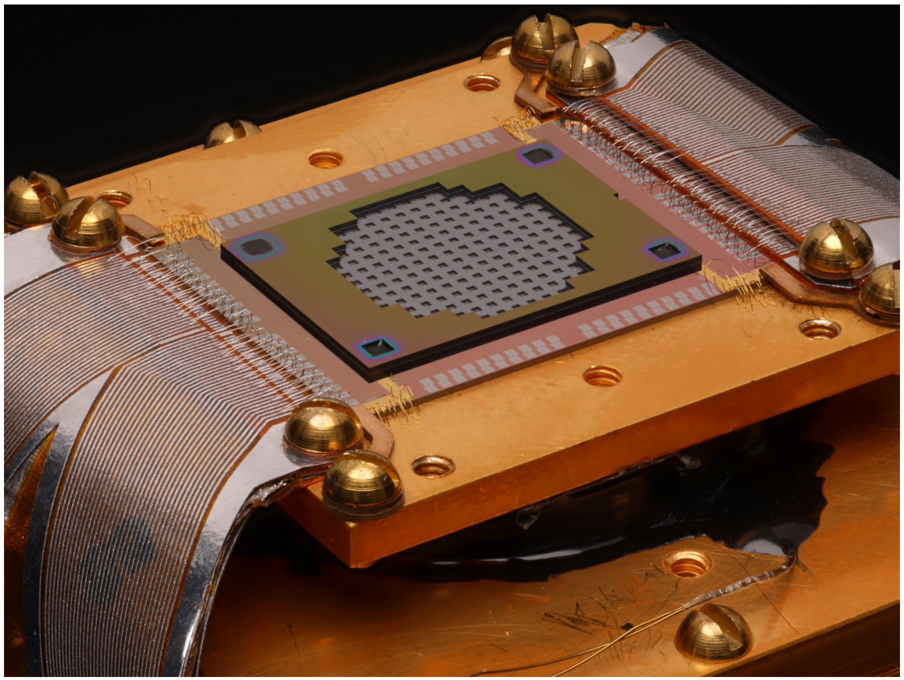
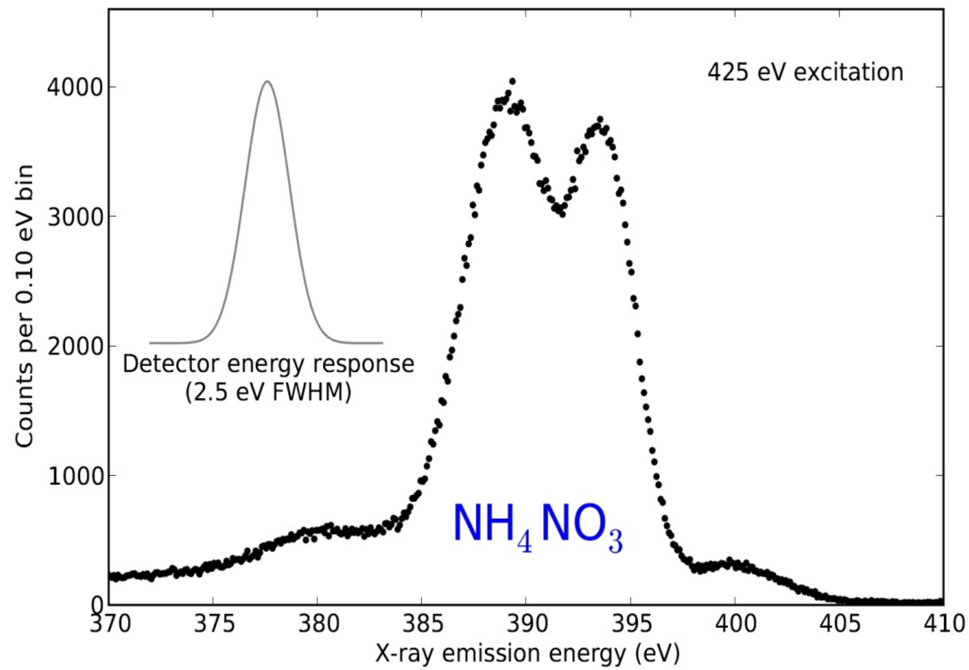
first synchrotron observations by the NIST soft x-ray transition-edge sensor spectrometer

Daniel Swetz, NIST (Boulder, Colorado)





1) x-ray spectroscopy and x-ray detectors
2) NIST TES spectrometer at NSLS beamline U7A
3) initial synchrotron spectroscopy results

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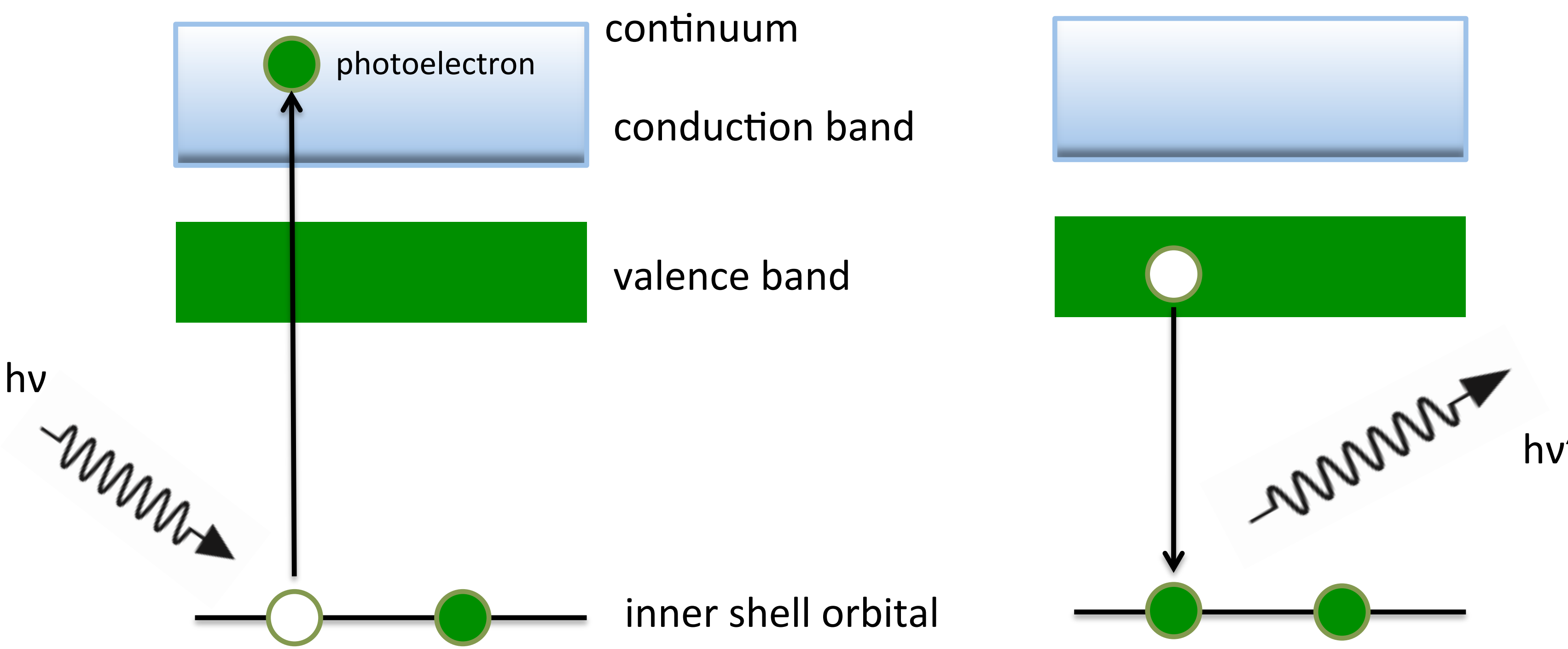
why care about x-rays?

- x-rays = powerful tools for molecular and materials analysis
  - penetrating; sensitive to buried material
  - nm wavelengths provide good spatial resolution
  - elemental and chemical sensitivity

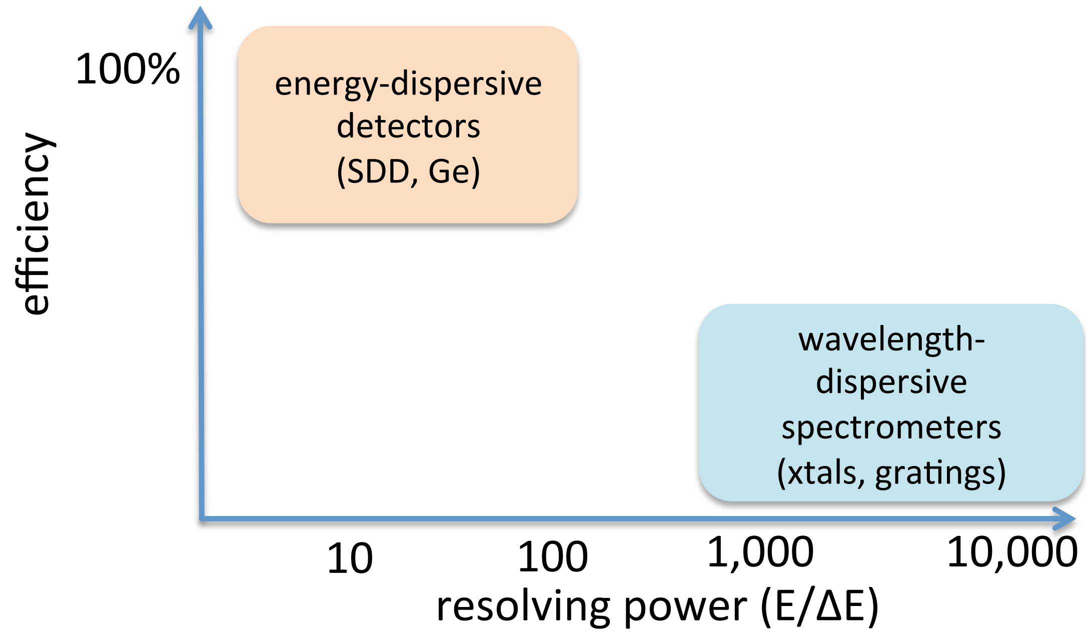


x-ray absorption spectroscopy (XAS)
-element-specific absorption edge
-probes unoccupied density of states (DOS)

x-ray emission spectroscopy (XES)
-energy shift from chemical bonding
-probes occupied DOS

why care about x-ray detectors?

- historical limits of x-ray detectors for synchrotron beamline science



high-resolution x-ray spectroscopy (crystals and gratings):

- requires brightest synchrotron beamlines (10^{12} –10^{14} photons/sec/0.1eV)
- can easily damage sample
- coverage gap between 1.5 – 2 keV
- some applications (eg: time-resolved) *still* photon starved

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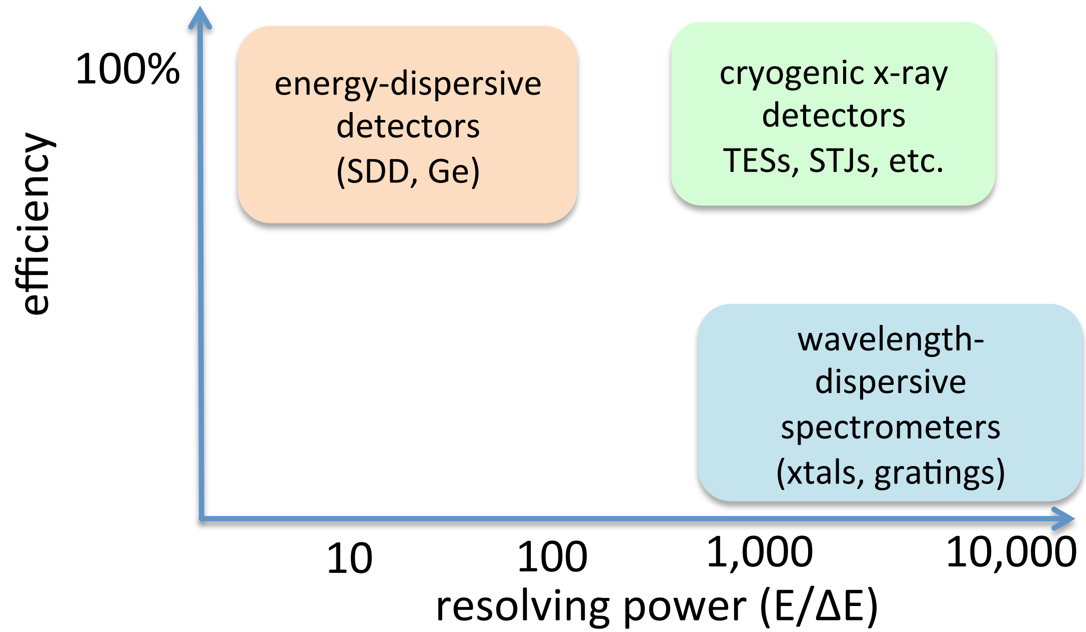

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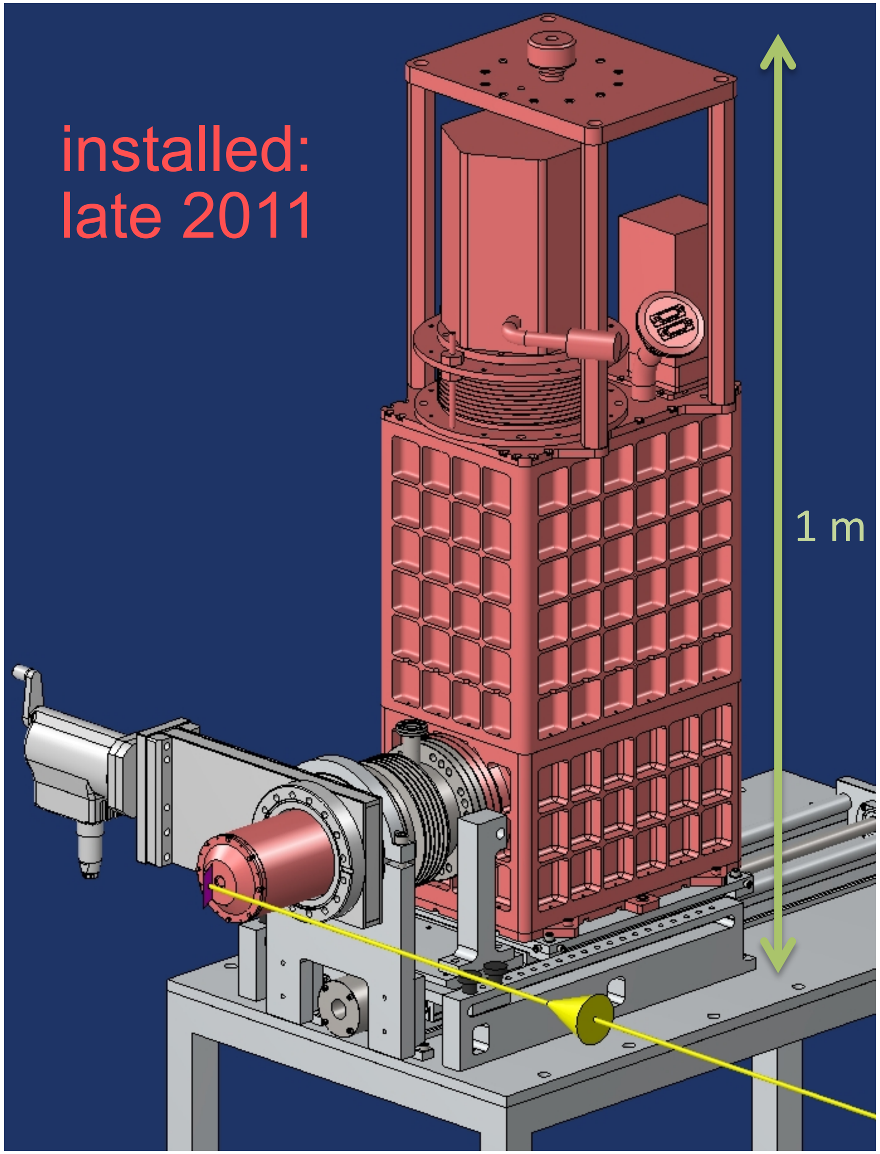
TES spectrometers provide a unique combination of spectral resolution and efficiency

