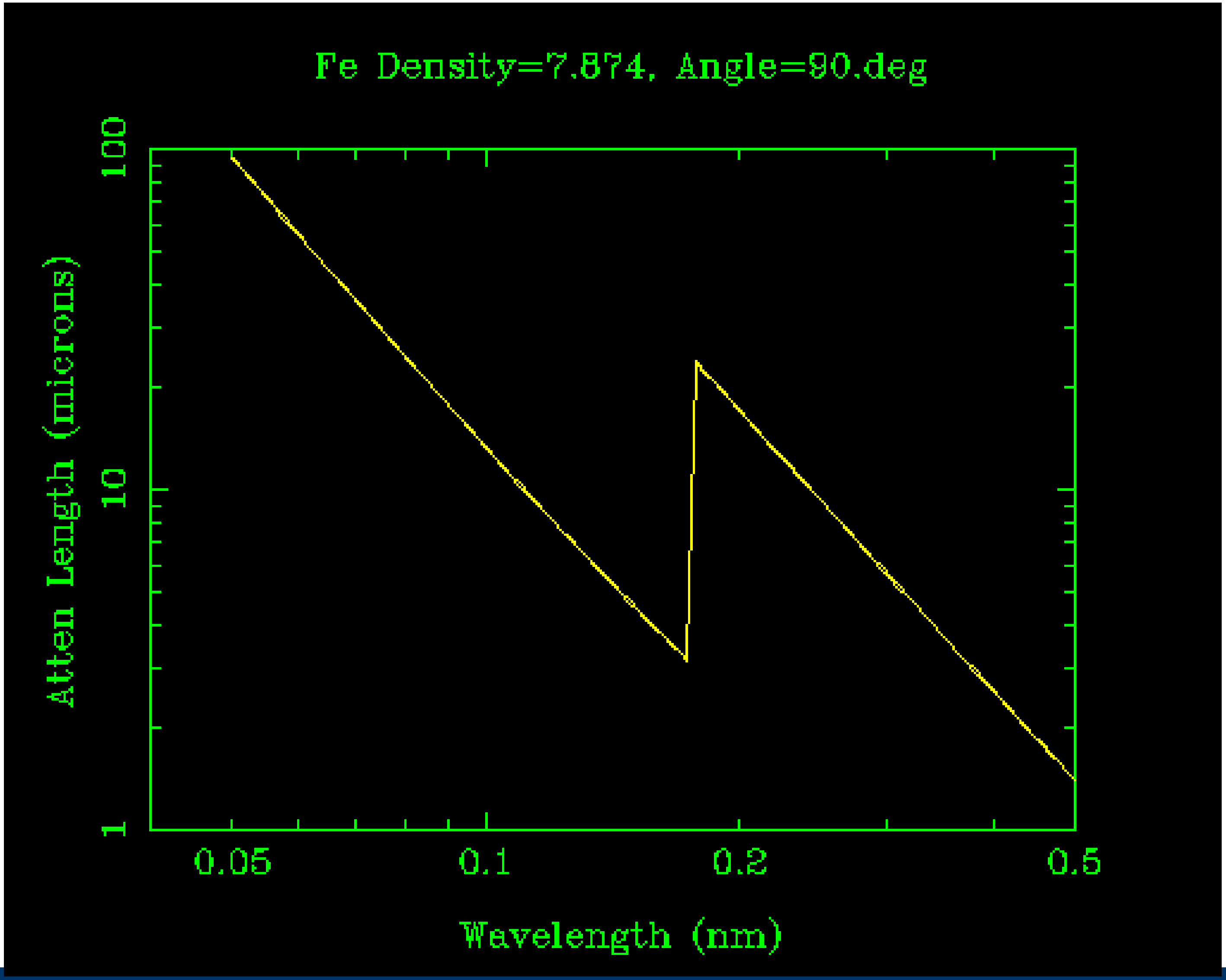


От кристалла до данных

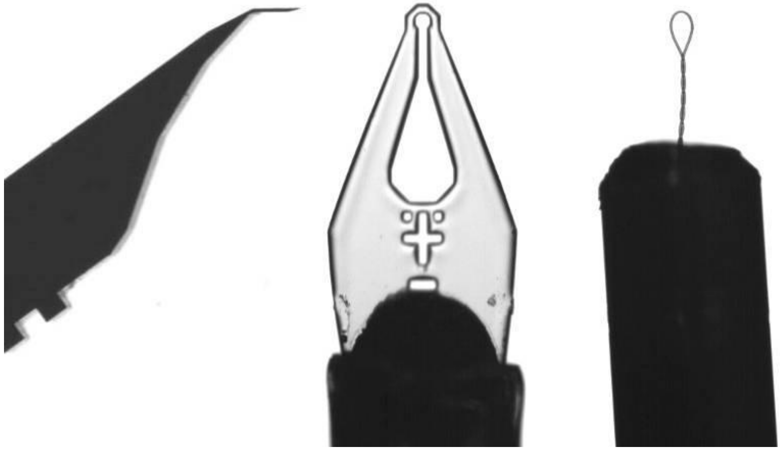
- Выбор кристалла
- Кристалл на гониометре
- Выбор детектора
- Стратегия измерений
- Температура и давление

