# Collect your own spectra …….

# (Conducting Nuclear Resonant Scattering Experiment at 3ID, APS)

**Jiyong Zhao**

*Advance Photon Source,*
*Argonne National Laboratory*

**Workshop on Nuclear Resonant Scattering**
**APS, November 11-13, 2016**

# To plan for an experiment of NRS

## 1. What can be measured?

NRIXS: thermal dynamics

SMS: hyperfine interactions

## 2. What's available at the beamline

how strong the beam, how small the beam size,

how low/high the temperature or field etc.

what else?

## 3. How and when to apply the beam time

# To plan for an experiment for NRS

1. What can be measured?

   NRIXS: thermal dynamics

   SMS: hyperfine interactions

2. What's available at the beamline

   how strong the beam, enriched or natural,

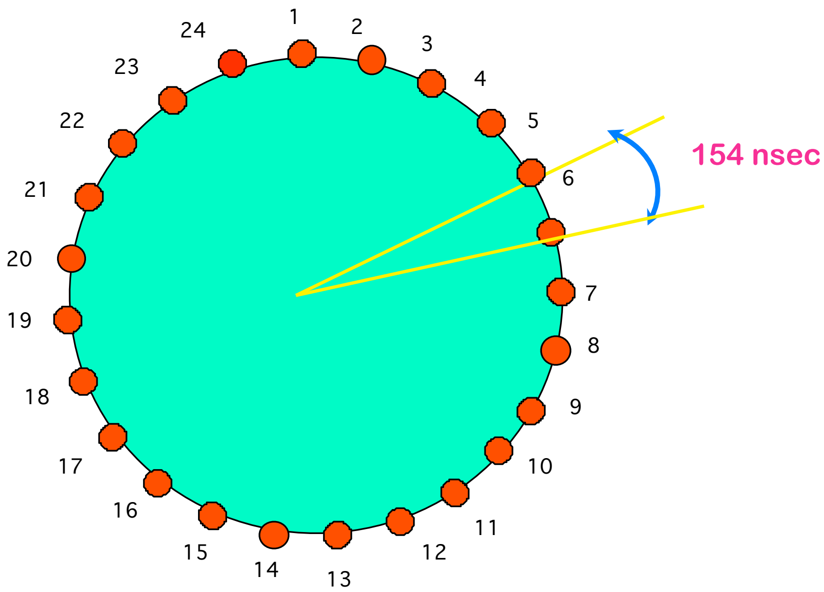
   how small the beam size,

   how low/high the temperature or field etc.

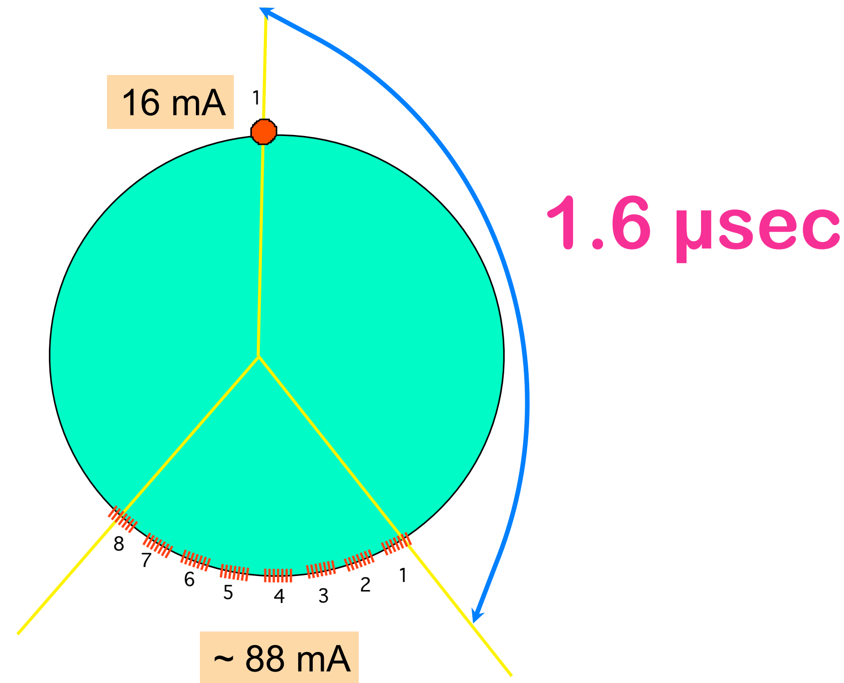
3. How and when to apply the beam time

# Nuclear resonance beamlines around the world, 2016

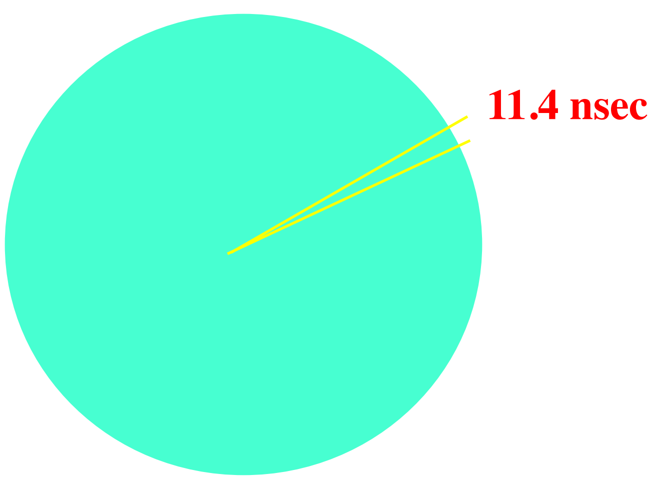
# APS storage ring filling pattern




24-bunch mode, 4.25mA/bunch, 65%



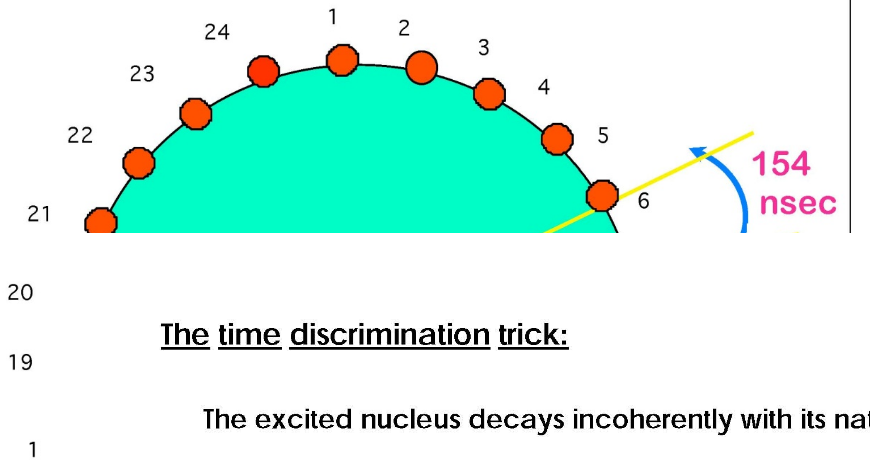

Hybrid mode 1+8X7-bunch, 15%




324-bunch mode, 0.3 mA/bunch, 20%

1296 buckets, 2.84 nsec separation

# Standard Time structure @ APS

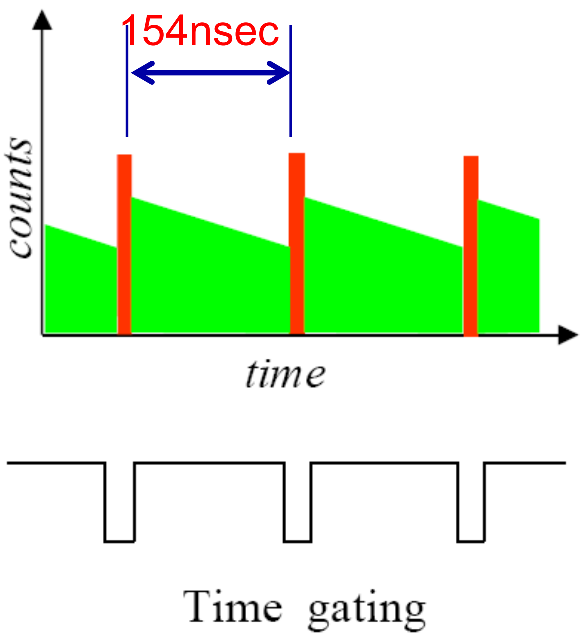


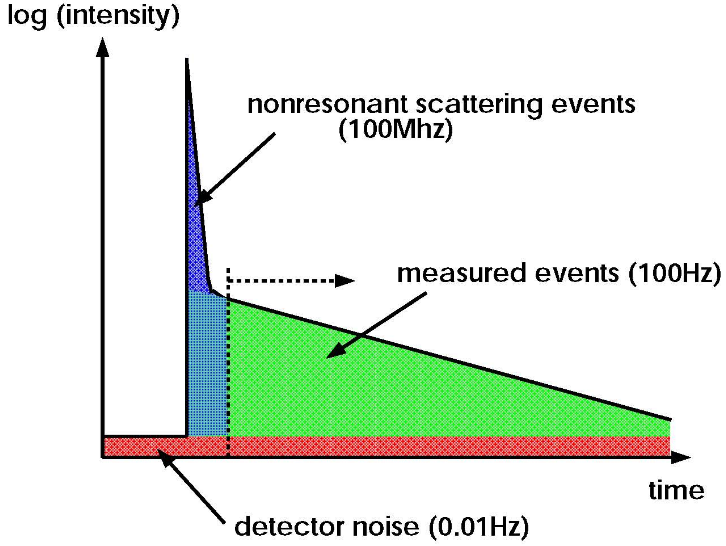

**The time discrimination trick:**

The excited nucleus decays incoherently with its natural life time $\tau$.