the TES spectrometer

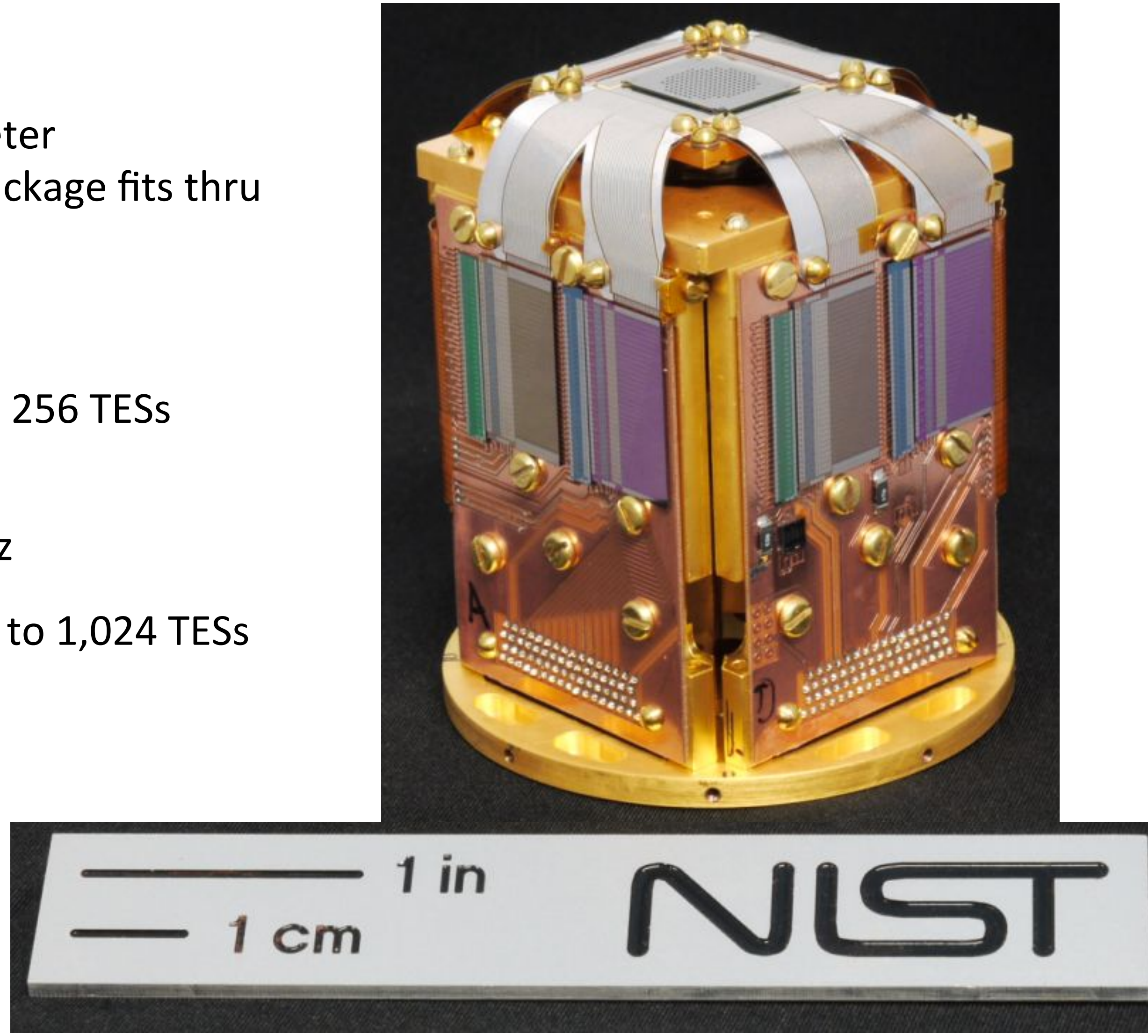
soft x-ray TES spectrometer at the NSLS

- TES X-ray spectrometer deployed to National Synchrotron Light Source (NSLS)
- NSLS U7A (bending magnet) beamline:
    - Soft x-ray: 200-1400 eV
    - 1 x 10^{10} photons/sec/0.1eV



50 mK detector "snout"

- 50 mK stage: 2.3 inch diameter including three IR shields, package fits thru 4" ID of 6" conflat flange
- TES array on top
- TDM/CDM readout for up to 256 TESs (8col x 32row) around sides
- array count rate up to 25 kHz
- architecture could be grown to 1,024 TESs (32c x 32r) straightforwardly

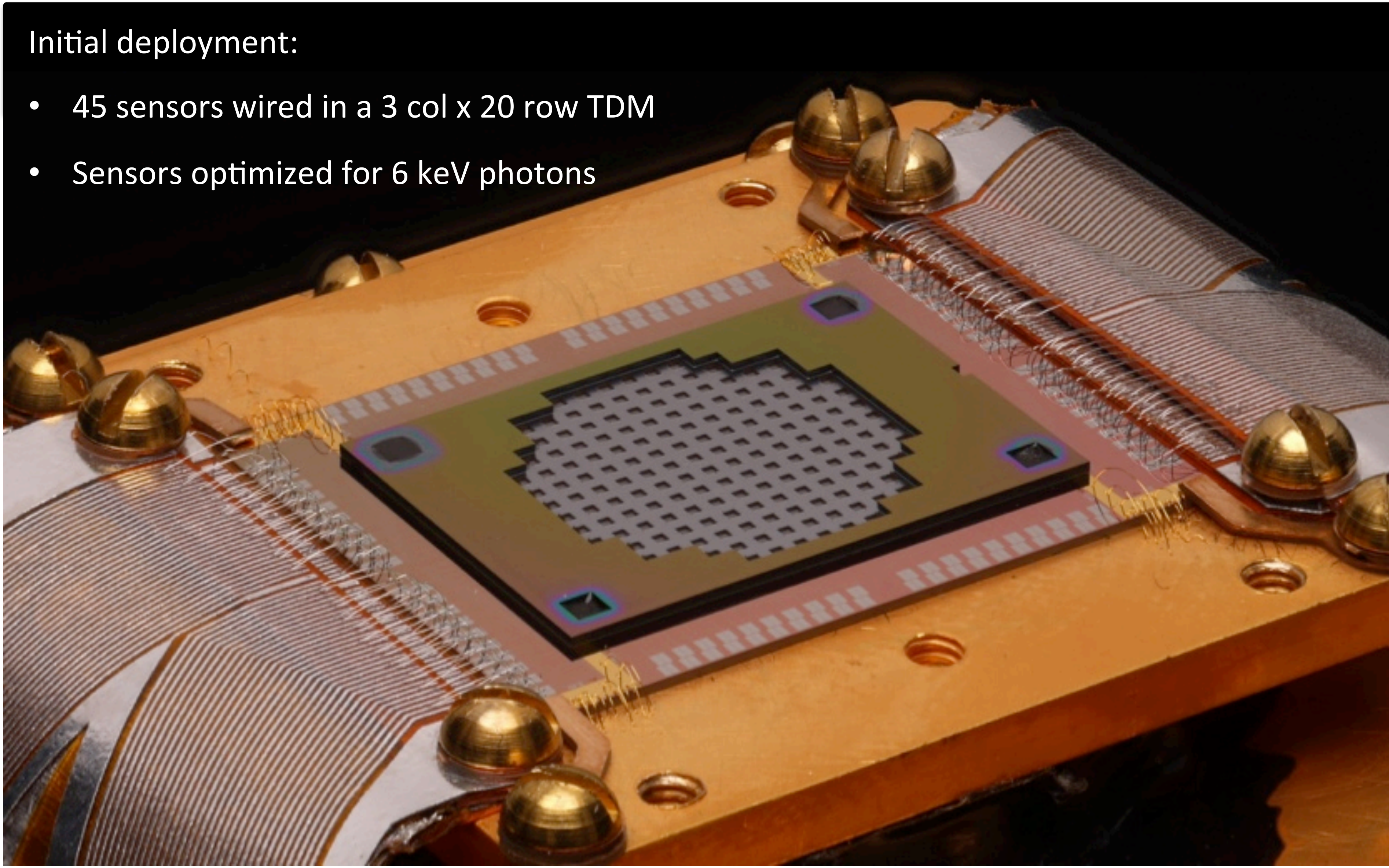


Deployed to NSLS, Jyväskylä, Lund, GSFC, NIST, soon APS

TES detector plane

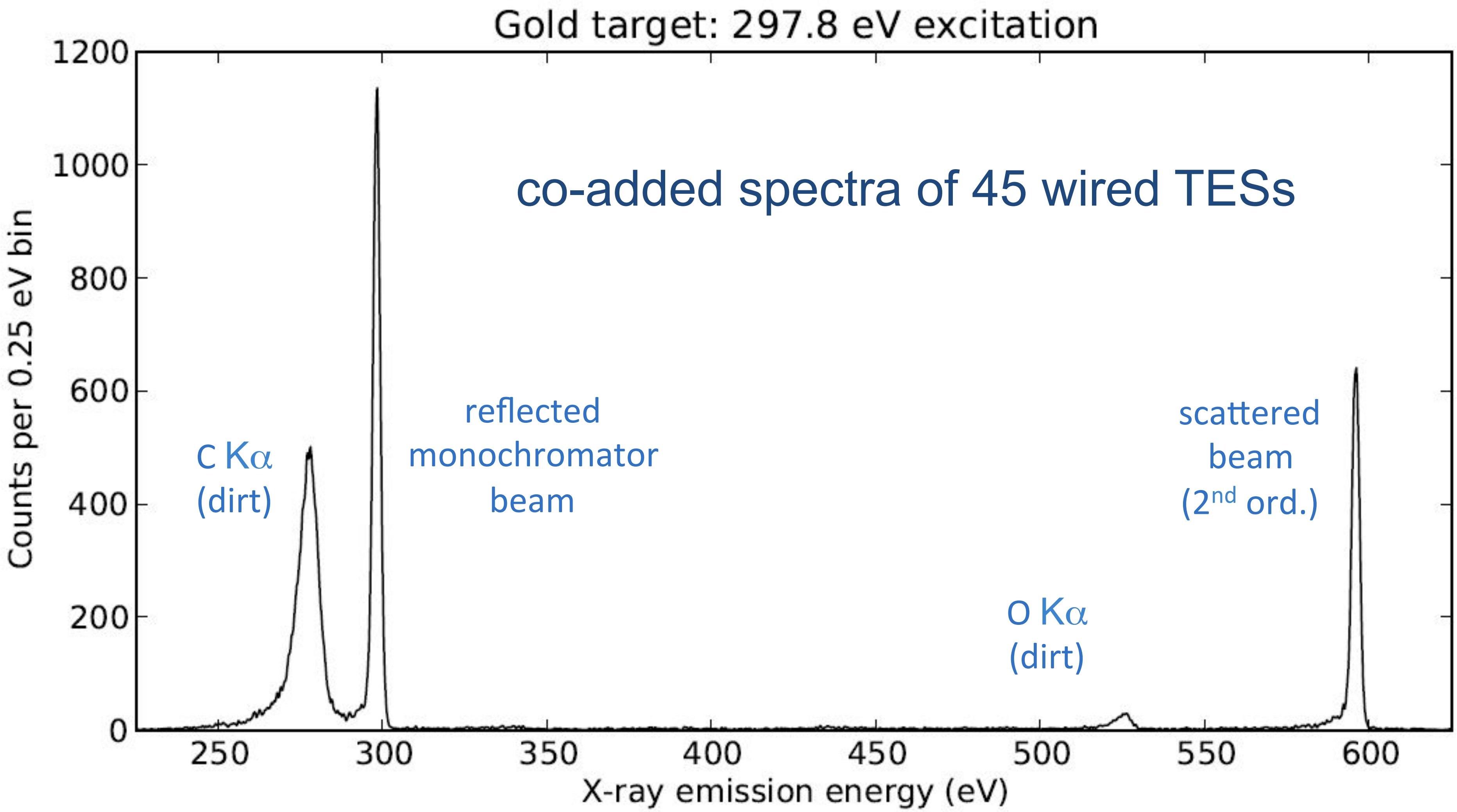
Initial deployment:

- 45 sensors wired in a 3 col x 20 row TDM
- Sensors optimized for 6 keV photons

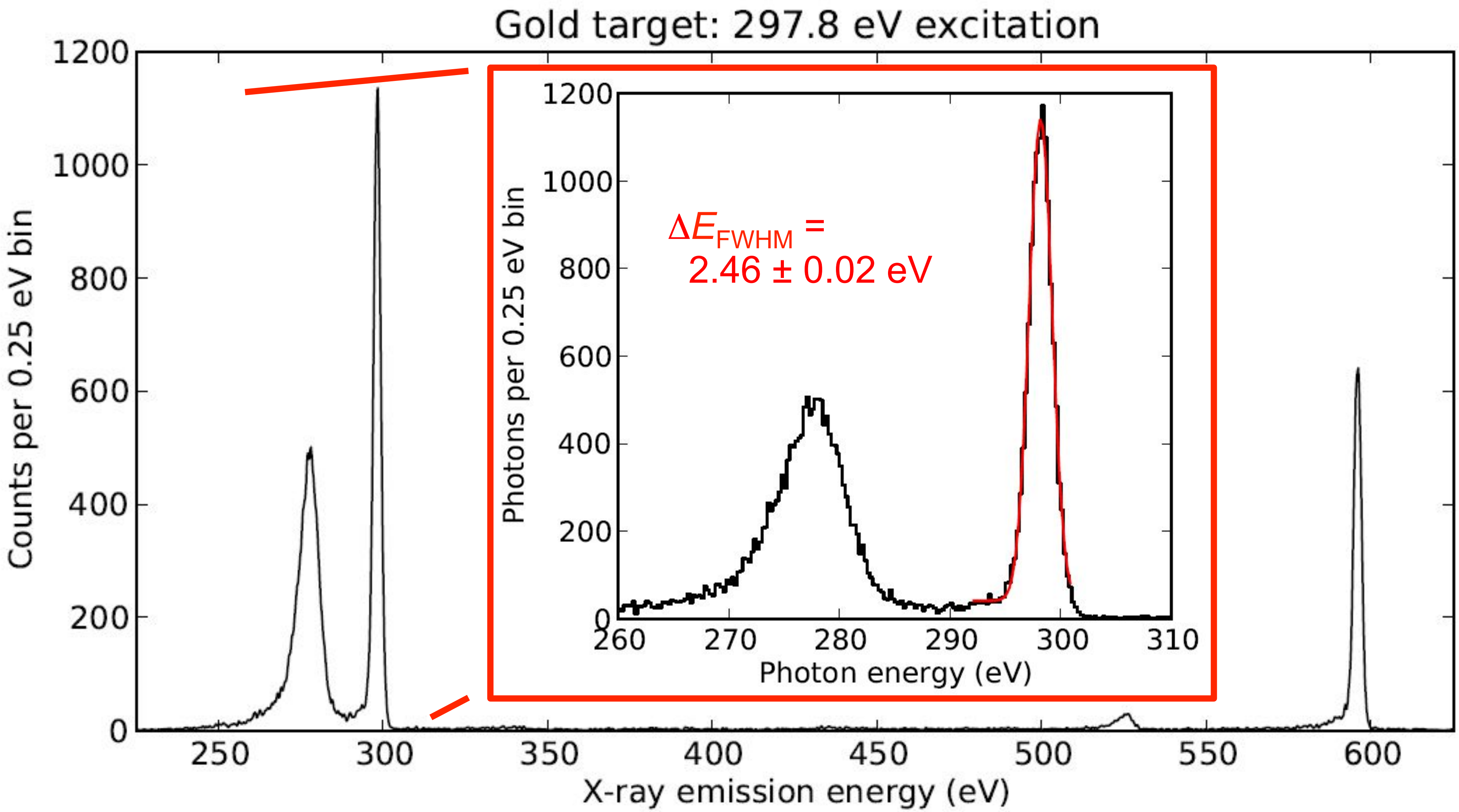


Package versatile - other sensors can be substituted for other applications

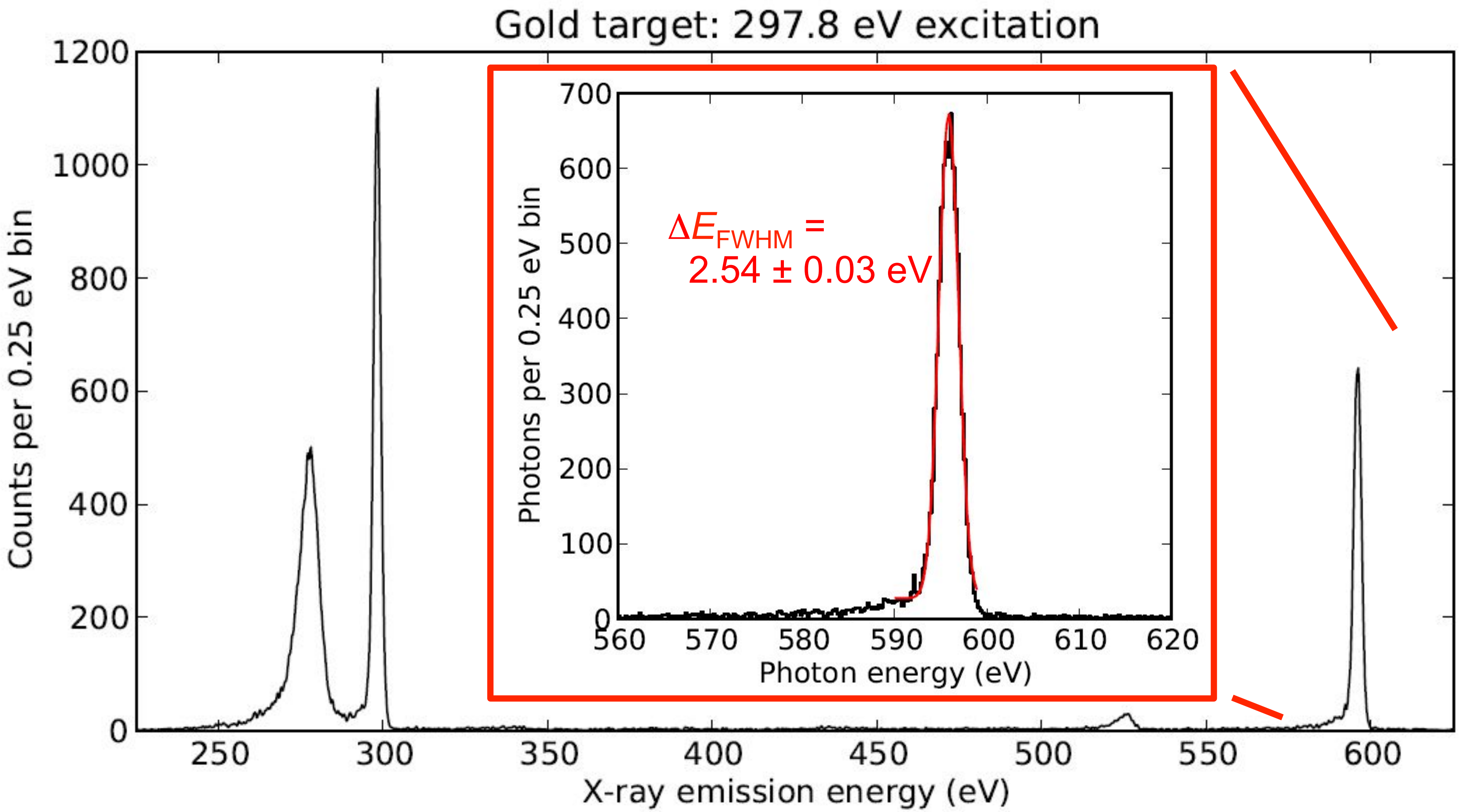
achieved energy resolution



achieved energy resolution



achieved energy resolution



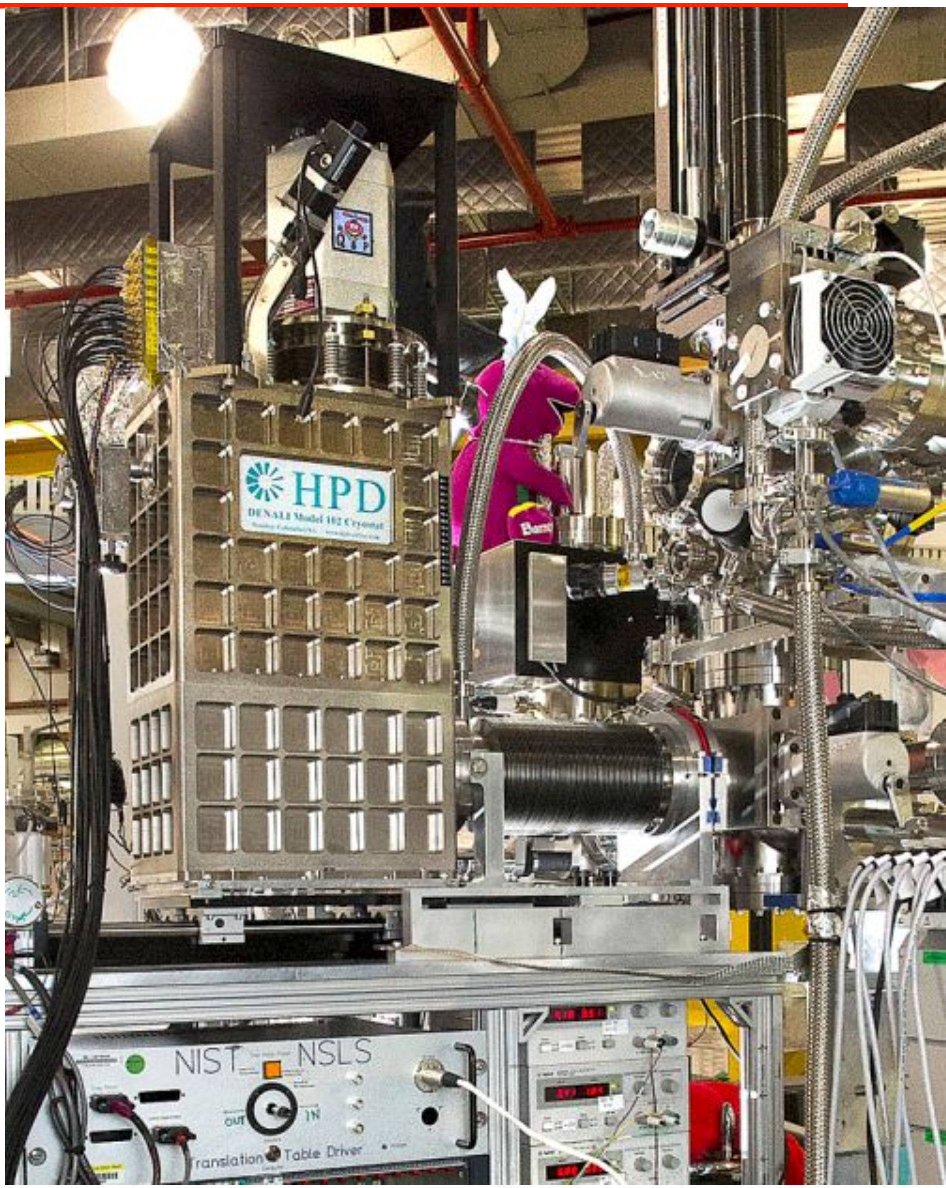
applications

Immediate applications of prototype NSLS spectrometer:

- partial-fluorescence-yield absorption spectroscopy (chemistry of unoccupied valence states)

- XES of eV-scale chemical shifts (chemistry of occupied valence states)

Let's see an example of each!



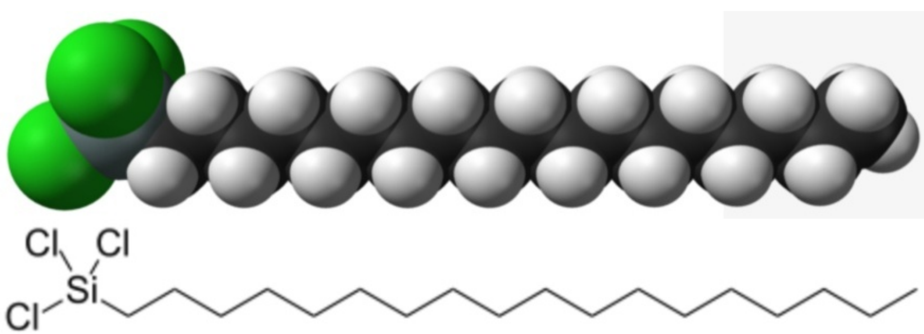
PFY-NEXAFS

first application example

partial-fluorescence-yield
near-edge x-ray absorption fine structure
(PFY-NEXAFS)

map unoccupied D.O.S.

PFY-NEXAFS

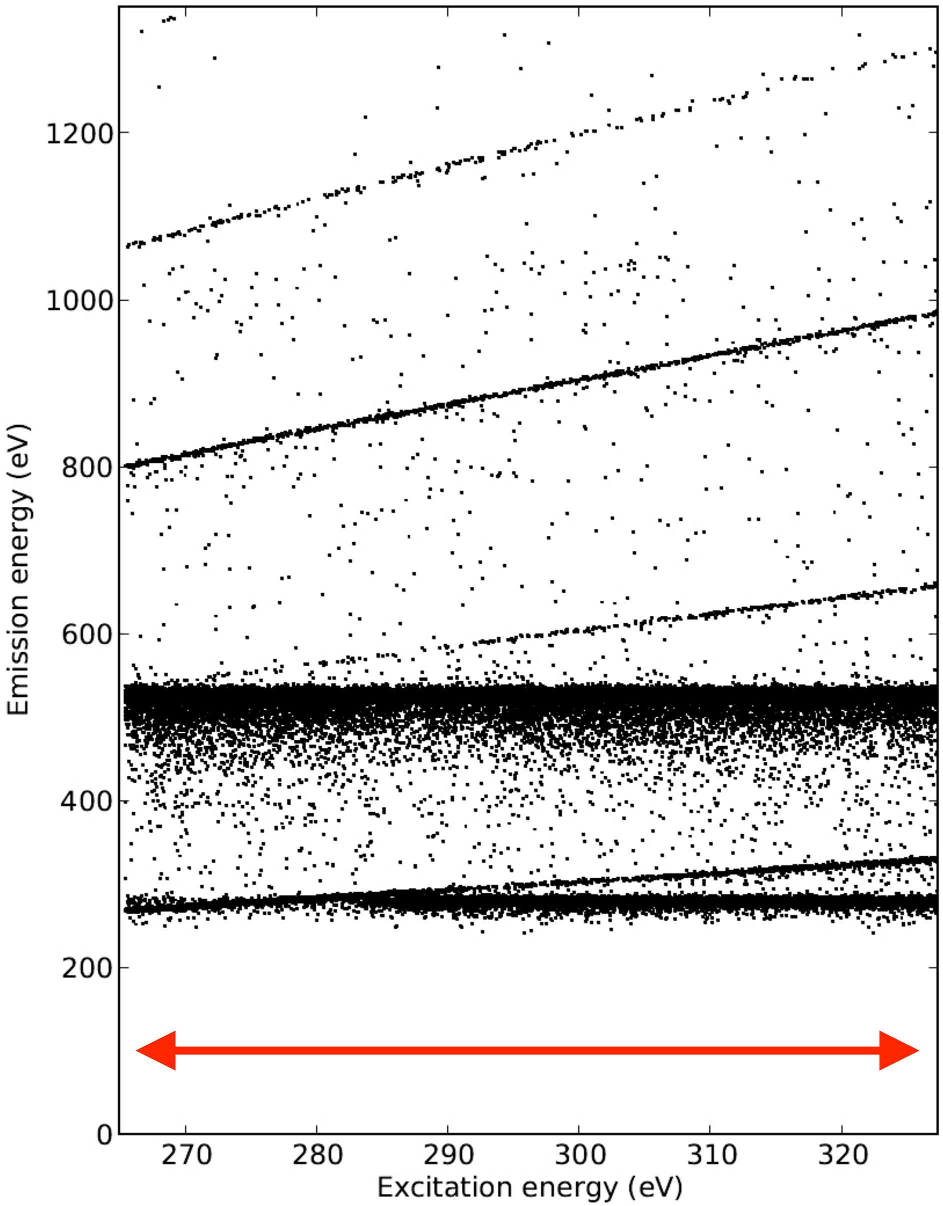


A dilute sample: NIST standard reference material (SRM) 1216-I

octadecyltrichlorosilane (OTS) at 0.7% C by mass in porous microparticulate SiO_2 (particle diam. = 20 μm)

Goal: carbon-edge absorption spectroscopy

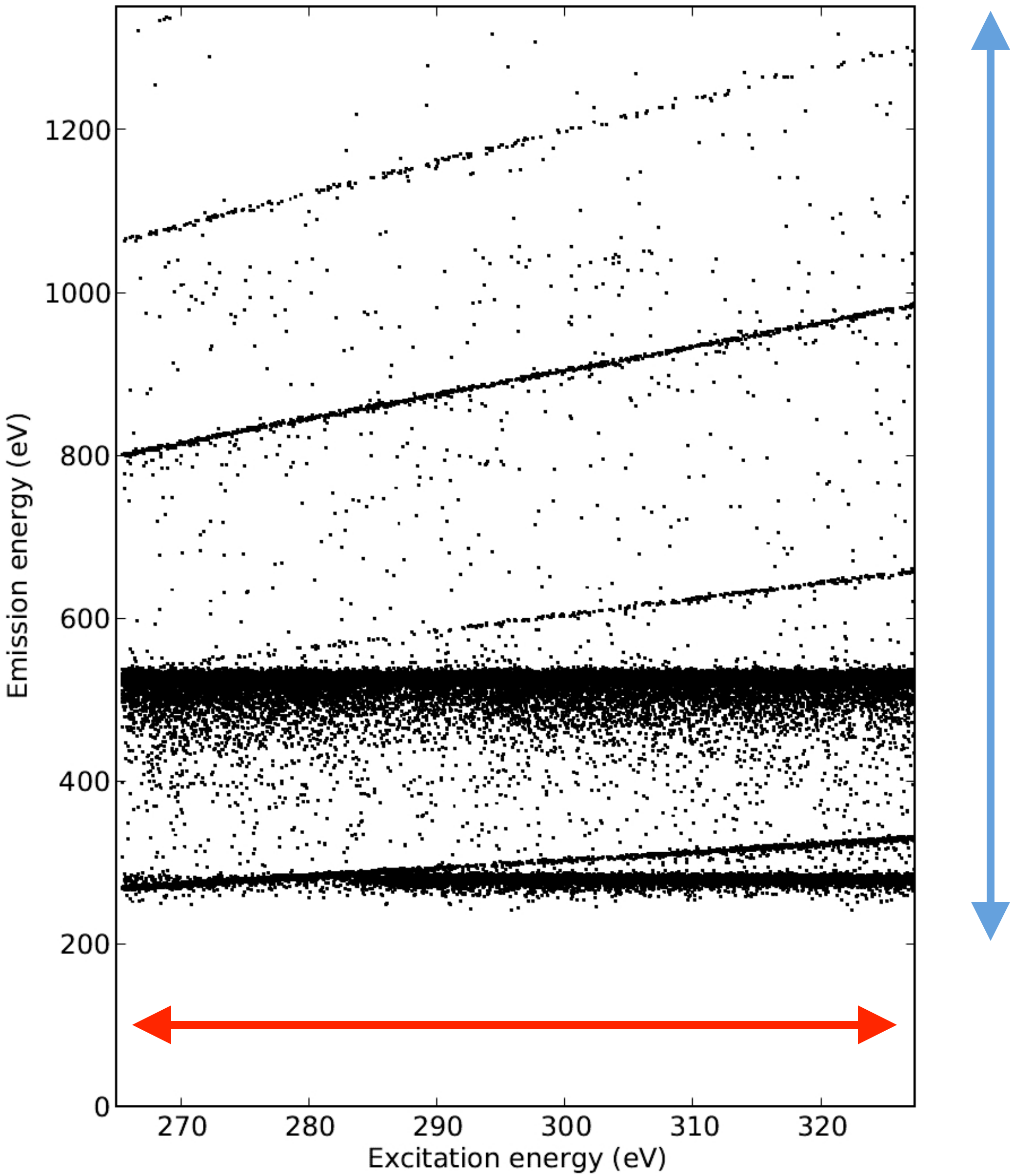
PFY-NEXAFS



beamline monochromator scanned from 265 to 327 eV

(across the Carbon K-edge)