Visual inspection under polarized light

Crystal size



Crystal mounting



2D detectors

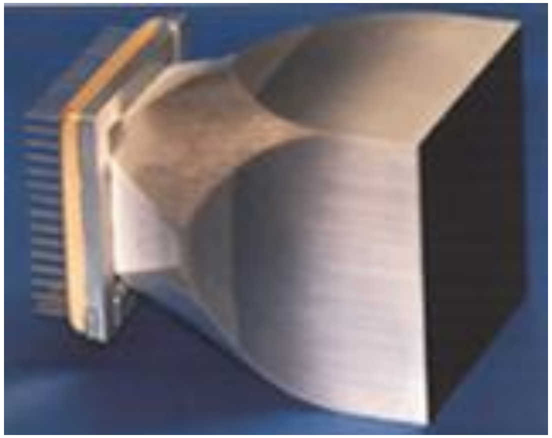
Характеристики идеального детектора

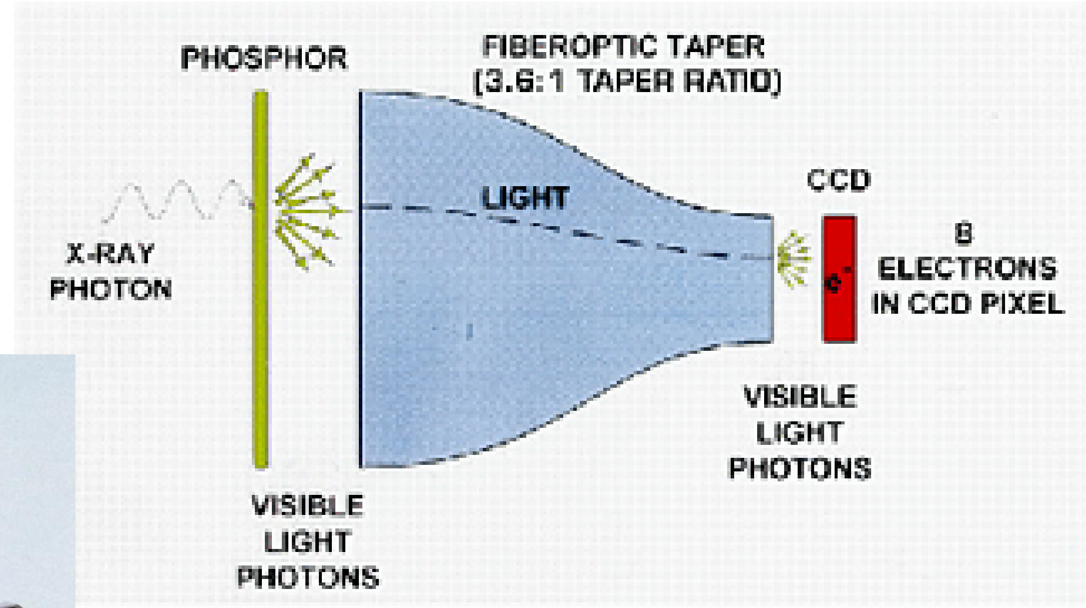
- 100% эффективность
- Идеально однородный отклик
- Линейный отклик
- Отсутсвие паразитных шумов
- Неограниченный динамический диапазон
- Простые и понятные характеристики

Реальные детекторы

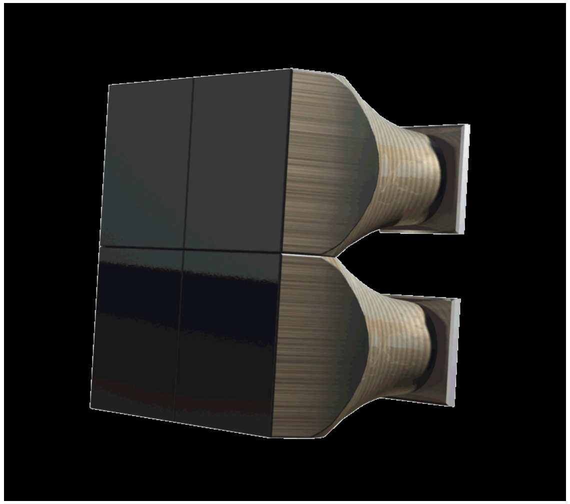
- CCD
- Image Plate
- Imaging Panel
- Pixel Area Detectors

CCD




PHOSPHOR
FIBEROPTIC TAPER
(3.6:1 TAPER RATIO)
LIGHT
CCD
X-RAY
PHOTON
8
ELECTRONS
IN CCD PIXEL
VISIBLE
LIGHT
PHOTONS
VISIBLE
LIGHT
PHOTONS


mar



Technical data

Overall dimensions	243 x 178 x 234 mm
Weight	16 kg
Active area	Ø 135 mm
CCD chip	Kodak KAF4320 2048 x 2048 pixels
Pixel size on scintillator	48 µm
Scintillator material	‘Super plus’
Fibre optic reduction	2:1
Peltier cooling	-40ºC
Temperature stability	± 0.05ºC (micro-processorised PID)
Analogue-to-digital resolution	True 18 bit
System noise (so-called read noise)	<10 e-RMS full frame
Dark current	<0.19 e-/pix.s
Communication	1 Gb ethernet
Gain	140 e-/X-ray (Mo)
Pixel sampling rate	5.6 MHz
Readout time	0.22s (4x4 binning), 0.46s (2x2 binning), 1.59s (1x1 binning)

- Быстрый детектор – хорош для изучения кинетики
- Ограниченный динамический диапазон
- Шумный
- Не терпит больших доз

Image Plate

Technical Specifications

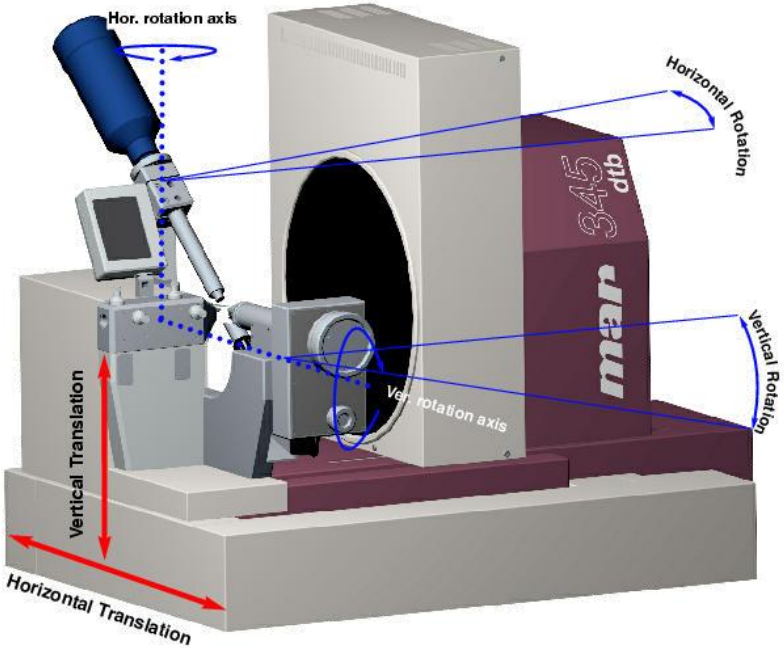
Type	Imaging plate
X-ray Sensitive Surface	Round, 345mm diameter (93,480mm^2)
Gain	1 ADU/8keV photon
Read Noise	1 to 2 8keV photon equivalent
Dynamic Range	17 bits (1:131,000)
Operating Temperature	Room temperature
Cooling	None required
Readout Mechanism	Spiral scan, laser-stimulated luminescence
Readout Electronics	Photomultiplier; 16-bit ADC, 2× oversampling

Readout Options (Software Selectable):

