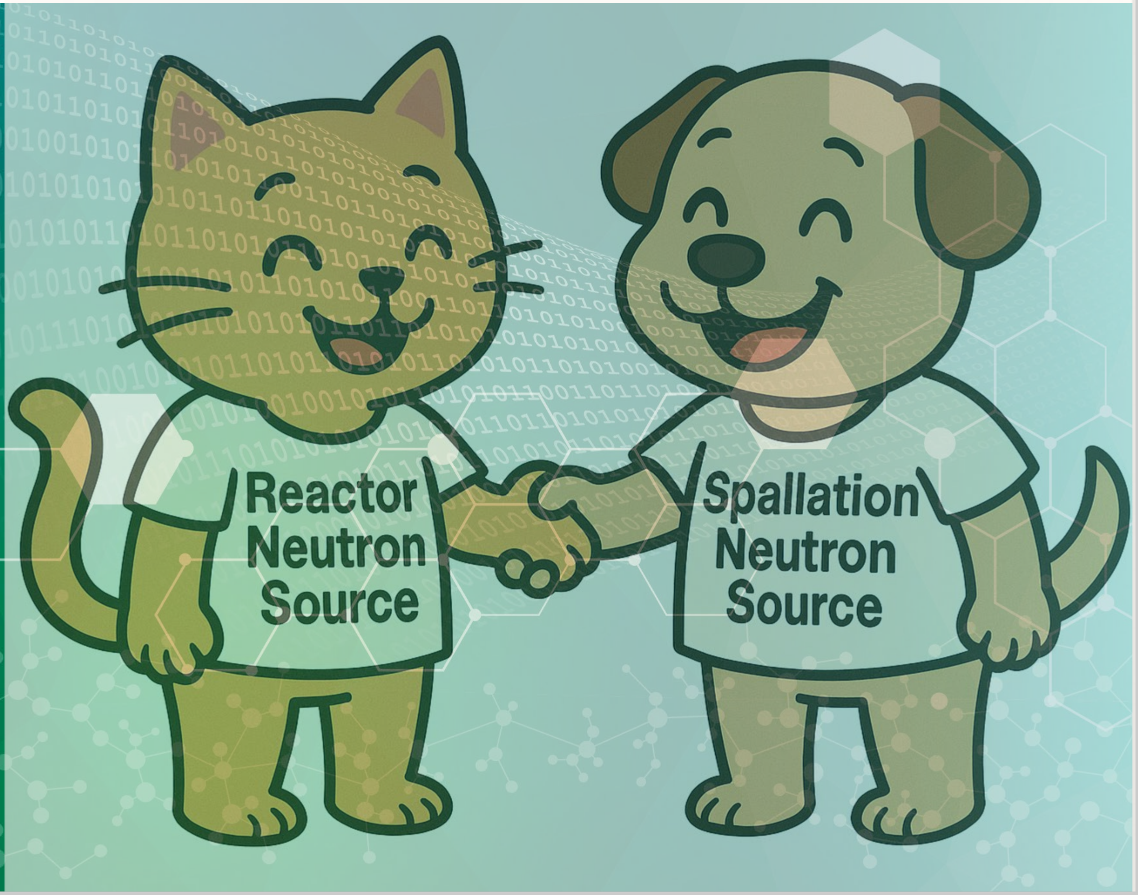
OAK RIDGE National Laboratory

Diffraction at Time-of-Flight (TOF) ~~vs~~ **and** Continuous Sources (CS)

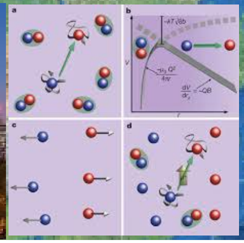


Stuart Calder

Neutron Scattering Division
Oak Ridge National Laboratory

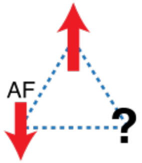
How I got here?

Undergraduate in Physics
University of Edinburgh



Postdoc at ORNL

PhD at University College London
Investigation of frustrated Magnetic Materials with neutron scattering



ISIS, UK

ILL, France

Current role:

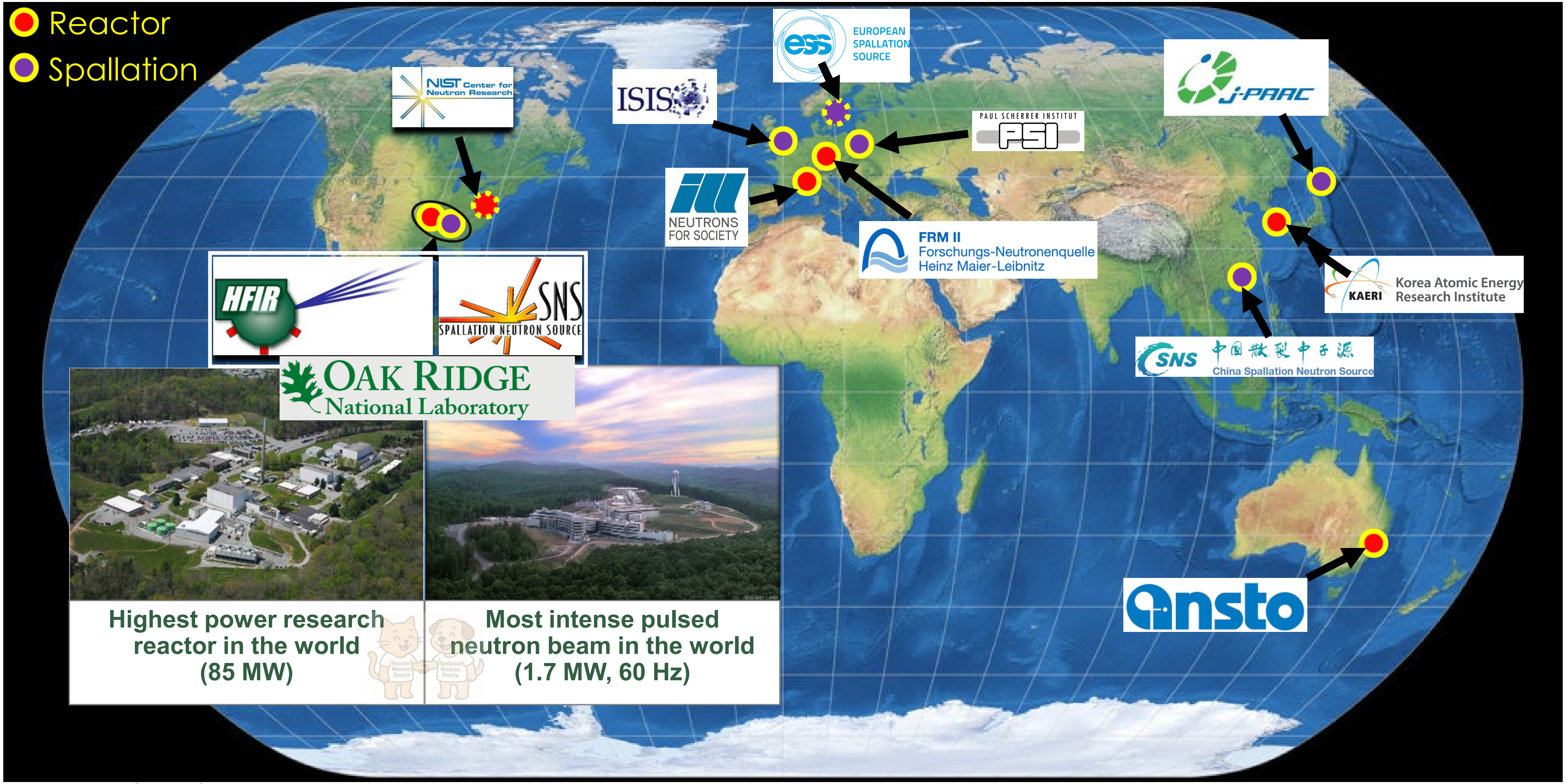
- **Instrument Scientist on HB-2A Neutron Powder Diffractometer**
- **Research on magnetic materials**
- **Utilize the diverse neutron instruments at SNS/HFIR**
- **Collected data on 8/12 instruments at HFIR and 9/18 at SNS.**

Presentation Overview: Diffraction at TOF ~~vs~~ and CS

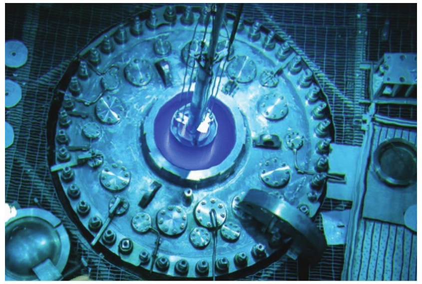
- General characteristics of TOF and CS
  - ORNL has both → use both!
- How the different sources impacts diffraction instruments
- Strengths for different experiments
- Consider which source is best suited to your science