PFY-NEXAFS



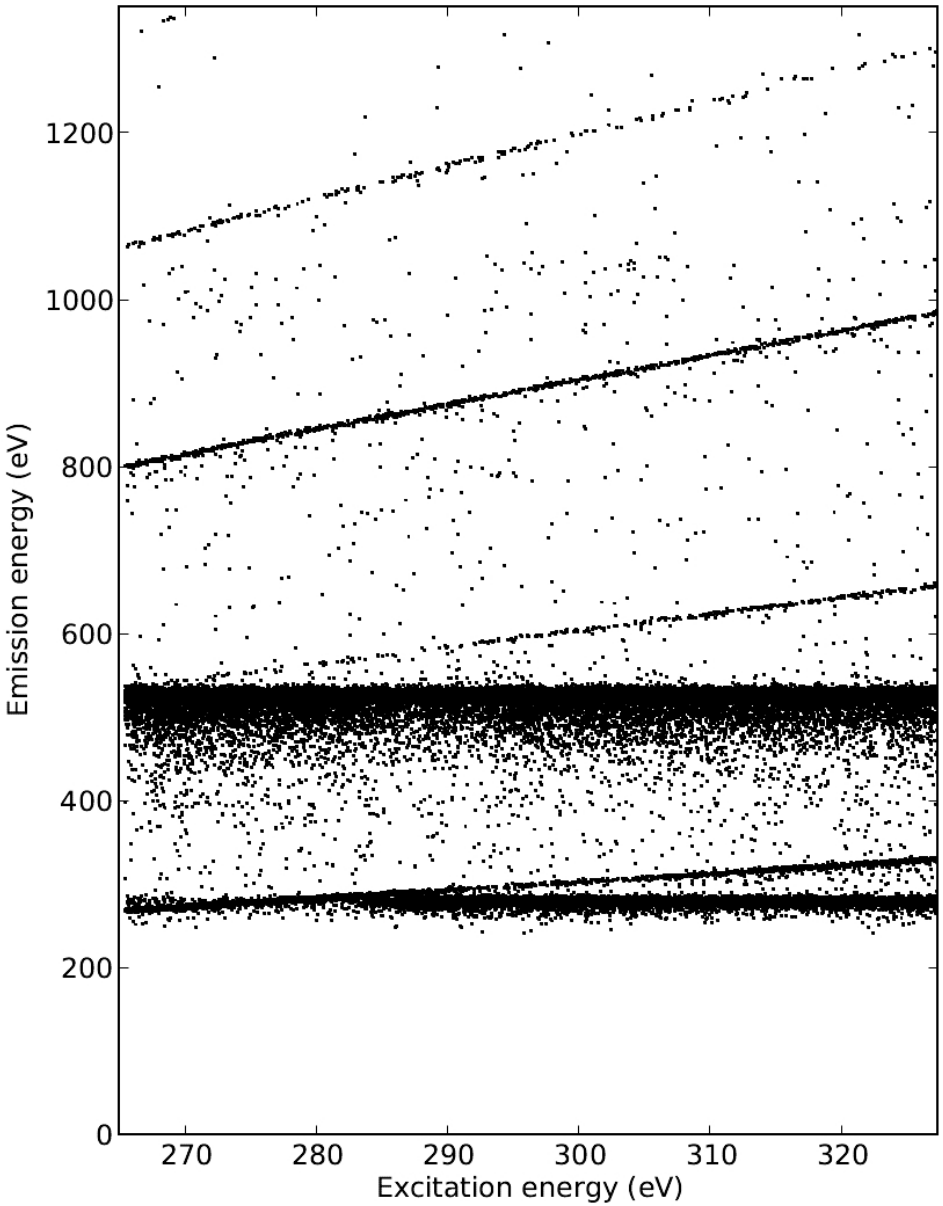
emission collected simultaneously from 200 eV to 1400 eV by TES array

(beamline produces no photons above 1400 eV)

beamline monochromator scanned from 265 to 327 eV

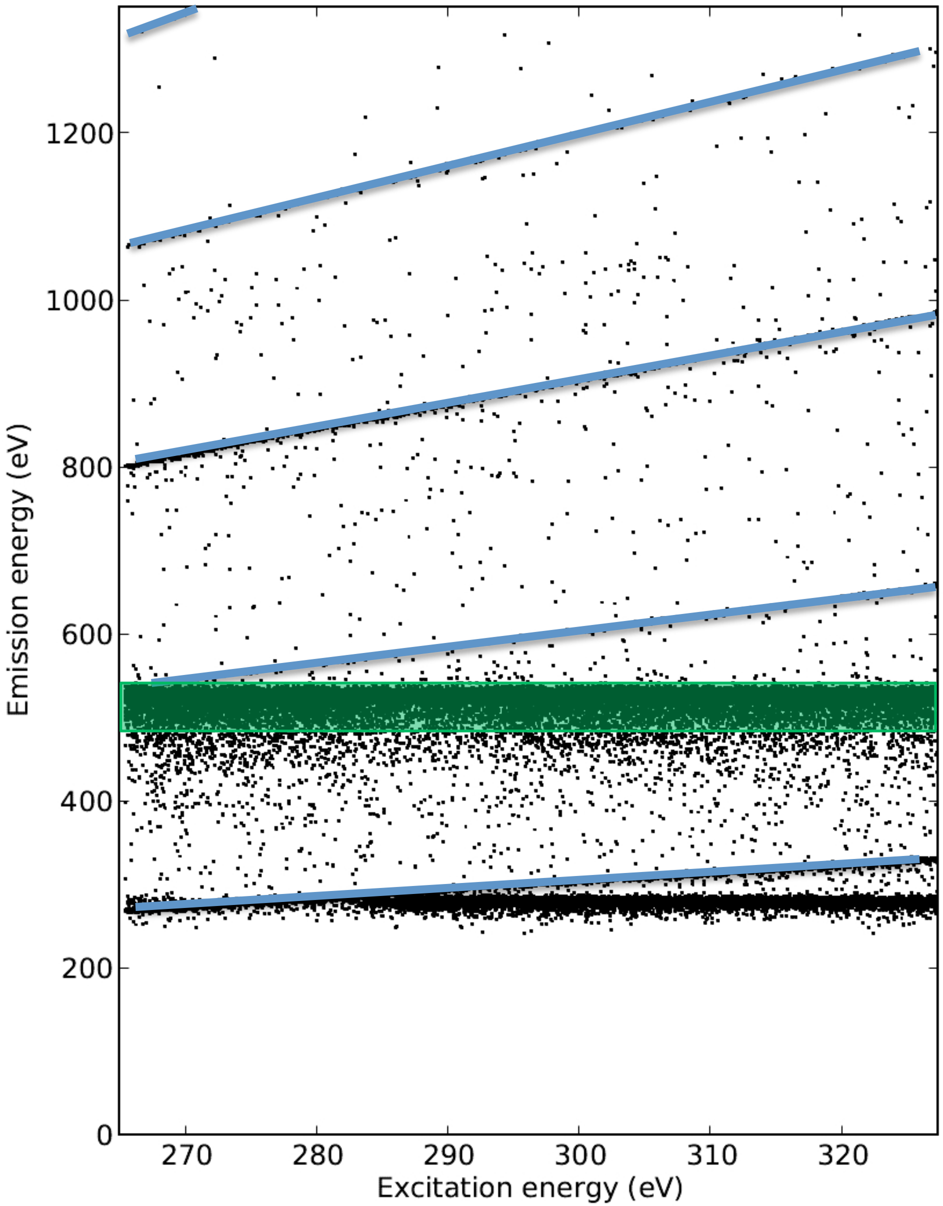
(across the Carbon K-edge)

PFY-NEXAFS



- each point is an x-ray
- 1.6 million x-rays collected over 20 min.
- < 10% of total data plotted for clarity
- TES's broadband spectral resolution allows "total knowledge" of what's coming off sample – exceedingly useful for diagnostics

PFY-NEXAFS

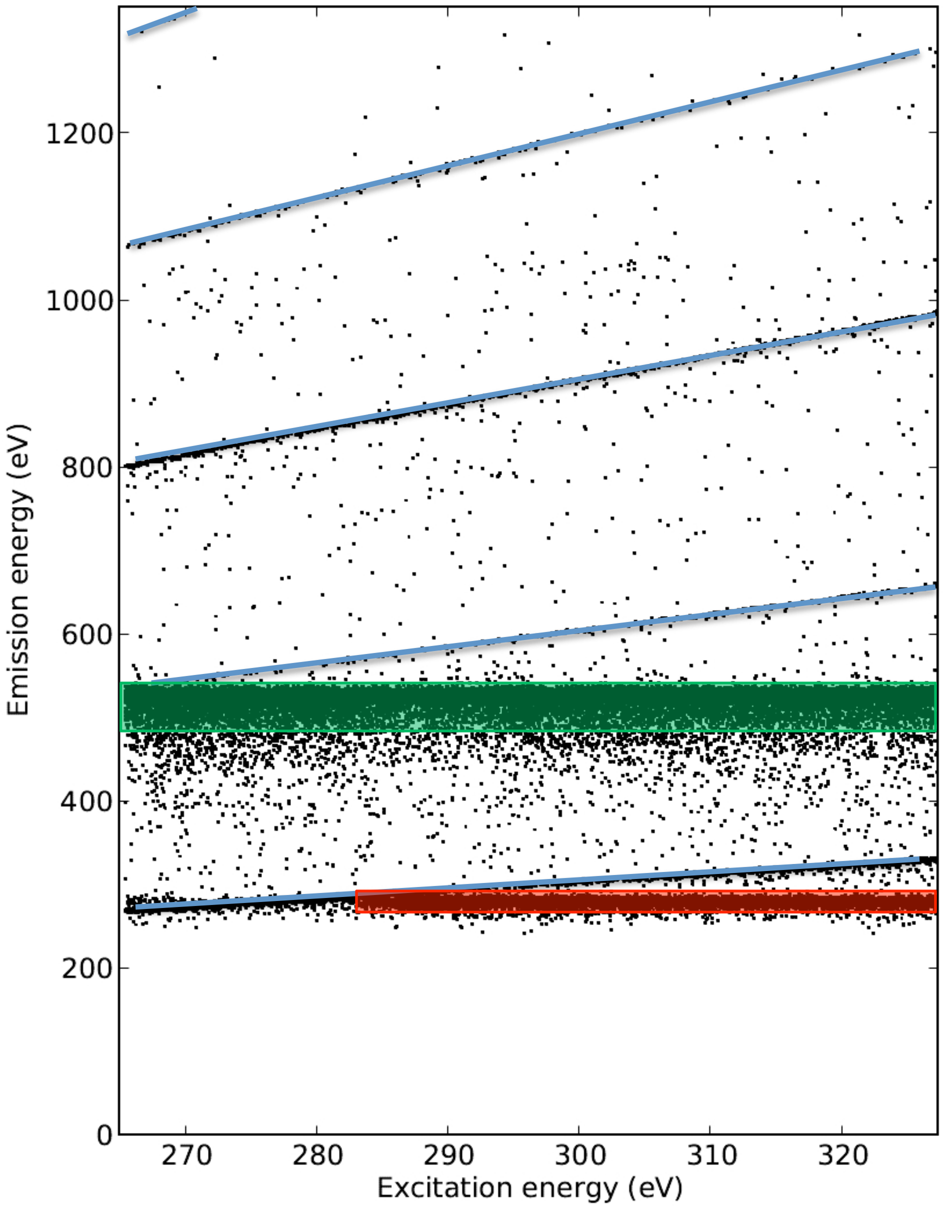


backgrounds:

harmonics 1 – 5 of scattered beam

O Kα (excited by harmonics)

PFY-NEXAFS



backgrounds:

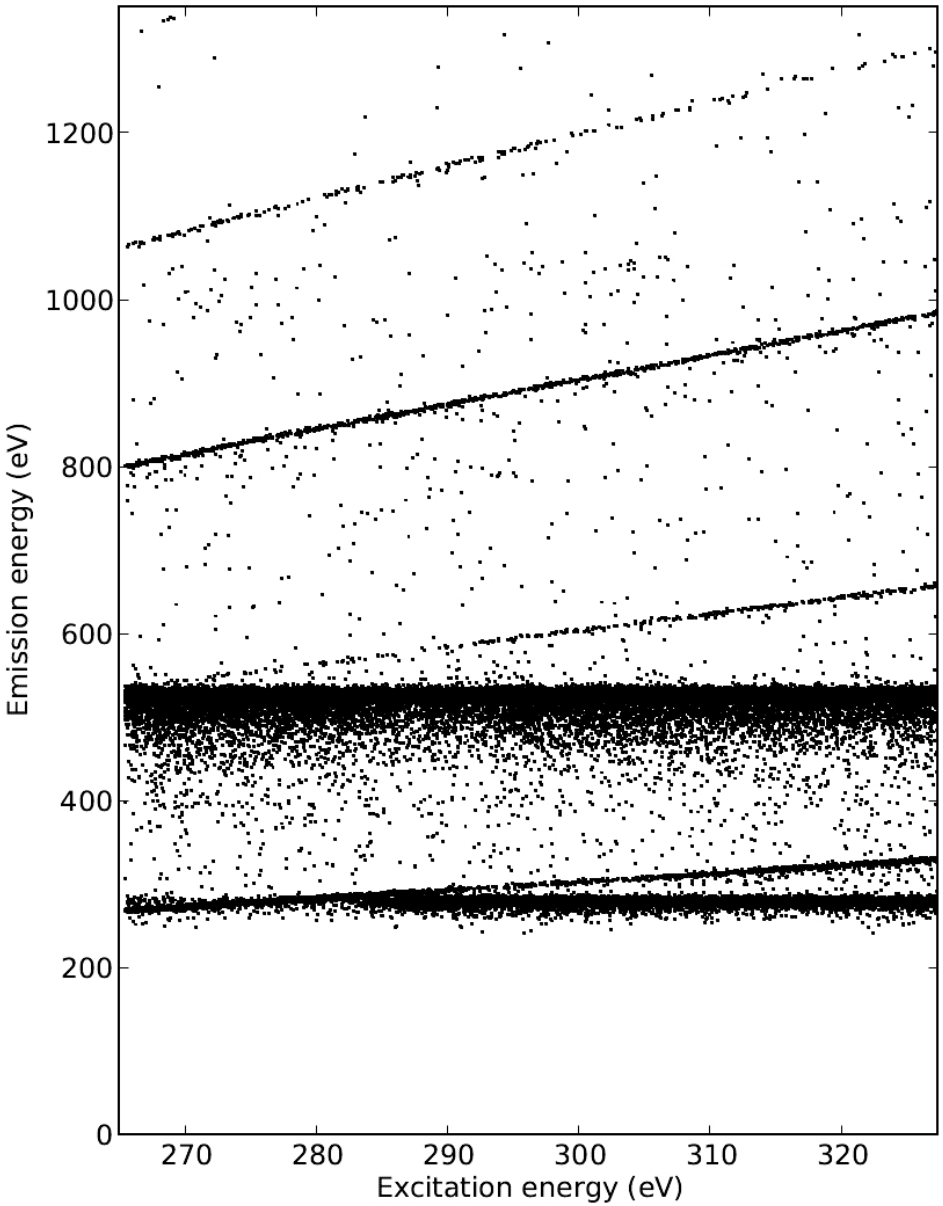
harmonics 1 – 5 of scattered beam

O Kα (excited by harmonics)

C Kα: desired signal (track strength vs. E_{beam})