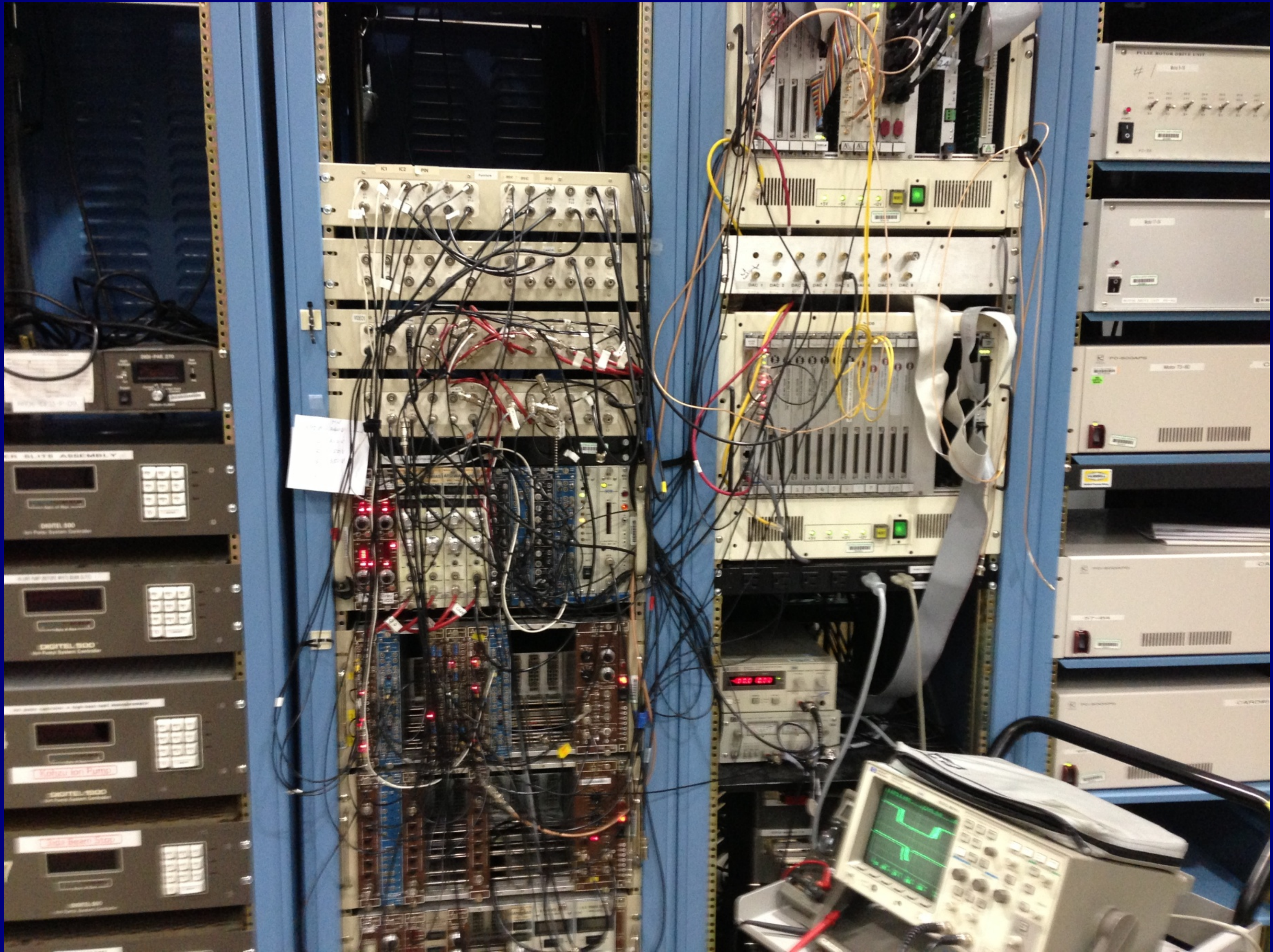
$\tau = \hbar / \Gamma$

141 ns for $^{57}Fe$

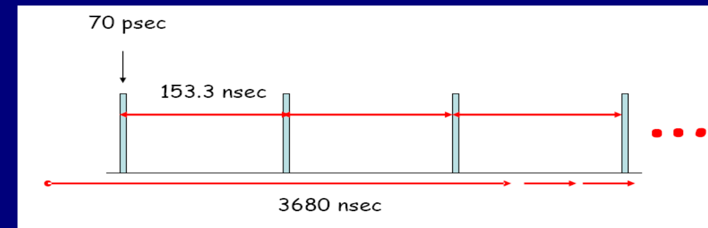


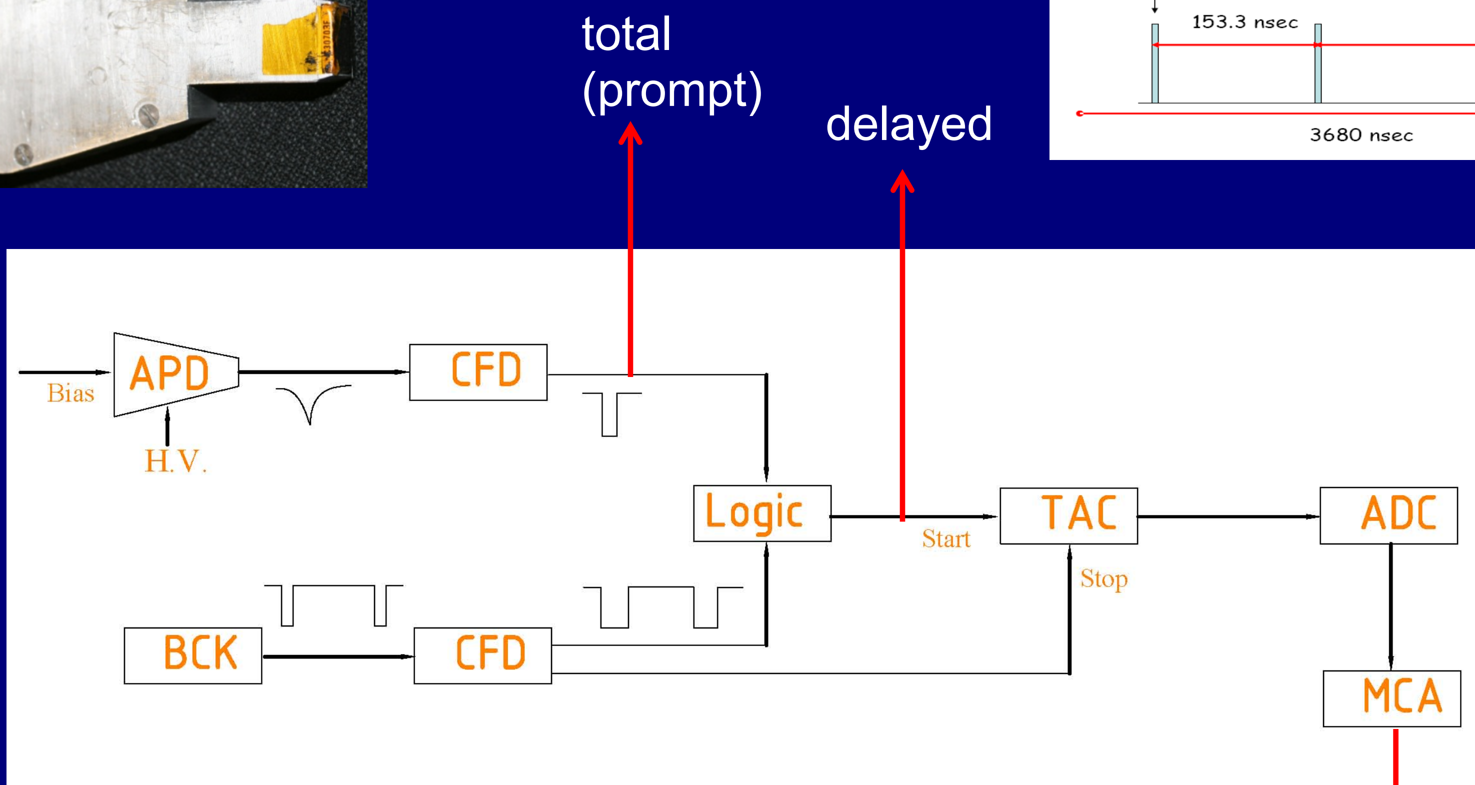



# Timing technique to select NRS delayed signal from a strong electronic scattering background

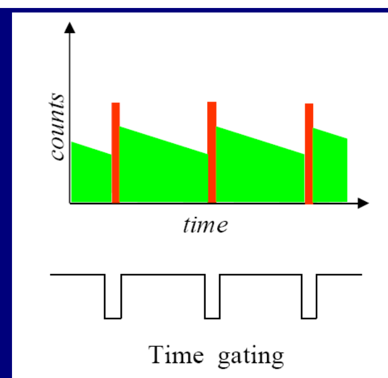


# Timing technique

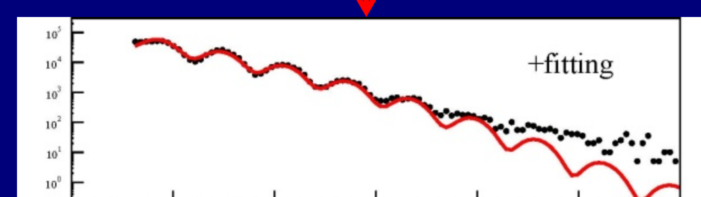


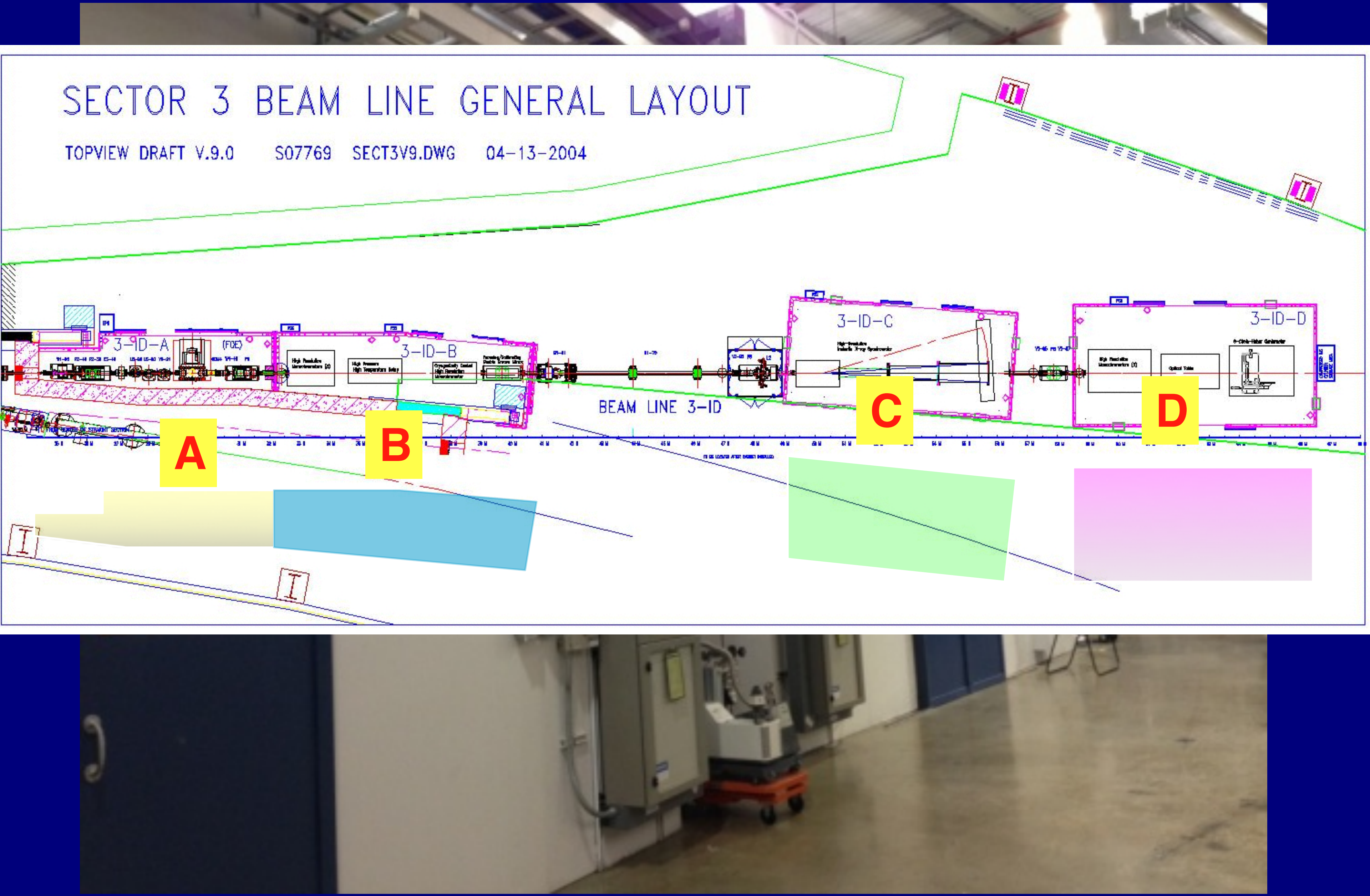

Avalanche photodiode (APD):
100μm Si diode with HV
Efficiency@14keV: 14%
Time resolution: 1ns
Dynamic range: $10^9$
Noise: $10^{-2}$