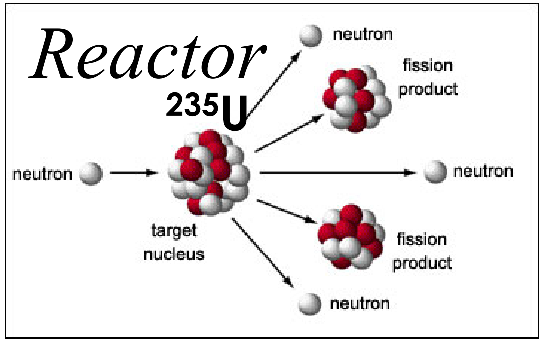
Always talk to an instrument scientist

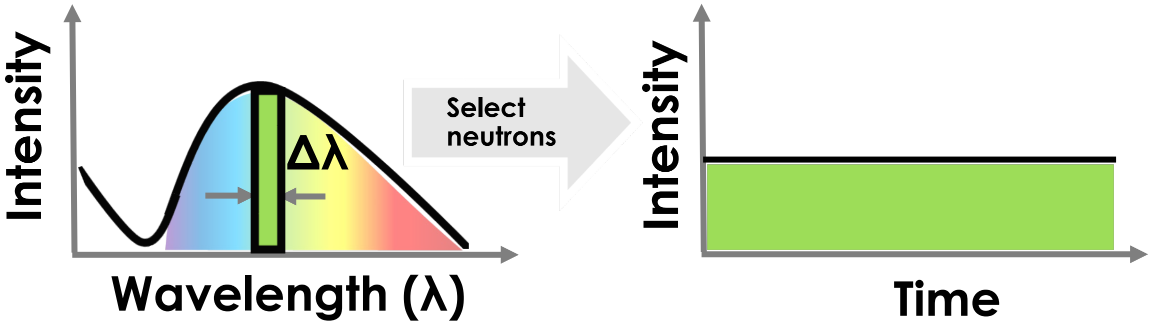
Neutron Sources around the world



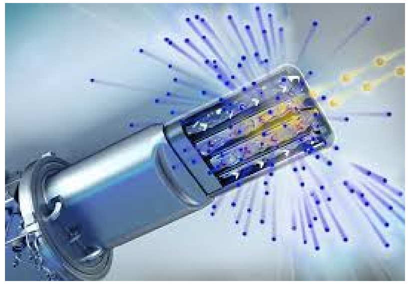
Neutron sources:
Reactor (CS) and Spallation (TOF)

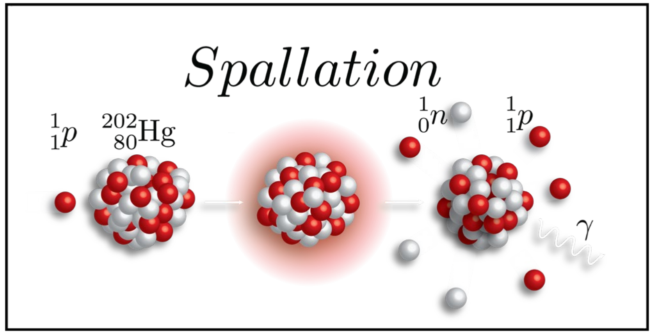


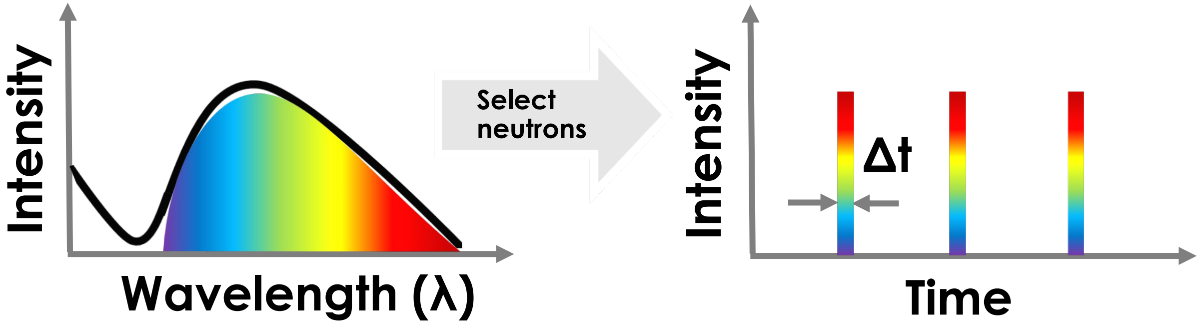




Small Δλ used, but source on all the time







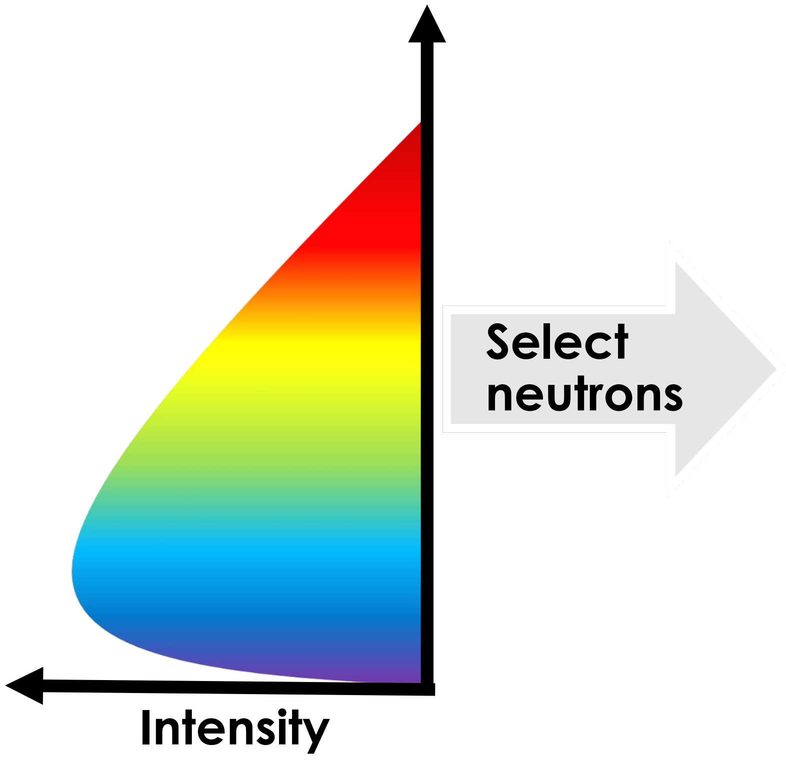
Each pulse of neutrons contains a broad spectrum of energy (λ)

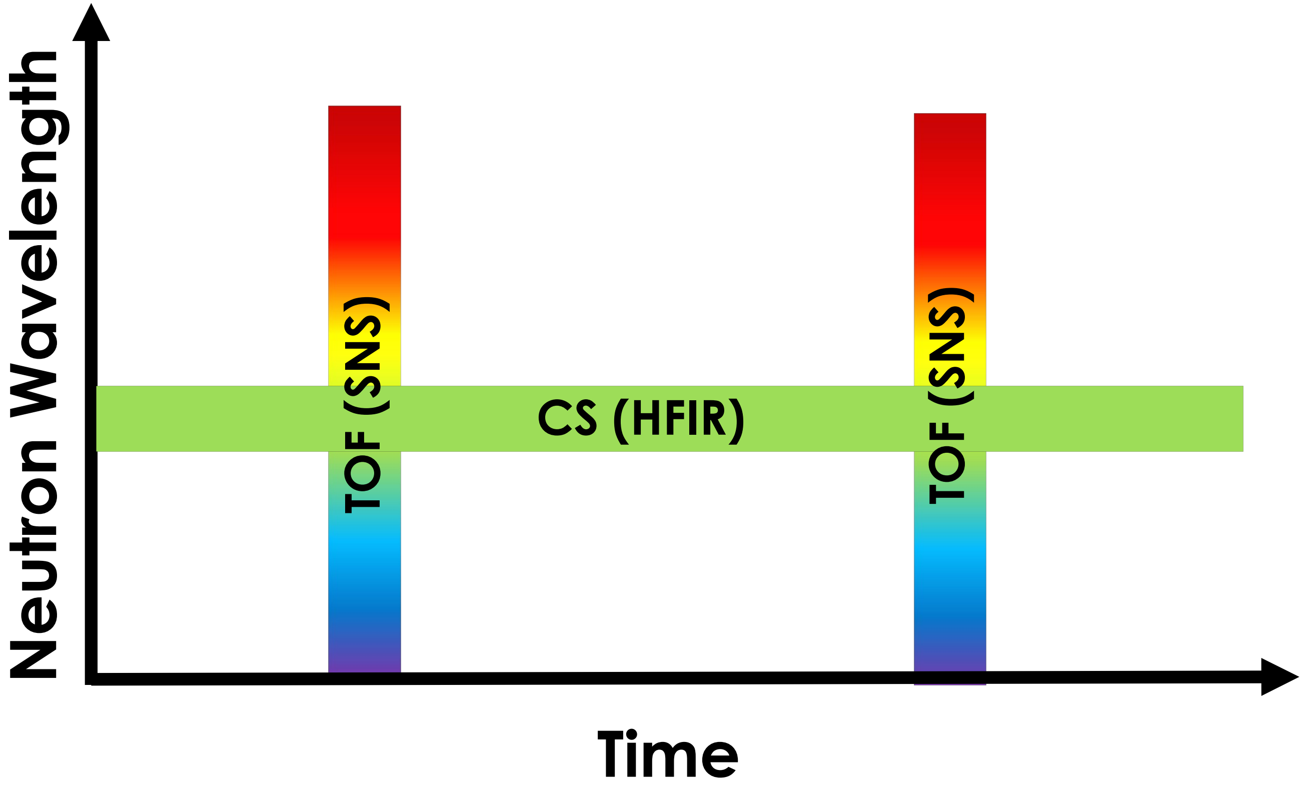
Pulse of neutrons ~60 times per second

How do you like your neutrons?