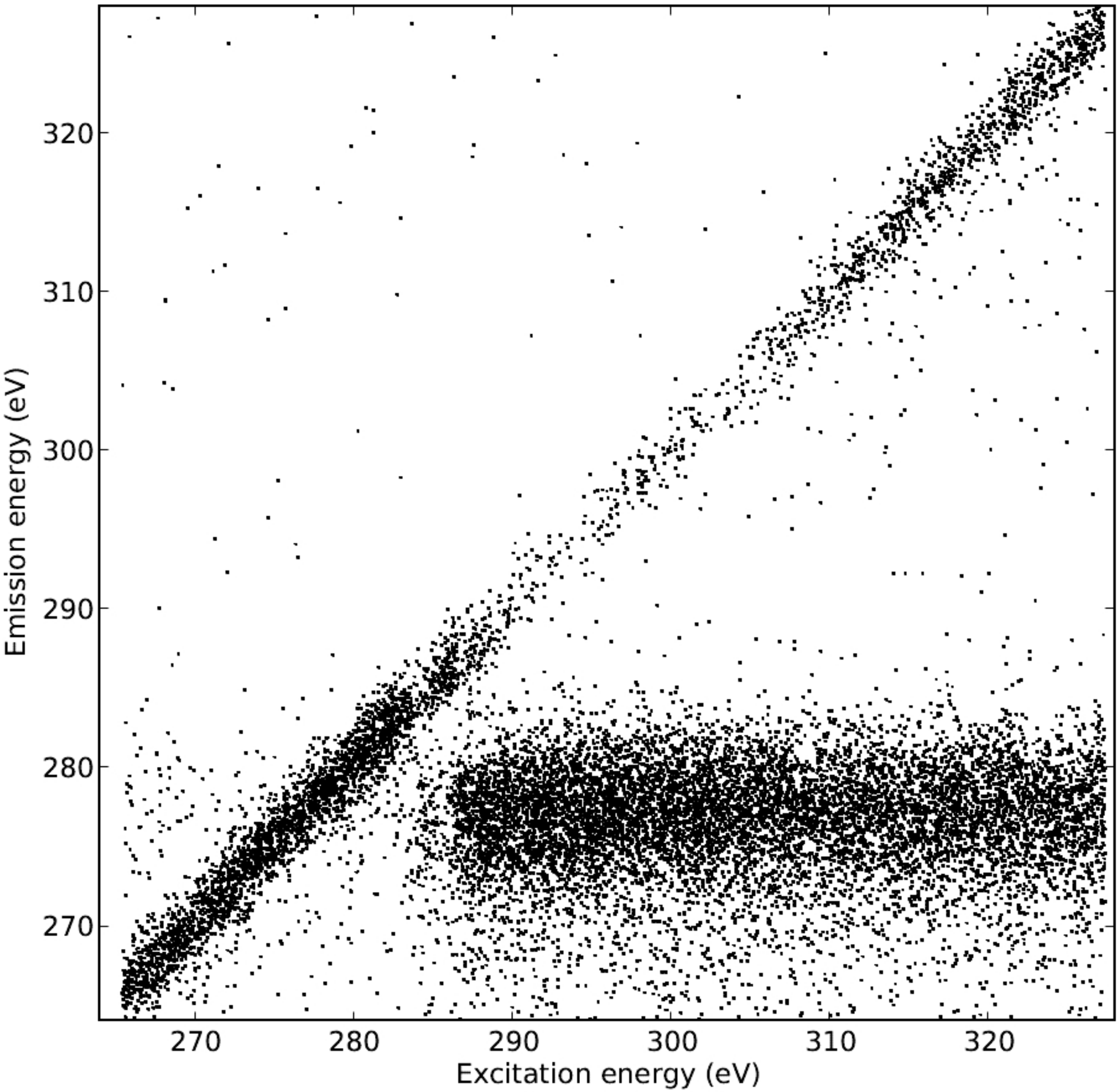
TES spectrometer's broadband spectral resolution allows separation of backgrounds from signal

PFY-NEXAFS



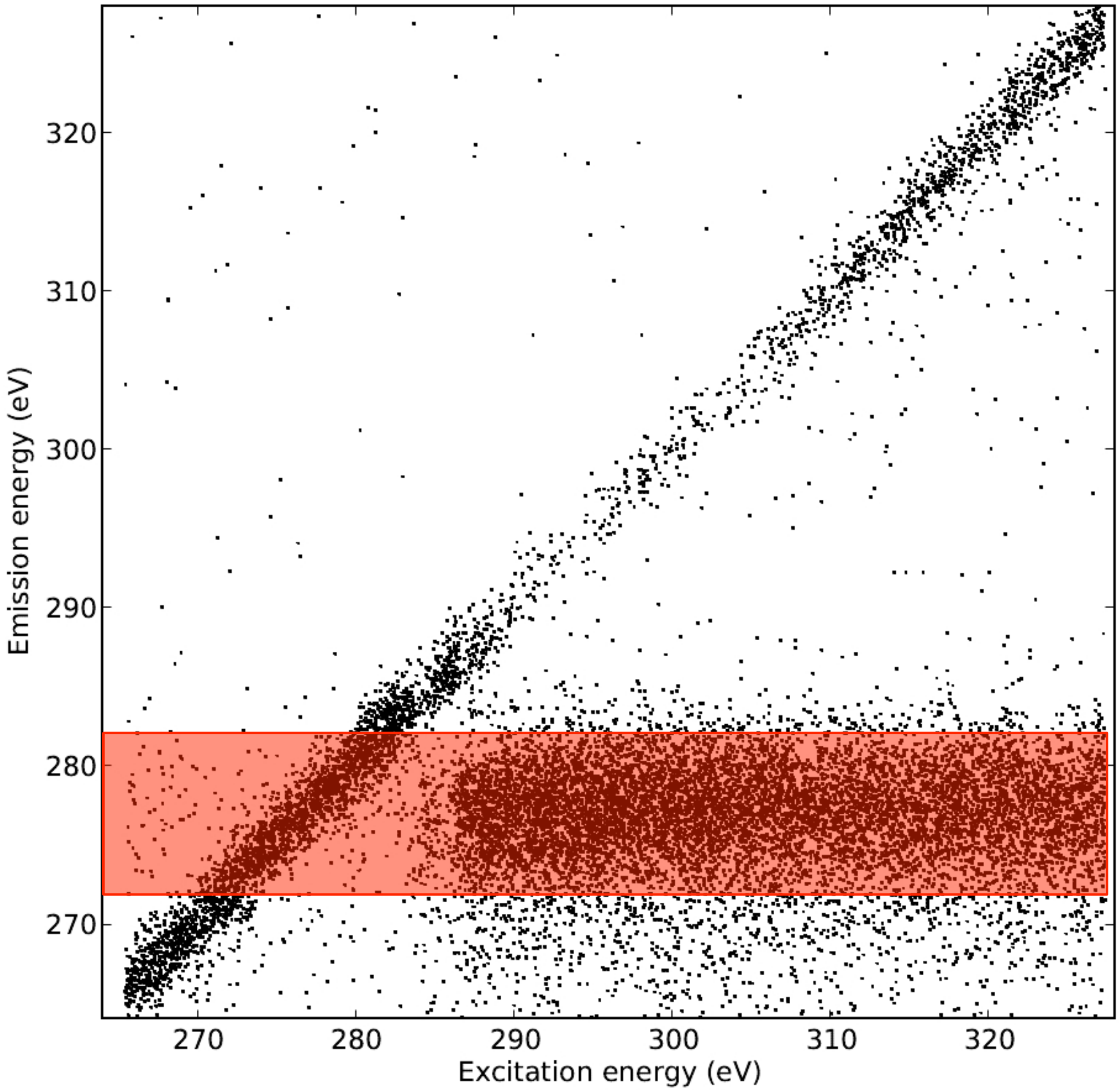
zoom

PFY-NEXAFS



zoom

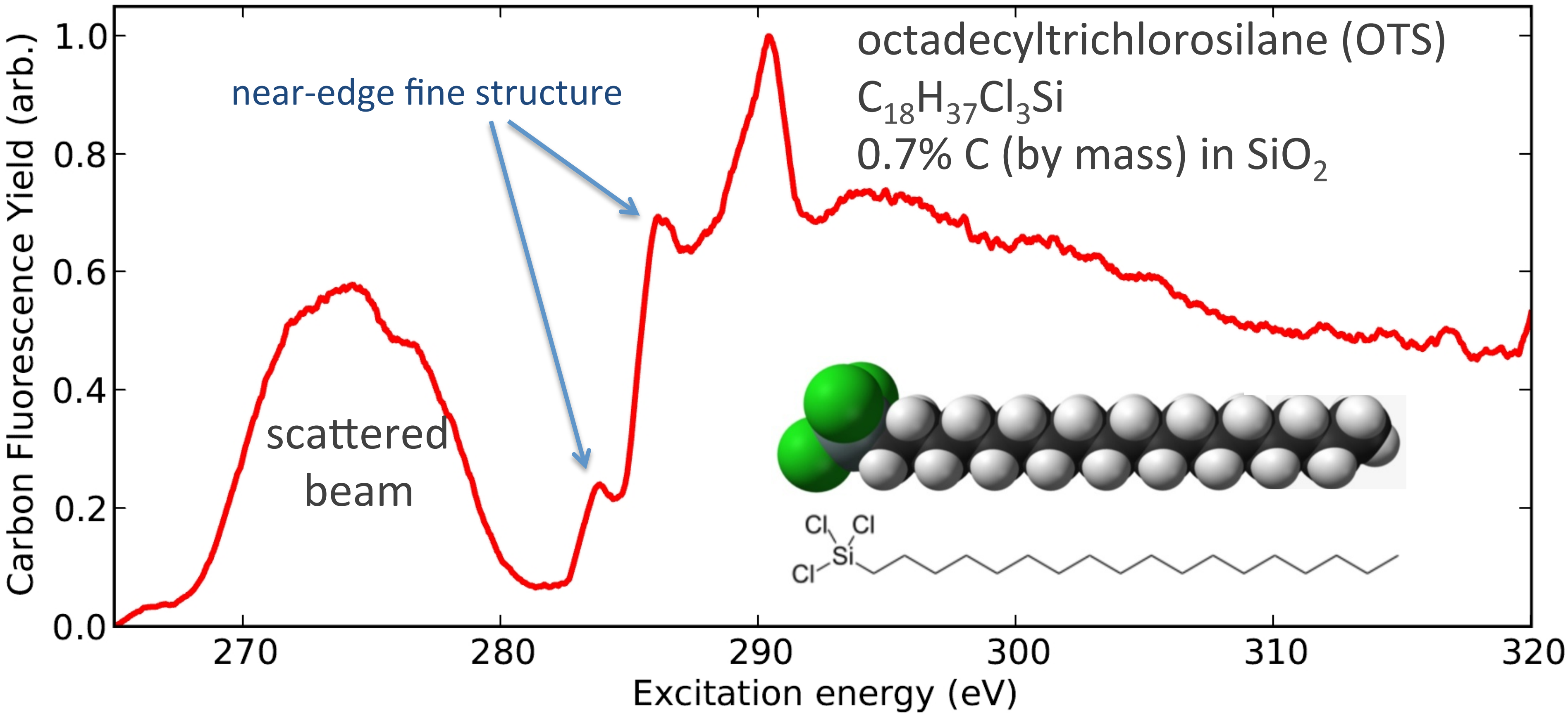
PFY-NEXAFS



window on C Kα, histogram

(can adjust window after acquisition)

PFY-NEXAFS Spectrum



resulting NEXAFS spectrum

- better than MultiLayer Mirror (MLM) spectrum of same sample
- unlike MLM, also works at N, O, … all other edges

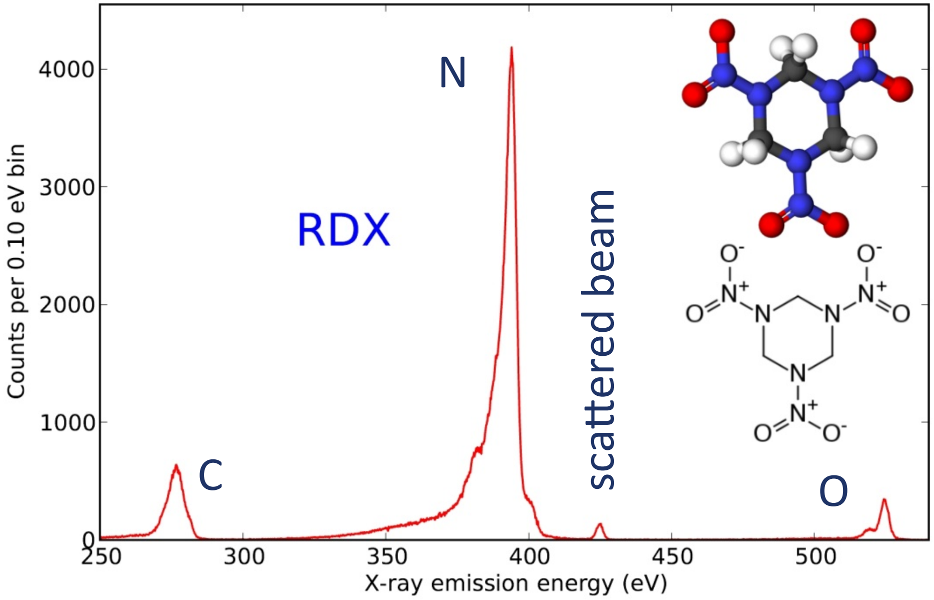
chemical-shift XES

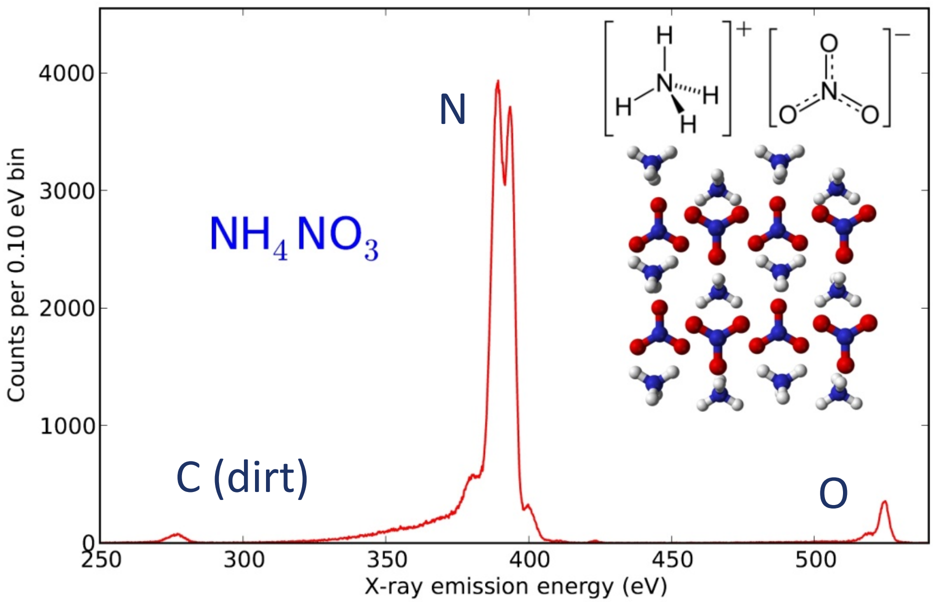
second example application

X-ray emission spectroscopy (XES)
for chemical analysis

map the occupied D.O.S.

chemical-shift XES





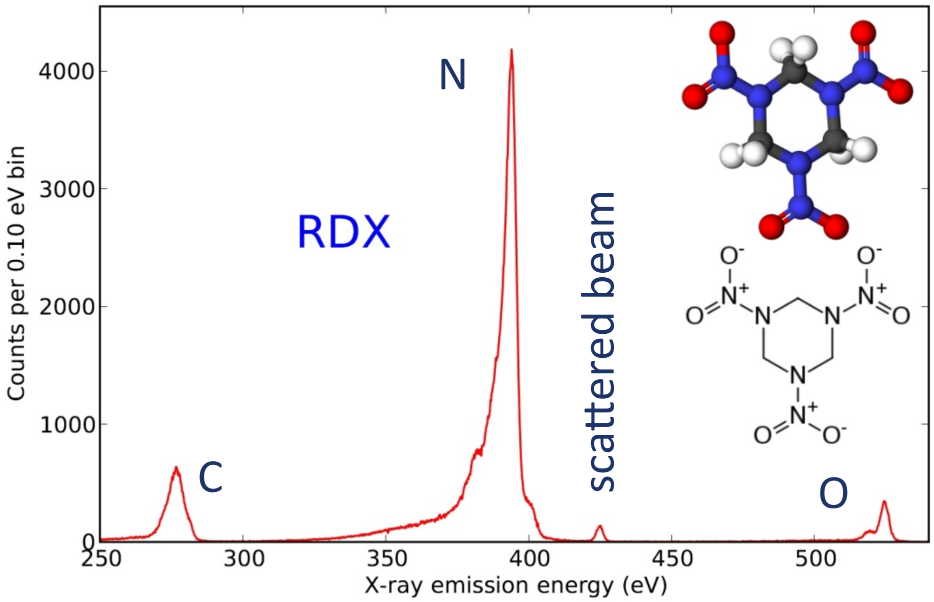
NIST is cataloging emission spectra of "energetic nitrogen compounds" for SEM analysis of criminal forensic samples.

