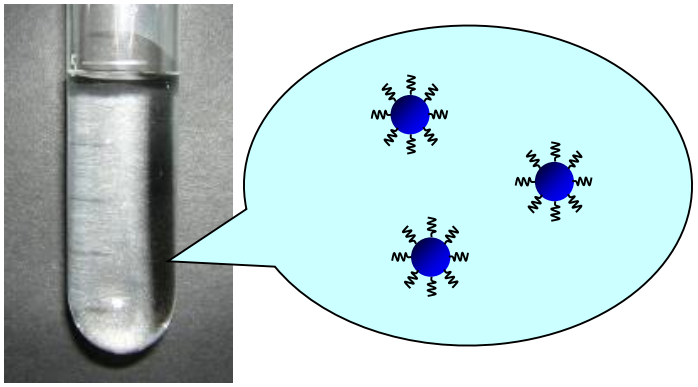
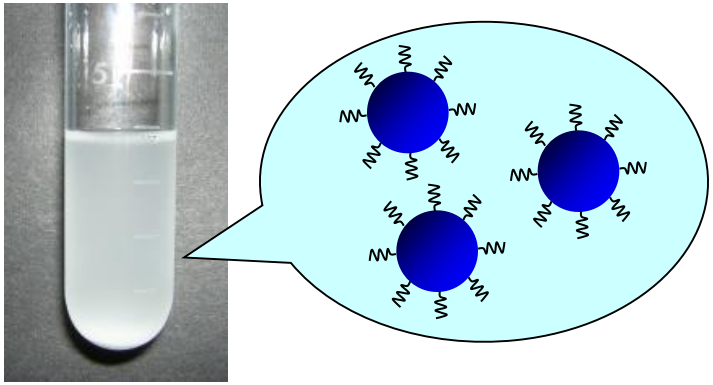
The Latest X-ray Equipment and Method For Nanotechnology (SAXS)

Purpose of SAXS

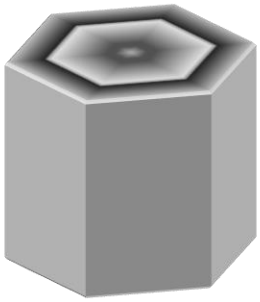
Small Angle X-ray Scattering

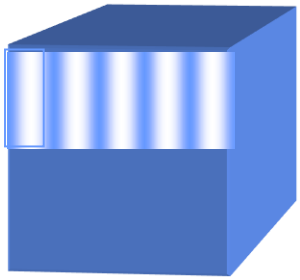
- Particle size estimation



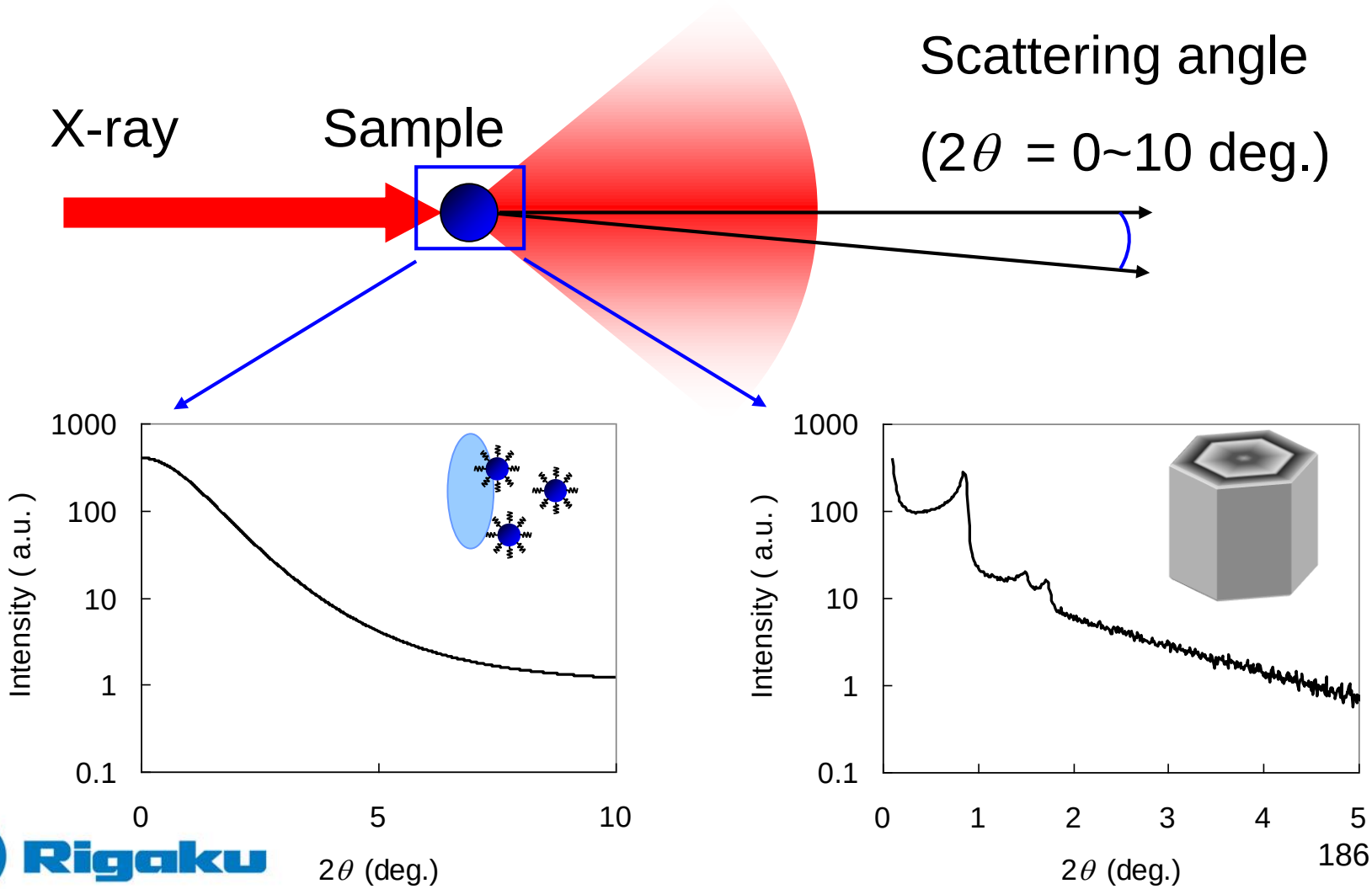


- Phase identification

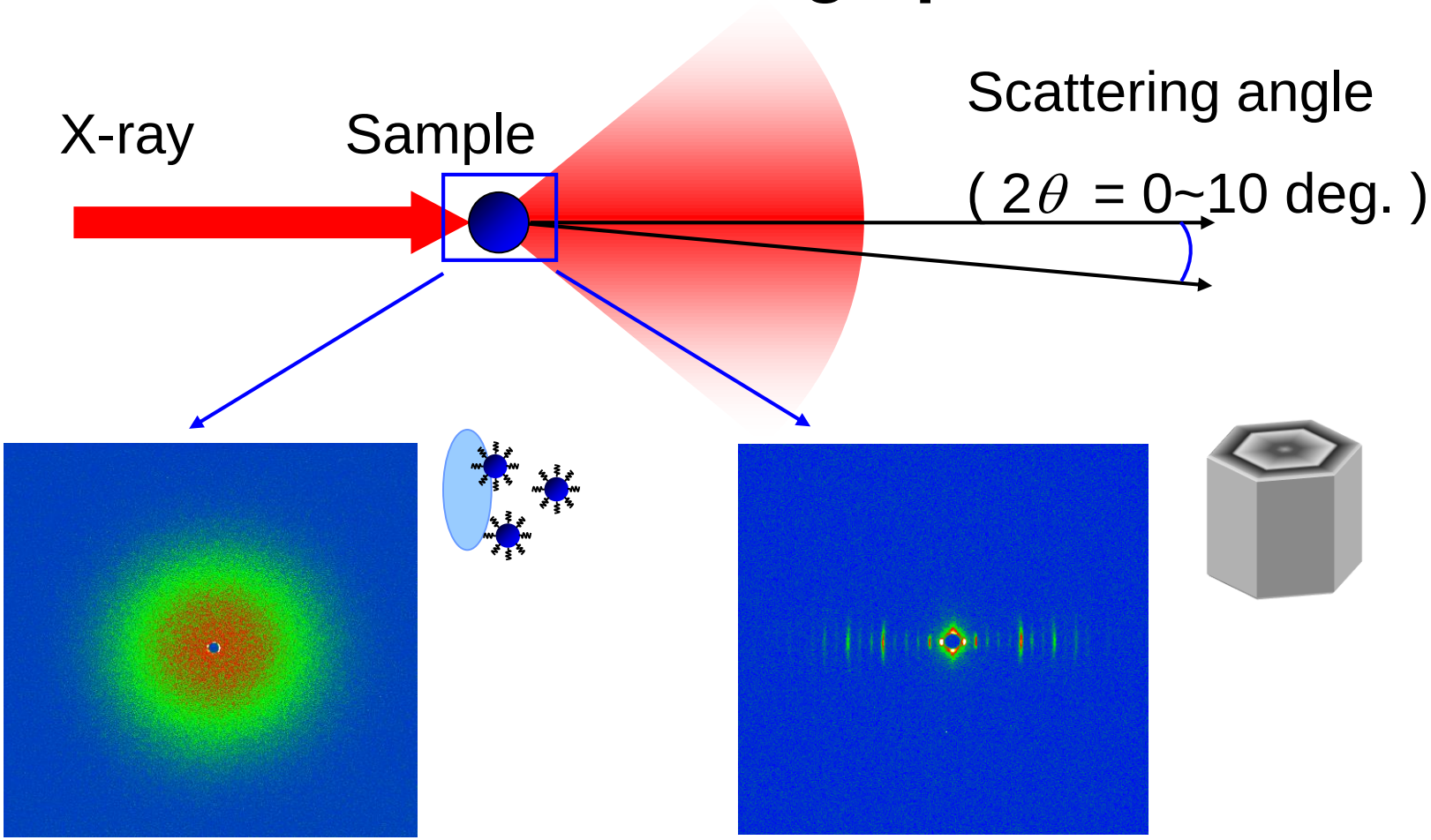




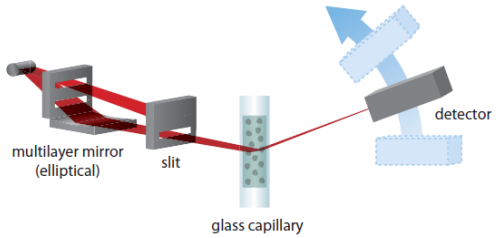
Principle of SAXS
~ Line focusing optics ~