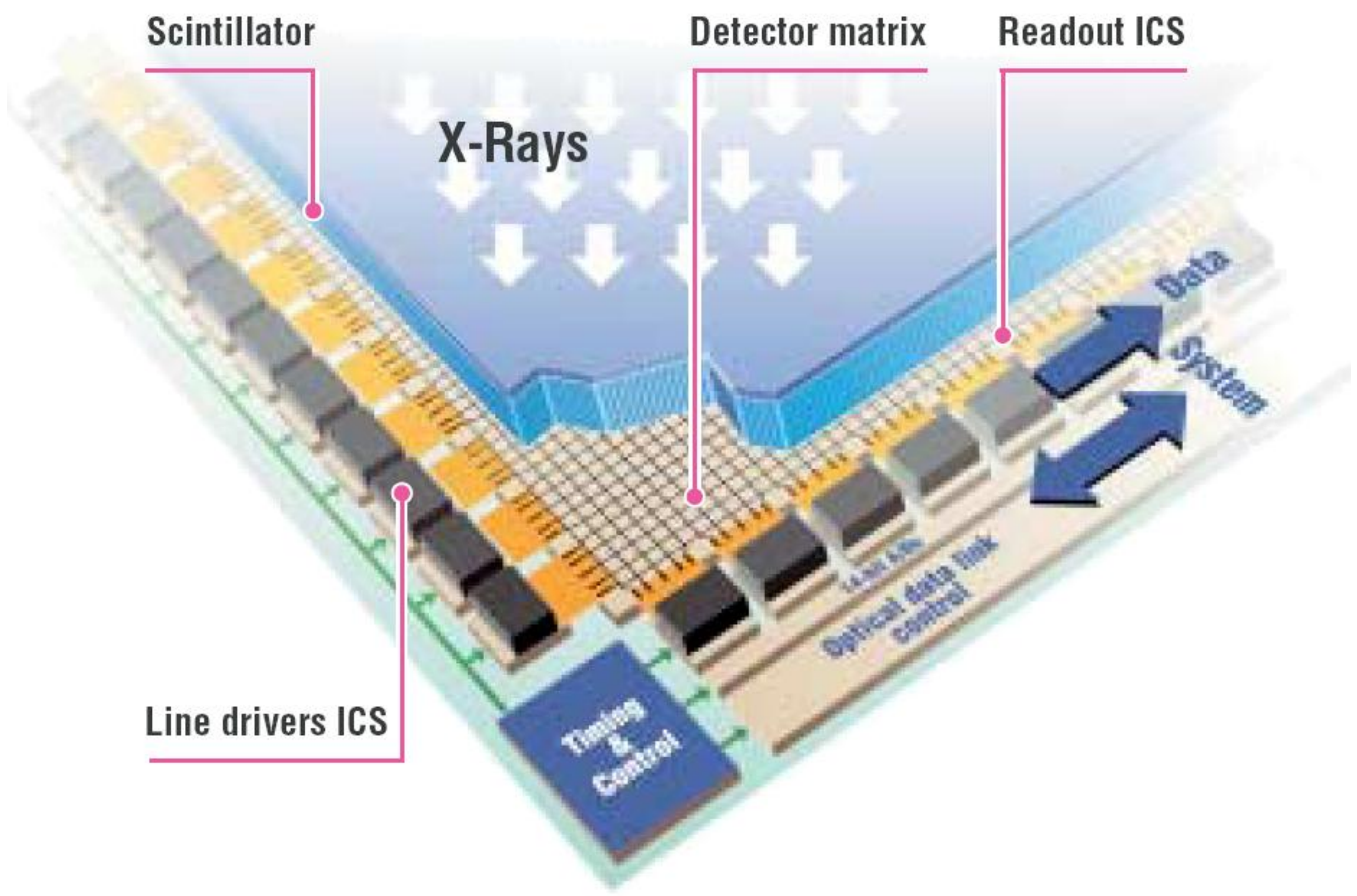
Resolution	Diameter	No. of Pixels in Image	Pixel Size	Read Time (sec.)	Erase Time (sec.)	Total Time (sec.)
High	345mm	3450 × 3450	100μm	96	12	108
	300mm	3000 × 3000	100μm	77	10	87
	240mm	2400 × 2400	100μm	53	9	62
	180mm	1800 × 1800	100μm	34	8	42
Standard	345mm	2300 × 2300	150μm	68	12	80
	300mm	2000 × 2000	150μm	56	10	66
	240mm	1600 × 1600	150μm	39	9	48
	180mm	1200 × 1200	150μm	26	8	34

Computer Interface	10BaseT Ethernet
Physical Dimensions (h × w × d)	50cm × 40cm × 36cm; weight: approx. 53kg

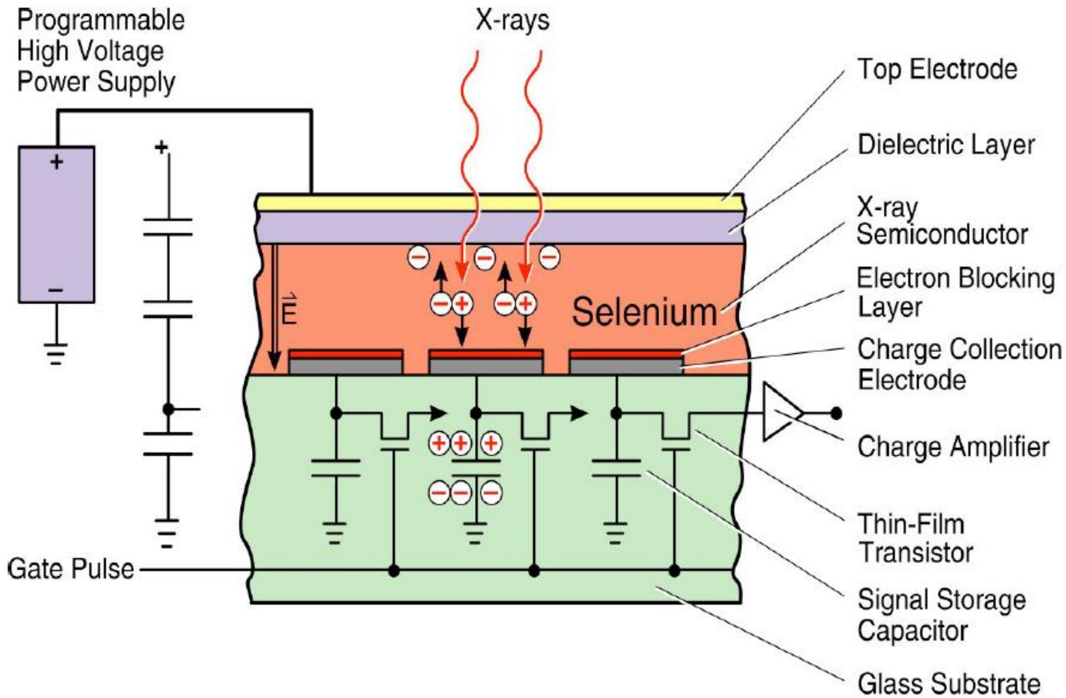
- Высокая надежность
- Большой динамический диапазон
- Хорошо переносит большие дозы
- Медленный….



Flat Panel



Scintillator
Detector matrix
Readout ICS
X-Rays
Data
System
Line drivers ICS
Timing & Control
Optical data link control



- **Energy of x-ray photons is directly converted to electric charges**
- **Charges move along the electric field towards pixel electrode**

SPECIFICATIONS OF THE *mar555*

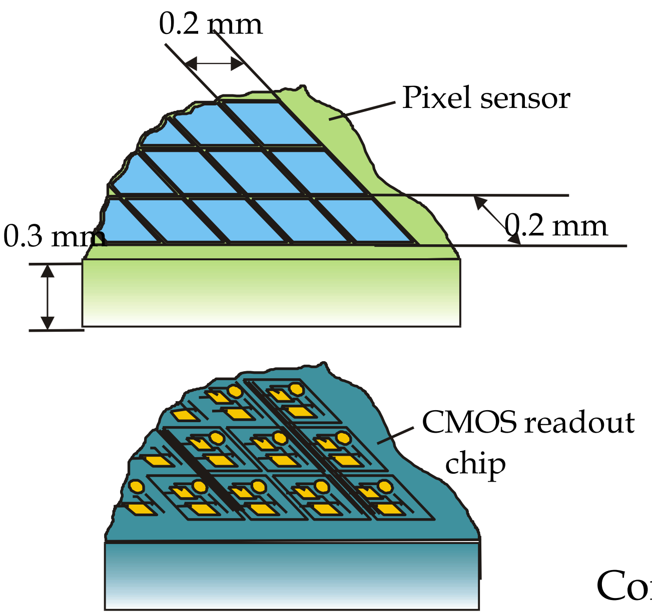
Active area	430mm x 350mm
Diagonal	555mm
Pixel size	140µmx140µm
	3072x2560 pixels
	7.8 Mpixels
Dynamic Range	16 bit, 18 bit with expansion
Operated @	+25°C
Read-out time	0.6 sec + PCI overhead= 1 sec
Signal Charge	~120e- @ 12keV
Amplifier noise	500e- rms
Total noise	750e-rms or 6 x-rays@12keV
	(including kTC noise of Cpixel = 2pF)