- RDX: major component of C4 plastic explosive
- ammonium nitrate (fertilizer; can be used to build fertilizer bombs)

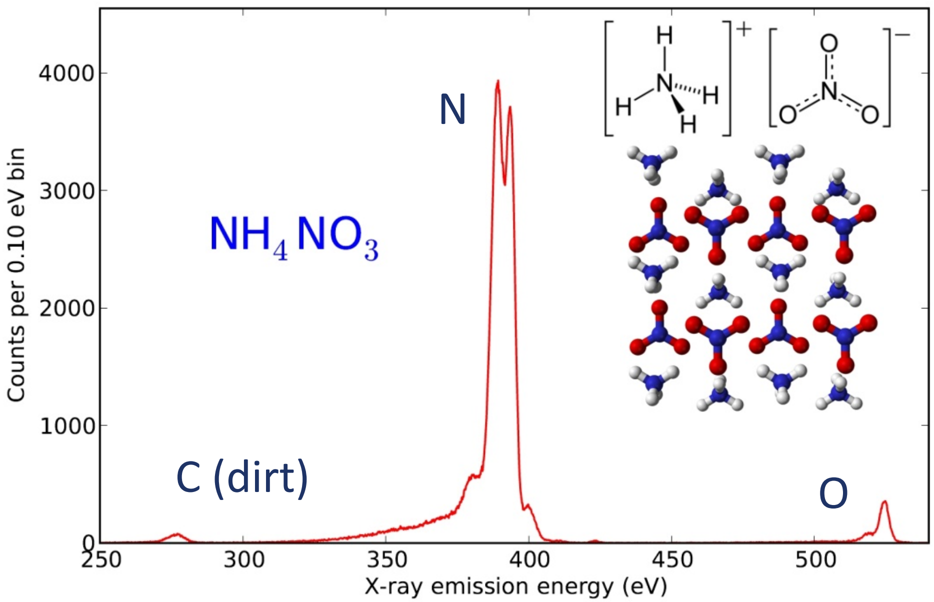
excite @ 425 eV

(well above N edge).

chemical-shift XES

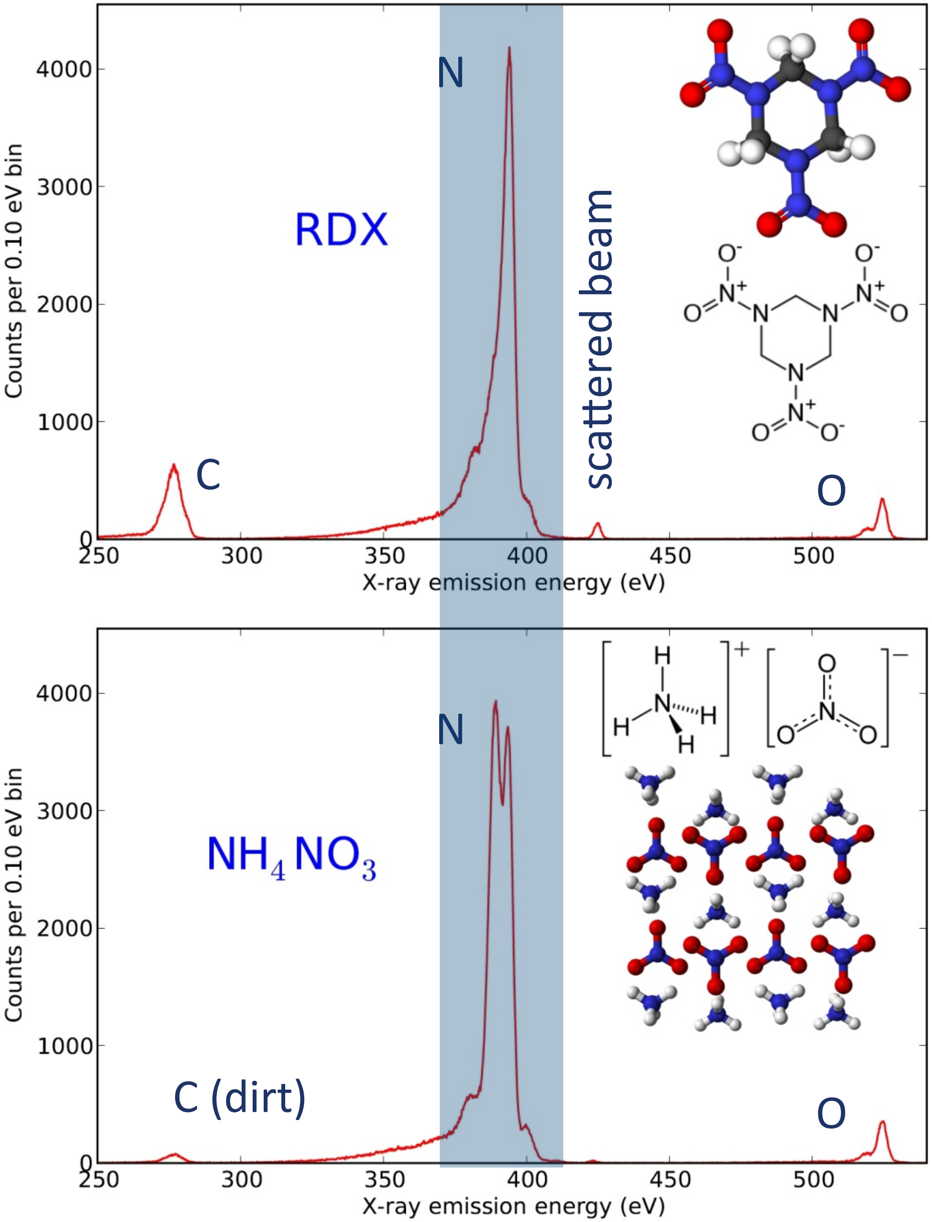


RDX spectrum acquired in 22 min.



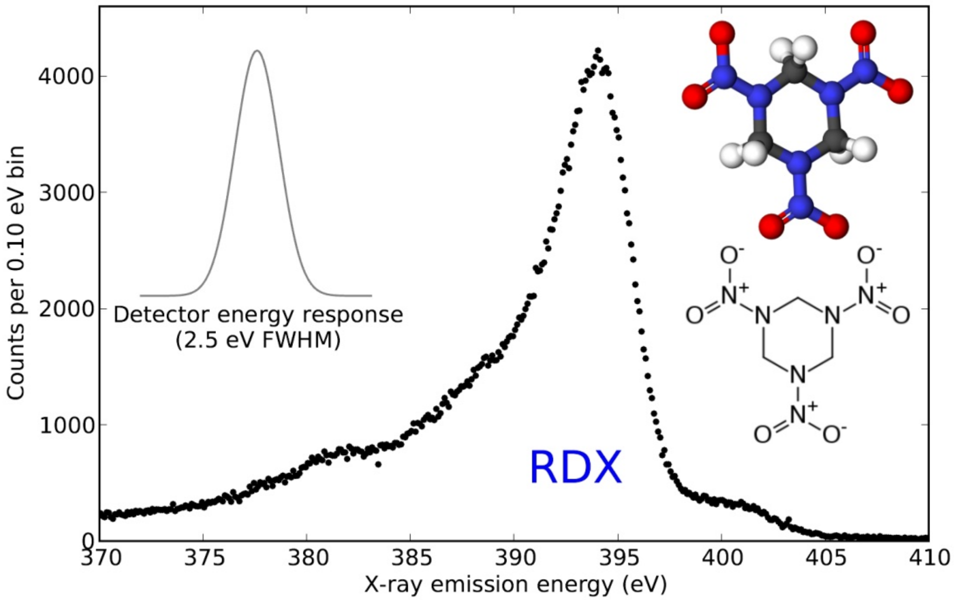
NH_4NO_3 spectrum acquired in 29 min.

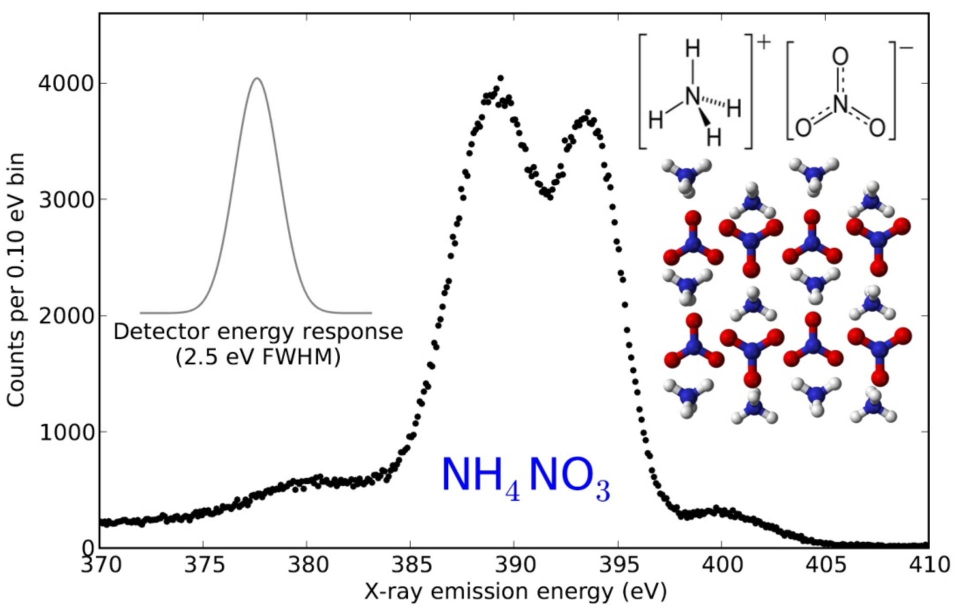
chemical-shift XES



zoom in on nitrogen peak in each spectrum:

chemical-shift XES





RDX is clearly distinguishable from NH_4NO_3.

XES is probing the nitrogen chemical environment

TES spectrometers for beamline science: status

successful commissioning of a TES spectrometer at NSLS

- 2.5 eV ΔE at a synchrotron facility by an energy-dispersive x-ray spectrometer
- chemical-shift emission spectroscopy
- fluorescence-yield absorption spectroscopy

TES spectrometers for beamline science: status and future

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larger spectrometers under development

- ΔE ~ 0.5—1 eV (E_γ < 700 eV)
- 256 pixels later this year
- 1,024 pixels near term
- 100—300 kHz count rate

permanent general-user instrument for NSLS-II (2015)

spectrometer visit to Advanced Photon Source (Argonne National Lab)--fall 2013

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more generally, TES spectrometers provide:

- very high E/ΔE
- broadband spectral coverage
- much higher efficiency (more than 100x higher at ~5 keV)
  - new science at dimmer beamlines
  - ability to probe radiation-sensitive samples
  - time-resolved measurements

fin

WARNING: YOU ARE ABOUT TO ENTER THE BONUS MATERIAL SECTION
ENTER AT YOUR OWN RISK

comparison to state-of-the-art grating spectrometer

ALS 8.0.2 VLS working at S $L_{2,3}$, C K, N K, O K (150-525 eV)

	ALS 8 VLS	**10^3 pixel TES**
Solid angle	1.6×10^{-5} of 4π sr (less for any single E)	2×10^{-3} of 4π sr
Efficiency	about 5%	7.5% at C K 33% at N K 58% at O K
E/ΔE	> 1.2×10^3	about 5×10^2

grating #'s from Fuchs, RSI, 2009

comparison to state-of-the-art crystal spectrometer

ESRF ID26 working near 6 keV

	ESRF ID26	10^3 pixel TES
Solid angle	1.6×10^{-3} of 4π sr	2×10^{-2} of 4π sr
Efficiency	about 10% (also, must scan)	100%
$E/\Delta E$	around 10^4	$3\text{-}4\times10^3$

crystal #'s from P. Glatzel

strengths of x-ray TESs for beamline science

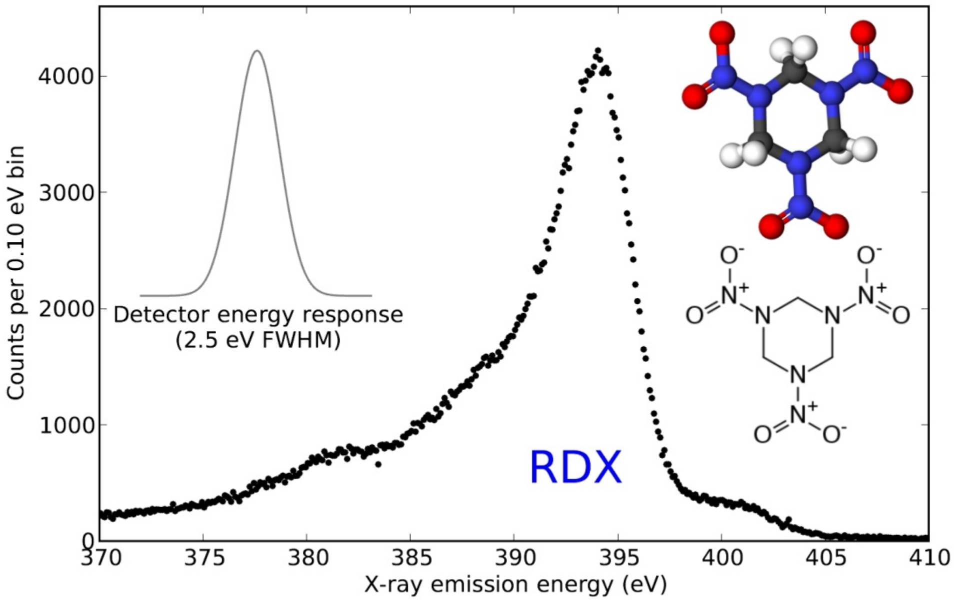
- E/ΔE not quite as good as xtals and gratings, but often good enough

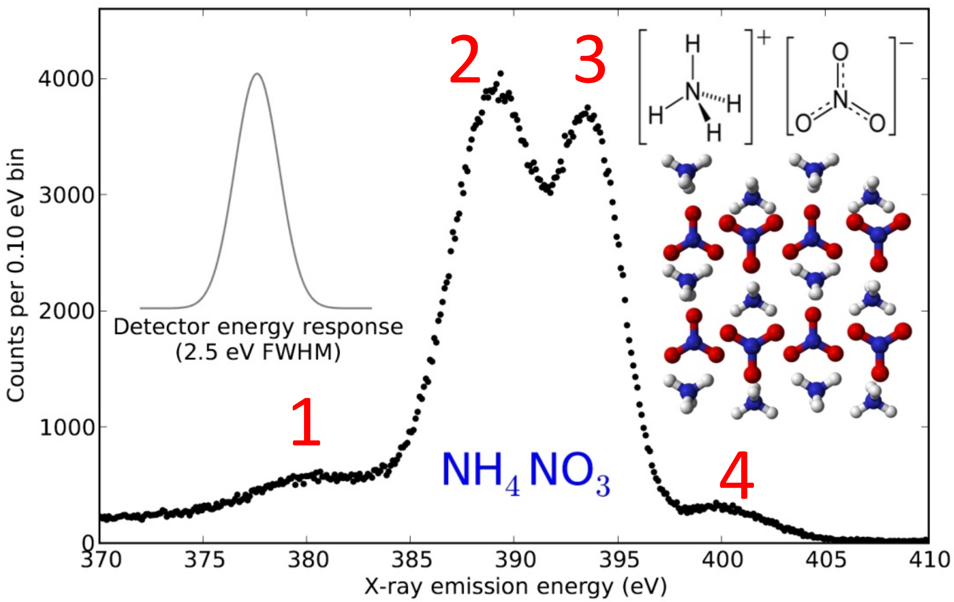
- Much higher efficiency than xtals or gratings (more than 100x higher)
  - New science at dimmer beamlines
  - New science on radiation sensitive samples
  - Time-resolved measurements