Principle of SAXS
~ Point focusing optics ~



SAXS geometry

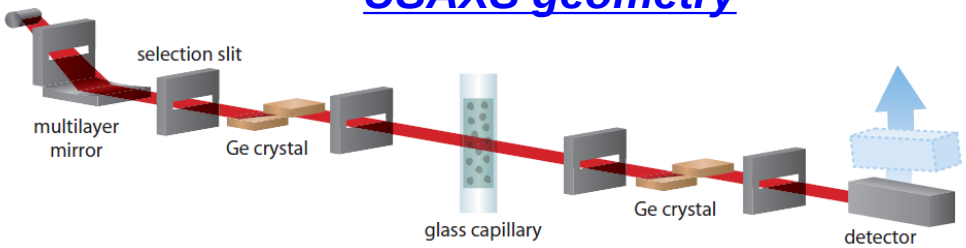


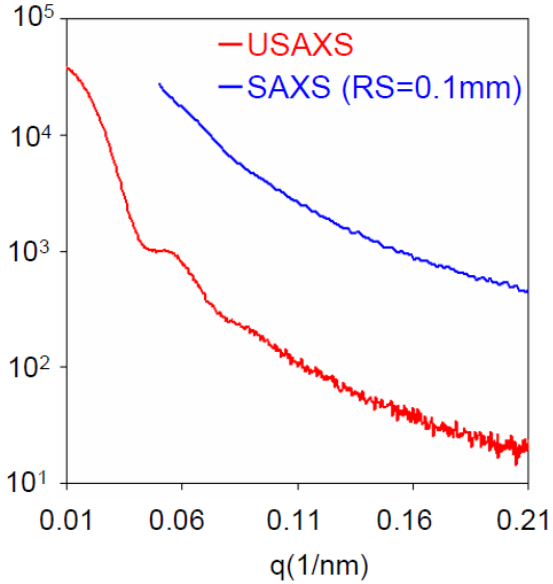
$$A(\mathbf{q}) = \int_V \Delta\rho(\mathbf{r}) \exp(i\mathbf{q}\mathbf{r}) d\mathbf{r}$$

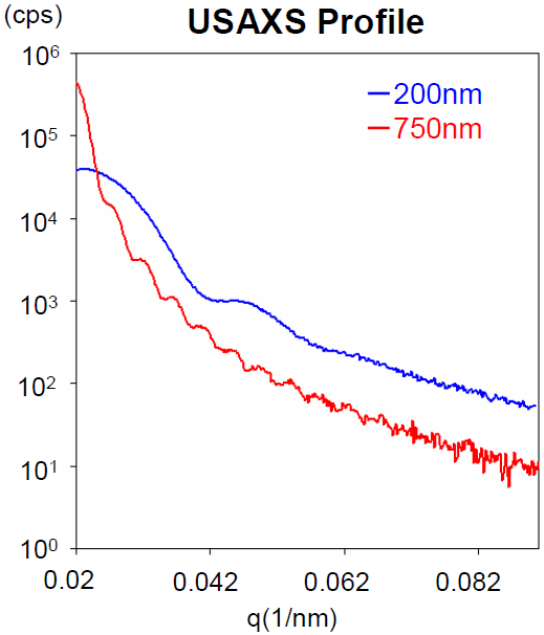
$$q = \frac{4\pi}{\lambda} \sin\frac{2\theta}{2}$$

Scattering amplitude from electron density fluctuations
Is given as FT of the density fluctuations