CS (HFIR):
Some of the neutrons λ all the time?

TOF (SNS):
All the neutrons λ some of the time?





Neutron sources:
Reactor (CS) and Spallation (TOF)

CS (HFIR):
High flux over a chosen region.

TOF (SNS):
Large coverage.

What does this mean for diffraction experiments?

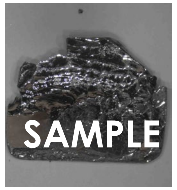
A typical CS diffractometer (HFIR)

Reactor

Monochromator:

- **selects λ.**
- **Control resolution.**

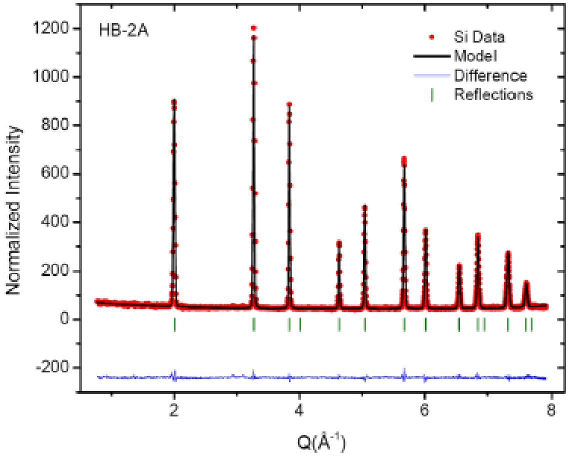
Single wavelength



Detector at 2θ measures single Q

λ = 2d sin θ

Q range limit is 4π/λ

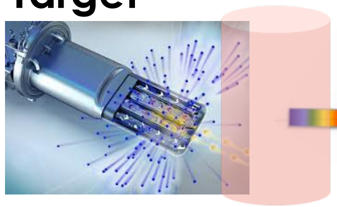


A typical TOF diffractometer (SNS)

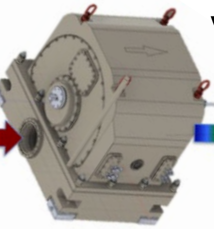
De Broglie wavelength: λ = h/mv = ht/mL

Bragg's law: λ = 2d sin θ = constant * t

Target



Moderator



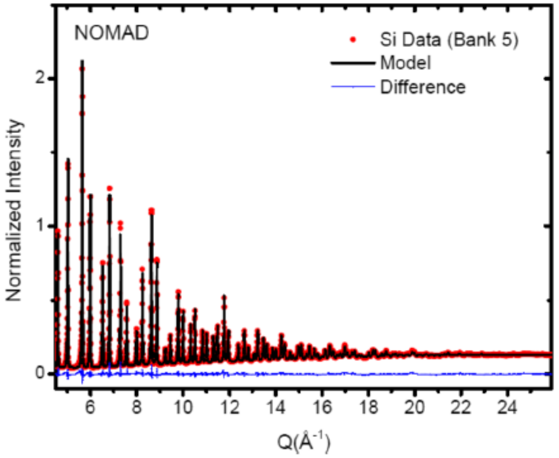
Chopper

Multiple wavelengths

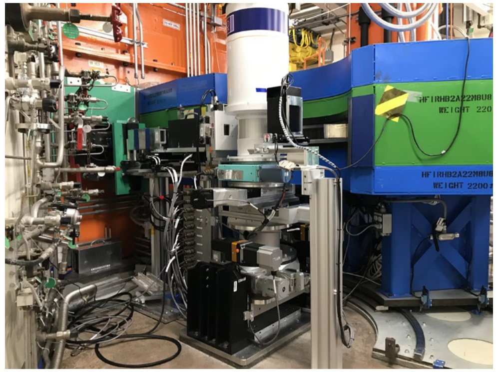
Detector at 2θ measures multiple Q

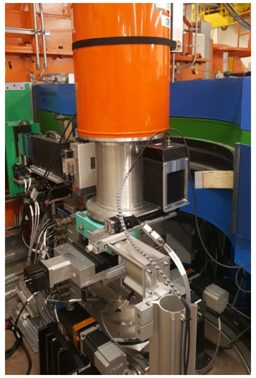
Resolution depends on moderator and t,L, and 2θ

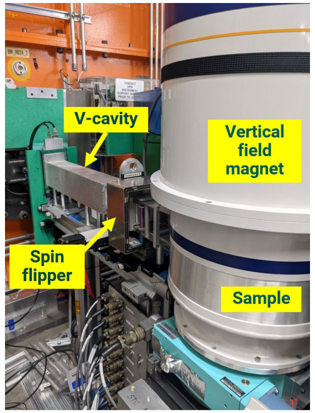
Q range determined by λ_{min}, λ_{max} and 2θ



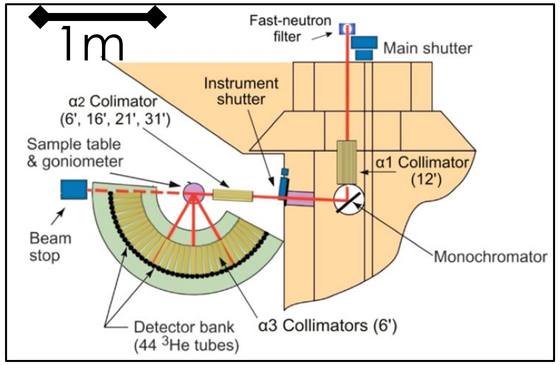
A typical CS diffractometer (HB-2A, HFIR)





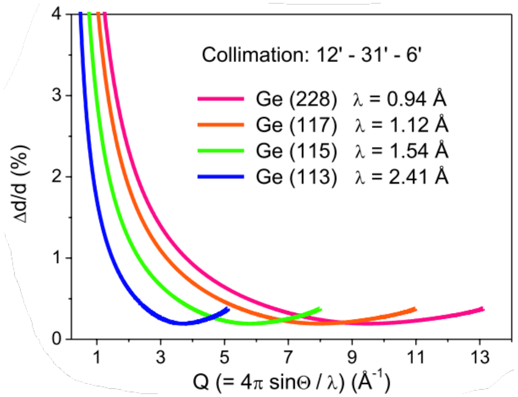


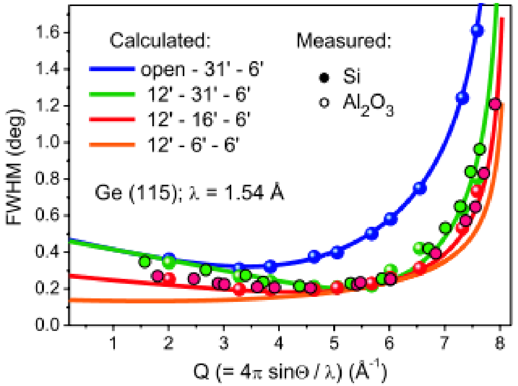
Versatile: open sample space and compact



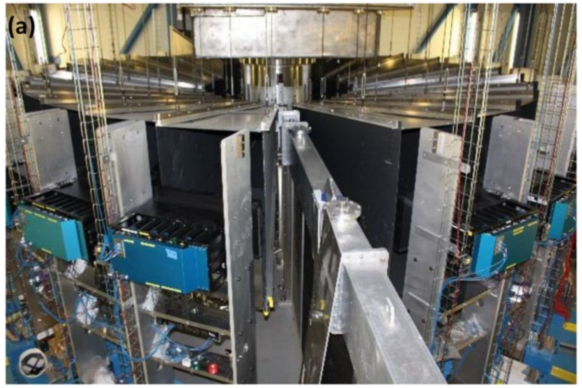
Ge(hkl)	λ (Å)	Q ($Å^{-1}$)	Flux (n/cm^2s)
(113)	2.41	0.2 - 5.1	5 x 10^6
(115)	1.54	0.35 - 7.9	1 x 10^7
(117)	1.12	0.5 - 10.9	4 x 10^6