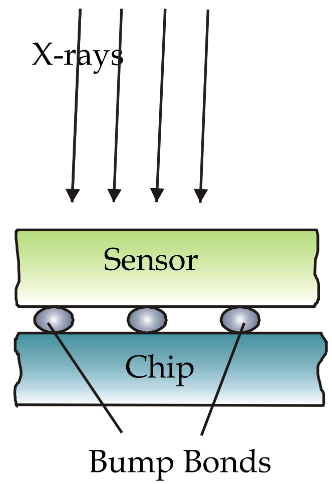
IP, CCD, Flat Panel record X-ray intensity by integrating the energy deposited by X-ray photons.

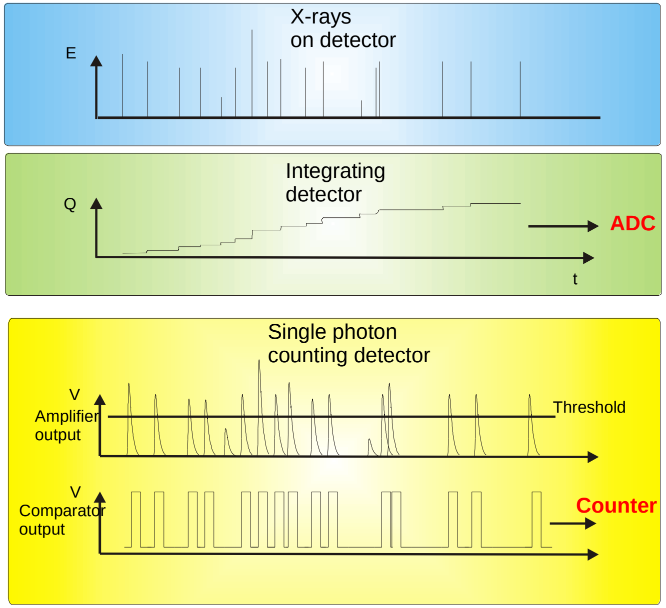
Conventional Si, Ge, and NaI detectors are still essential instruments, when fluorescence background has to be rejected by energy discrimination, for example.

The readout time of CCD is in the second range, and that for imaging plate is minutes. It is often so inefficient and so time consuming.

- In this respect, the single photon counting pixel detector is regarded as a new generation of X-ray detectors. The most important features are the following.
  - No dark current, no readout noise and energy discrimination, resulting in maximum dynamic range.
  - High quantum efficiency.
  - Short readout time.



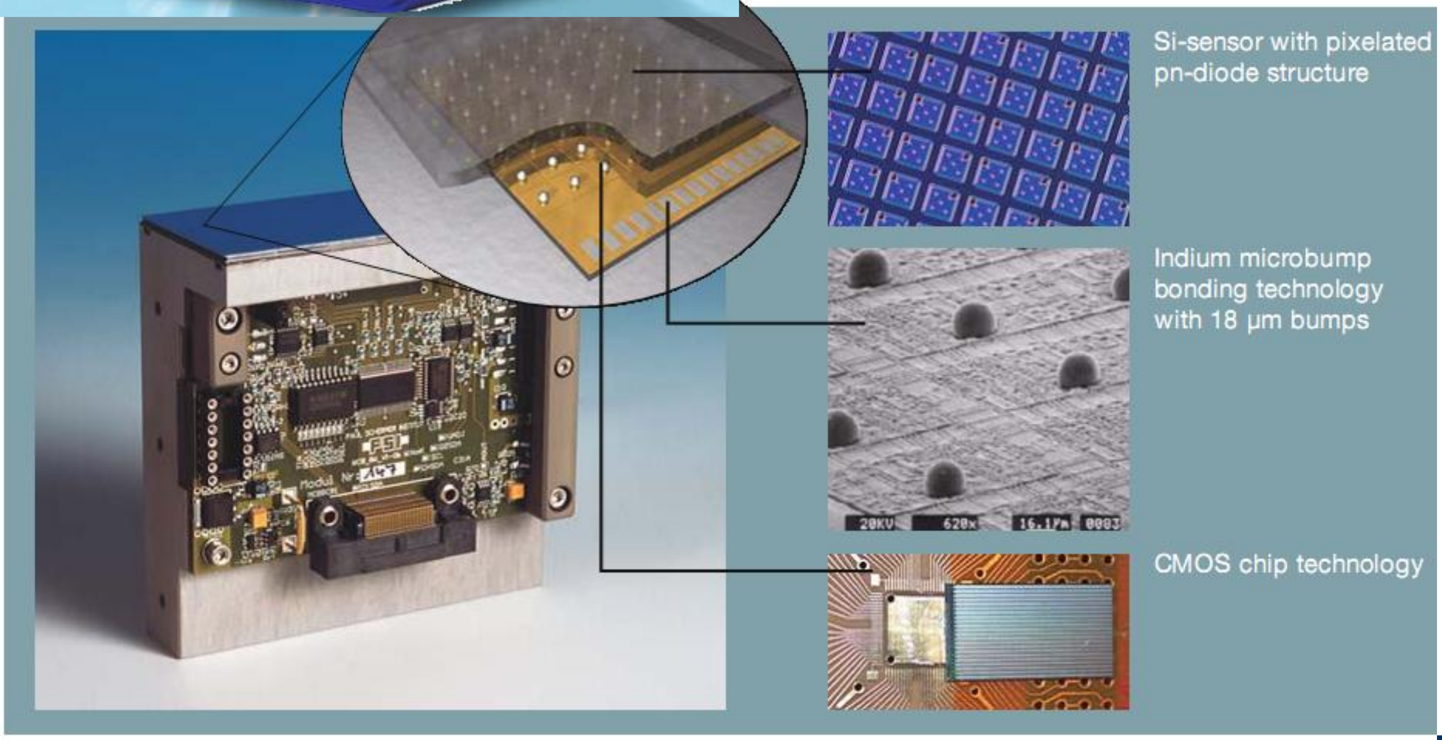




Complementary metal–oxide–semiconductor (CMOS)