USAXS geometry

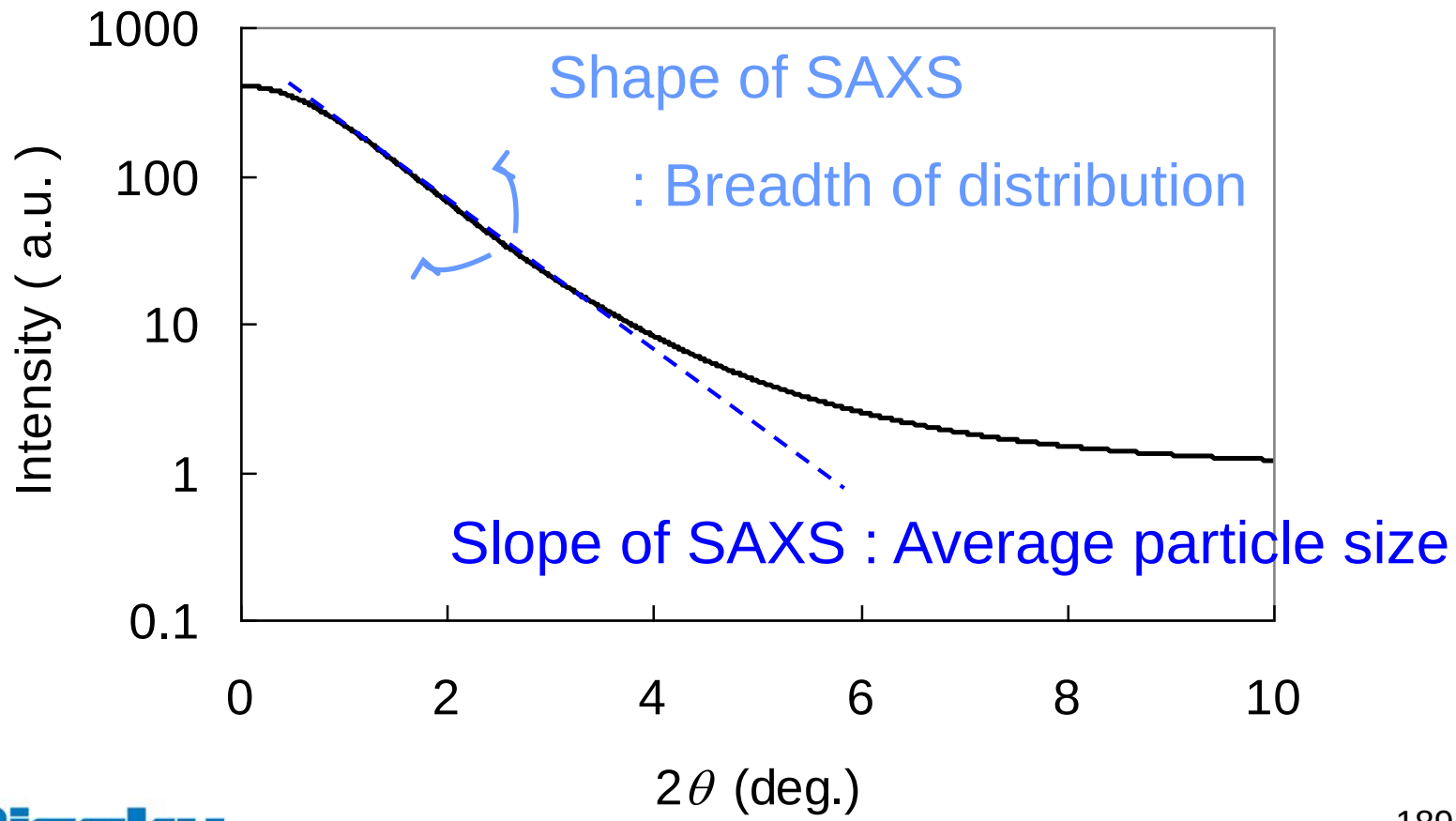




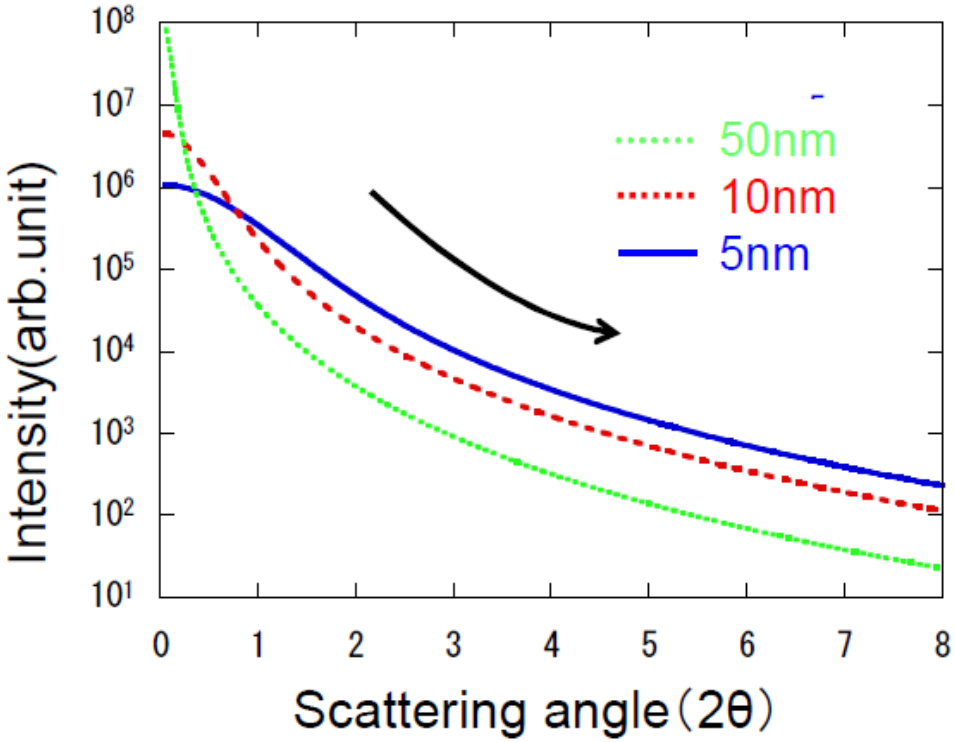


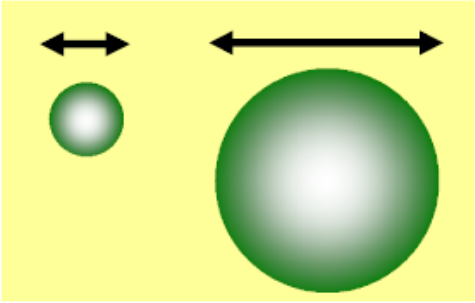
Particle information

- Particle size and particle distribution



Particle size and SAXS profile

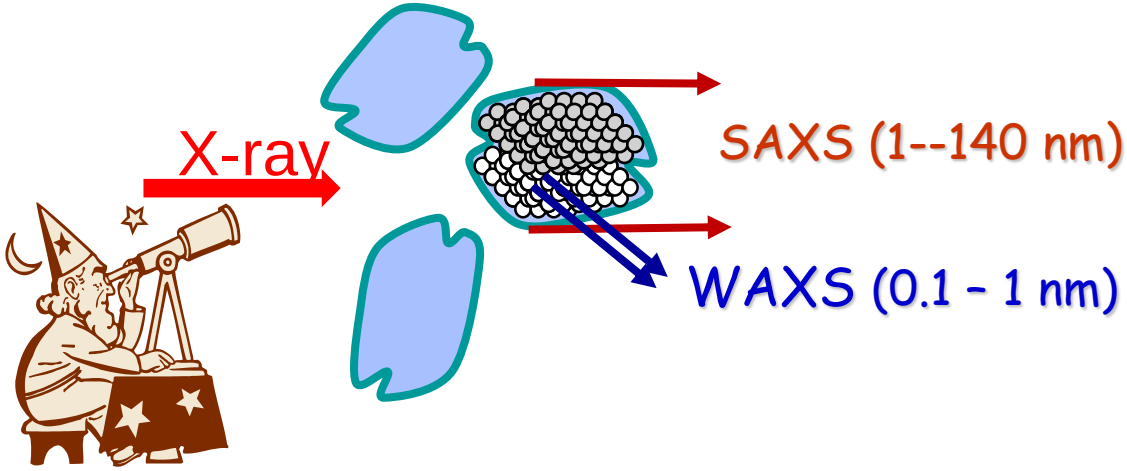




Shape of SAXS: Breadth of distribution

Slope of SAXS : Average particle size

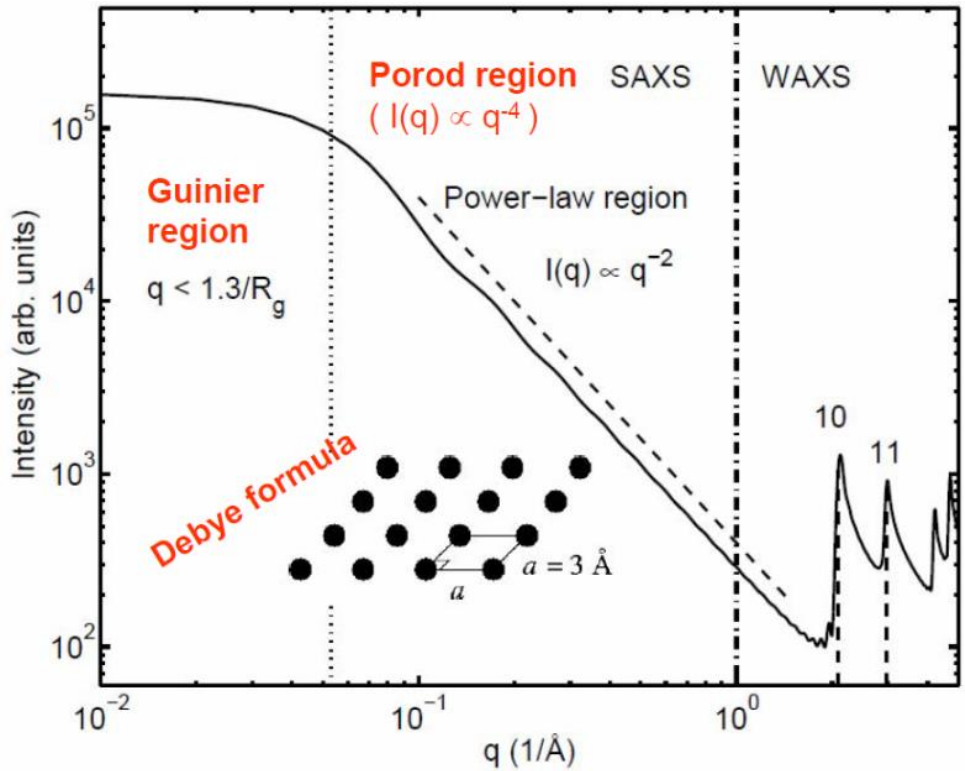
Scattering measurement range



SAXS – super molecular structure (macromolecules, molecular aggregates)

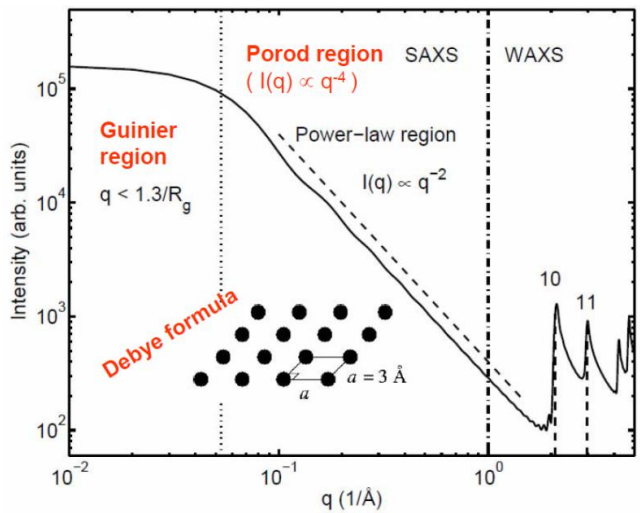
WAXS – Inter molecular distances

Guinier plot - *ln I(q)* vs q^2



Rigaku

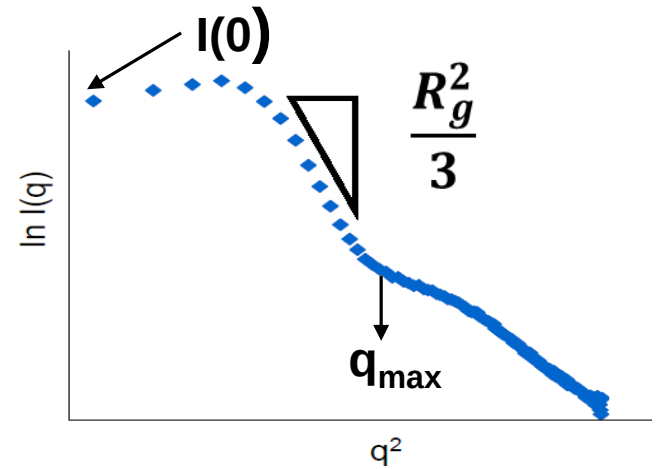
Scattering measurement range



Guinier`s law (low `q` limit)

$$I(q) = (\rho v)^2 \, exp\left(-q^2 {R_g}^2 / 3\right)$$

$$\ln I(q) = \ln(\rho v)^2 - \left(\frac{{R_g}^2}{3}\right) q^2$$

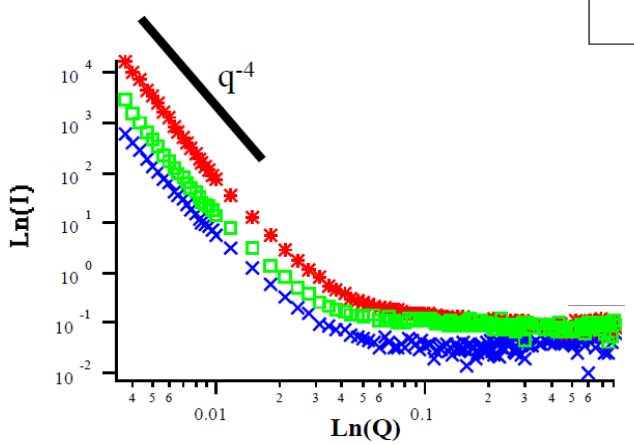


Porod law (high `q` limit)

at large q,

$$I(q) \propto Q^{-n}$$

n = 4 : smooth
1 < n < 3 : mass fractal
3 < n < 4 : surface fractal



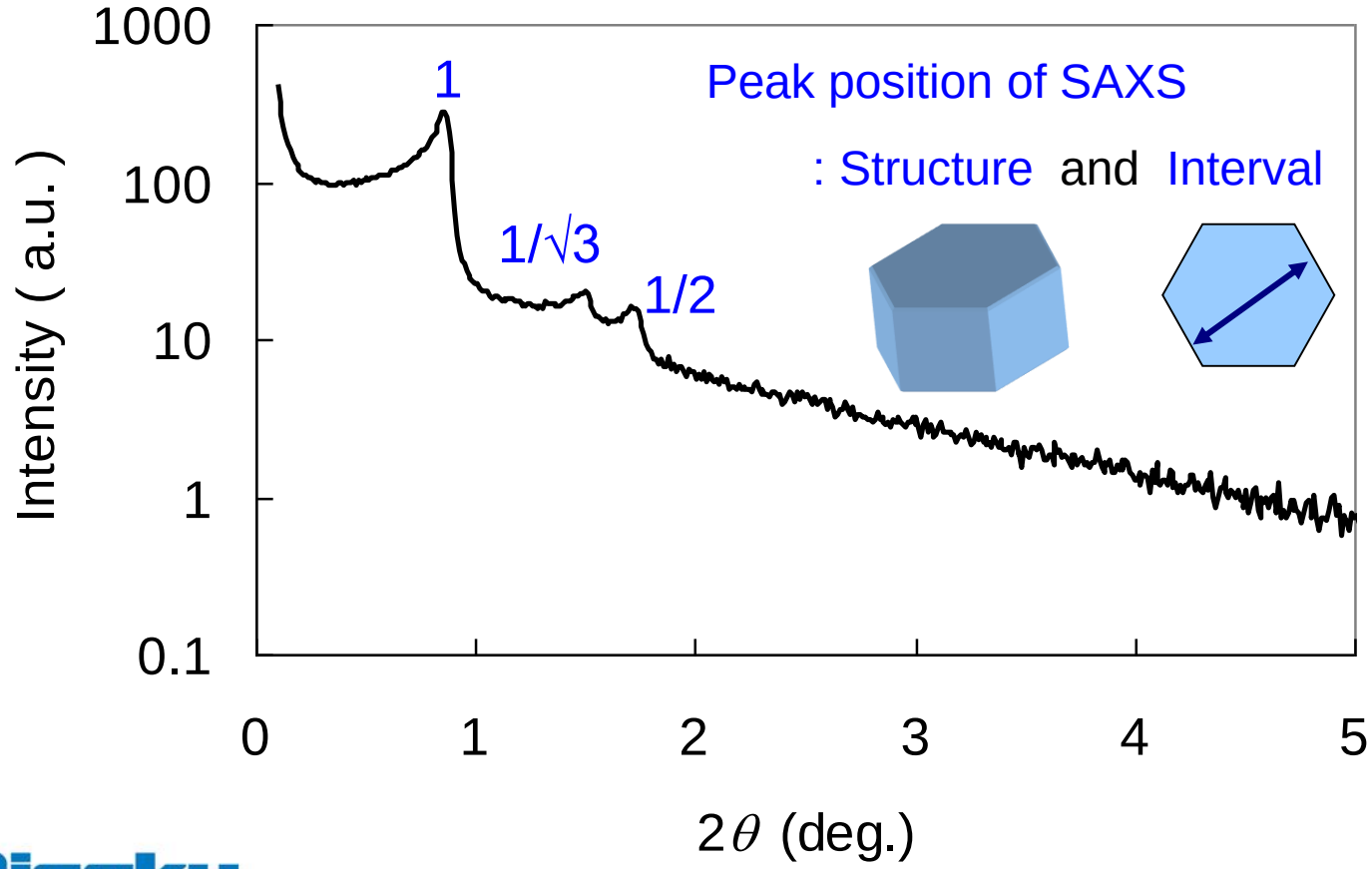
$$I(q)_{q\to\infty} = 2\pi S(\rho_1 - \rho_2)^2 q^{-4}$$