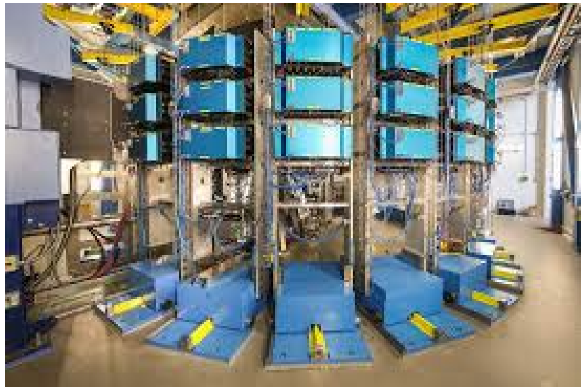
Resolution:
best resolution Δd/d: 1x10^{-3}
Dependent on wavelengths and *d*





A typical TOF diffractometer (POWGEN, SNS)

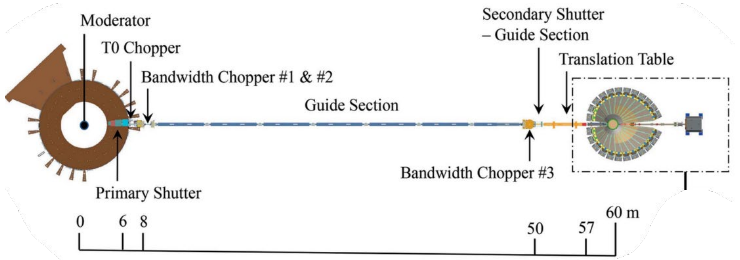


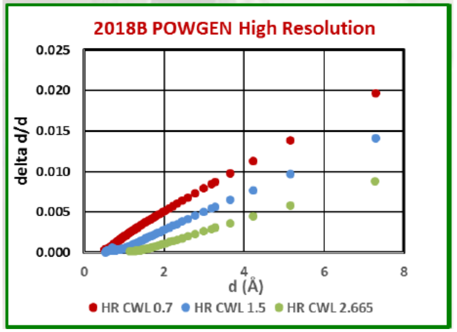


Larger, more detector coverage

Freq (Hz)	WL center	WL min	WL max	dmin	dmax	Qmin	Qmax	Bank
60	0.533	0.15	1.066	0.075	7.50	0.82	83.45	0
60	0.800	0.27	1.333	0.134	8.00	0.76	46.88	1
60	1.500	0.97	2.033	0.485	13.00	0.48	12.95	2
60	2.665	2.13	3.198	1.070	21.00	0.30	5.87	3
60	4.797	4.26	5.33	2.140	38.00	0.17	2.94	4

Resolution: *best resolution Δd/d: 1x10^{-4}*
Dependent on wavelengths and *d*





CS ~~vs~~ and TOF: Factors to consider for a diffraction experiment

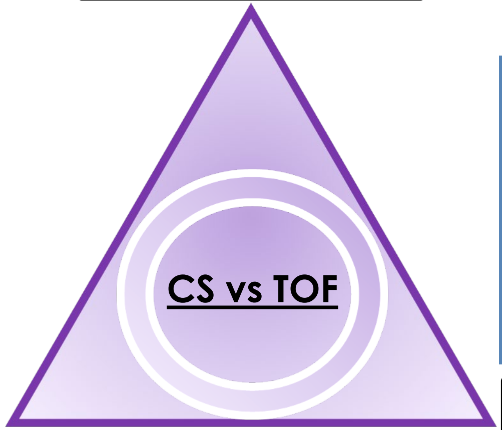
Intensity (CS)

- High flux
- Tune intensity with monochromator, collimators

Intensity (TOF)

- High peak brightness
- Balance of instrument distance, bandwidth, coverage

Intensity



Q-resolution (TOF)

- Highest resolution
- Complex asymmetric peak shape related to moderator characteristics

Q-resolution

Q-resolution (CS)

- Tunable over Q-range
- Simple, symmetric peak shape function

Q-range (TOF)

- Wide Q coverage
- Q_{max} can be very high (>100 Å^{-1})
- Good coverage with even limited detectors

Q-range

Q-range (CS)

- Q range limited, but tunable
- Limited Q_{max} (~20 Å^{-1})
- Need wide/continuous detector coverage

https://addie.ornl.gov/data_viz_hfirestimate

Do simulations. Lots of tools!

ADDIE | Home | About | Scattering Tools | Instrument Tools | General Tools | External Link

ExperimentSimulator

Given a cif file and necessary sample information, estimate the signal level (with uncertainties) for HFIR powder diffraction instruments.

The underlying GSAS-II engine can ONLY process properly structures with origin choice 2. Please make sure your uploaded structure is with the proper choice of origin.

Upload Data

Select CIF file: Choose File MnO_Fm-3m.cif

Select an instrument config: HB2A-Ge113-WL2p41-CollOut

Container Inner Diameter (cm): 0.6 Show/hide notes

Container Material: V

Sample Mass (g): 20

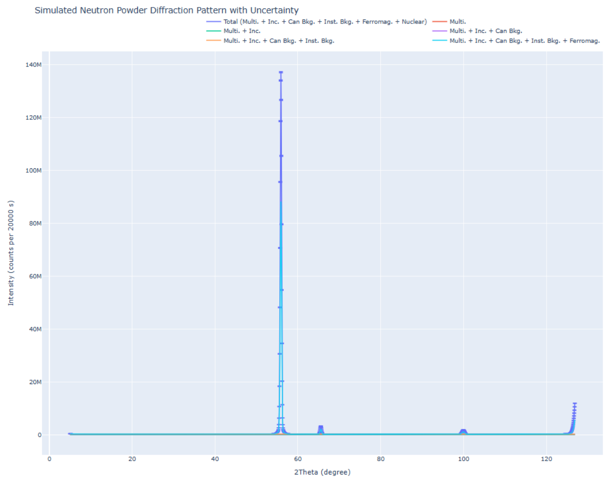
Counting Time (hour): 1

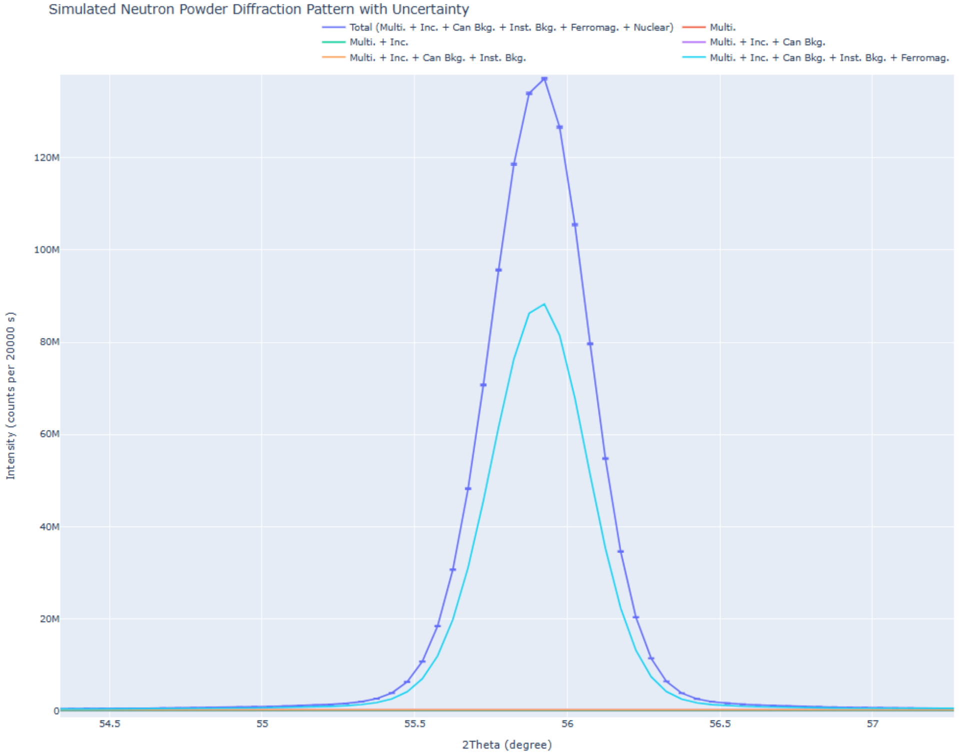
Number of Magnetic Atoms (per unit cell): 4 Show/hide notes

Magnetic Moment (per atom, in muB): 5

Submit

***The backend engine for current interface is developed by Dr. Joe A. Paddison. Frontend interface is implemented by Dr. Yuanpeng Zhang.**





Ask an instrument scientist what tools are available

Some other strengths for diffraction experiments?