Time spectrum



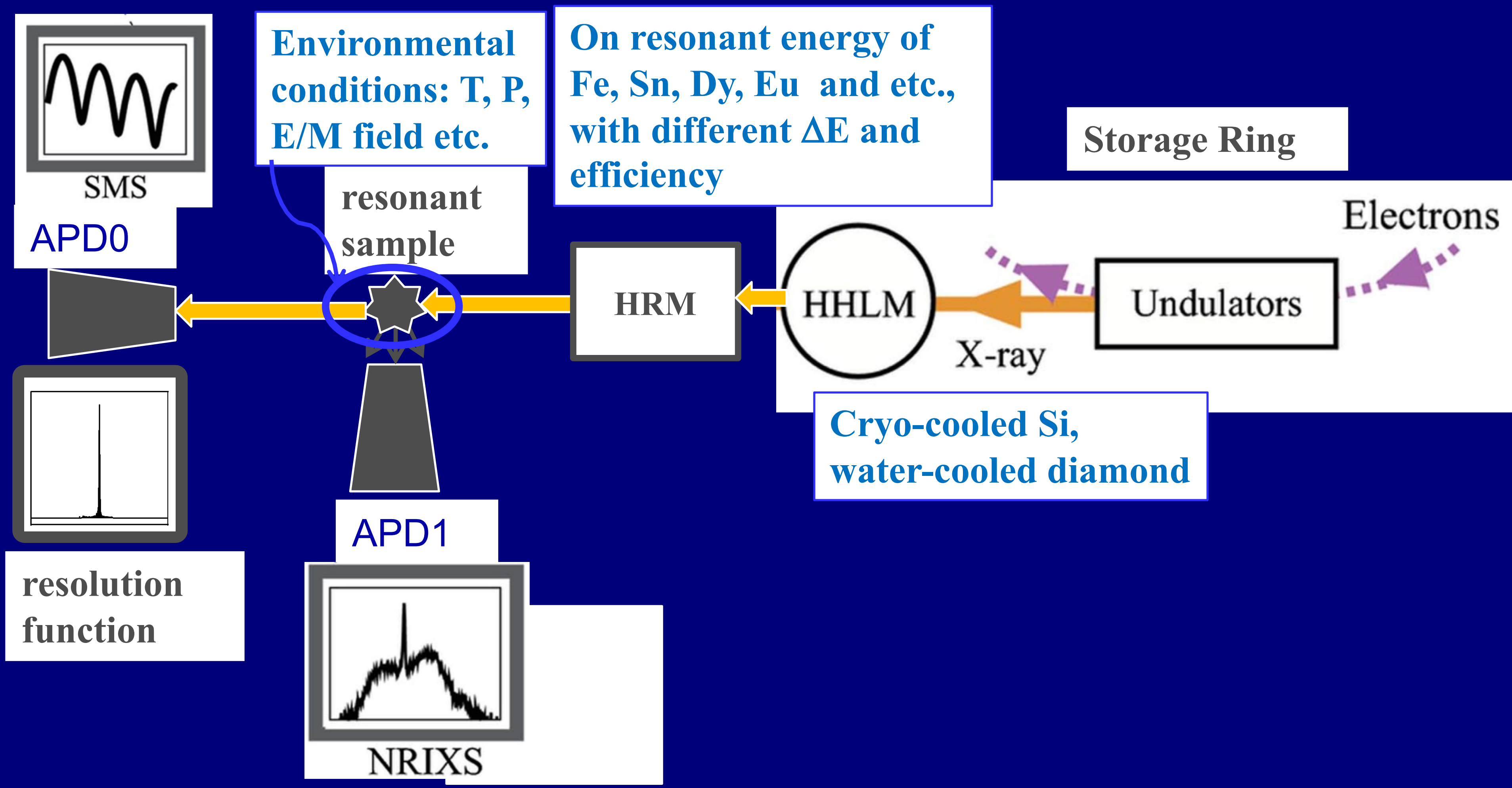
# 4 stations: **A-B-C-D at 3ID, APS**



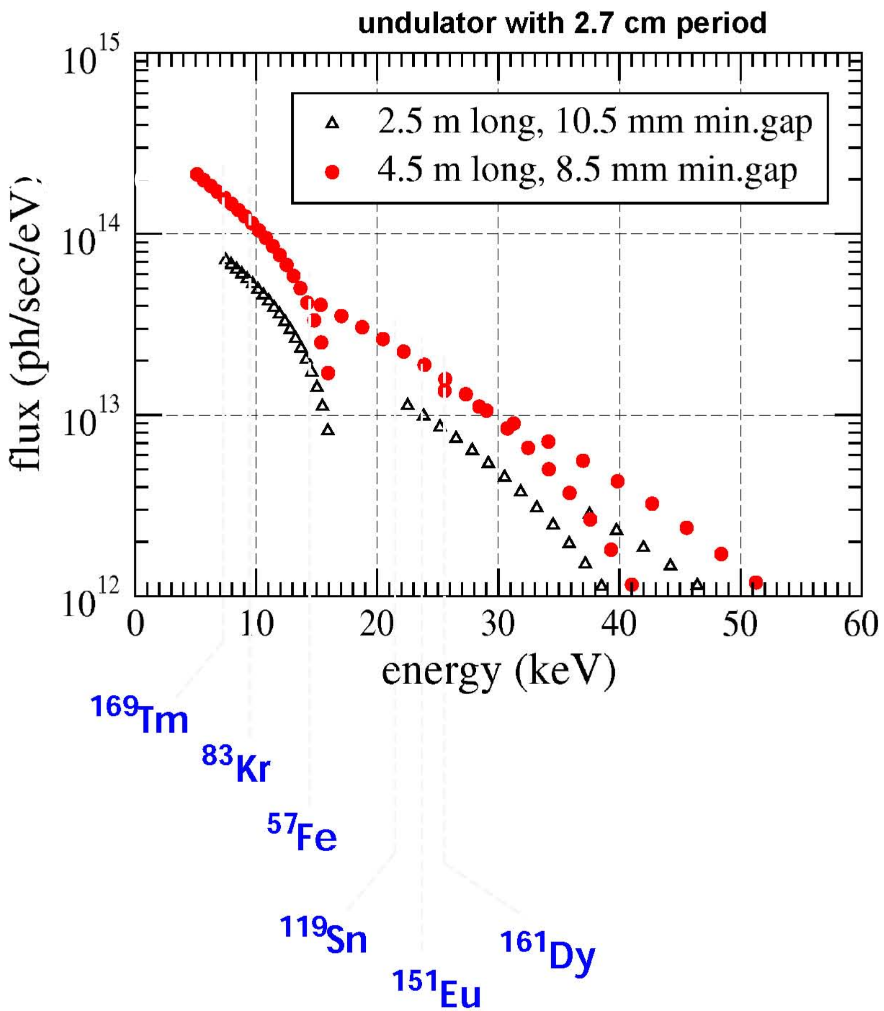
# X-ray Source and Instruments for NRS

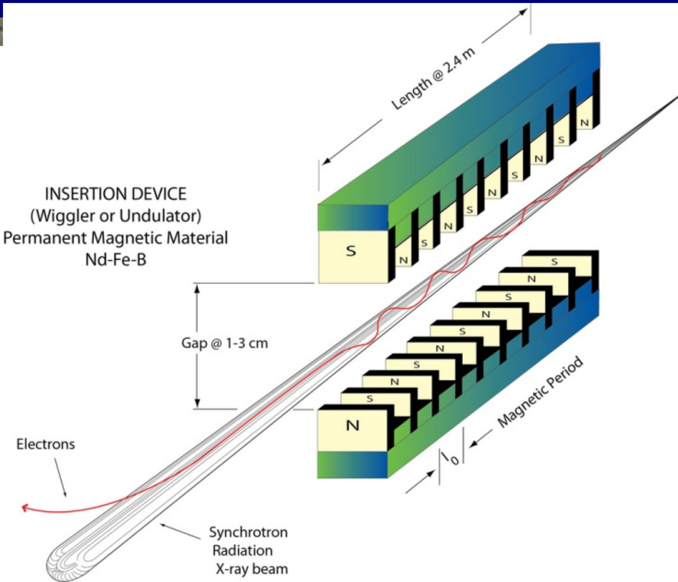
1. SR Source (undulator)
2. Monochromator (HHLM, HRM)
3. Focusing (KB, toroidal mirror, CRL)
4. Environments (HT, HP, LT,E/M-field)

# Setup for a synchrotron radiation nuclear resonant scattering experiment



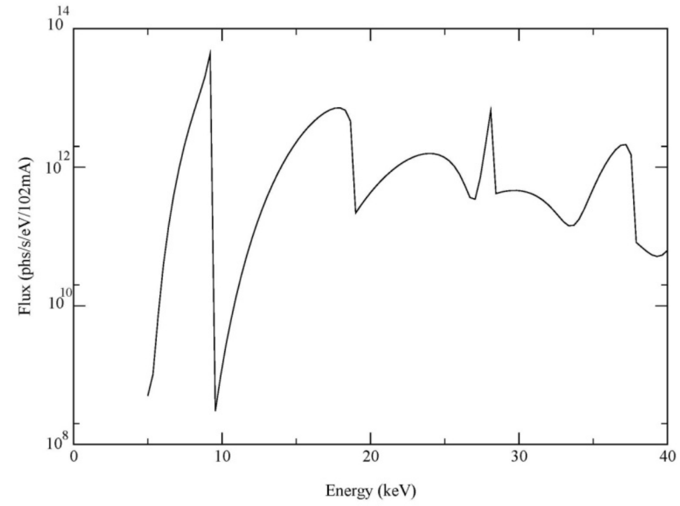
## Synchrotron radiation at the Advanced Photon Source:




At 3ID, there are two 2.4 m long undulators, with 2.7 cm period

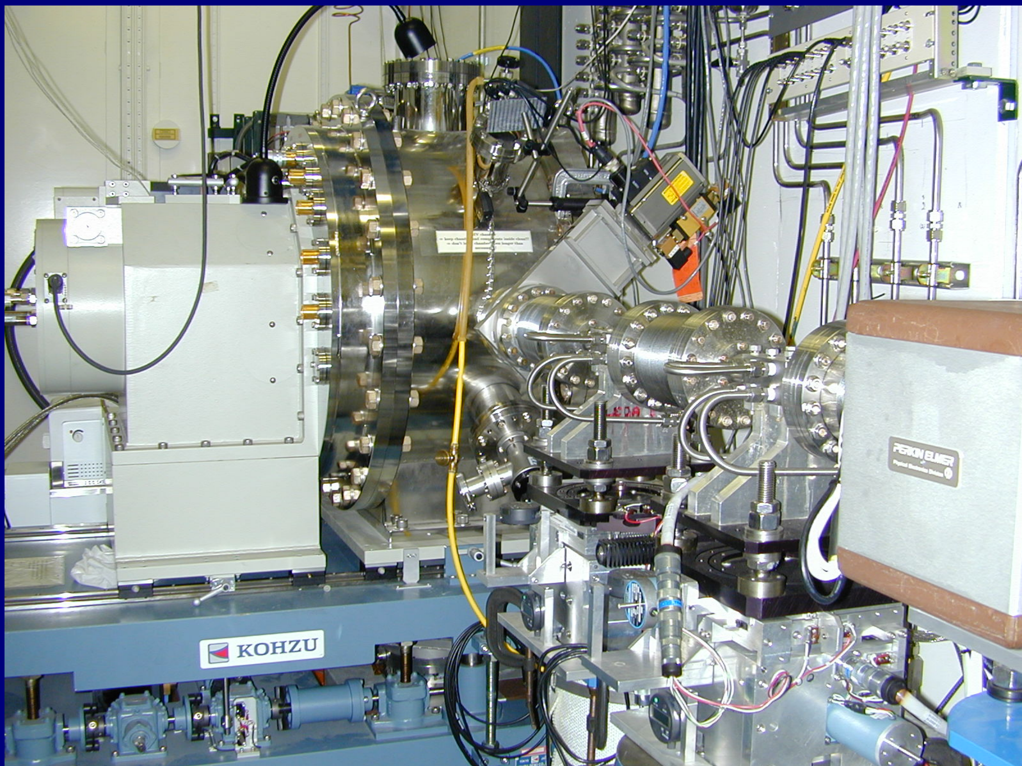
# 3ID undulator and HHLM

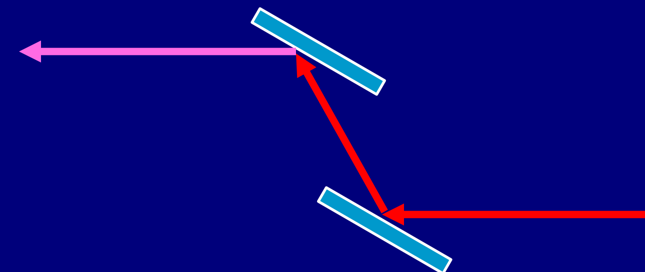


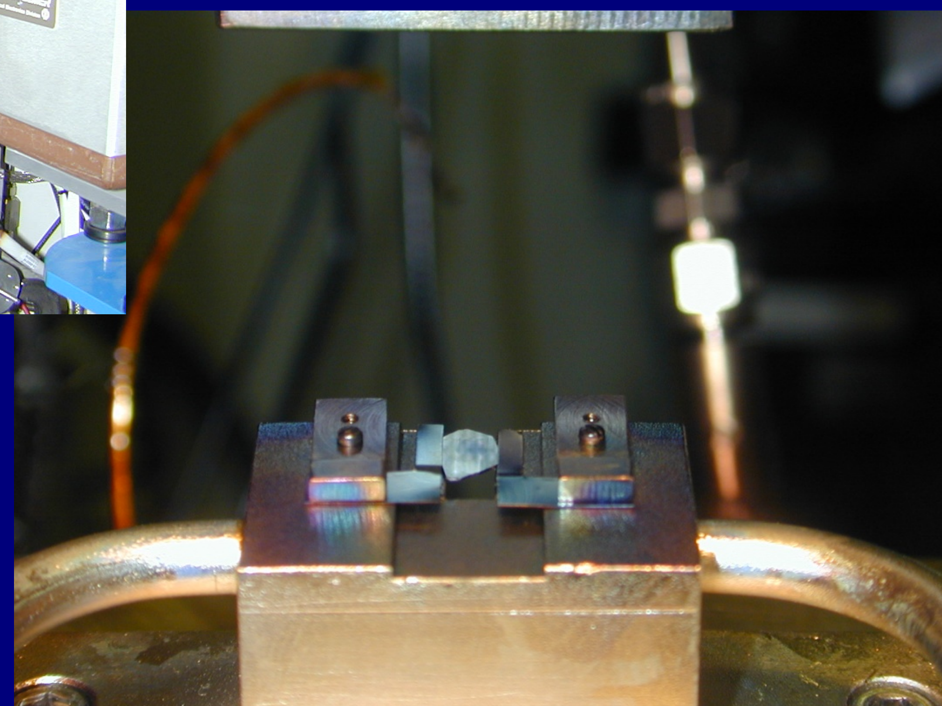

Parameters for running 3ID_undulators
Two undulators: 2.7cm, 88 periods
& Performance of Diamond (111) HHLM

| Energy (KeV) | Isotopes | Ky | Gap (mm) | Vertical Divergence of 3ID_U (μrad) | HHLM acceptance (μrad) | | ΔE after HHLM (eV) (measured) | | Flux after HHLM while WBS 0.4mmX3mm THz (phs/sec/100mA) | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Calculated | Measured | Caculated | Measured | Calculated | Measured |
| 9.403 | $^{83}$Kr | 1.3 | 12.7 | 15.6 | 19.3 | | 0.7 | | 70 | |
| 14.413 | $^{57}$Fe | 0.6 | 19.5 | 13.7 | 12.3 | 14.4 | 0.82 | 0.93 | 29 | 20 |
| 21.657 | $^{151}$Eu | 1.7 | 10.7 | 12.4 | 8.0 | 9.5 | 1.23 | 1.57 | 27 | 10 |
| 23.880 | $^{119}$Sn | 1.5 | 11.5 | 12.1 | 7.6 | 8.0 | 1.29 | 2.7 | 23 | |