S surface area per unit volume

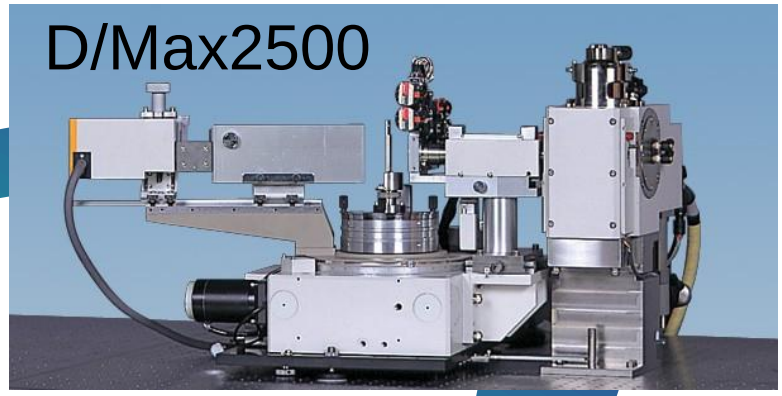
Rigaku

Phase information

- The structure of long period

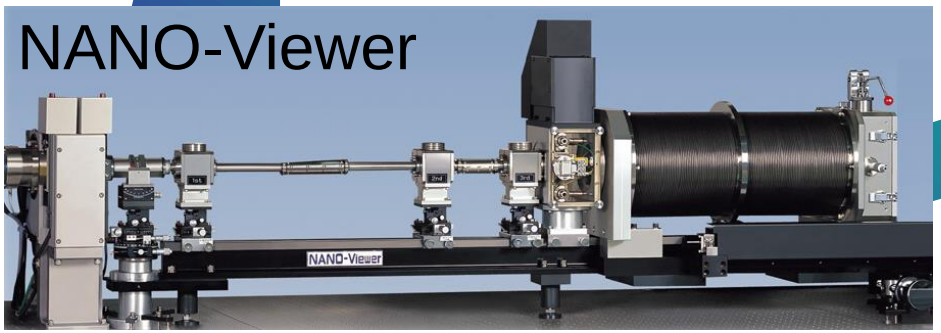


SAXS Optics



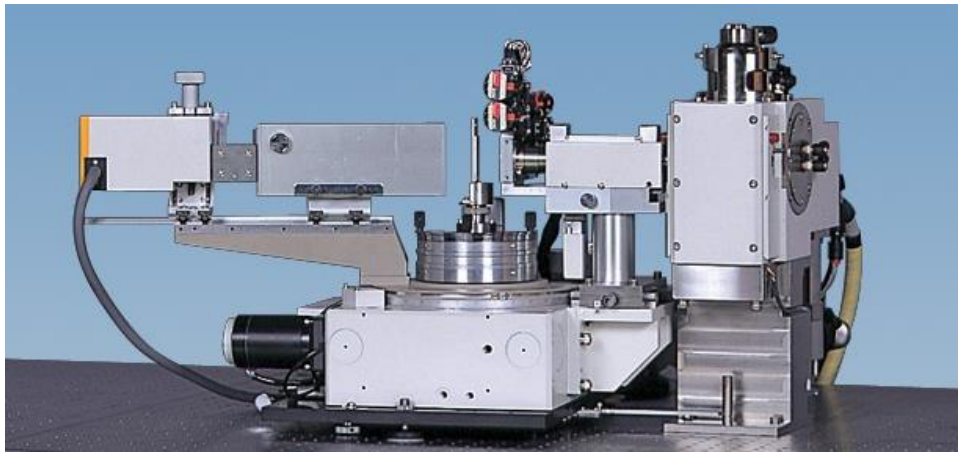
SAXS optics

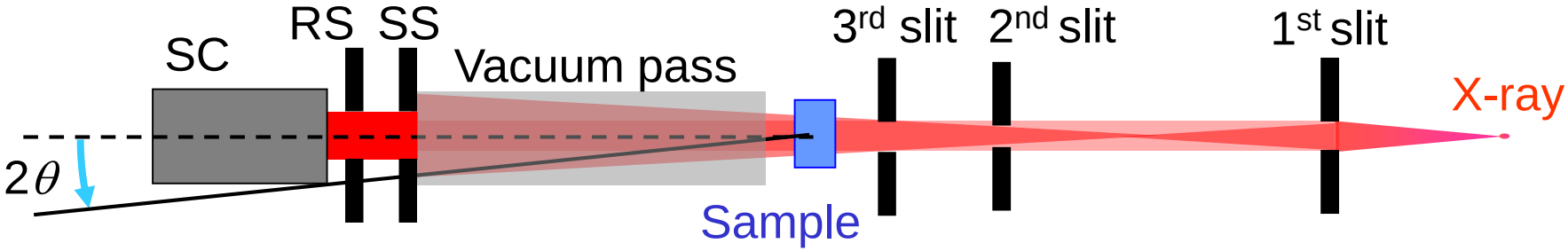
Line focusing optics
(D/Max series, Ultima IV, TTRAXIII)
Point focusing optics
(NANO-Viewer)



Line focusing optics

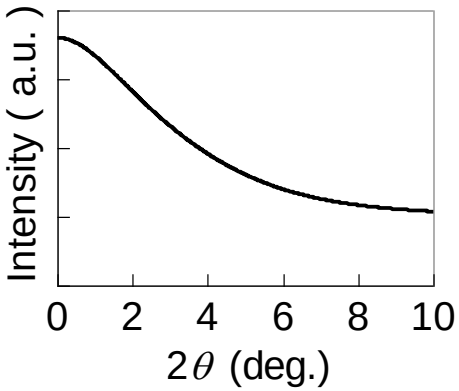
- **D/Max 2200 & D/Max 2500**
  - You can obtain high resolution SAXS profiles

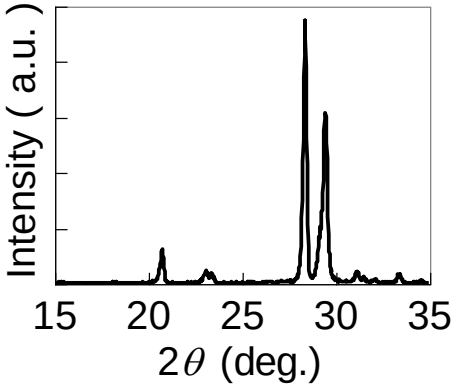


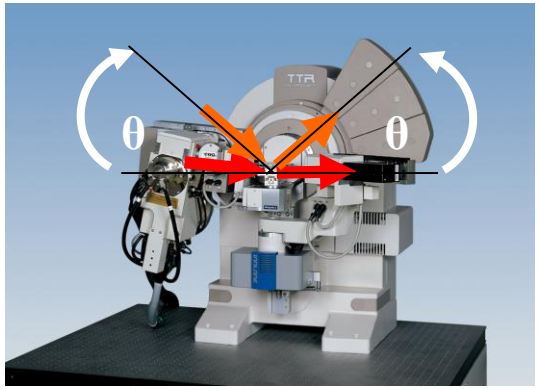


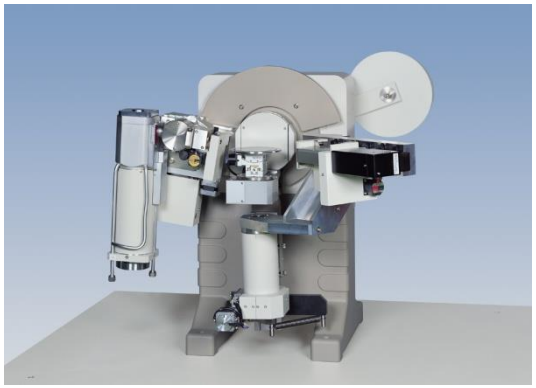
Line focusing optics

- **SmartLab, Ultima IV & TTRAX III**
  - You can switch SAXS and Wide angle X-ray diffraction





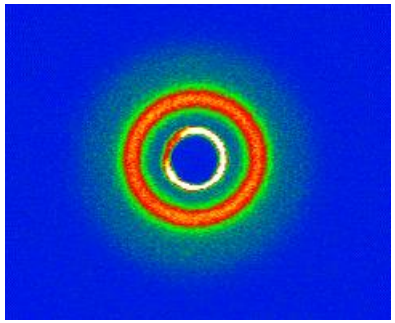


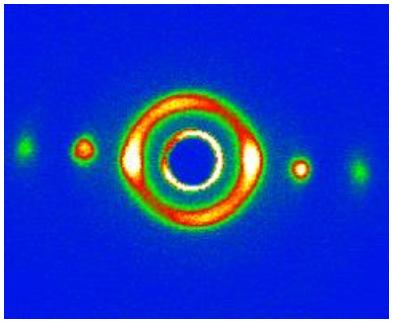


Point focusing optics

- **NANO-Viewer (2D-SAXS System)**
  - You can evaluate Nano-structures

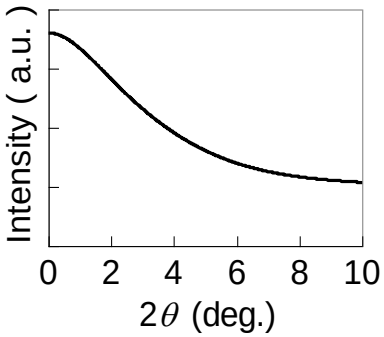
2D detector





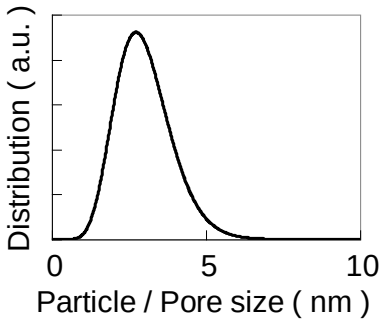
NANO-Solver

- Automatic particle size & distribution estimation!