CS diffractometer at HFIR	TOF diffractometer at SNS
Main: High Flux beam, versatility	Main: Resolution and wide Q-range
Simple beam profile corrections - Absorbing, attenuating samples and holders	Count rate - Typically larger detector coverage, optimized beams
Open instruments with variable sample environment	High peak brightness
Beam is always on - Time dependent measurements	Pump probe measurements
Polarization is easier	Newer facility, STS

Sample Environment considerations

Other factors

An instrument is only useful if the sample can be measured under the desired conditions.

https://neutrons.ornl.gov/sample

To only show available items for specific beamline, use this filter.

HB-2A

ID	Sample Space Dia.	Temp Range	Instruments	Associated Resources	Subcategory	Description
ULT-E	40mm	0.03 to 300 K	CG-2, CG-3A, CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3	CRYO-A, CRYO-C, CRYO-O, MAG-B, MAG-E, MAG-G	Insert	Dilution Refrigeration Insert
ULT-G	40mm	0.3 to 300 K	CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3, HB-3A	CRYO-A, CRYO-C, CRYO-N, CRYO-O, MAG-B, MAG-I	Insert	Helium-3 Insert
ULT-H	40mm	0.03 to 300 K	CG-2, CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3, HB-3A	CRYO-A, CRYO-C, CRYO-N, CRYO-O, MAG-B, MAG-E, MAG-G, MAG-I	Insert	Dilution Insert
ULT-J	60mm	0.03 to 300 K	CG-4C, HB-1, HB-1A, HB-2A, HB-2C, HB-3	CRYO-D, CRYO-J, CRYO-K, CRYO-L, CRYO-M	Insert	Dilution Insert
ULT-K	40mm	0.3 to 300 K	CG-2, CG-4C, HB-1, HB-1A, HB-2A	CRYO-A, CRYO-N, CRYO-O, MAG-E, MAG-G, MAG-I	Insert	Helium-3 Insert
ULT-M		0.3 to 300 K	HB-2A, HB-2C, HB-3, HB-3A		Bottom Loading	

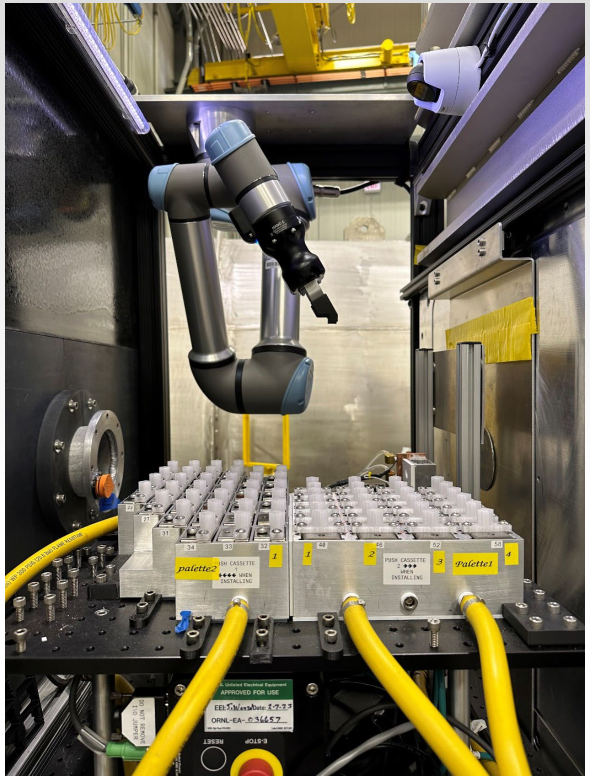
Equipment

- Low Temperature & Magnetic Fields
- High Temperature & Engineering Materials
- High Pressure & Gas Handling
- Special Environments

Ask an instrument scientist what is available

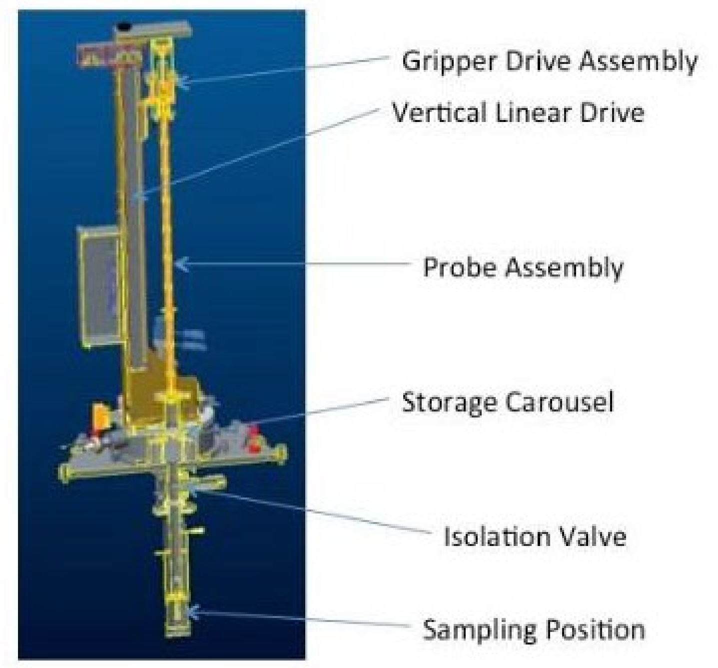
Types of Sample Changers

External Sample – External Manipulation



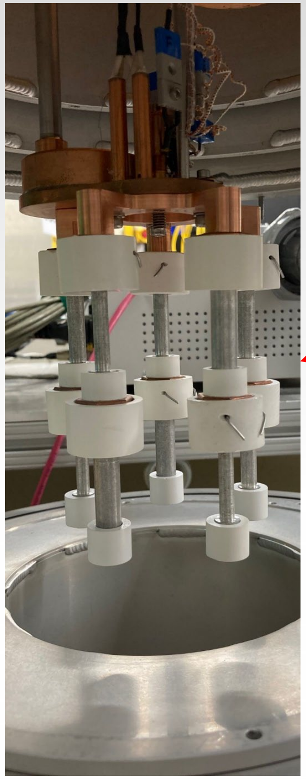
- 90 K to 573 K
- Open air environments
- Fast to load

Internal Sample – Internal Manipulation



- 20 K – 300 K
- Only CCR range (not below 1 K)
- Slow to load

Internal Sample – External Manipulation



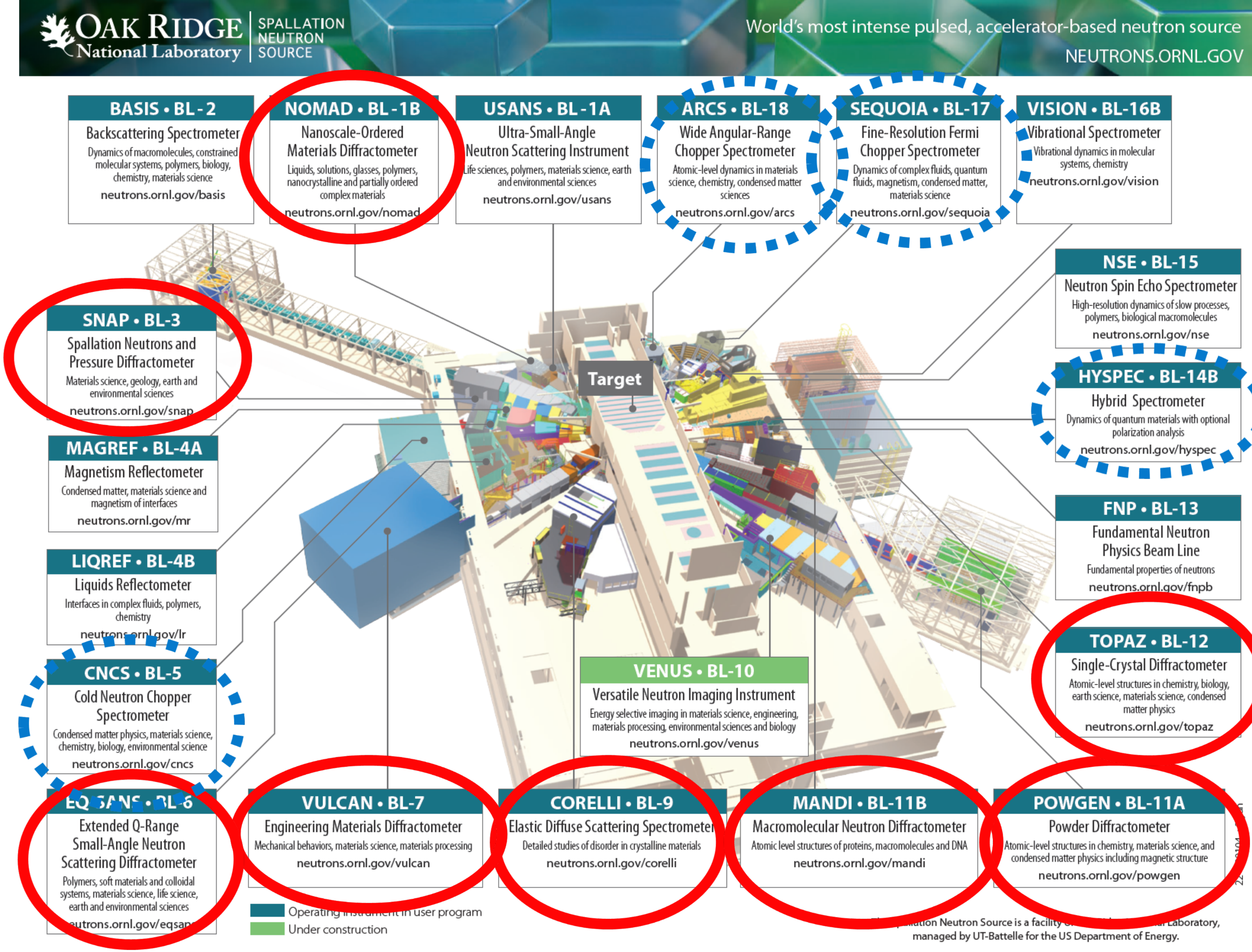
- 0.04 K to 700 K
- Full range plus magnets
- Slow to load, do it offline