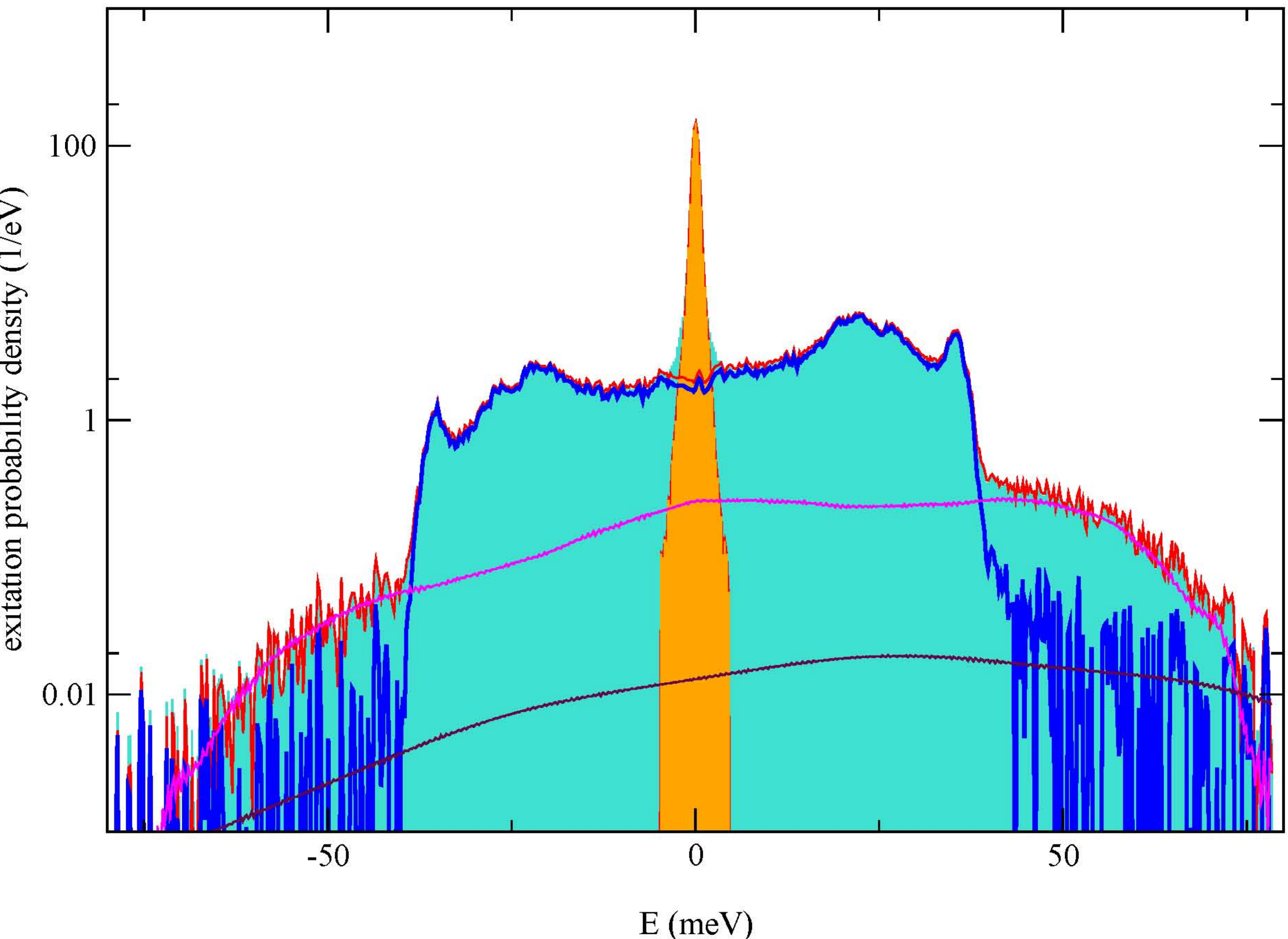
# ▪ 3ID-A:  High heat-load monochromator



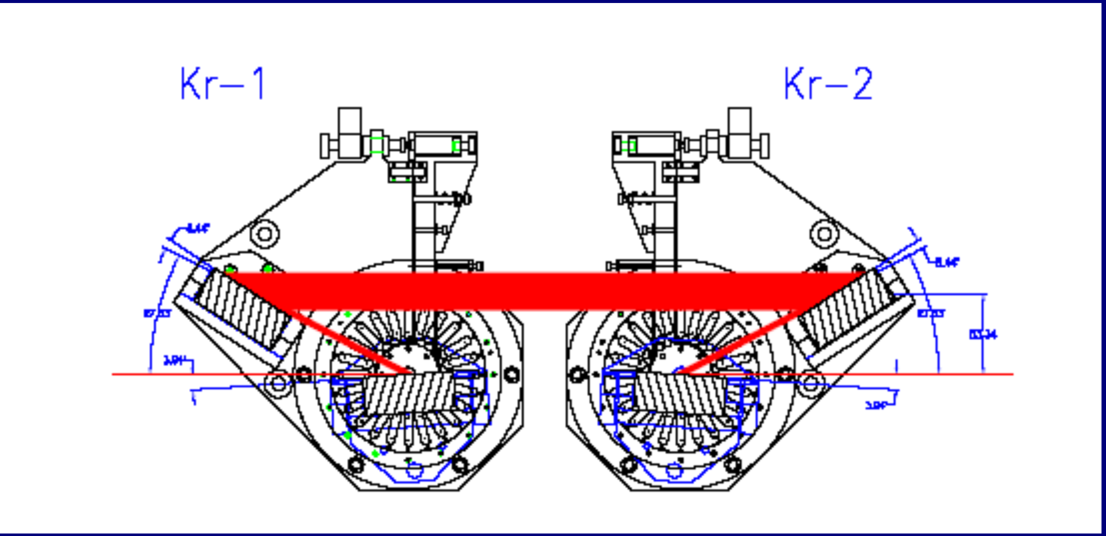


Kohzu high-heat-load monochromator consists of two water-cooled Diamonds

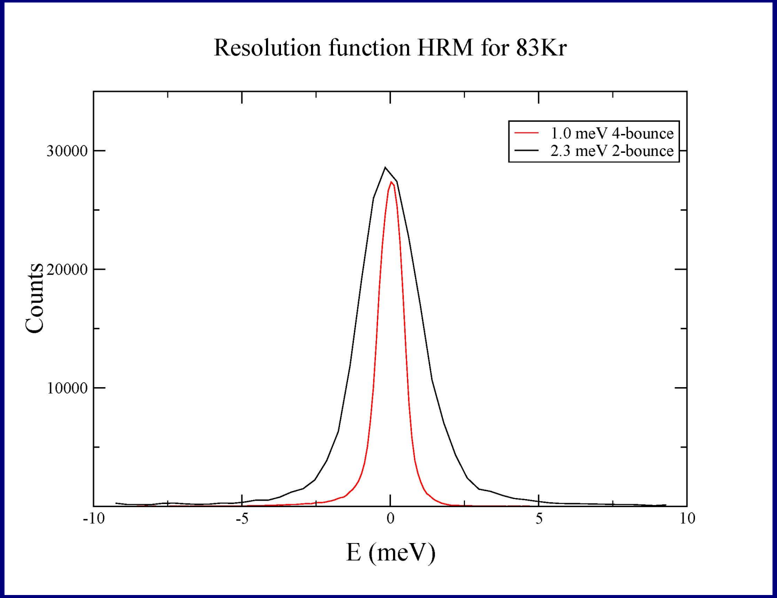


100
1
0.01
-50
0
50
E (meV)
exitation probability density (1/eV)

# High-energy resolution monochromator (HRM)



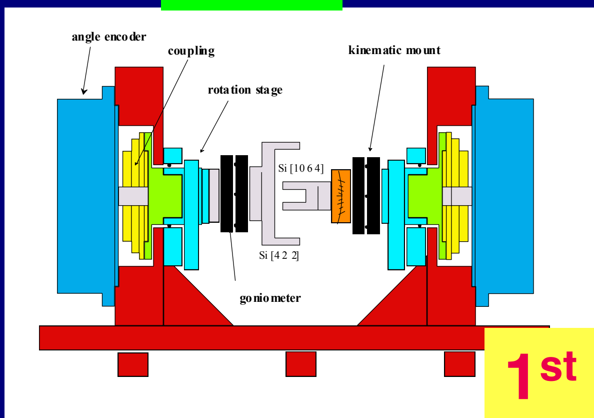

Designed by Toellner



# 3ID-B: High energy-resolution monochromator and focusing optics

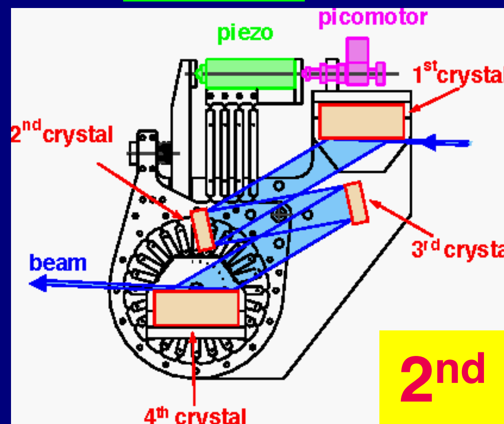
## Generations of high-resolution monochromators

1992



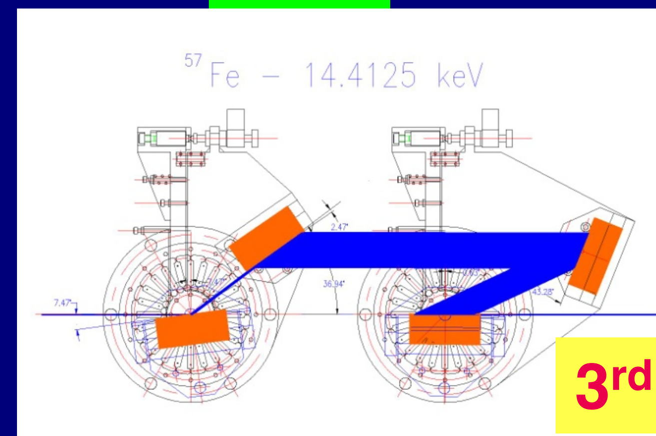

1st

1999



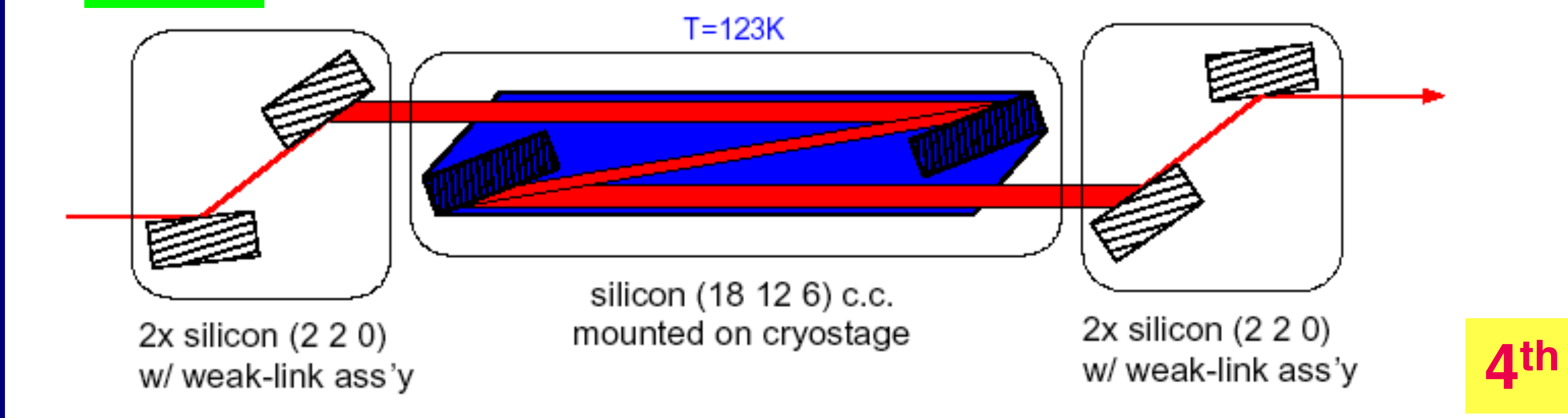

2nd

2002




3rd

2004




4th

# HRM at Sector 3

| | | |
|---|---|---|
| $^{57}Fe$, | 14.4 keV, | HRM: 1/0.8/2.3/5 meV |
| $^{151}Eu$, | 21.541 keV, | HRM: 0.8 meV |
| $^{119}Sn$, | 23.880 keV, | HRM: 0.85/0.14 meV |
| $^{161}Dy$, | 25.651 keV, | HRM: 0.5 meV |
| $^{83}Kr$, | 9.404 keV, | HRM: 2.3/1.0 meV |