- Possible saturation (1-3 $\times 10^5$ cnts/sec) so may not be needed at brightest beamlines with rad hard samples

- Ease-of-use much better than xtals or gratings
  - Physically small, and can approach sample
  - Broadband - don't need different crystals for each element, or K_α, K_β
    can easily work near 2 keV between grating and xtal ranges
  - Same instrument can do emission or absorption spectroscopy

chemical-shift XES





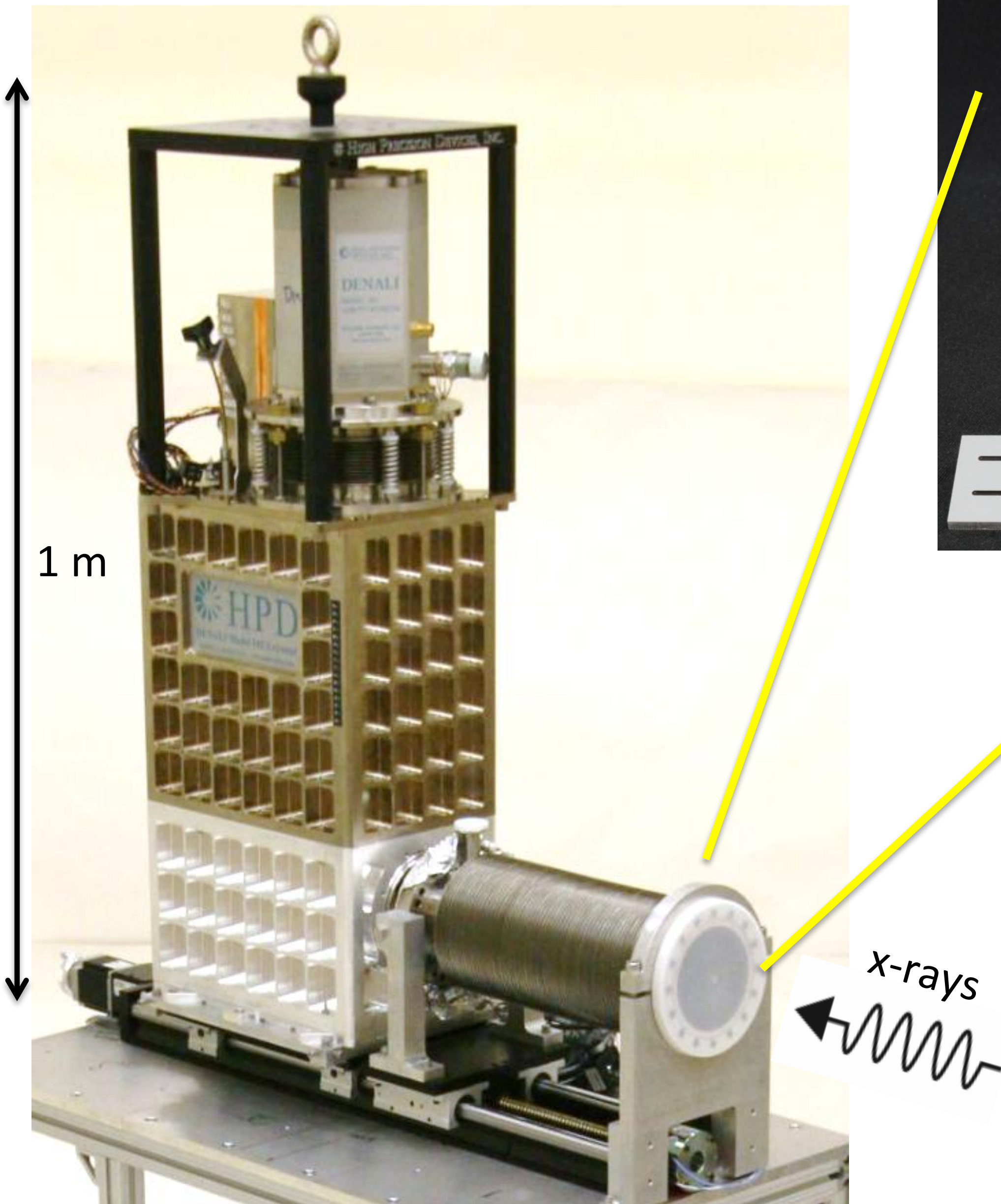
further, NH_4NO_3 has four resolvable features that are associated with:

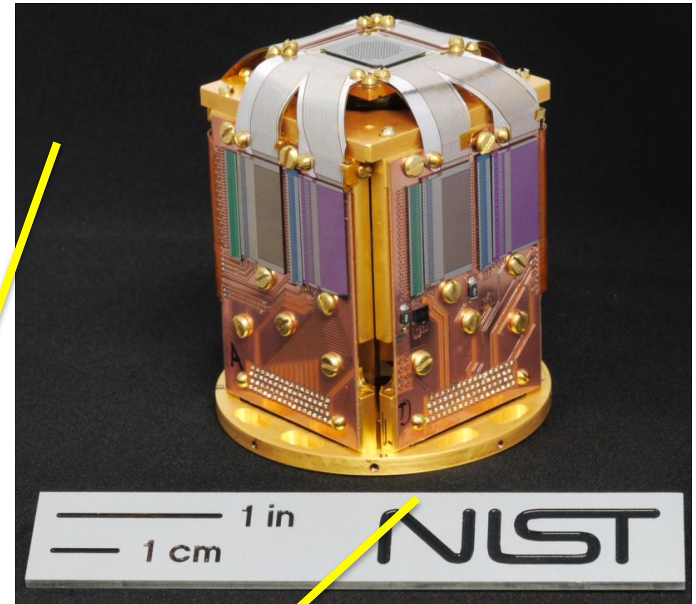
- NH_4^+ (highly reduced) (2, 3)
- NO_3^- (highly oxidized) (1, 3, 4)

(feature ID's from F.D. Vila, et al., *J Phys. Chem. A*, 115, 3243-3250 [2011])

XES is probing the local chemical environment

TES soft x-ray synchrotron spectrometer

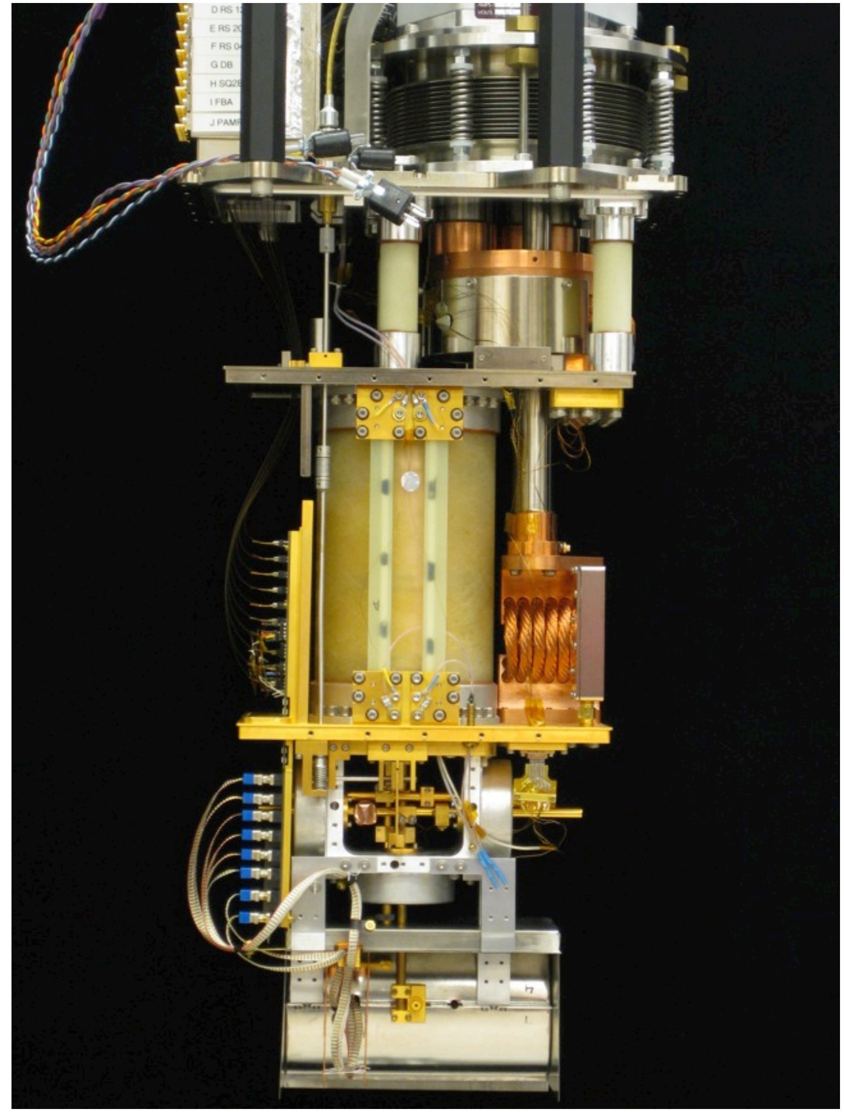




- compact 50 mK refrigerator
- pulse tube cooled ADR
- no liquid cryogens
- translation stage and bellows
- mates to a 4” ID of 6” conflat flange

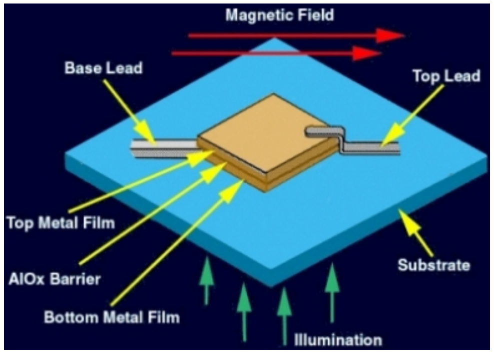
Commercialization of robust refrigerators

- Simple, robust cryogenics required for dissemination of cryogenic sensors
- Designed & commercialized pulse tube-cooled adiabatic demagnetization refrigerator compatible with up to 256 pixels
- Several 10's of units sold worldwide
- Taking steps towards next-generation refrigerators compatible with kilopixel arrays



Types of low temperature detectors

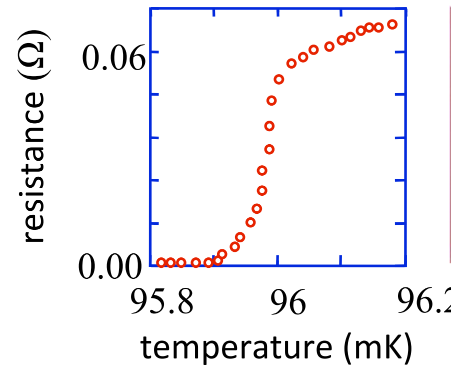
Superconducting Tunnel Junctions (STJs)

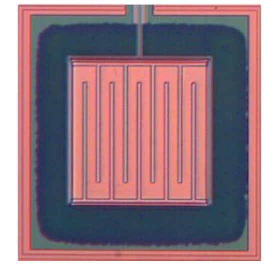


- absorption of photon breaks Cooper pairs: Δ ~ 1 meV
- junction formed from pair of superconducting electrodes separated by an insulator
- signal = electronic tunneling across junction
- ~40 pixel measurements from LLNL group, 100 pixels at AIST

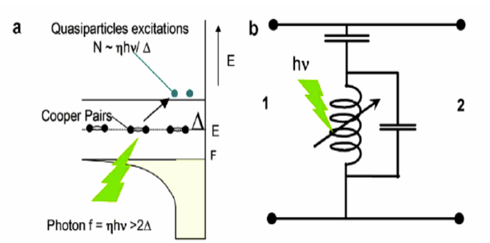
Transition Edge Sensors (TESs)

Thermal sensors

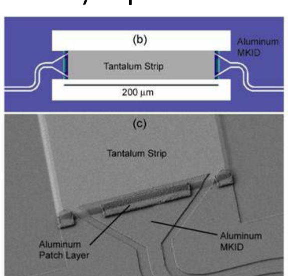




Microwave Kinetic Inductance Detectors (MKIDs)



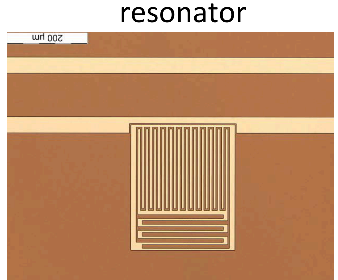
UCSB (Mazin) separate absorber



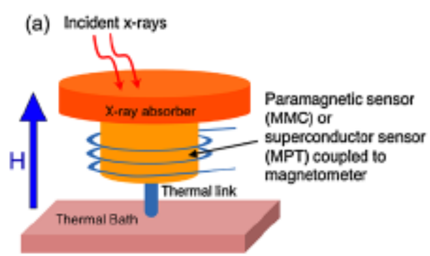
-pair breaking changes surface impedance of superconductor

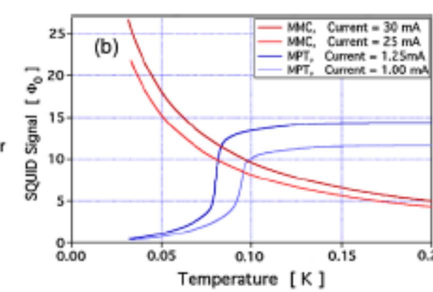
-film embedded in µwave resonator

APS (Miceli) lumped element resonator



Magnetic MicroCalorimeters (MMCs)





- Deposited energy changes paramagnetic or diamagnetic response
- SQUID sensor sees this as change in flux
- No Johnson noise

Types of low temperature detectors

Superconducting Tunnel Junctions (STJs)

- ΔE ~ 5 eV @ 1 keV, > 10 eV above 1 keV
- High per pixel count rates: ~ 10 kHz
- No multiplexing technology:
  arrays limited to ~10^2 pixels
- No leverage from other applications

Transition Edge Sensors (TESs)

- Best achieved resolutions:
  ΔE =1.6 eV @ 6 keV, 22 eV @ 97 keV
- Per pixel count rates: ≤ 300 Hz
- Largest achieved arrays: 256 (X) 10^4 pixels (Far IR)
- Compatible w/ µwave readout: path to 10^5 pix arrays
- Most efficient use of readout bandwidth:
  hybrid multiplexer architecture: 10^6 pix arrays
- More complicated fabrication
- Extensive leverage from other applications

Microwave Kinetic Inductance Detectors (MKIDs)