Lots of diffraction instruments at HFIR/SNS

SNS (TOF)

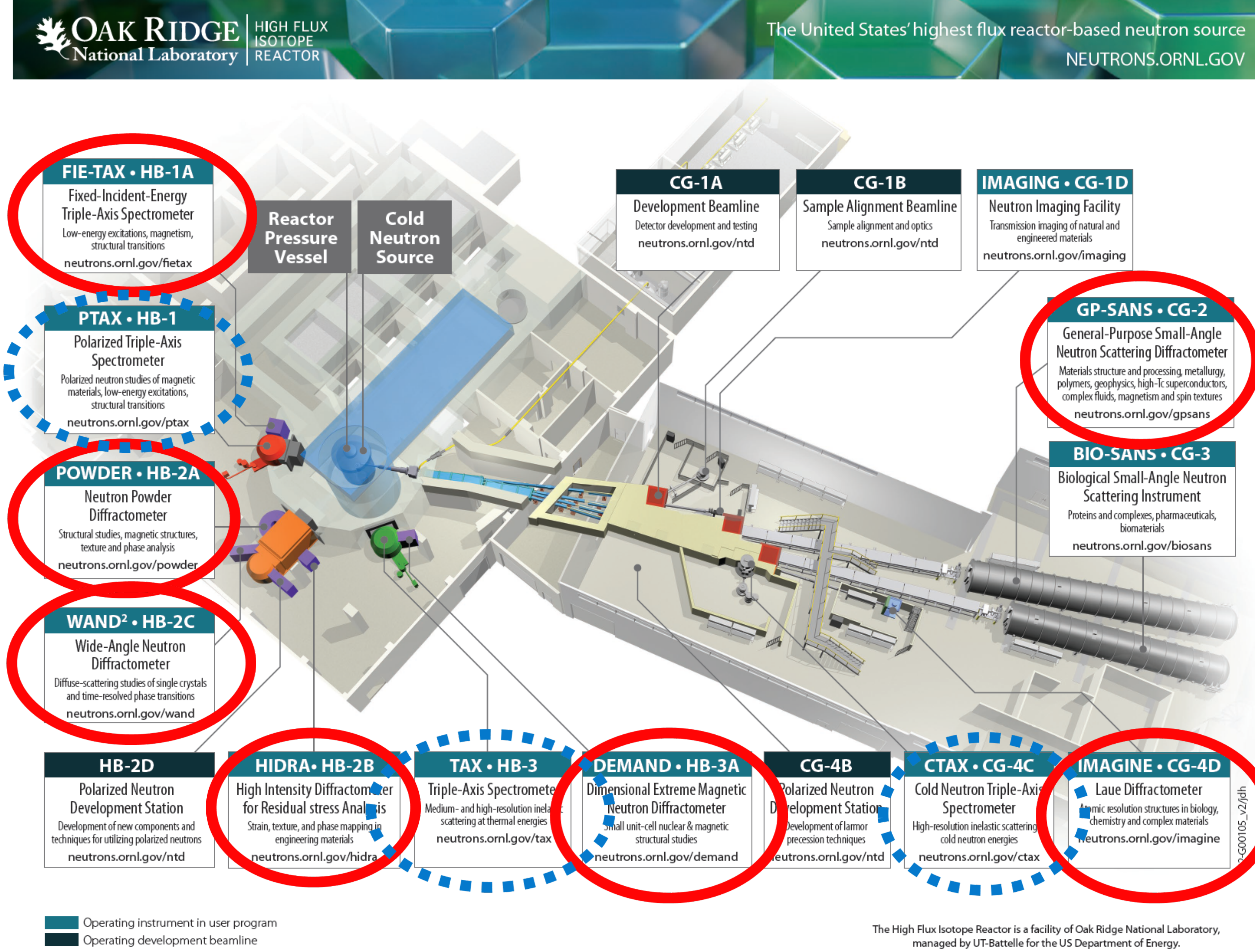
- POWGEN
- NOMAD
- TOPAZ
- CORELLI
- MANDI
- SNAP
- VULCAN
- EQ-SANS

  - HYSPEC
  - ARCS
  - SEQUOIA
  - CNCS



HFIR (CS)

- HB-2A
- DEMAND
- IMAGINE
- $WAND^2$
- VERITAS (HB-1A)
- HIDRA
- GP-SANS
  - HB-1
  - HB-3
  - CTAX



 https://neutrons.ornl.gov/suites/diffraction Created in 2019

Instrument	Type	Methods	Applications	T (K)	P (GPa)	H (T)	Special	Resolution (Δd/d)	Q-range (Å-1)	Beam size H x V (mm)
HFIR HB-2A POWDER	constant wavelength	Rietveld	Magnetic structures	0.05 - 1800	< 0.6 gas cell < 2 clamp cell	< 7	Electric field	2 x 10-3 (variable)	0.2 - 5.1 and 0.35 - 8	30 x 60
HFIR HB-2C WAND 2	constant wavelength	Rietveld, single-crystal	General purpose powder / single-crystal	0.05 - 1800	< 0.6 gas cell < 2 clamp cell	< 6	Humidity chamber	5 x 10-3	0.1 to 8.2	30 x 40
SNS BL-11A POWGEN	t-o-f	Rietveld, PDF	General purpose powder	10 - 300 (PAC) 2 - 300 (Orange cryo & Slim Sam) 5 - 500 (JANIS) 30 - 750 (JANIS) 300 - 1450 (MICAS) 300 - 1100 (with gas insert) (MICAS)		< 5	24 position sample changer (10 - 300 K), Gas Handling with pO2 and RGA	0.8 x 10-3	0.76 - 46.9 in one frame 0.17 - 46.9 with multiple frames	10 x 40
SNS BL-1B NOMAD	t-o-f	PDF, Rietveld	Disordered materials, glasses, liquids; high flux powder	90 - 1500 (Ar cryostream) 2 - 300 (Orange cryo) 425 - 11450 (ILL furnace, limited measuring time above 1100C) 1000 - 3500 (levitator)	0.02 - 0.2 gas cell (TiZr cell, high background)		Cryostream sample changer; gas handling, batteries	5 x 10-3 to 5 x 10-2	Typical 0.2 - 31.41, low Q limited to 0.4 in furnace and Orange cryo.	Typically 6 x 6 FWHM, Gaussian beam profile, generally 2cm filling height required.
SNS BL-3 SNAP	t-o-f	Rietveld	High pressure studies powder/ single-crystal	85 - 1350 (Paris Edinburgh) 300 - 1500 Paris Edinburgh (high T insert) 2 - 350 Clamp cell (Orange cryo) 10 - 1350 Diamond Anvil Cell (DAC) (bottom loader cryo) 10 - 350 Gas cell / Clamp	< 20 PE cell < 6 PE cell < 2 clamp cell 0.6 Gas cell < 40 DAC			8 x 10-3 (at 2θ = 90°)	0.8-30	Typical, 1 cm2 < 1 mm2 for DACs