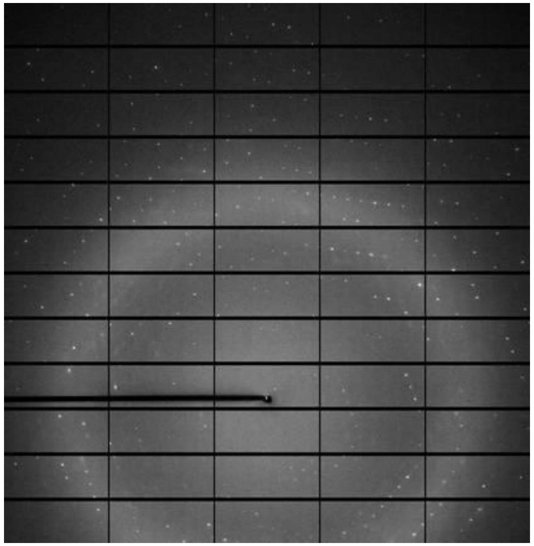
Pilatus

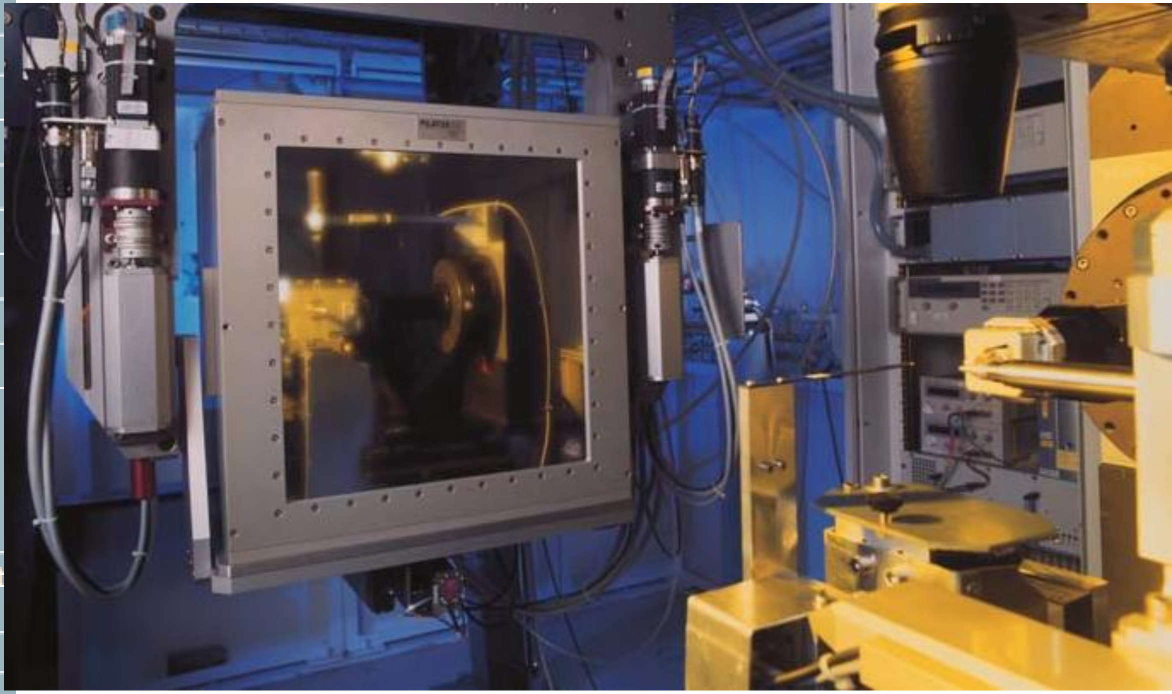
dectris.com



PILATUS (Pixel Apparatus for the SLS) is a challenging project to develop a large area single photon counting pixel detector for synchrotron radiation experiments by the PSI

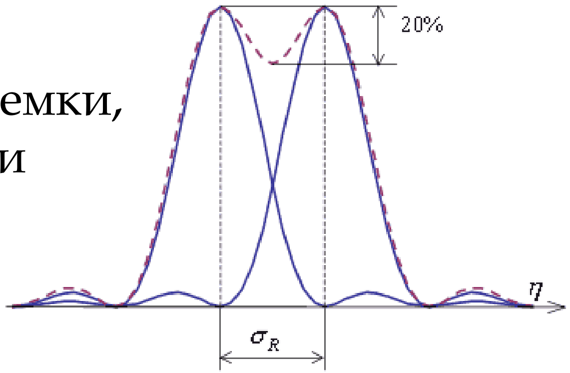
Number of modules	3 x 8 = 24
Sensor	Reverse-biased silicon diode array
Sensor thickness	320 µm
Pixel size	172 x 172 µm²
Format	1475 x 1679 = 2,476,525 pixels
Area	254 x 289 mm²
Intermodule gap	x: 7 pixels, y: 17 pixels, 8.0% of total area
Dynamic range	20 bits (1:1,048,576)
Counting rate per pixel	> 2 x 10⁶ X-ray/s
Energy range	3 – 30 keV
Quantum efficiency (calculated)	3 keV: 80% 8 keV: 99% 15 keV: 55%
Energy resolution	500 eV
Adjustable threshold range	2 – 20 keV
Threshold dispersion	50 eV
Readout time	3.6 ms ←
Framing rate	30 Hz
Point-spread function	1 pixel
Data formats	Raw data, TIF, EDF, CBF
External trigger/gate	5V TTL, 3 different modes
Software interface	Through socket connection; clients for EPICS, SPEC and stand-alone operation are available
Cooling	Close-circuit water cooling unit for temperature stabilization
Power consumption	200 W
Dimensions (WHD)	384 x 424 x 458 mm
Weight	42 kg

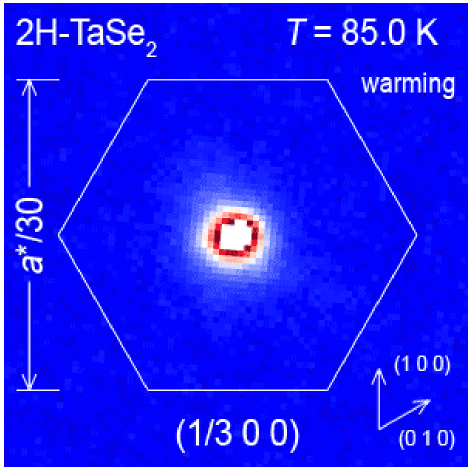


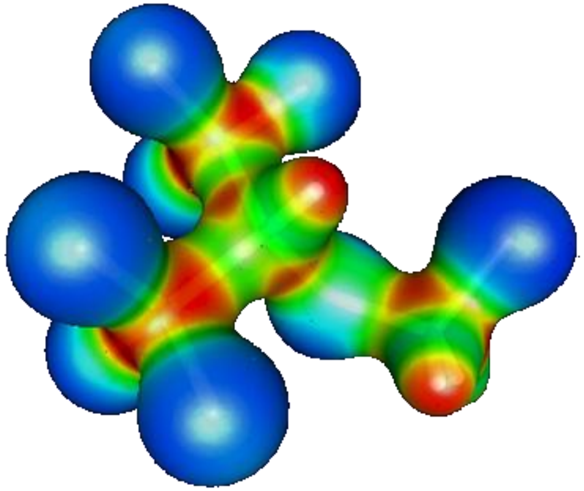


Разрешение детектора и дифракционного эксперимента

- Разрешение детектора – разрешающая способность по Релею.
- Определяется геометрией съемки, размером пикселя, свойствами люминафора
- Определяет детальность дифракционной картины







- Разрешение дифракционного эксперимета определяет детальность структуры (электронной плотности)
- Определяется максимальным углом Брегга где еще наблюдается дифракция

Разрешение 2D детектора

We start from the Bragg's low for a reflection HKL detected at radius R for a sample of diameter δD at the distance D.