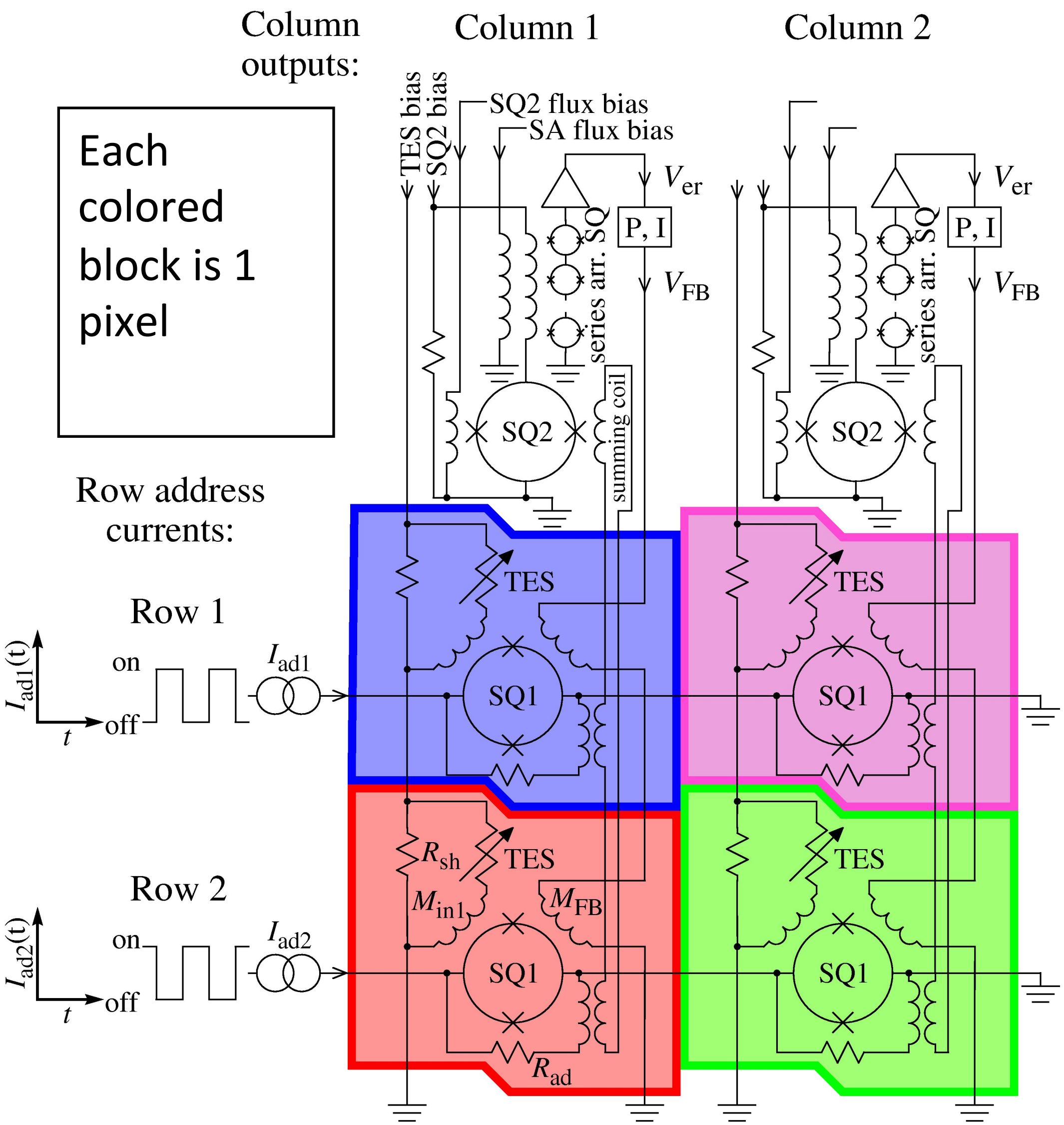
- Successful in Far IR and visible
- Achieved X-ray resolution: 62 eV @ 6 keV
  obstacles to very good ΔE, solvable?
- Per pixel count rates: 500-1000 Hz
- Achieved arrays: few (X), 10^3 (visible, Far IR)
- Compatible w/ µwave readout:
  path to 10^5 pix arrays
- Simpler fabrication

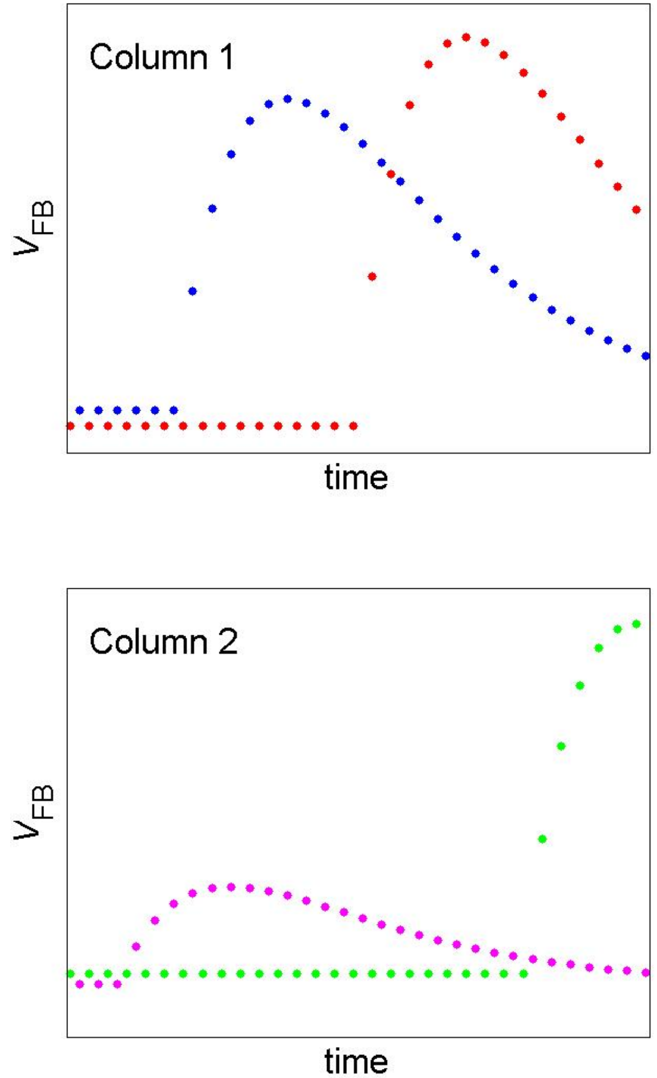
Magnetic MicroCalorimeters (MMCs)

- Achieved resolutions similar to TES
- Resolution limits ~ 2x better than TES
- Count rates similar to TES
- Very early multipixel demonstrations
- Multiplexing possible, harder than TESs
- Readout easily degrades resolution
- Compatible w/ µwave readout: 10^5 pix arrays
- Fabrication similar to TES

The NIST TDM architecture

Each colored block is 1 pixel





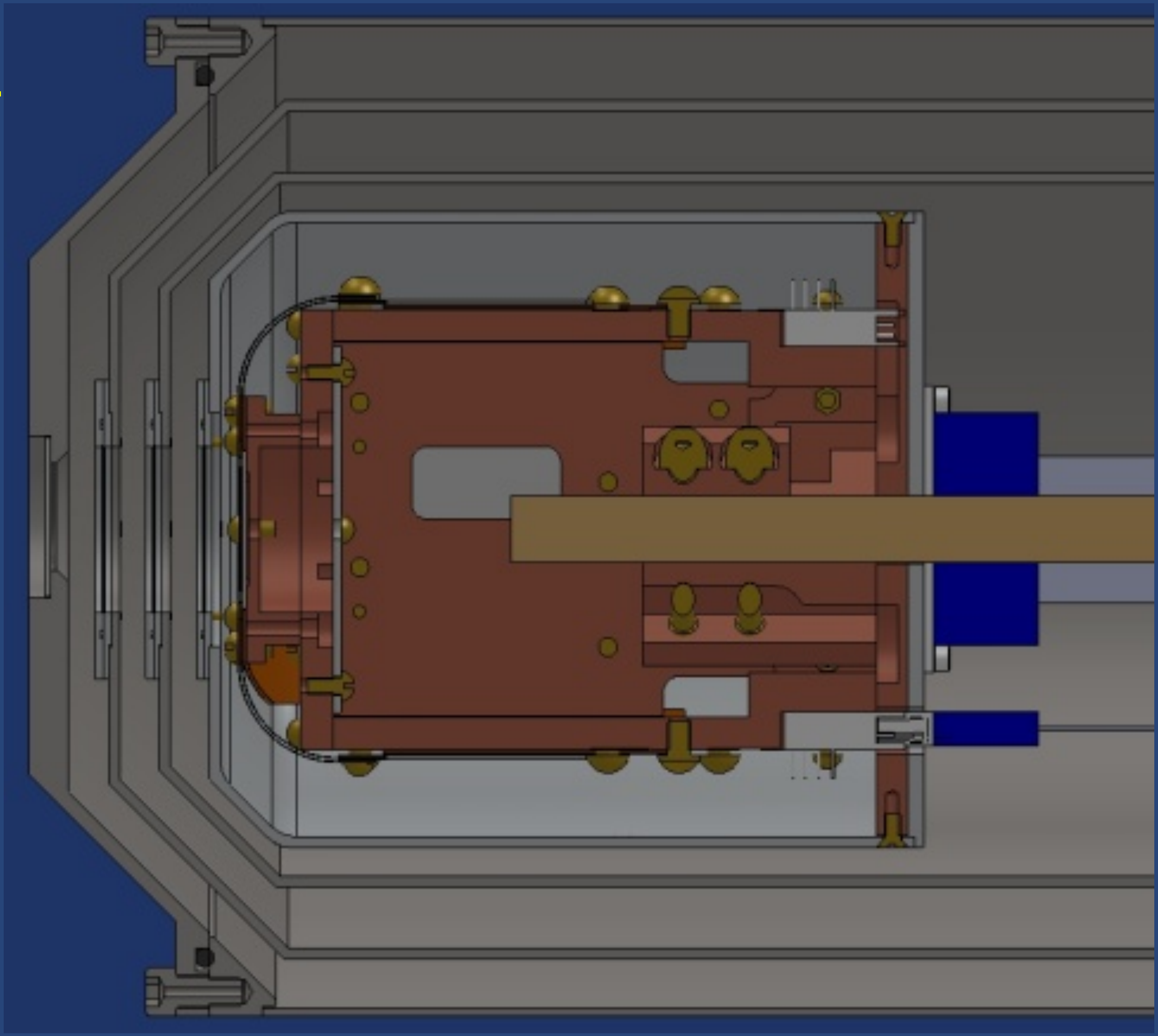
IR filters and vacuum window

<u>Al IR-blocking filters:</u>

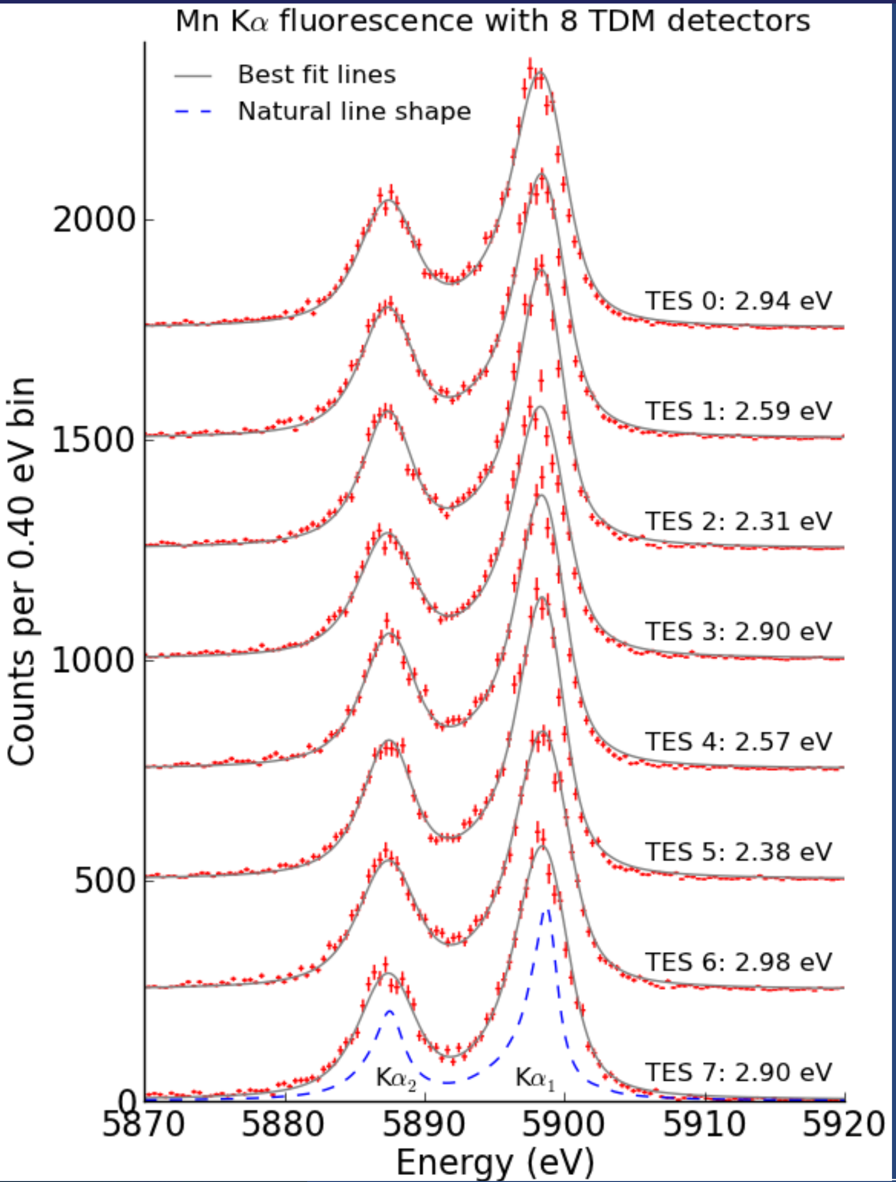
- 3x 1000Å
- 50 mK, 3K, 60K
- free-standing (no polymer)
- Luxel

<u>vacuum window:</u>

- Moxtek AP3.3 (polymer)
- will be removed soon



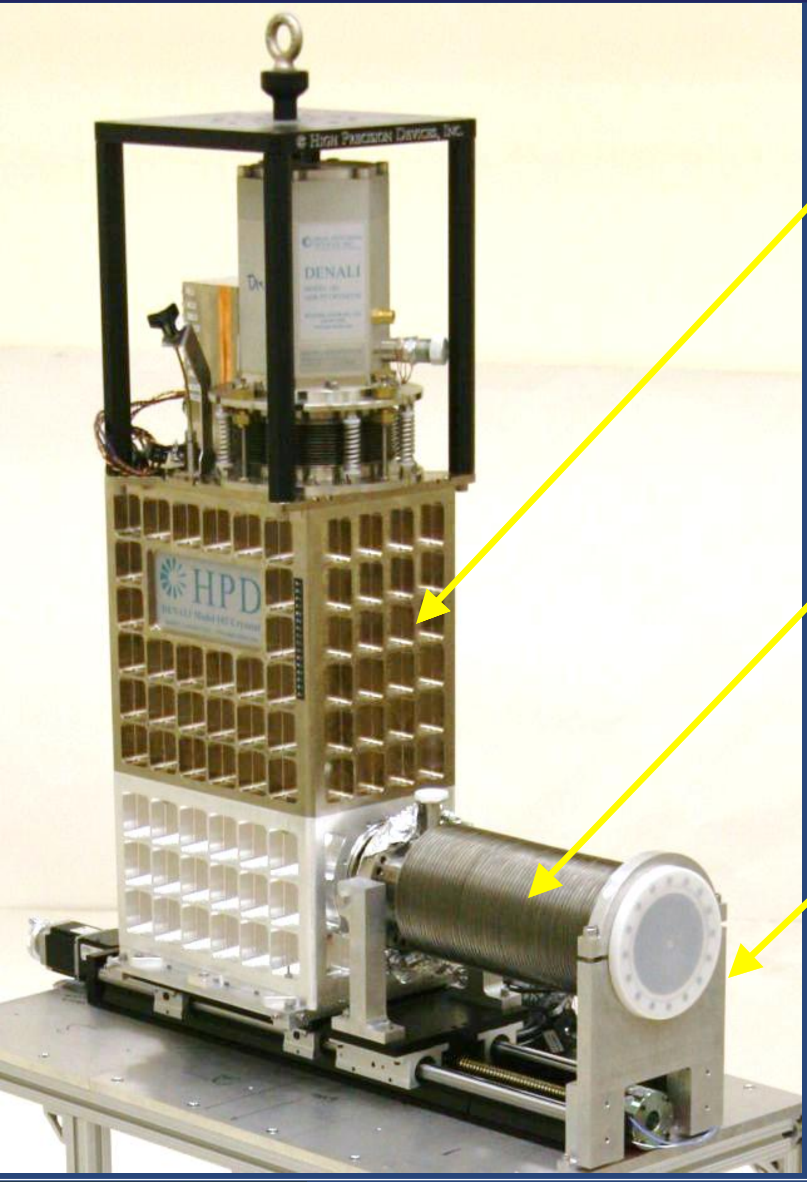
achieved energy resolution



testing with X-ray tube source (fluoresced Mn; 5.9 keV) in our lab in Boulder:

- 8 individual sensors: $\langle \Delta E_{FWHM} \rangle = 2.7$ eV
- best TES: 2.3 eV

cryostat integration with beamline



- High-precision devices (exhibitor here at ASC2012) built pulse-tube cryostat with two-stage (FAA / GGG) ADR.
- bellows-mounted on translation table for entry into UHV sample chamber
- 256 pixel 50 mK stage and 3 concentric radiation shells go through 6” OD (100 mm ID) ConFlat flange