https://neutrons.ornl.gov/suites/diffraction

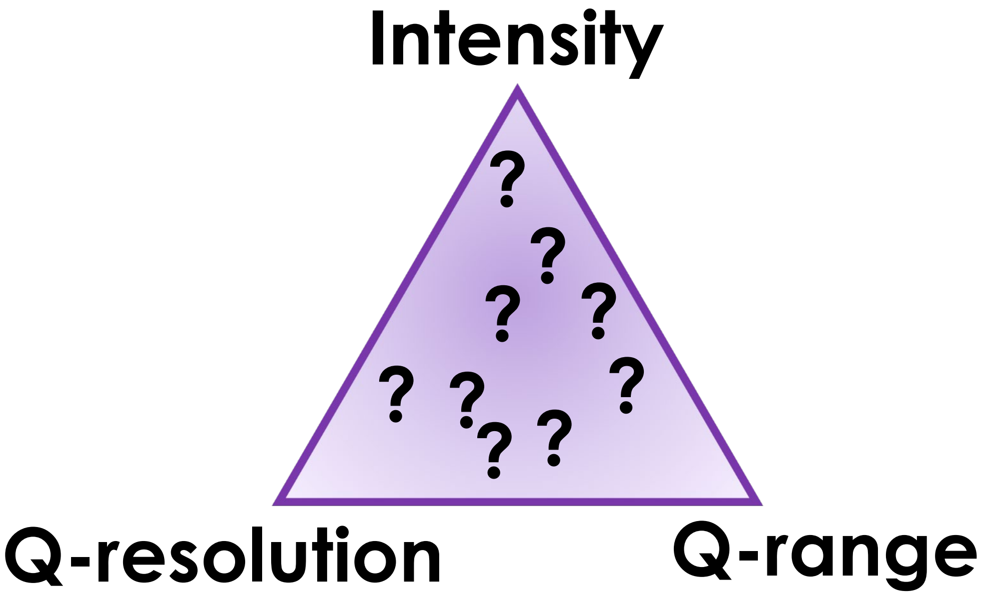
Created in 2019

SINGLE CRYSTAL INSTRUMENTS

Instrument	Type	Applications	T (K)	P (GPa)	H (T)	Special	Q-range (Å-1)	Unit cell size (Å)	Beam size H x V (mm)
HFIR HB-3A DEMAND	constant wavelength	Magnetic / nuclear structures; small unit cells	4 - 1700 CCR 1.6 - 300 OC 0.05 - 300 DF	< 2 clamp cell < 10 DAC*	< 6	Electric current, Tunable beam divergence	0.12-12	< 100	6 mm in diameter
HFIR HB-2C WAND 2	constant wavelength	General purpose powder/single-crystal; small unit cells	0.05 - 11800	< 0.5 gas cell < 2 clamp cell < 5 Cubic anvil cell < 20 PE cell (Room T)	< 6	Humidity chamber	0.1-8.2	< 100	Variable: 10 x 40 to 30 x 60
SNS BL-12 TOPAZ	t-o-f	General purpose; small & large unit cells; diffuse scattering	90 - 1450 LN2 5-300 CCR	None	None	Electric current	0.4-25	< 75	2 , 3 and 4 in diameter
SNS BL-11B MaNDi	t-o-f	Structural biology, macromolecules, small molecules	80 - 400	None	None	Tunable beam divergence 0.12 - 0.8°	0.6 Å max resolution	10 - 300	1, 3 and 5 in diameter
HFIR CG-4D IMAGINE	quasi-Laue	Structural biology, supramolecular chemistry, magnetic / nuclear structures	4 - 1450 CCR 10 - 1300 (DAC)	< 10 DAC	None	Kappa goniometer	1.2 Å max resolution	< 150	3.2 × 2
SNS BL-9 CORELLI	t-o-f	Magnetic / nuclear diffuse scattering	6 - 750 CCR 1.6 - 300 OC 303 - 1873 (MICAS) 0.3 - 300 3He insert	< 1.8 clamp cell < 10 DAC < 4 GPa McWhan	< 5 < 30 Pulsed	Elastic discrimination			10 x 10
SNS BL-3 SNAP	t-o-f	High pressure studies powder / single-crystal	85 - 1300 (PE 10 GPa) 300 - 1500 (PE 6 GPa) 2 - 350 (gas and clamp) 10 - 350 (DAC)	< 40 DAC** < 20 PE cell < 6 PE cell < 2 clamp cell 0.7 Gas cell	None				

Which instrument to choose? TOF or CS or both?

- It depends on the question to be answered in your science
  - Speak to an instrument scientist!
  - Do some homework (simulations, read papers, etc)



Other factors
Sample environment
Polarization
Pump-probe
Source stability
Sample absorption
Source availability
Mail-in
….

Let's consider some common questions diffraction can answer and match to strengths of sources

- Measure nuclear structure
- Measure magnetic structure
- Measure disorder in structure
- … Lots more cases for you to consider!

Measure a crystalline structure

Need
- To collect lots of Bragg reflections

Instrument should have
- High Q-resolution **(TOF/CS)**
- Large Q-range **(TOF)**
- High Q **(TOF)**

- **TOF is typically best, especially for powders.**
- **Single crystal: TOF for largest unit cells, but CS can work well.**

Other factors?

Specialized SE → **TOF/CS**

Absorbing, H →
- **CS easier**
- **TOF feasible**

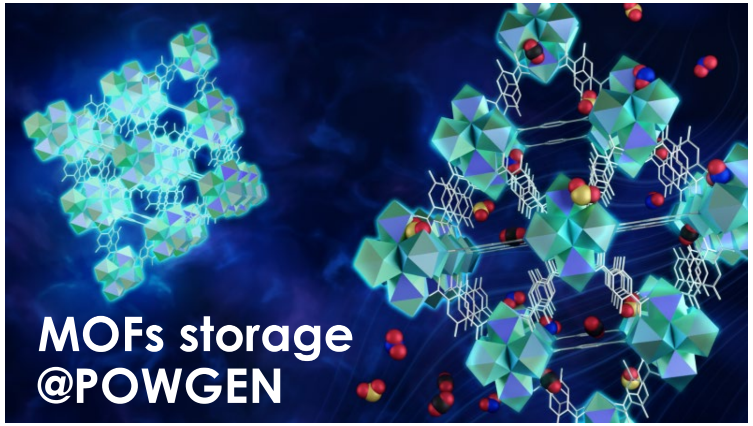
Specific Q region → **CS, unless very high Q**

Small sample → **TOF/CS**

Measure the crystal structure

Instruments at HFIR/SNS

- Powder samples
  - **POWGEN (TOF):** High resolution and Q coverage
  - **NOMAD (TOF):** High flux and Q coverage
  - **HB-2A (CS):** Smaller unit cell, complex SE, absorbing samples.



- Single crystals
  - **DEMAND (CS):** Smaller unit cell inorganic materials
  - **TOPAZ (TOF):** High resolution and coverage for inorganic/organic and larger structures
  - **IMAGINE (CS):** Quasi-Laue for macromolecules
  - **MANDI (TOF):** Protein crystallography

DEMAND (10^4 Å^3)
TOPAZ (10^5 Å^3)
IMAGINE (10^6 Å^3)
MaNDi (10^7 Å^3)

Measure the Magnetic structure

Need
- To measure where magnetic scattering is strongest.
- Good signal to noise.
- Variety of sample conditions.

Instrument should have
- Low Q **(CS/TOF)**
- High intensity **(CS)**
- Low background **(CS/TOF)**
- Low T and magnets **(CS)**

- **CS is typically best, but TOF has ever increasing options.**

Other factors?

Short-range order
TOF/CS Consider low Q or mPDF options

Specialized SE
CS/ TOF

Polarization
CS

Absorbing, H
- **CS easier.**
- **Consider TAS.**
- **TOF feasible**

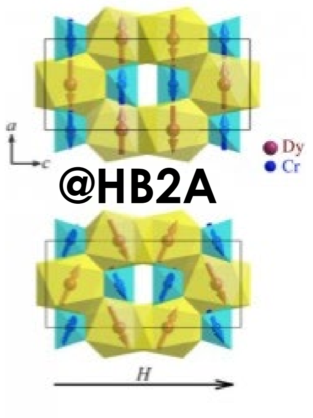
Specific Q region
TOF/CS
- **TOF: If need both high/low CS:**
- **One region**

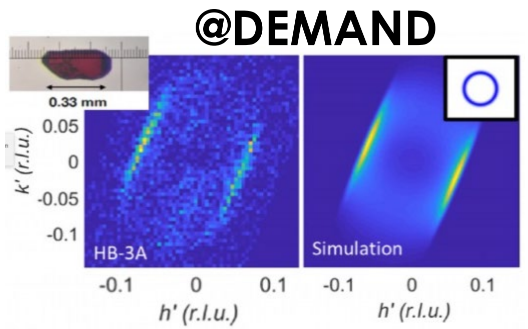
Small sample
TOF/CS

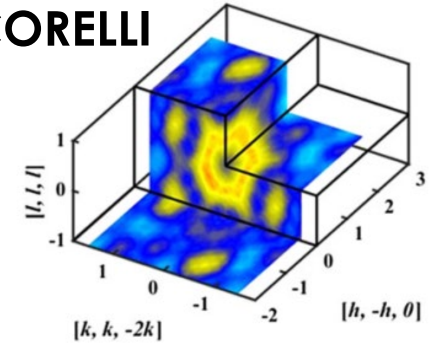
Measure the magnetic structure

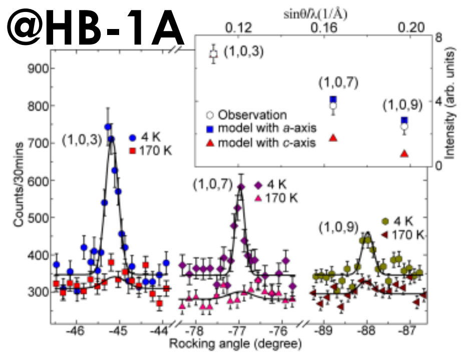
<u>**Instruments at HFIR/SNS**</u>

- Powder samples
  - **HB-2A (CS):** Access low Q. Low Temperature and magnets. Polarization.
  - **POWGEN (TOF):** High resolution and Q coverage.
- Single crystal
  - **DEMAND (CS):** Low Q coverage and variety of sample environments. Polarization.
  - **TOPAZ (TOF):** Wide coverage in Q. New 5 K option.
  - **CORELLI (TOF):** Diffuse scattering. Variety of sample environments.
  - **GP-SANS (CS):** Very low Q for large spin textures (e.g. Skyrmions).
- Both Powder and single crystal
  - **WAND² (CS):** High flux → Long range and Diffuse signals
  - **HB-1A (CS):** Excellent signal-to-noise for weak signal