SAXS

profile

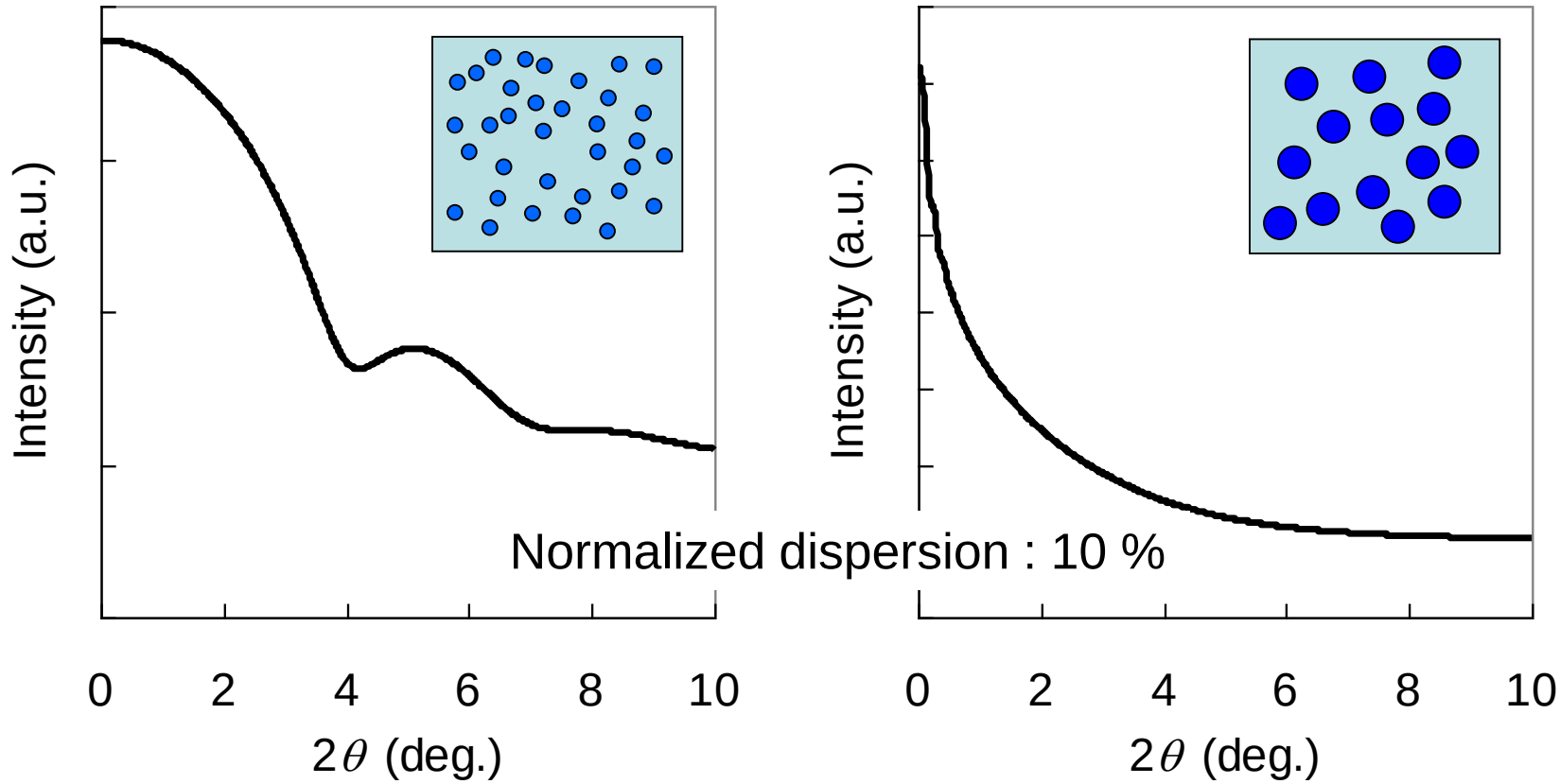


❖size

❖distribution

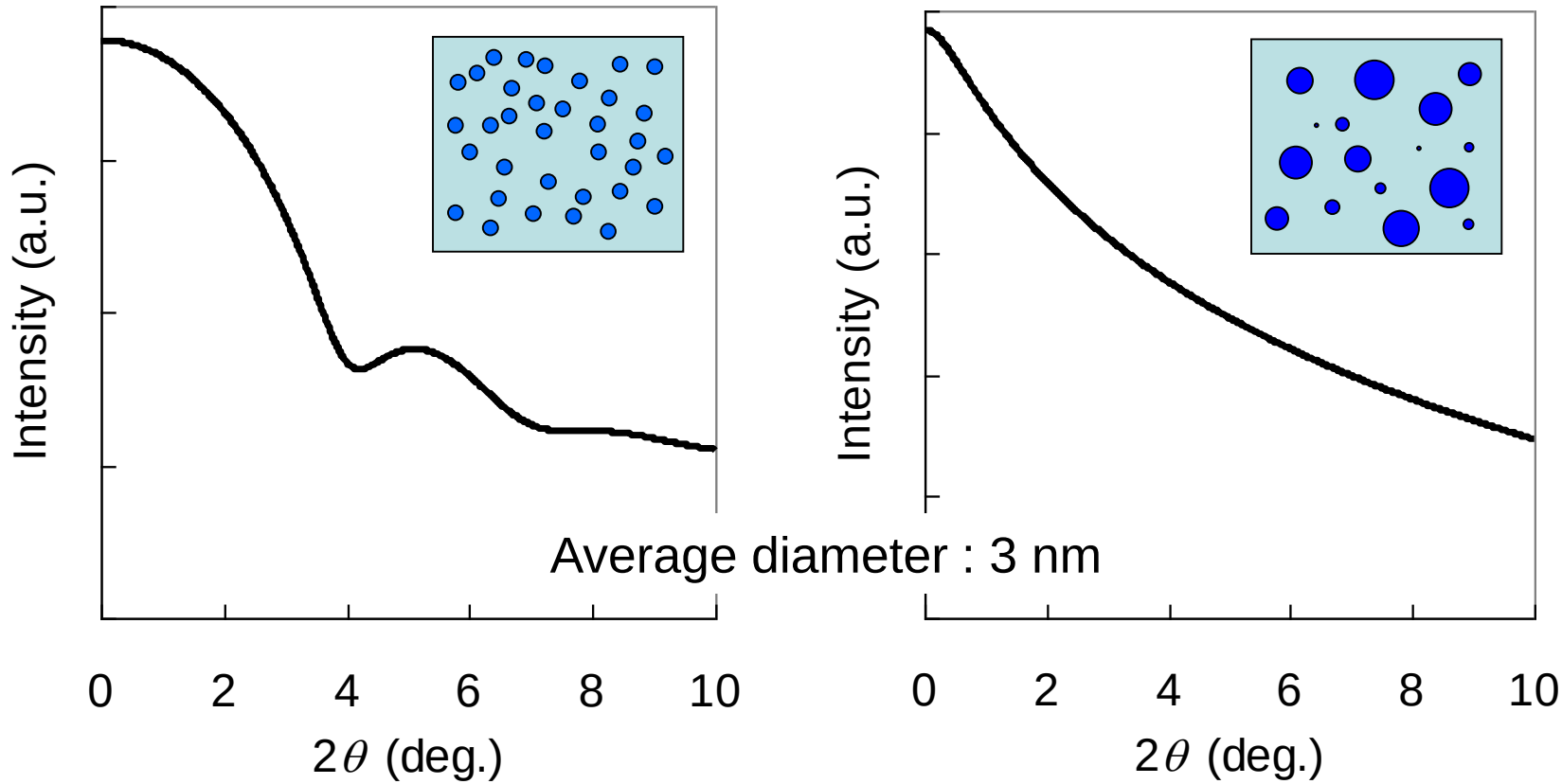
Relations of size and profiles

- Size: 3 nm
- Size: 60 nm



Relations of dispersion and profiles

- Dispersion: 10 %
- Dispersion: 90 %



The advantages of SAXS

- Easy sample preparation（non-destructive measurement)
- There is no limit of a sample state（solution, powder, bulk）
- A particle does not have to be a crystalline structure
- Quick measurement and quick analysis
- The average particle size and distribution information in the sample can be estimated
- The particle/pore size you can analyze is about 1~100 nm (in particular, sensitivity is high in less than 10 nm)

The disadvantages of SAXS

- Cannot distinguish between particles and pores
- Cannot distinguish particle information of an individual component, in case of many components
  - ❖ Cannot distinguish the scatter by SAXS profile
- A low density sample is difficult
  - ❖ Depends on an electronic density difference of a particle and a matrix
- Cannot measure the sample that X-rays cannot penetrate it (Transmission method)
  - ❖ When the solvent is with a big absorption coefficient
  - ❖ When sample thickness can not be changed