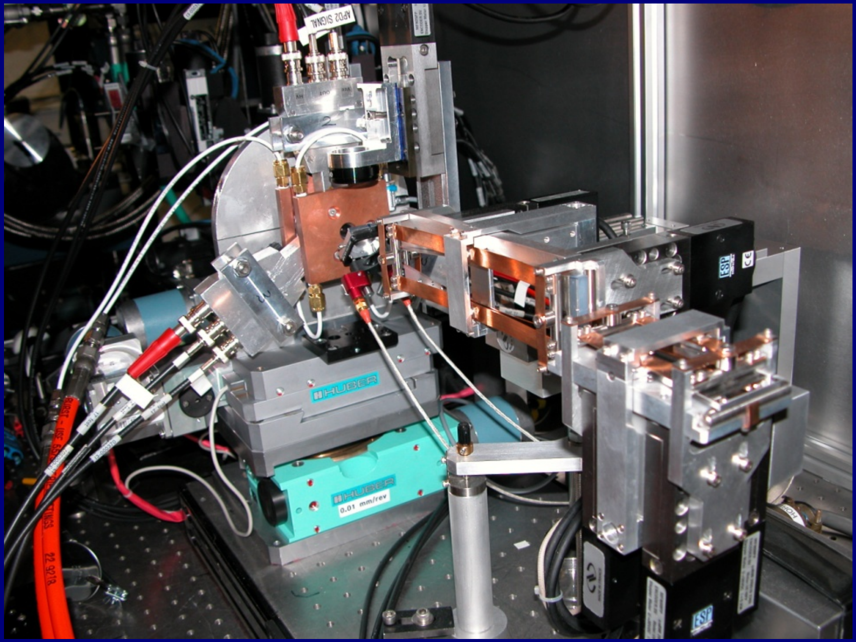
# Nuclear data for Mössbauer isotopes

| Isotope | Energy E(keV) | Life time $t_{1/2}$(ns) | Energy width $\Gamma$(neV) | Natural abundance(%) | Internal conv. coefficient $\alpha$ | Cross section $\sigma_0$($cm^2$ $10^{-18}$) | Recoil energy $E_R$ (meV) | Type |
|---|---|---|---|---|---|---|---|---|
| $^{181}$Ta | 6.22 | 6800 | 0.067 | 99.99 | 46 | 1.6 | 0.116 | E1 |
| $^{169}$Tm | 8.41 | 3.9 | 1.17 | 100 | 268 | 0.31 | 0.24 | M1 |
| $^{83}$Kr | 9.40 | 147 | 3.1 | 11.5 | 19.9 | 1.1 | 0.56 | M1 |
| $^{73}$Ge | 13.26 | 4 $10^3$ | 0.11 | 7.8 | 1000 | 0.0076 | 1.29 | E2 |
| $^{57}$Fe | 14.41 | 97.8 | 4.7 | 2.15 | 8.21 | 2.57 | 1.95 | M1 |
| $^{151}$Eu | 21.53 | 9.7 | 0.47 | 47.9 | 28.6 | 0.23 | 1.66 | M1 |
| $^{149}$Sm | 22.49 | 7.1 | 0.641 | 13.9 | 50 | 0.0711 | 1.82 | M1 |
| $^{119}$Sn | 23.88 | 17.7 | 0.257 | 8.6 | 5.12 | 1.40 | 2.58 | M1 |
| $^{161}$Dy | 25.65 | 28.1 | 0.162 | 19.0 | 2.9 | 0.95 | 2.2 | E1 |
| $^{40}$K | 29.56 | 4.26 | 1.07 | 0.012 | 6.6 | 1.6 | 11.6 | M1 |

# Unique capability at 3ID for NRS

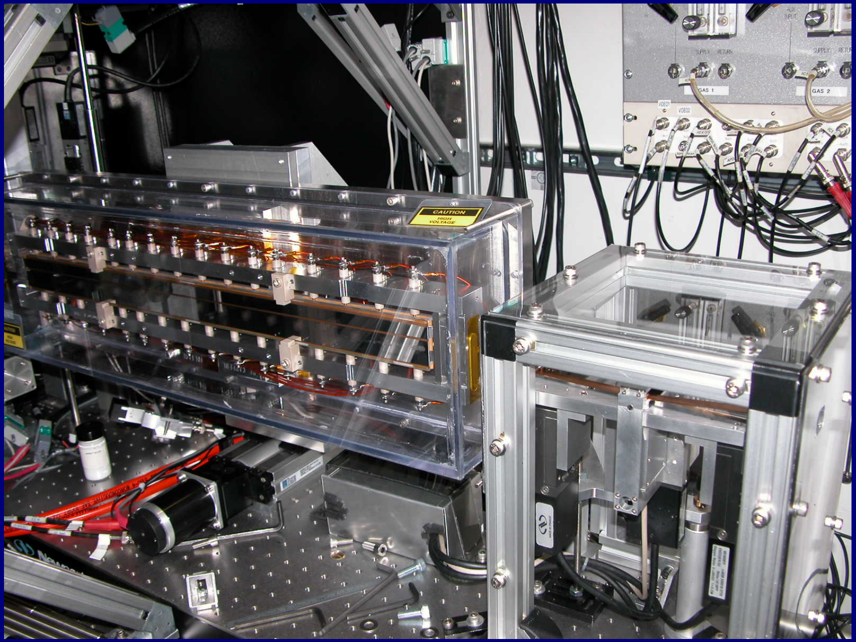
## Beam focusing at 3ID-B

K-B focusing mirror



Beam size: 6 μm x 7 μm

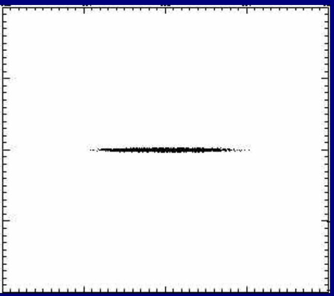
Acceptance: 0.4mm x 0.6 mm

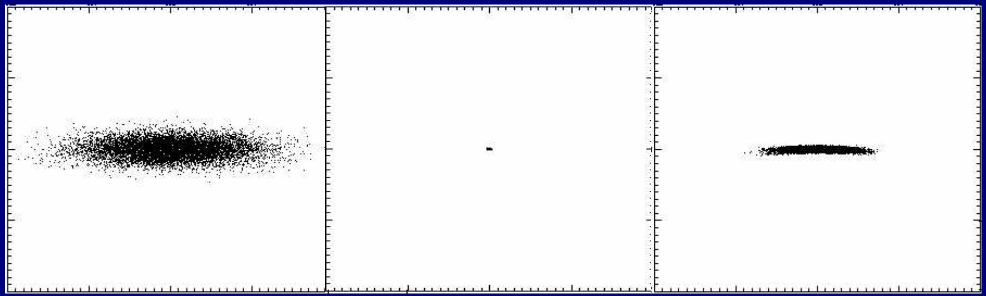


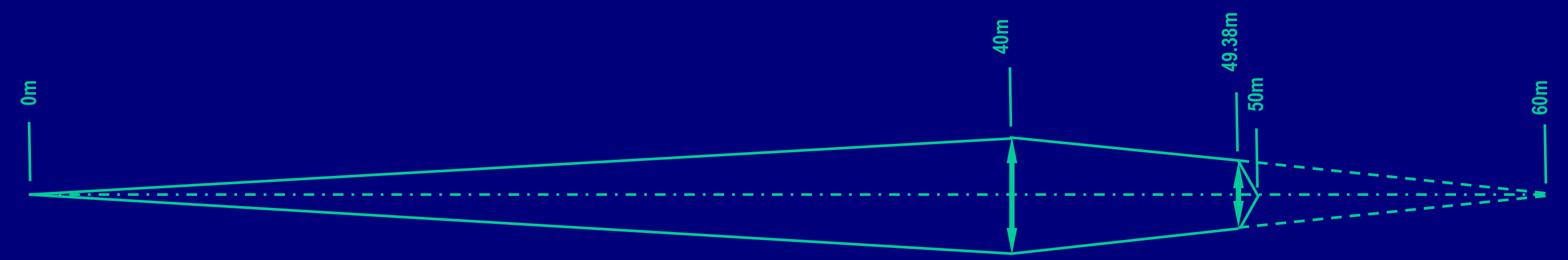
Beam size: 18 μm x 12 μm

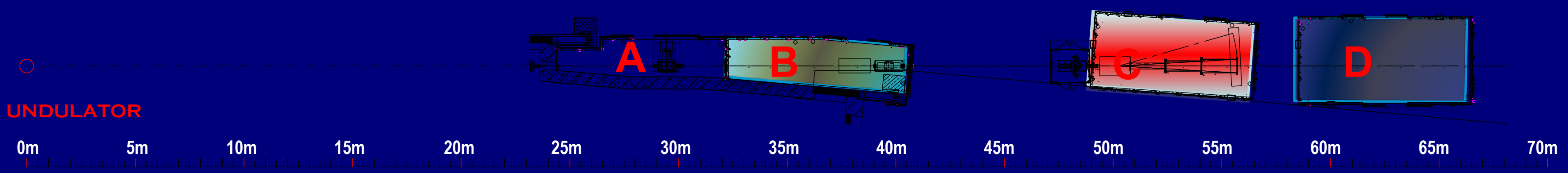
Acceptance: 0.4mm x 1.8 mm

# Toroidal + K-B tandem focusing at 3-ID-APS








Shadow simulations, A. Alatas

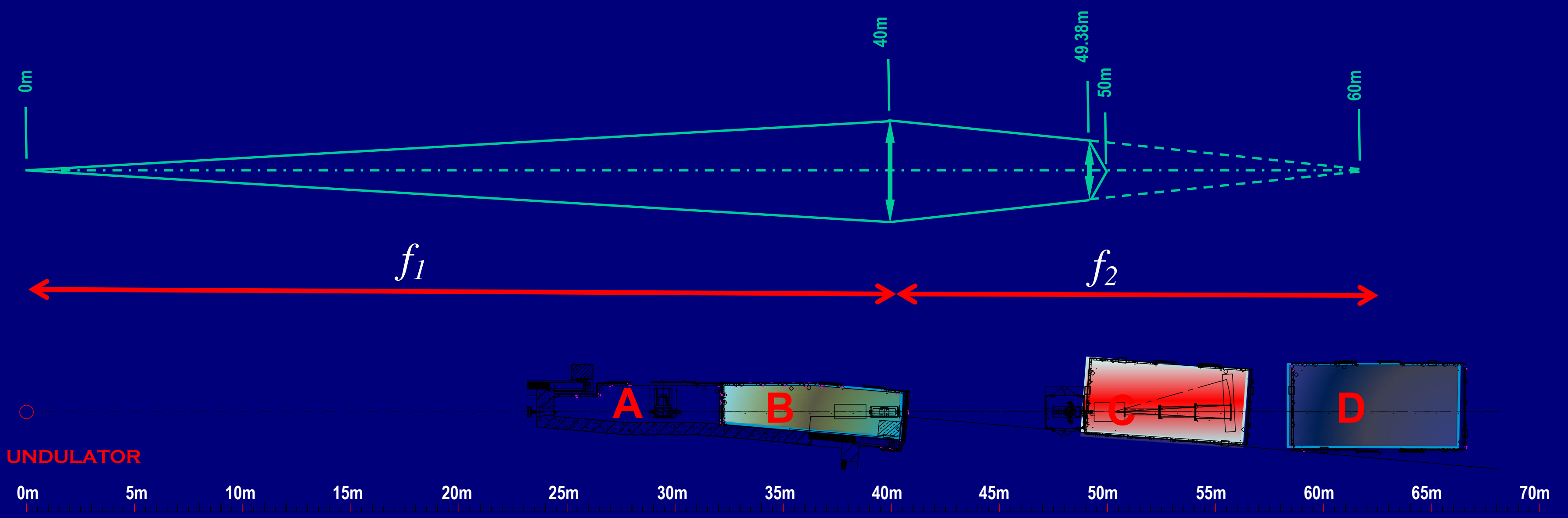
# Toroidal + K-B tandem focusing at 3-ID-C (IXS), and 3ID-D (NRS)

$$R_1 = \frac{2 f_1 f_2}{f_1 + f_2} \sin\theta$$

$$R_2 = \frac{R_1}{\sin^2\theta}$$

Sagittal focusing, horizontal, 46 mm

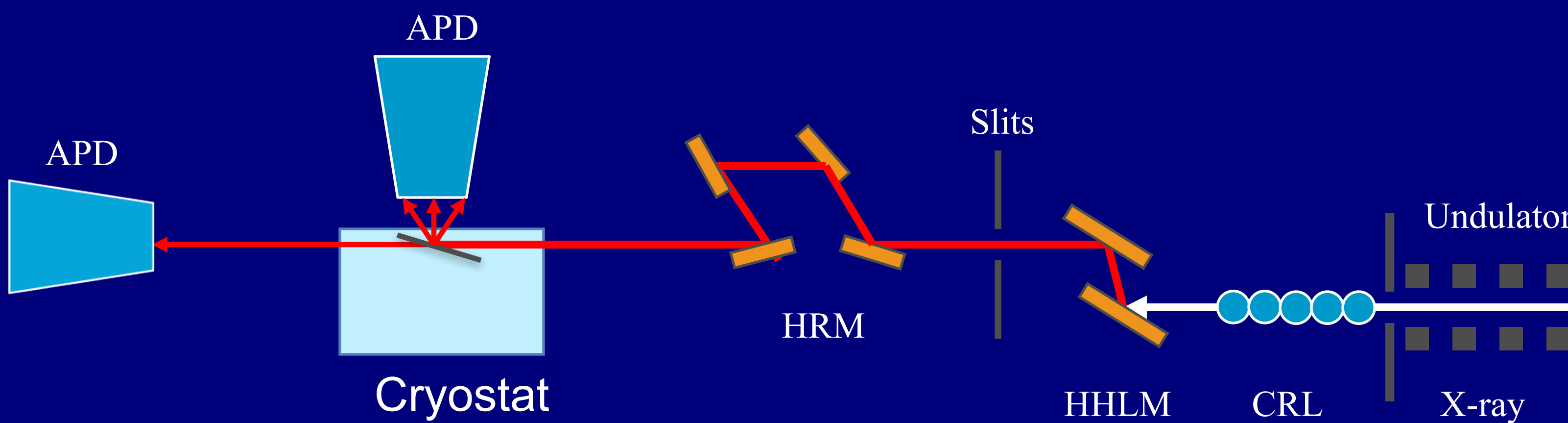
Vertical focusing, 11-33 km, $\theta = 1.6$ mrad