$$\Theta = \frac{1}{2}\cdot \operatorname{atan}\left(\frac{R}{D}\right)$$ is corresponding Bragg angle

$$d = \frac{\lambda}{2\sin\left(\frac{1}{2}\cdot \operatorname{atan}\left(\frac{R}{D}\right)\right)}$$ Bragg's law expressed via R and D

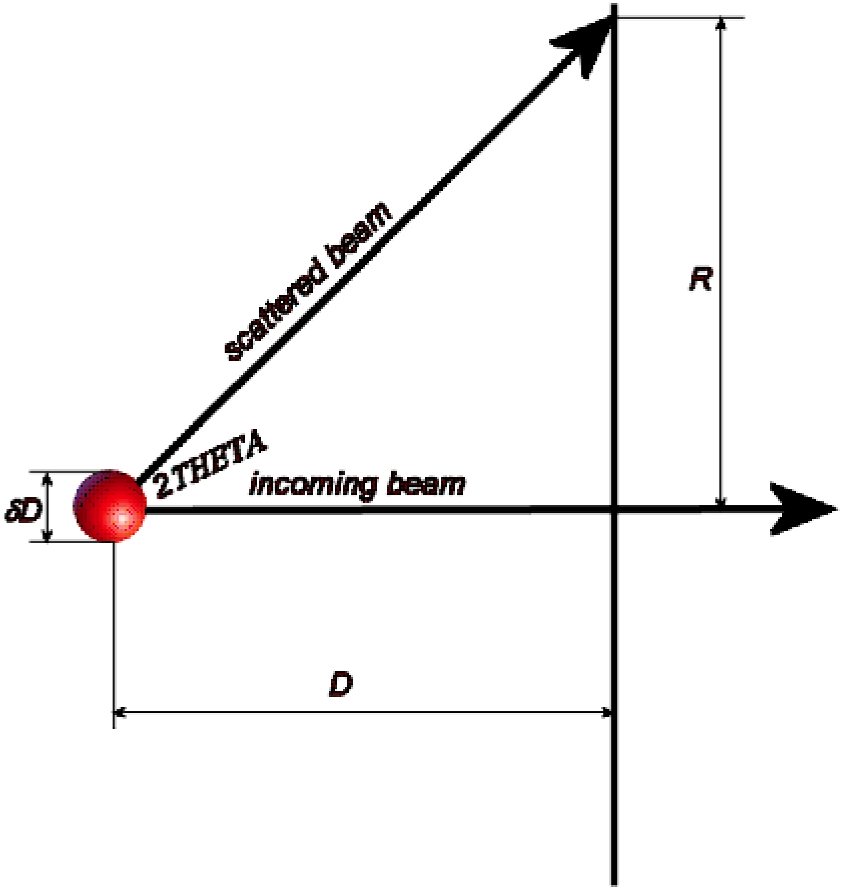
The uncertainty in d can be expressed as follows ("error propaga

$$\delta d^2 = \left(\frac{\delta d}{\delta D}\cdot \delta D\right)^2 + \left(\frac{\delta d}{\delta R}\cdot \delta R\right)^2 \qquad (1)$$

$$\frac{\delta d}{\delta D} = \frac{d}{2\tan(\Theta)\cdot D\cdot\left(1+\tan(2\Theta)^2\right)}$$

$$\frac{\delta d}{\delta R} = \frac{-d}{2\tan(\Theta)\cdot\left(1+\tan(2\Theta)^2\right)}\cdot\frac{R}{D}$$

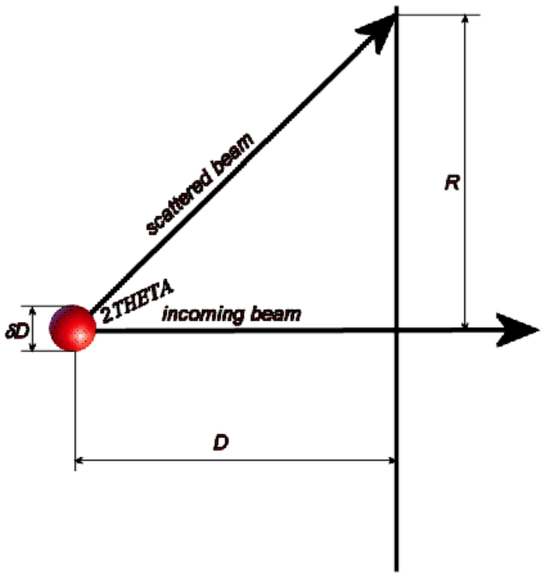
$$\delta d^2 = \left[\frac{d}{2\tan(\Theta)\cdot\left(1+\tan(2\Theta)^2\right)}\right]^2\cdot\left[\left(\frac{\delta D}{D}\right)^2 + \left(\frac{\delta R}{D}\right)^2\right]$$



The uncertainty in the crystal-to-detector distance is the size of the sample (diameter of th cappilary) or the size of the incoming beam, whatever is smaller.

δD =sample size ~ capillary

The uncertainty in the radial position of the reflection (radius of the Debye ring at 2Θ) is a sum of projection of the capillary onto the detector and the pixel size of the detector



$$\delta R = \frac{\delta D}{\cos(2\Theta)} + r$$ where r is the size of the pixel (2)

$$\left(\frac{\delta d}{d}\right)^2 = \left[\frac{1}{2\tan(\Theta)\cdot\left(1+\tan(2\Theta)^2\right)}\right]^2 \cdot \left[\left(\frac{\delta D}{D}\right)^2 + \left(\frac{\frac{\delta D}{\cos(2\Theta)} + r}{D}\right)^2\right]$$

$$\Delta 2\Theta = 2\frac{\delta d}{d}\tan(\Theta) = \frac{1}{1+\tan(2\Theta)^2}\cdot\sqrt{\left(\frac{\delta D}{D}\right)^2 + \left(\frac{\frac{\delta D}{\cos(2\Theta)} + r}{D}\right)^2}$$

$$\Delta 2\Theta = \frac{\delta D}{D}\cdot\cos(2\Theta)$$ which is the formula (6) from [P. Norby, J. Appl. Cryst. 1997, 30,