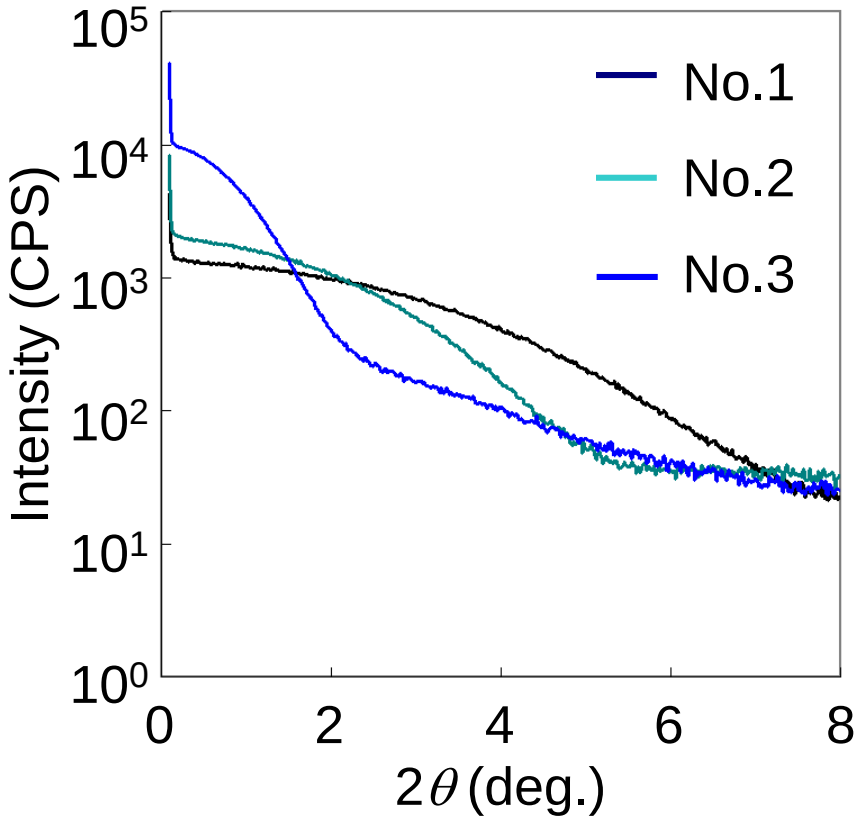
Applications of SAXS

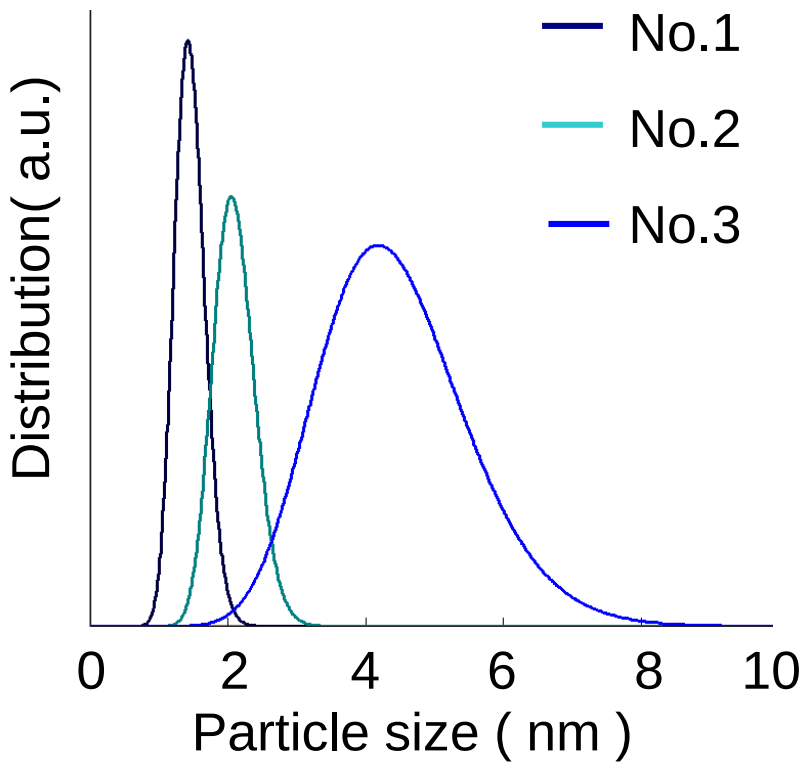
Particle size estimation

transmission mode & reflection mode

SAXS profiles and size distribution

- Au nanoparticles

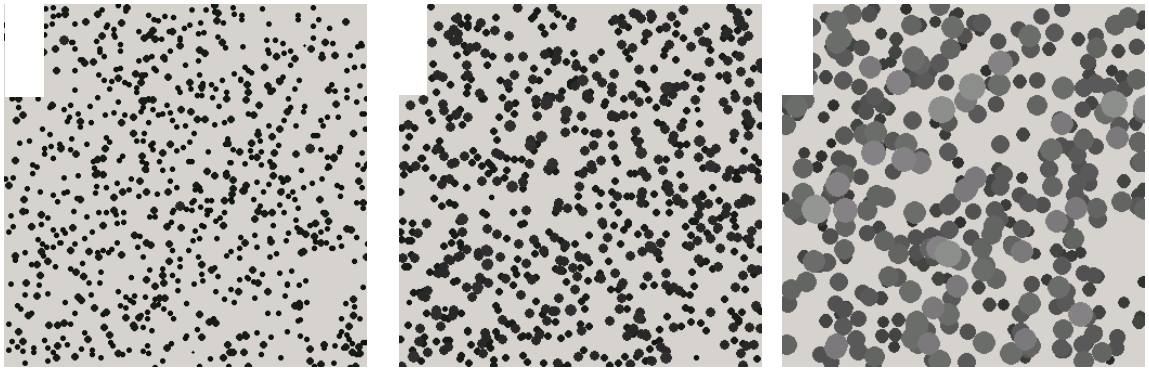




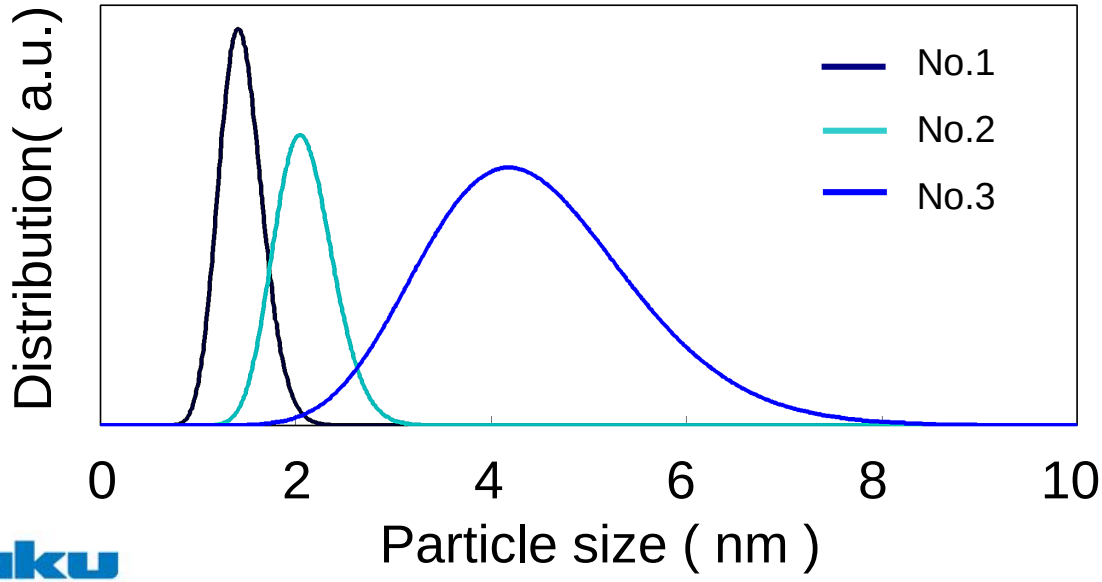
Comparison of TEM image

TEM image

No.1 No.2 No.3

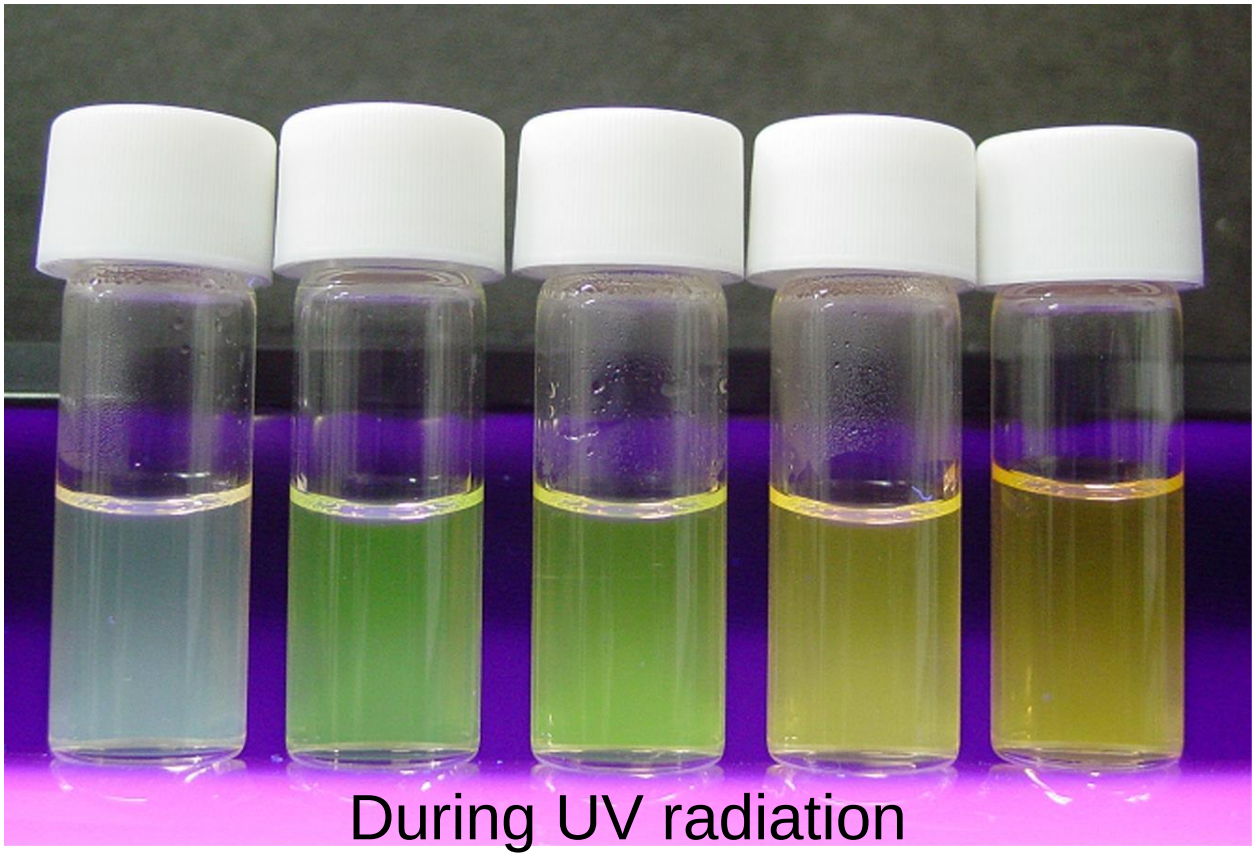


SAXS



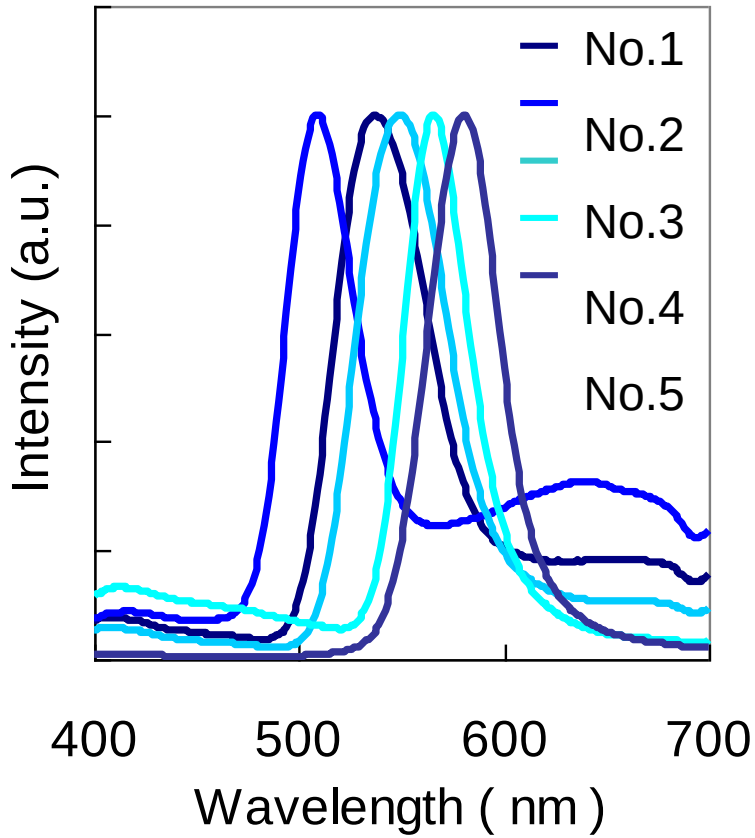
Nano size Cd-Se particles distribution

No.1 No.2 No.3 No.4 No.5