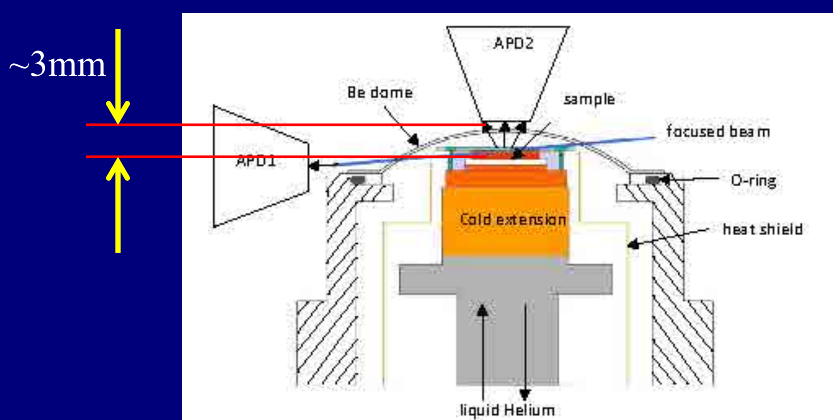


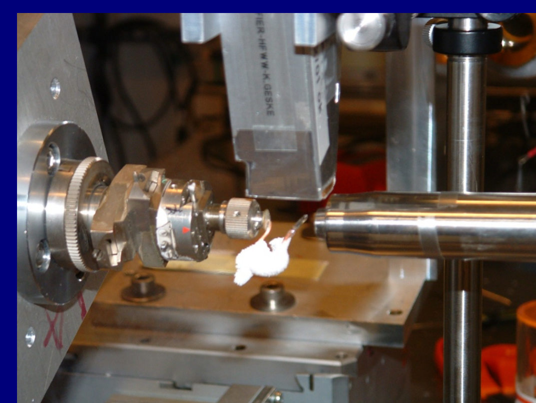
# Sample environment for NRS at 3ID

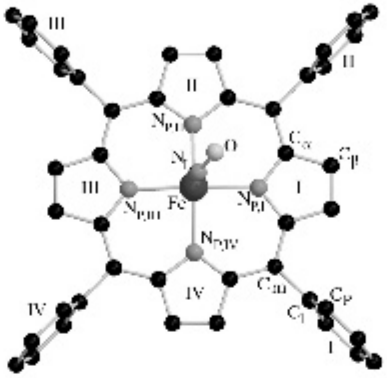
- Low temperature, flow cryostat
- High pressure and high temperature
- High pressure and low temperature

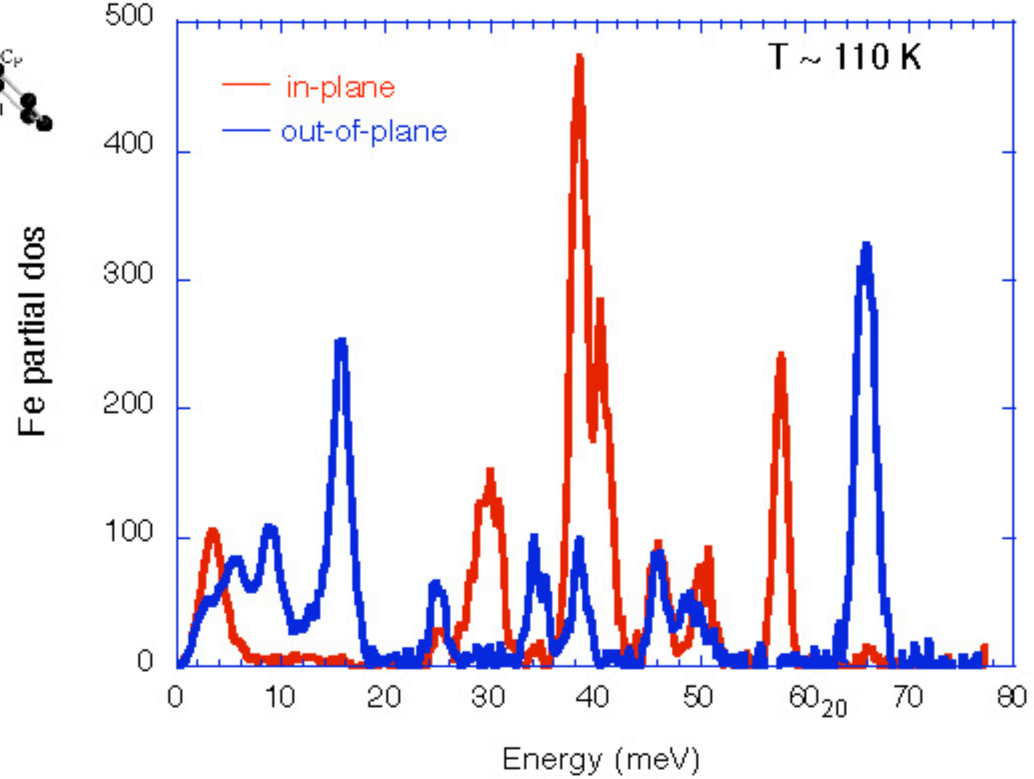
# Experimental Setup for Nuclear Resonant Inelastic X-ray Scattering under low temperature





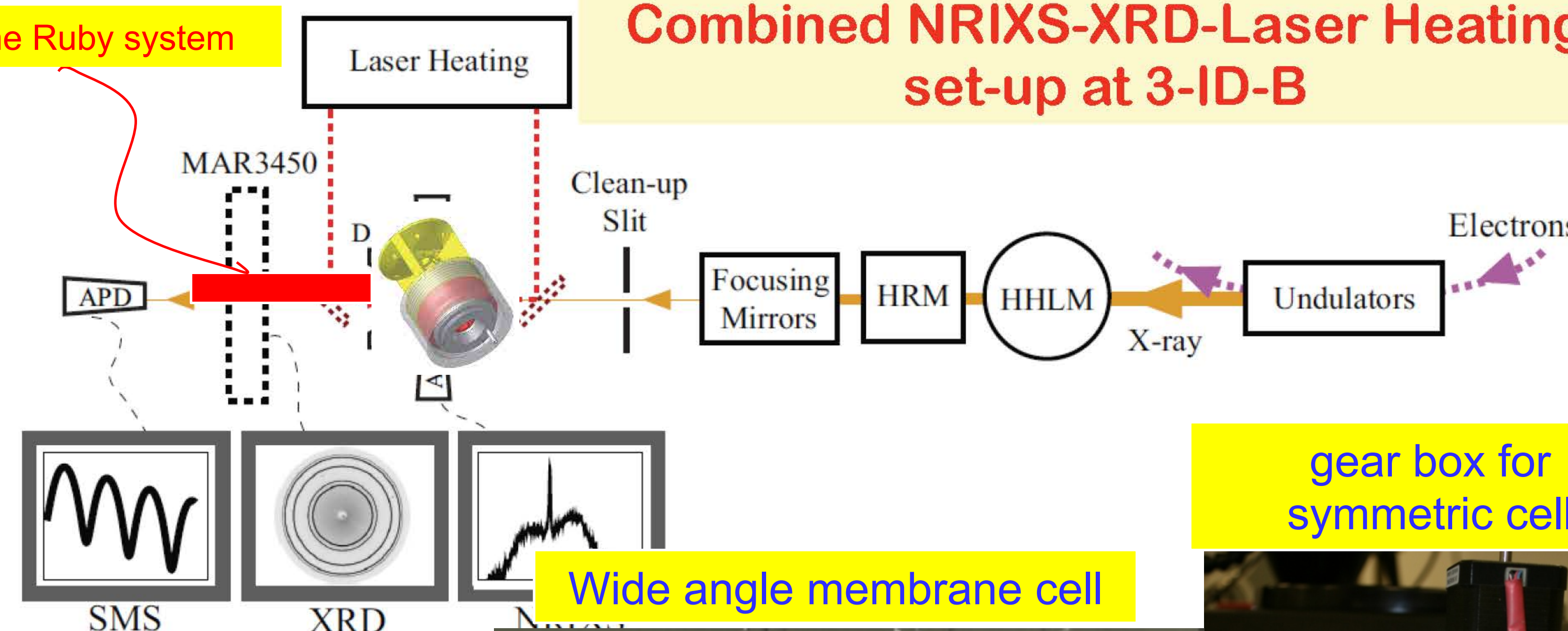


T ~ 110 K
in-plane
out-of-plane
Fe partial dos
Energy (meV)
0
100
200
300
400
500
0
10
20
30
40
50
60
70
80

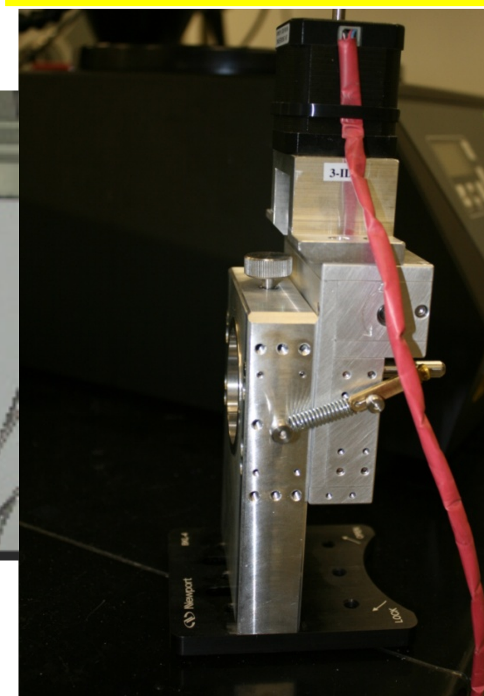
# Unique capability at 3ID: HP/HT for NRS