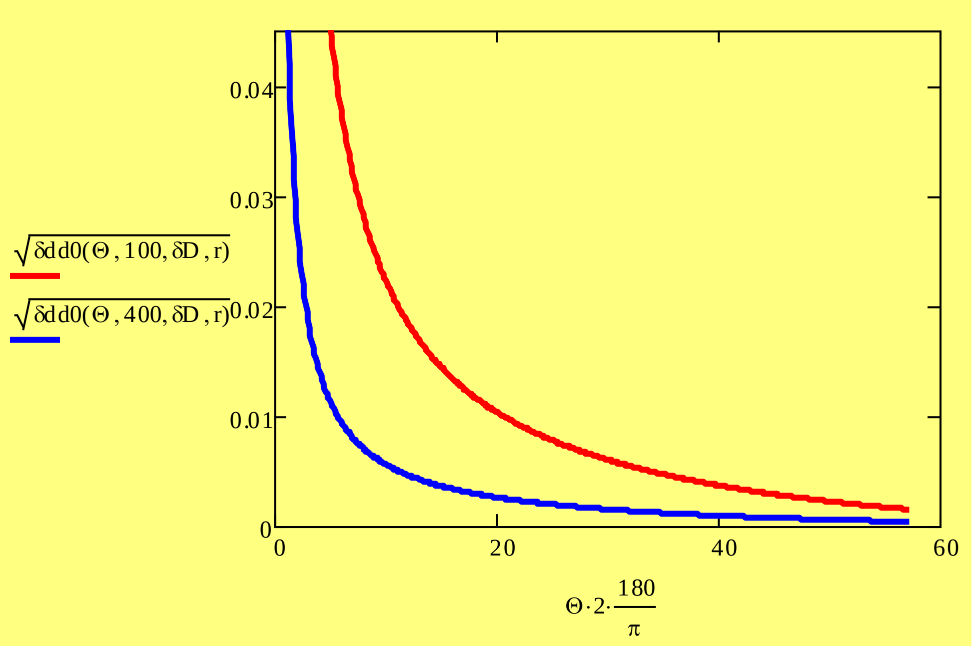
Numerical illustration

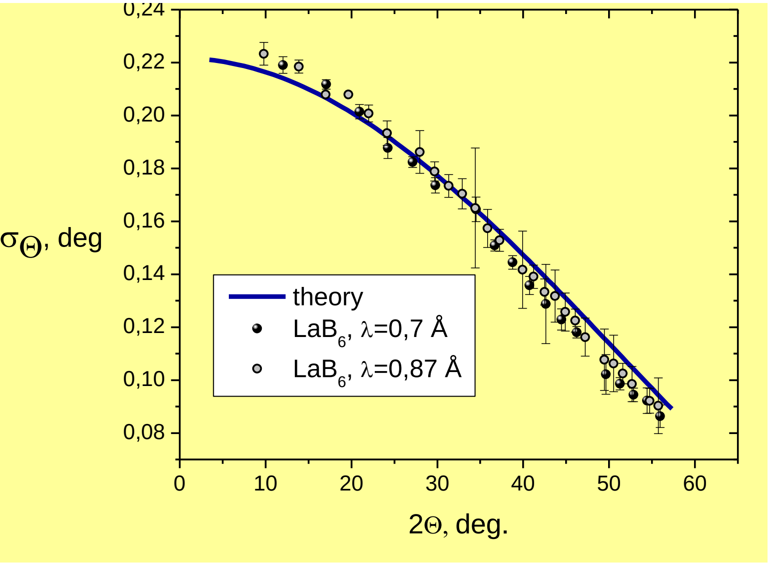
$r := 0.15$ pixel size

$\delta D := 0.2$ capillary size

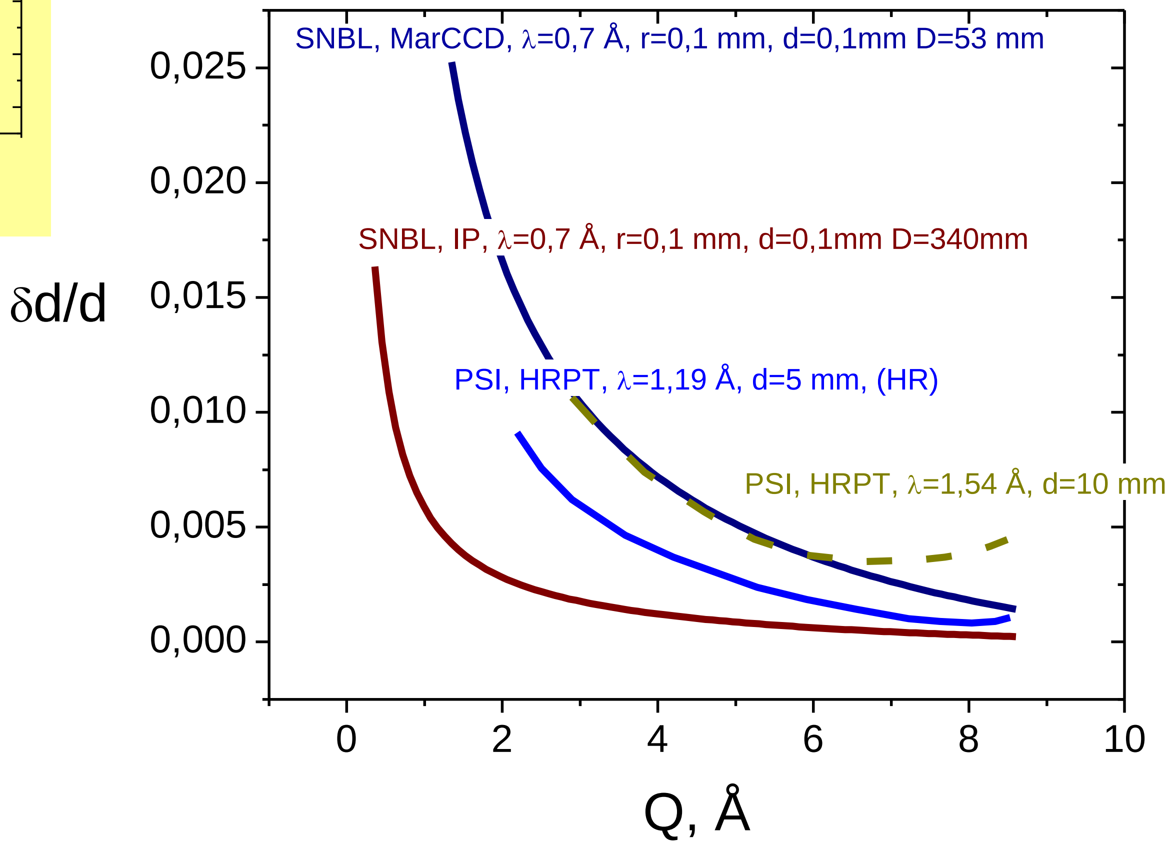
$\Theta := 0, 0.001 .. 0.5$

$$\delta dd0(\Theta, D, \delta D, r) := \left[\frac{1}{2\tan(\Theta)\cdot\left(1+\tan(2\Theta)^2\right)}\right]^2\cdot\left[\left(\frac{\delta D}{D}\right)^2+\left(\frac{\frac{\delta D}{\cos(2\Theta)}+r}{D}\right)^2\right]$$





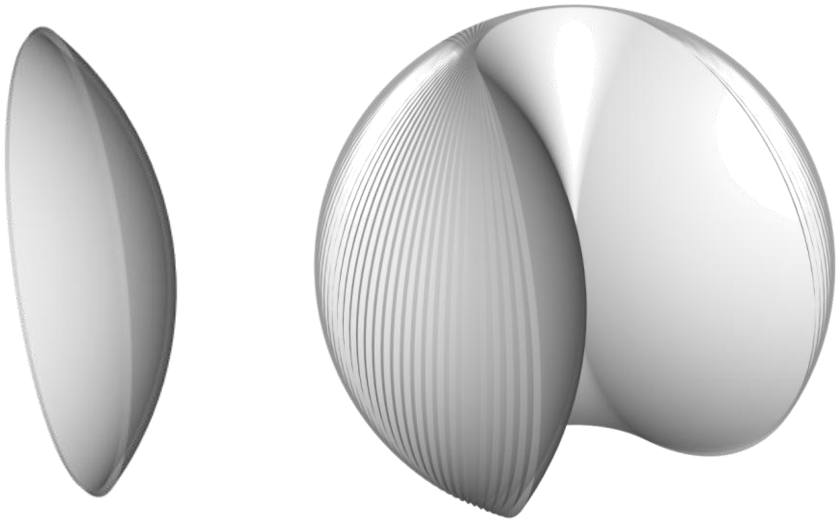
σΘ, deg
theory
LaB6, λ=0,7 Å
LaB6, λ=0,87 Å
2Θ, deg.