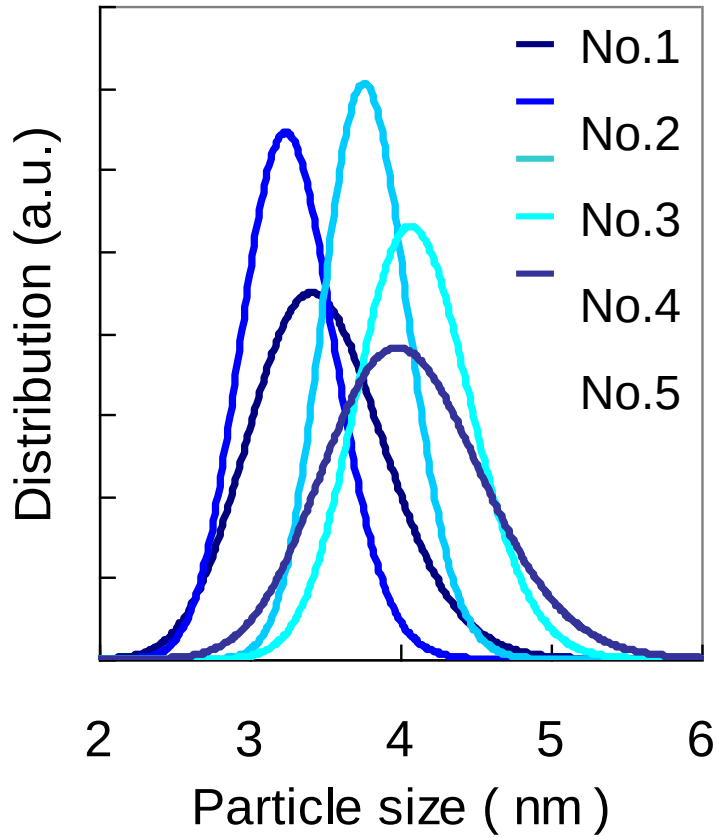


Correlation of fluorescence with SAXS

- Fluorescence

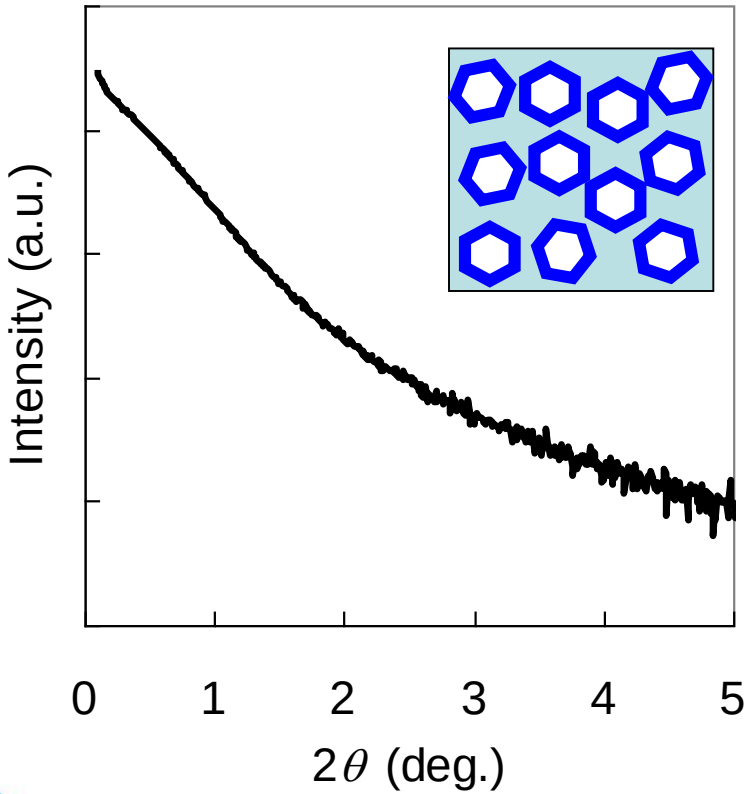


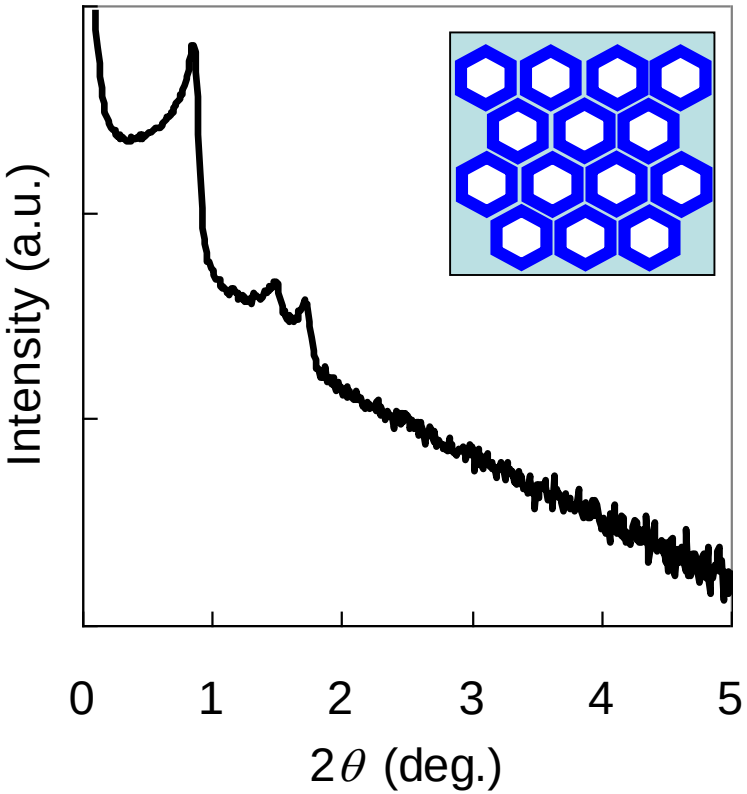
- SAXS



Nano size of porous silica

- Mesoporous silica

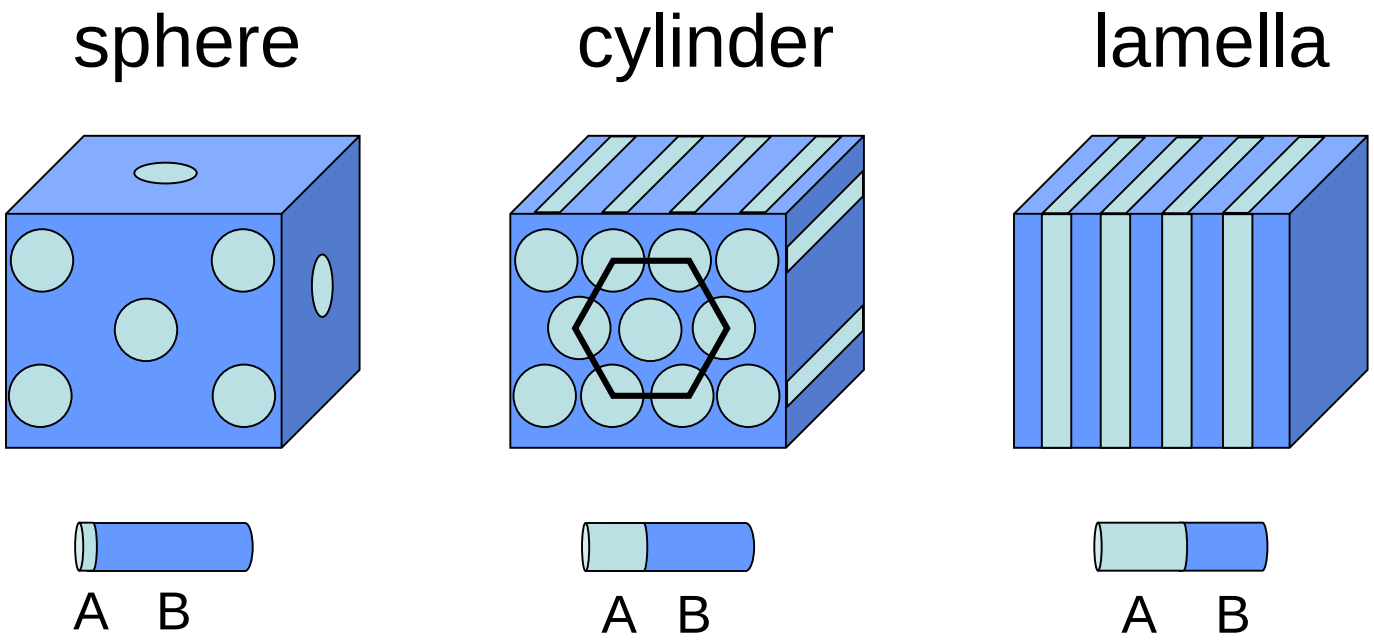




Applications of SAXS

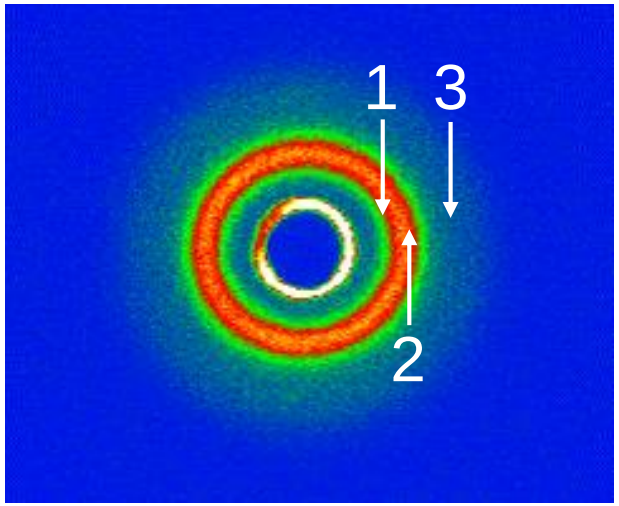
Phase identification

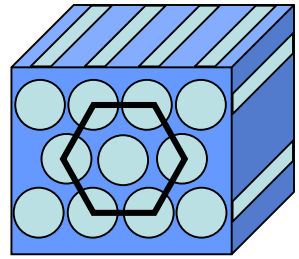
Block copolymer

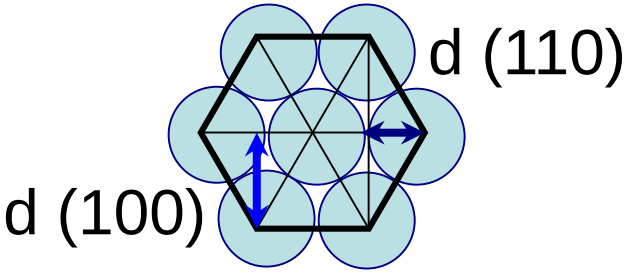


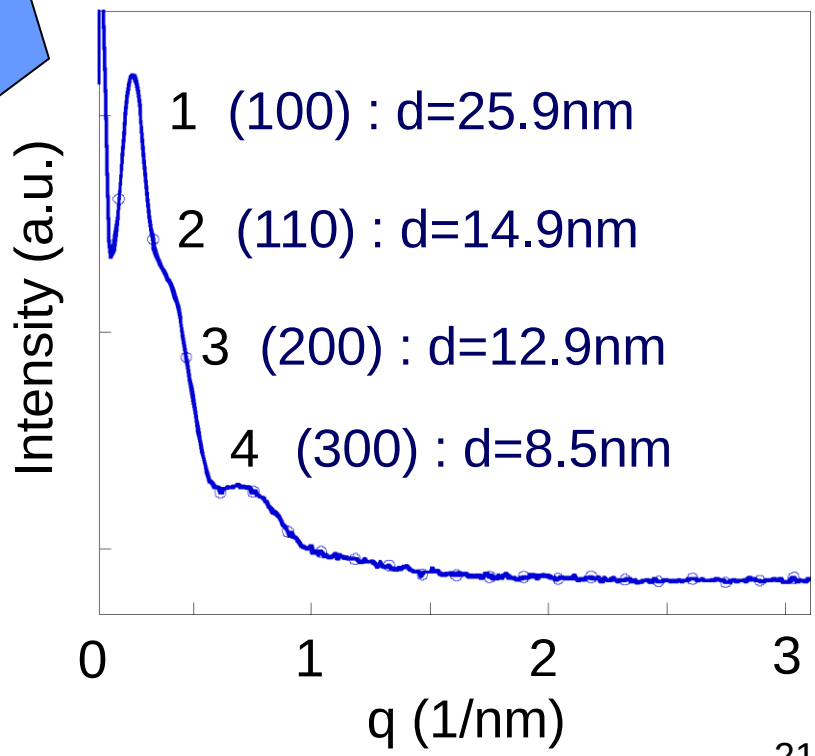
Block copolymer

- Cylinder structure