## Combined NRIXS-XRD-Laser Heating set-up at 3-ID-B

On-line Ruby system




gear box for symmetric cell

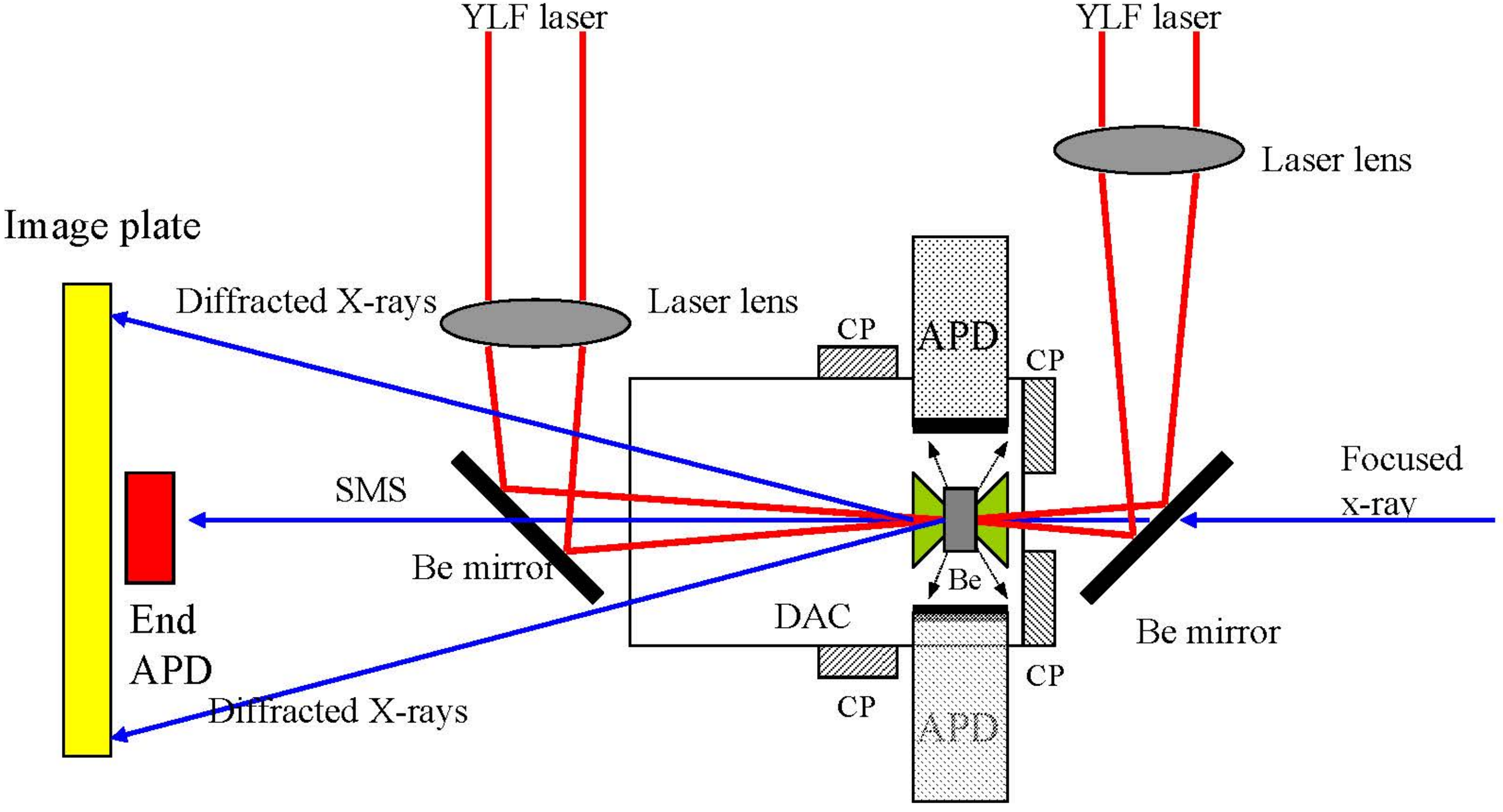


Wide angle membrane cell

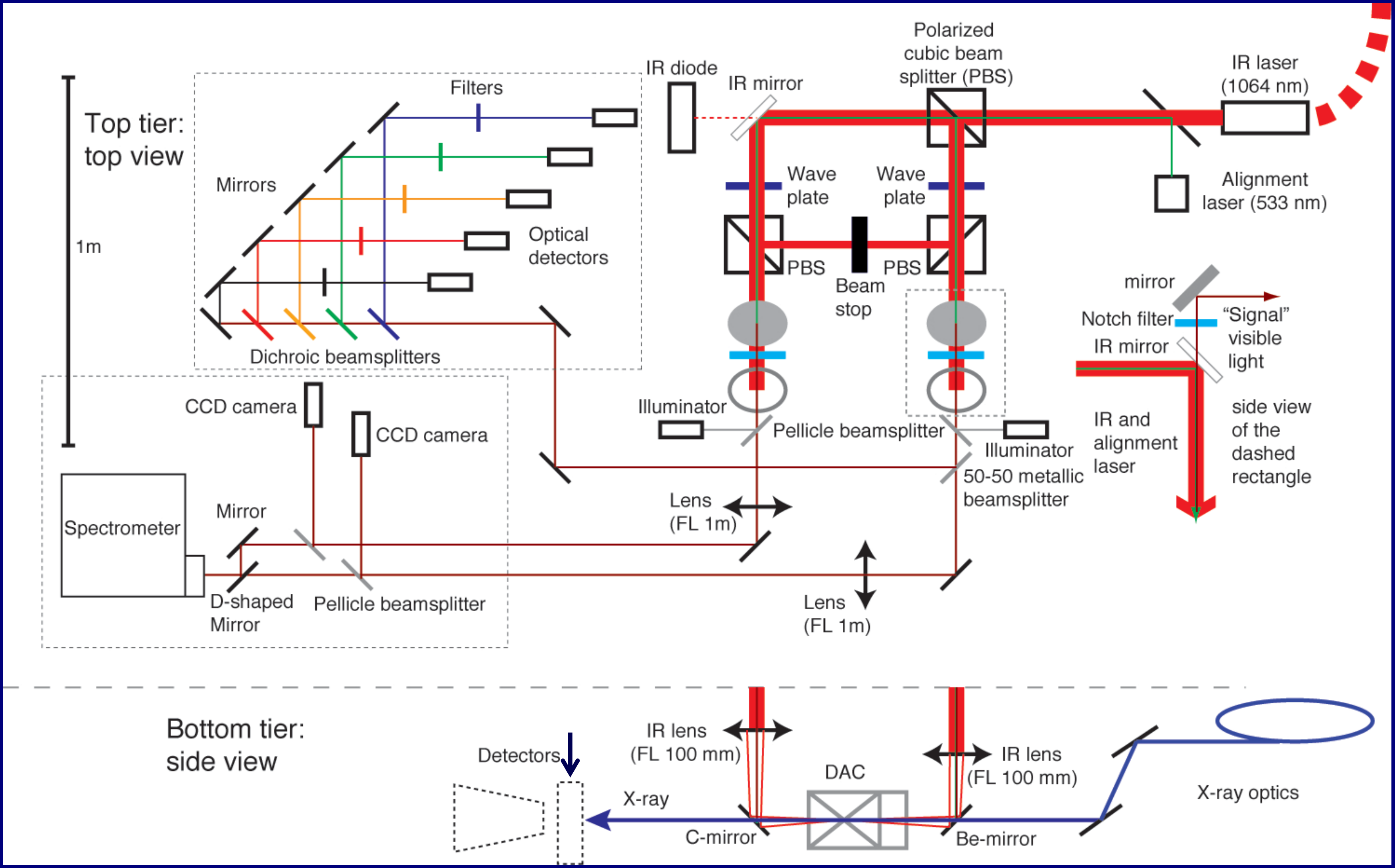
Glove box for DAC loading, $H_2O$: 1 ppm, $O_2$: 20 ppm

# NRIXS-SMS and diffraction

***In situ*** **X-ray diffraction, NRIXS, and SMS studies in a LHDAC provide structural (density), magnetic, elastic, vibrational, and thermodynamic information of the sample. This is also a powerful tool to detect melting.**



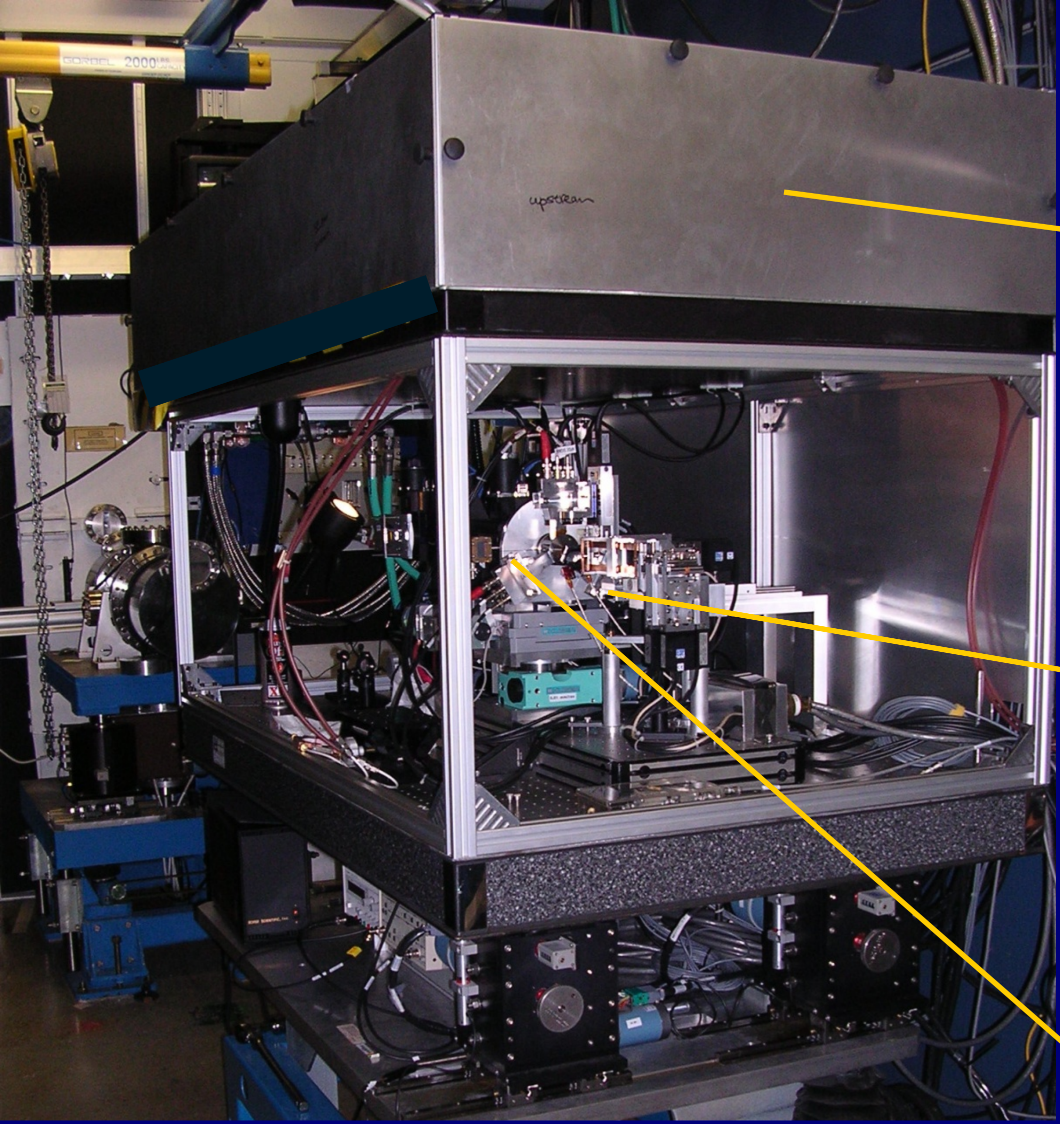
# HP-HT Nuclear resonant scattering

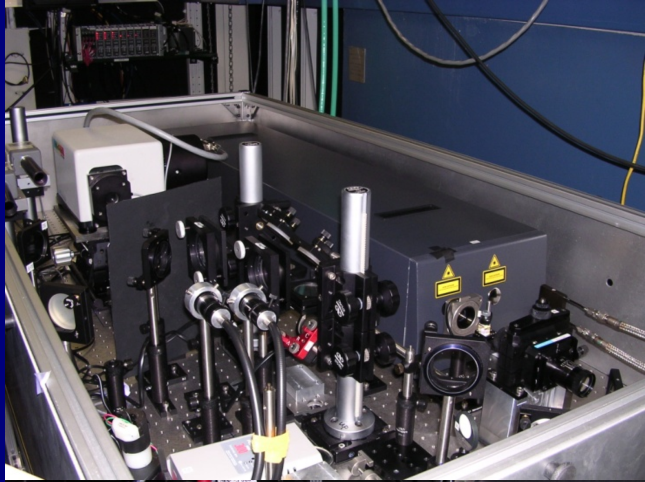


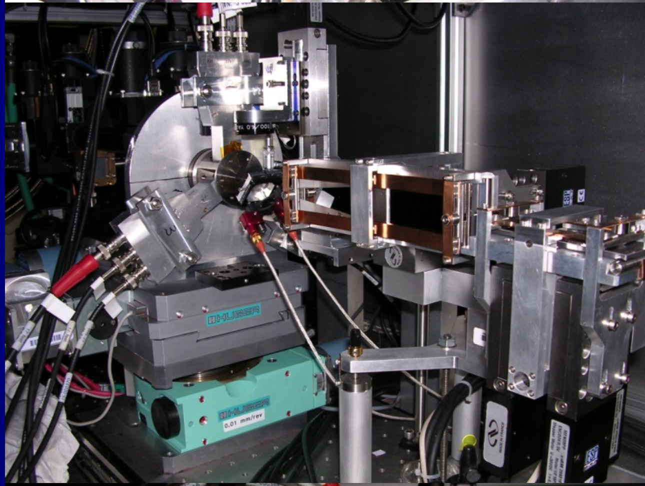

Fast Temperature Readout system(FasTeR)

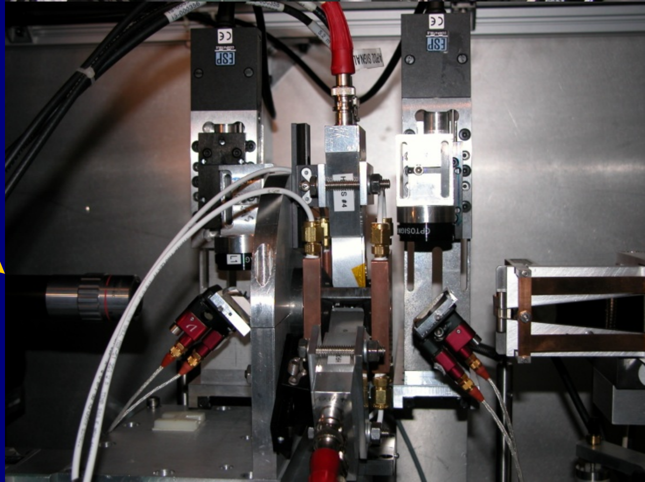
NRS+ Laser heating+ Spectroradiometer+ FasTeR spectrometer