δd/d
SNBL, MarCCD, λ=0,7 Å, r=0,1 mm, d=0,1mm D=53 mm
SNBL, IP, λ=0,7 Å, r=0,1 mm, d=0,1mm D=340mm
PSI, HRPT, λ=1,19 Å, d=5 mm, (HR)
PSI, HRPT, λ=1,54 Å, d=10 mm
Q, Å

Стратегия сбора данных монокристального эксперимента.

- Completeness
- Redundancy
- Resolution
- Time



Оптимизация измерений – CrysAlis Pro

Experiment Strategy (1.0.57)

User parameters

Resolution 0.7 ○ Laue group ◉ Other hemisphere

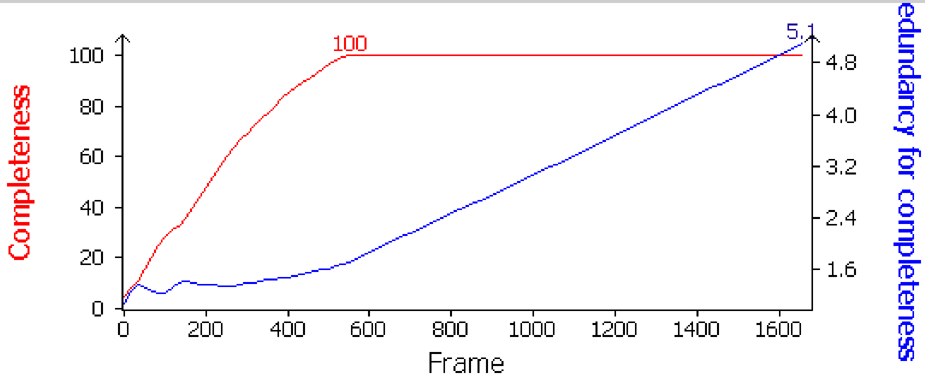
Detector Distance 100 | Advanced | Lattice Wizard | Options | CCD par

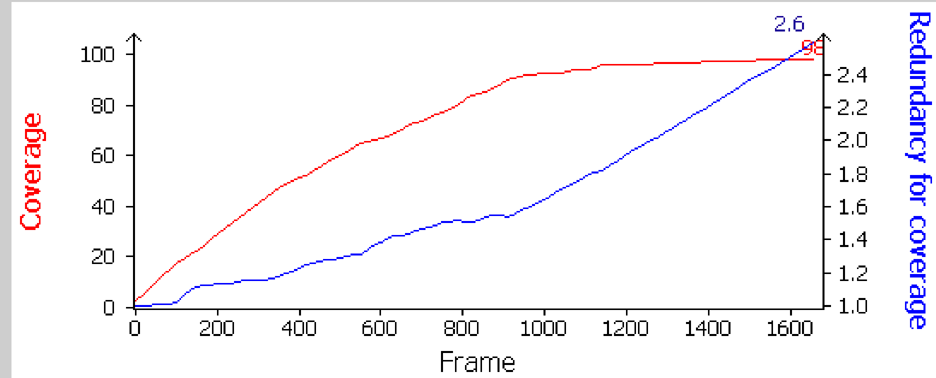
Strategy mode

Complete redundant data | limit 100.0 | IUCr limit | Max 100.00

Generates runs with global redundancy constraint and keeps

Redundancy constraint: 5.0





Res	Completeness (under Laue symmetry) #Data	#Theory	%	Redundancy	#Total	Coverage (under P1) #Data	#Theory	%	Redundancy	#Total
4.70- 1.49	65	65	100.00%	10.5	680	130	130	100.00%	5.2	680
1.49- 1.14	65	65	100.00%	7.9	513	130	130	100.00%	3.9	513
1.14- 1.03	65	65	100.00%	5.4	351	130	130	100.00%	2.7	351
1.03- 0.92	65	65	100.00%	4.7	308	129	130	99.23%	2.4	308
0.92- 0.87	65	65	100.00%	4.3	281	128	130	98.46%	2.2	281
0.87- 0.82	65	65	100.00%	4.1	265	130	130	100.00%	2.0	265
0.82- 0.78	65	65	100.00%	3.9	253	127	130	97.69%	2.0	253
0.78- 0.74	65	65	100.00%	3.7	239	126	130	96.92%	1.9	239
0.74- 0.73	65	65	100.00%	3.4	224	125	130	96.15%	1.8	224
0.73- 0.70	69	69	100.00%	3.2	224	133	138	96.38%	1.7	224
4.70- 0.70	654	654	100.00%	5.1	3338	1288	1308	98.47%	2.6	3338

runs: 25, frames: 1677

theta settings: 2, abs minimum theta: 0.00, abs maximum theta: 36.44, max resolution: 0.673

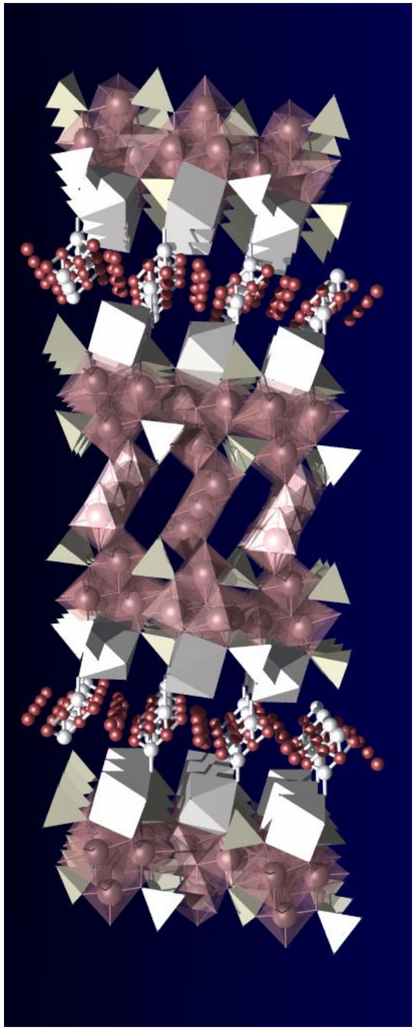
Approximate experiment time: 15h 29m, end of experiment Sun May 30 04:43:23 2010.

Current lattice 4.6998(4) 4.7011(4) 4.6999(4) 90.016(8) 89.994(7) 89.983(7) 103.842(16) User Bravais lattice type P

Edit runs | Find Experiment | Completeness | Help | Save experiment | Cancel

На что обратить внимание при выборе прибора для структурного эксперимента.

- Подготовка кристаллов, оптимизация размера
- Выбор детектора
- Выбор стратегии измерений
- Все это надо обсудить со специалистами ESRF <u>до подачи</u> предложения на эксперимент



Спасибо за внимание