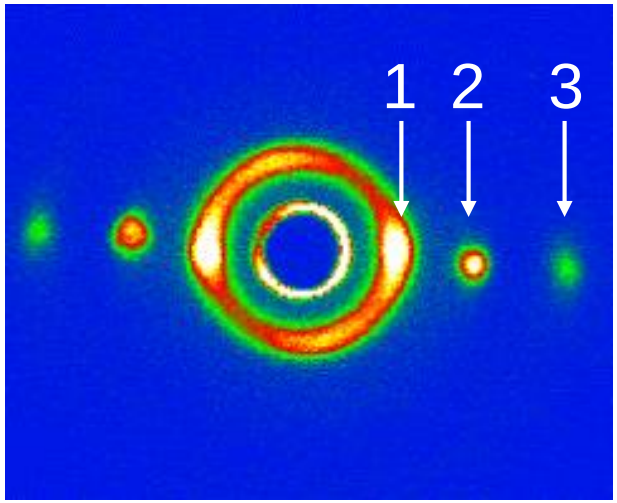


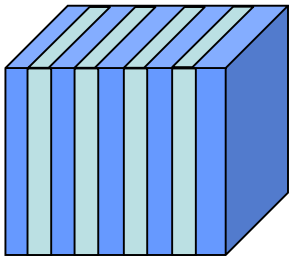


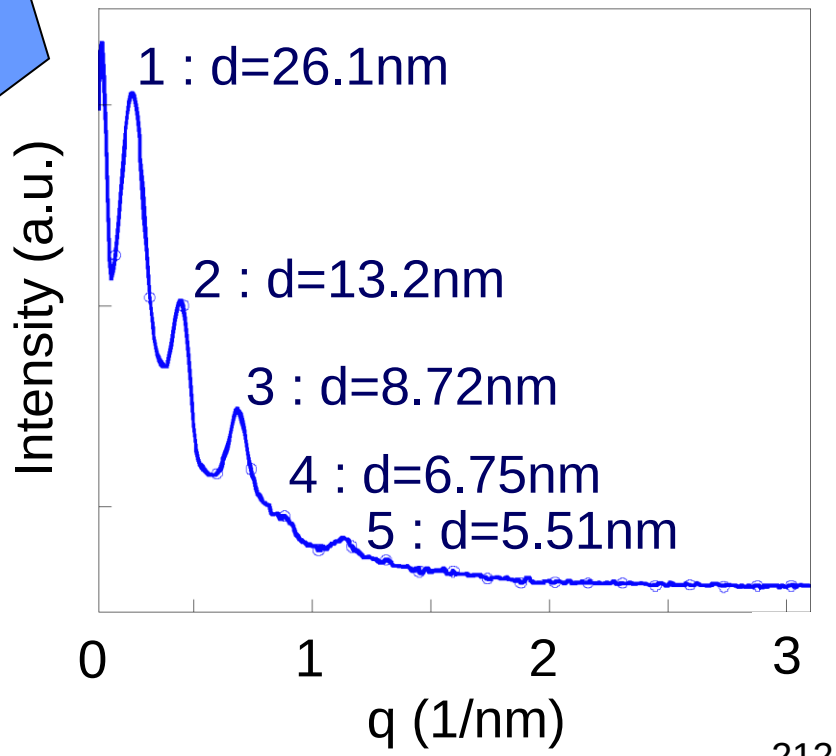


Block copolymer

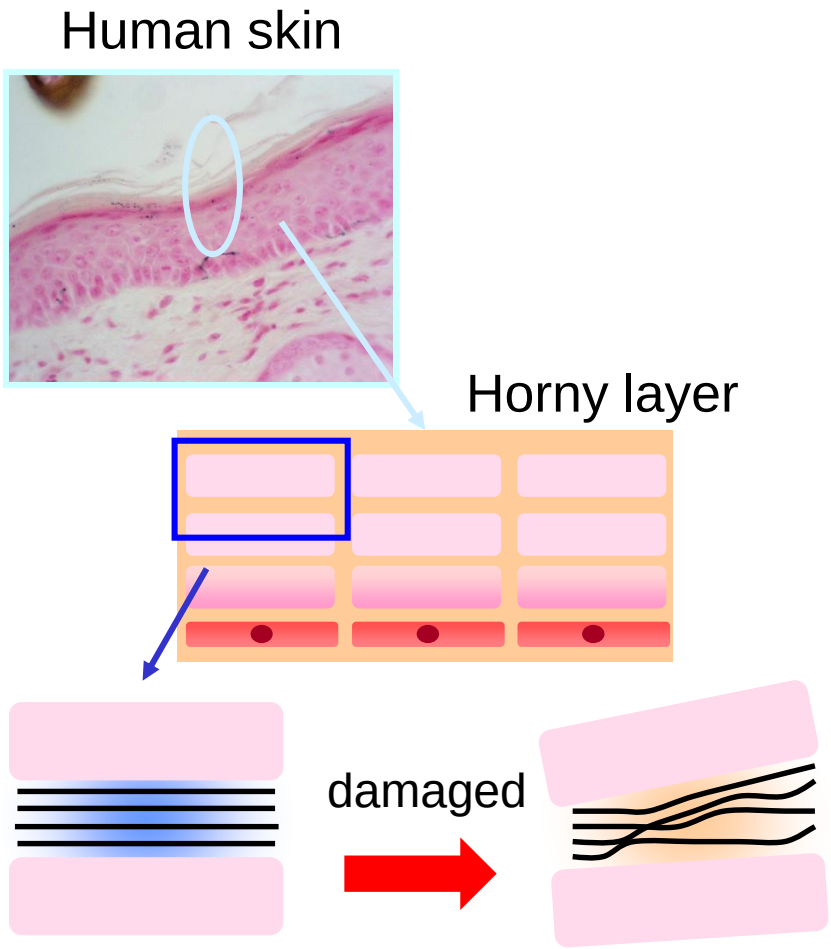
- Lamella structure

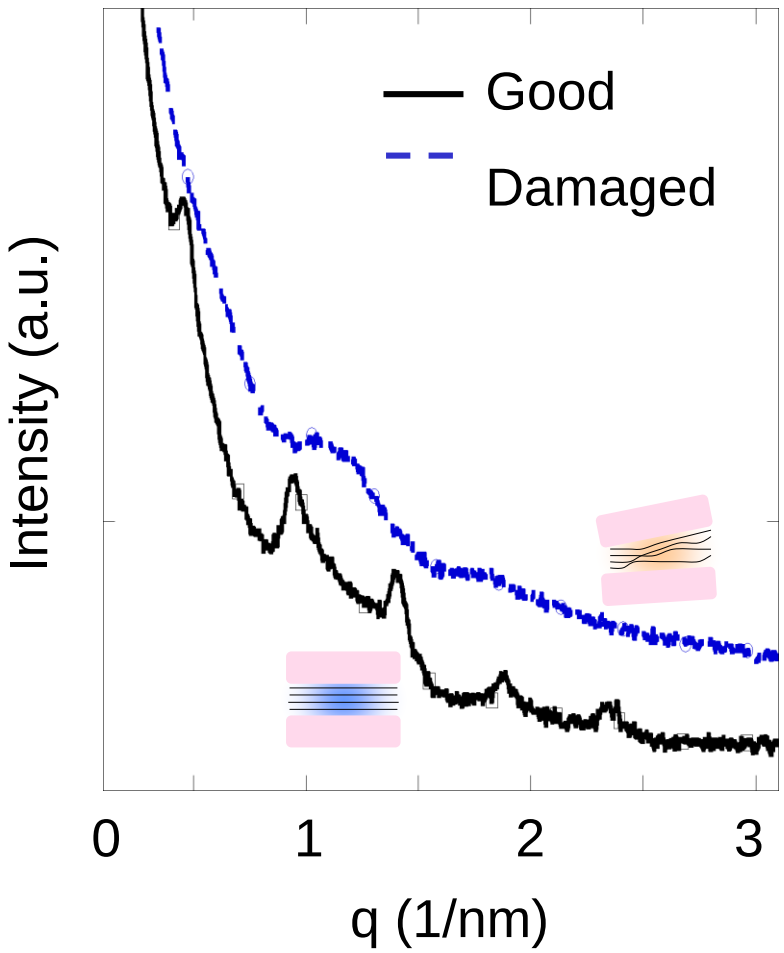






Changeable horny layer





Summary
Small angle X-ray scattering method

- Ultima IV, TTRAX III
  - Particle size and distribution
  - Phase identification

- Nano-viewer
  - Phase identification
  - Particle size and distribution

SAXS is for your Nano-technology development!