High pressure melting

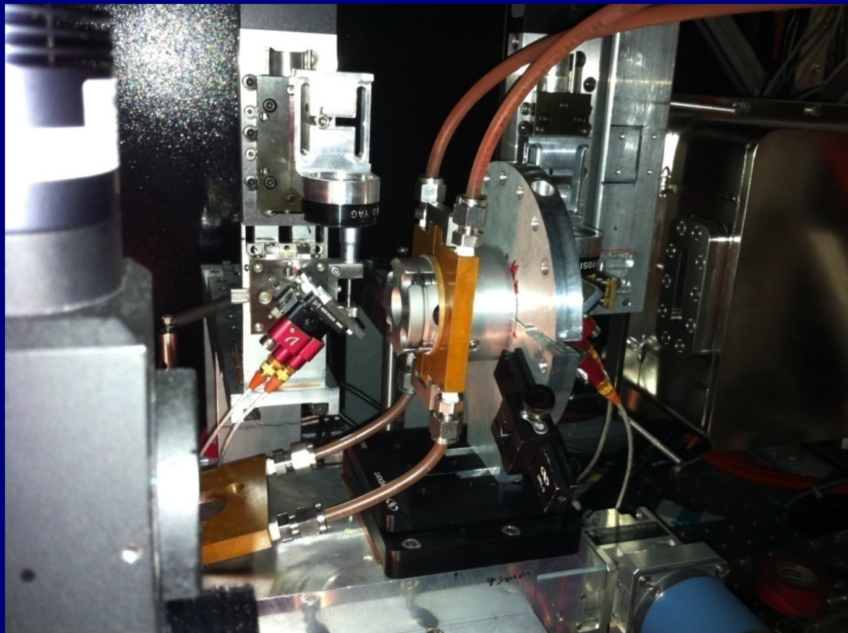

GORBEL 2000
upstream





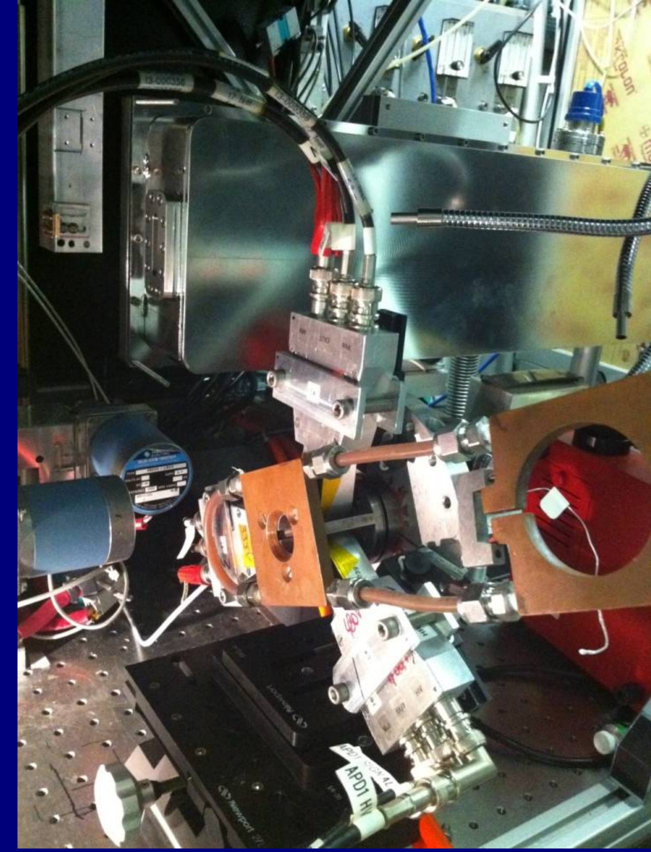


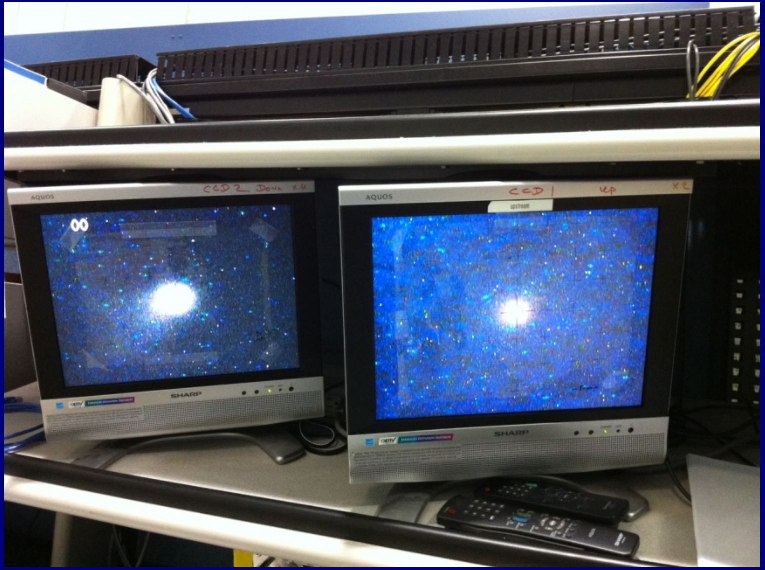
# NRS at HPHT setup



NRIXS ->

<- SMS



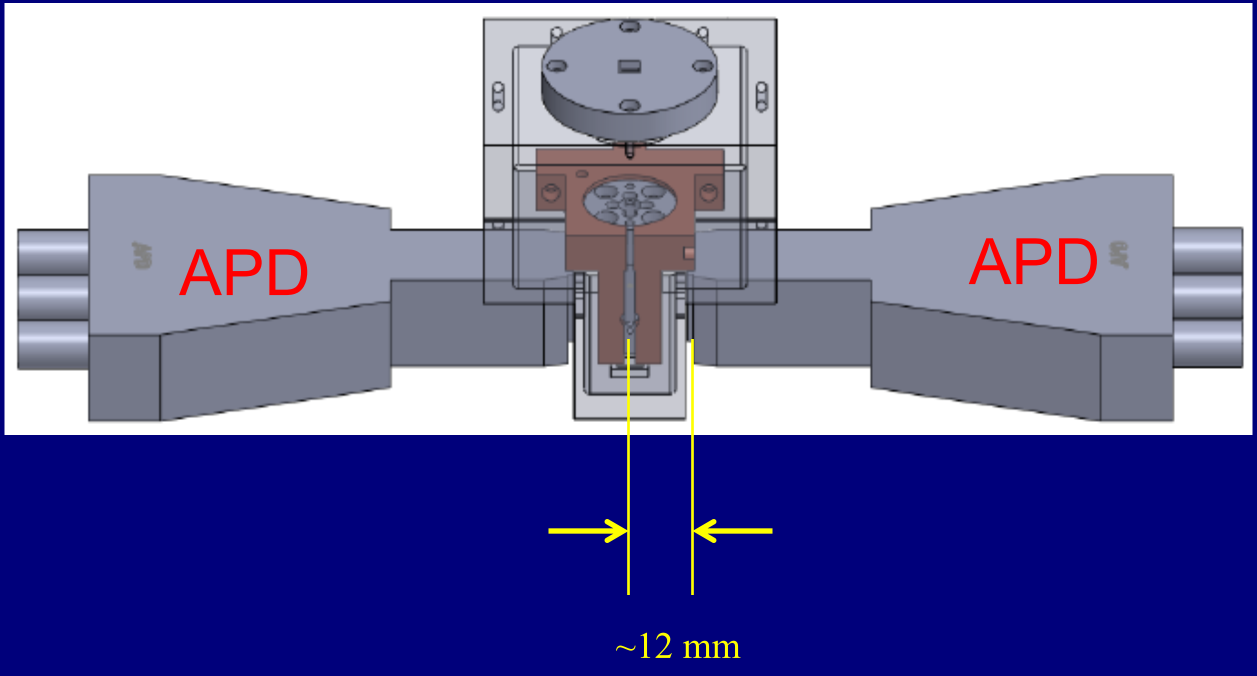


<- Hotspot

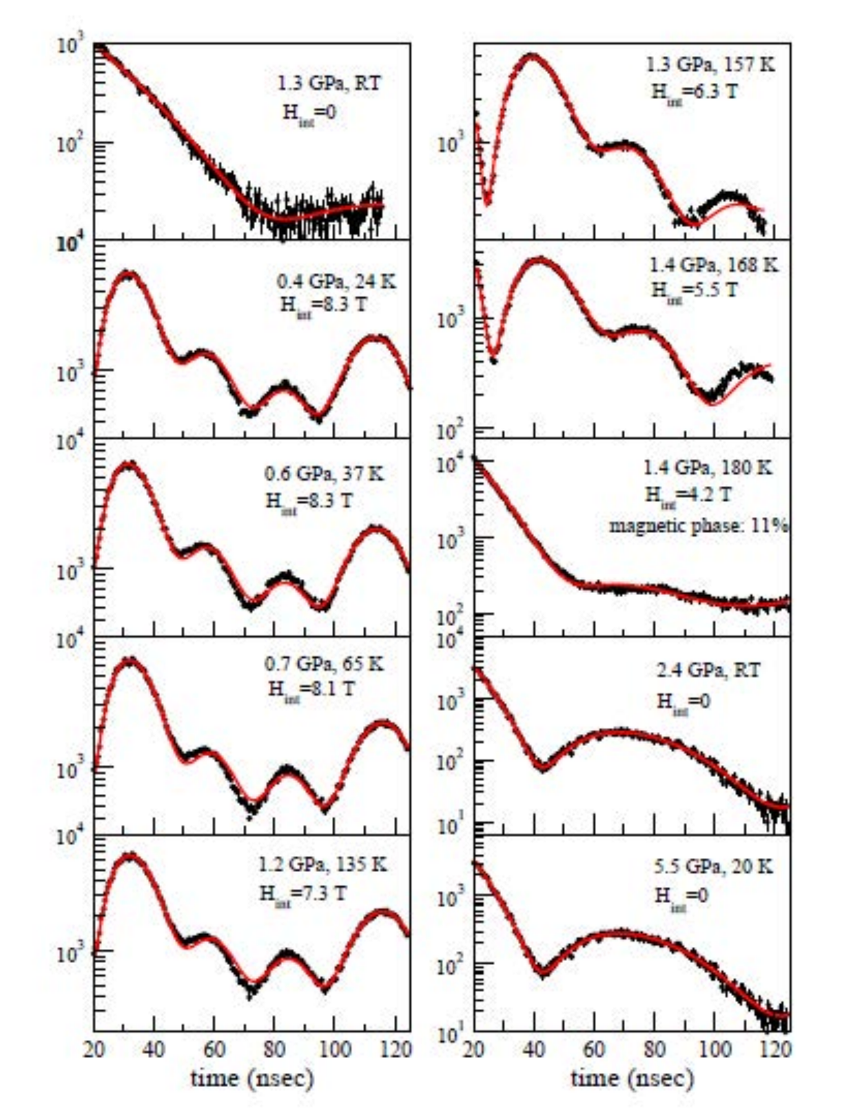
Example sample loading->

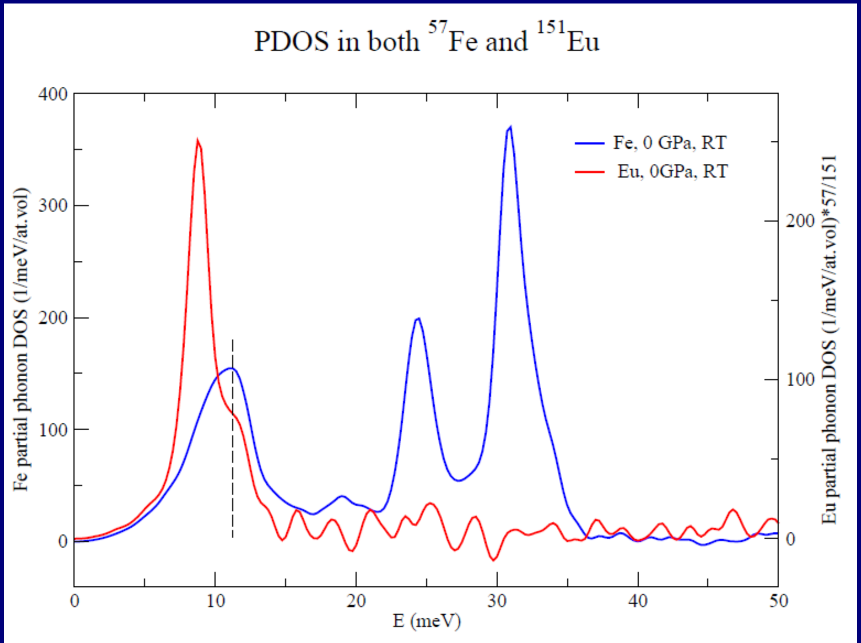
# NRIXS at High-P Low-T

Design of a miniature panoramic diamond anvil cell (DAC) .



# NRIXS and SMS of EuFe2As2 under LT and HP



# Active user programs at 3ID, APS with the following unique capabilities

1. A low temperature (4K) and high magnetic field (9T) and high pressure system for NFS. (since 2007)
2. A laser heated diamond anvil cell system (since 2002)
3. An In-situ diffraction system (since 2008)
4. An on-line Ruby system (since 2011)
5. Dynamic pressure adjusting system (gear box and gas-driven membrane cell). (since 2011)
6. Low temperature (9K) and high pressure (Mbar) system for NRIXS.

# To use the facility at 3ID, APS

- Nine months of running, in three periods
  - T1-period, Feb~Apr;
  - T2-period, Jun~Aug;
  - T3-period, Oct-Dec.

- Two type of proposals
  - GUP (General User Proposal)
    - effective for two years
  - PUP (Partner User Proposal):
    - Jointly developing new capability for the beamline, with guaranteed beam time each run

# To become a user at 3ID

- Plan ahead
- Talk to the beamline scientists
  - (Sample thickness, size, environment …)
- Apply through either
  - GUP (General User Proposal) or
  - PUP (Partner User Proposal)

Deadline: 2017-1, Oct-28-2016

2017-2, Mar-3-2017

2017-3, Jul-7-17

Thank you for your attention

and

See you at the beamline!