Disordered materials (PDF)

Need

- To access high Q and broad scattering

Instrument should have

- Wide Q-range **(TOF)**
- High Q_{max} **(TOF)**
- High intensity **(TOF/CS)**

- **TOF is typically best, especially for powders.**
- **But CS can work well.**

Other factors?

Specialized SE

TOF/CS

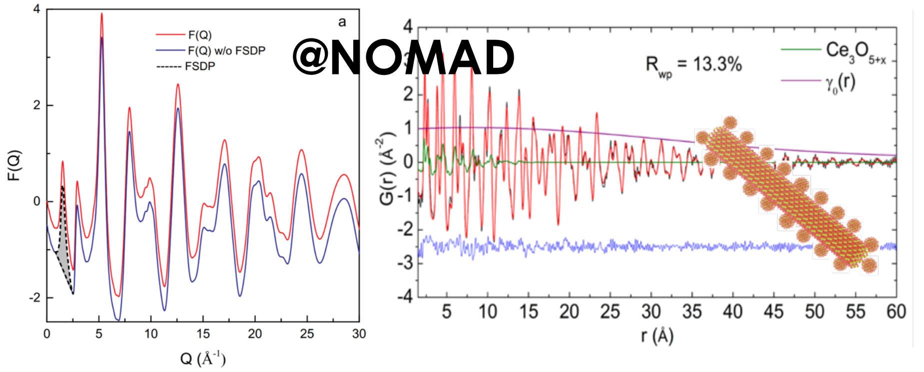
Absorbing, H

- **Hard for powders**

Small sample

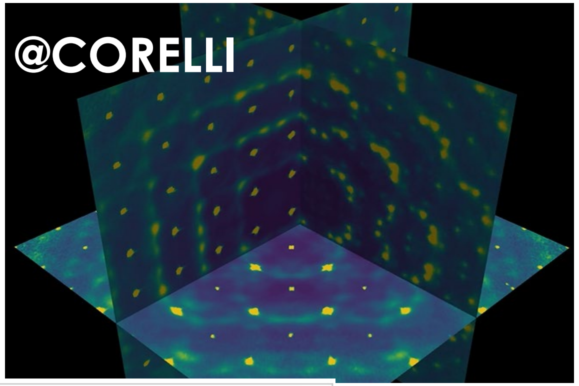
TOF/CS

Disordered material (PDF)

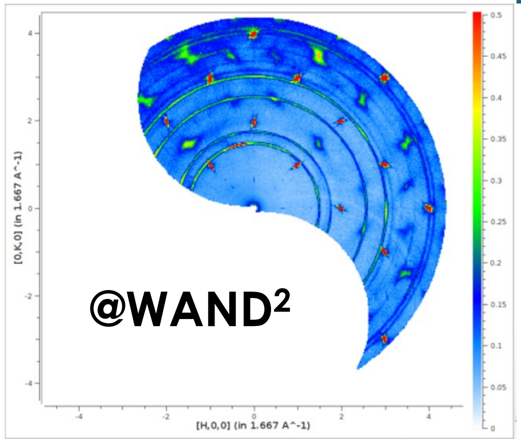


Instruments at HFIR/SNS

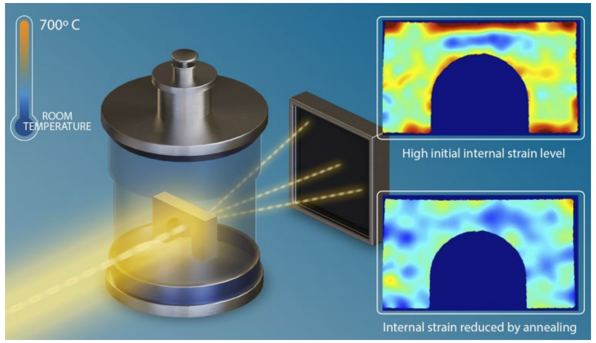
- Powder
  - **NOMAD (TOF):** Dedicated total scattering beamline.
  - **POWGEN (TOF):** Longer counting, but better resolution if needed.
  - **HB2A and WAND² (CS):** Magnetic PDF (mPDF)



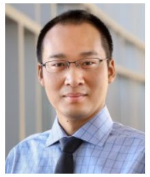
- Single crystal
  - **CORELLI (TOF):** Dedicated diffuse scattering beamline
  - **TOPAZ (TOF):** Large reciprocal space coverage
  - **WAND² (CS):** High flux and variety of sample environments



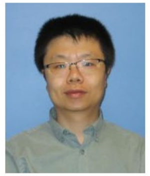
Many more diffraction experiments



Ke An
Instrument Scientist



Yan Chen
Instrument Scientist



Dunji Yu
Instrument Scientist

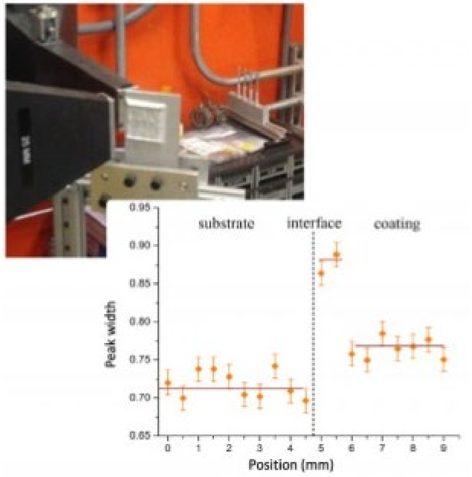
<u>Engineering diffraction</u>
<u>Measure strain/stress</u>

VULCAN (TOF)

- **In situ loading studies of crystalline/amorphous materials at high temperatures: phase transformation, fatigue damage, creep behaviors, and other deformation mechanisms in nanostructured materials, piezoelectric and shape-memory alloys.**

HIDRA (CS)

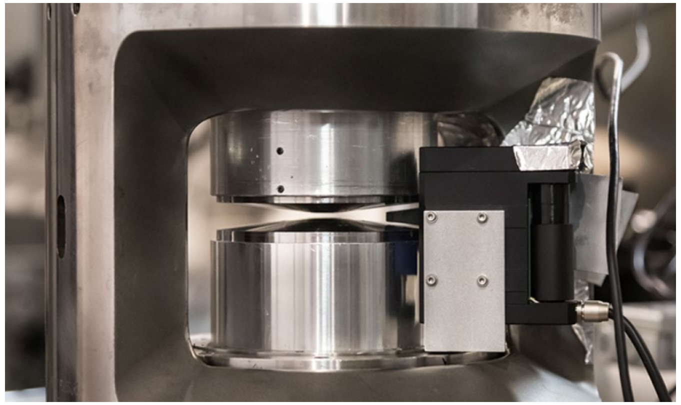
- **Optimized for strain measurement and determination of residual stress in engineering materials.**
- **Spatial resolution at a fraction of a millimeter is possible**



Jeffrey Bunn
Instrument Scientist

Chris Fancher
Instrument Scientist

Many more diffraction experiments



António M. dos Santos
Instrument Scientist

Chris Ridley
Instrument Scientist

High pressure diffraction

SNAP (TOF)

- Dedicated instrument for high pressure >100GPa.
- Powders and single crystals.
- Wide Q-coverage
- PDF options

WAND², DEMAND, HB-2A, TAS (CS) CORELLI (TOF)

- Variety of options for pressure measurements.

Dilip Bhoi

Review articles for the Diffraction Suite

REVIEW OF SCIENTIFIC INSTRUMENTS **89**, 092802 (2018)

A suite-level review of the neutron single-crystal diffraction instruments at Oak Ridge National Laboratory

L. Coates,[1,a)] H. B. Cao,[1] B. C. Chakoumakos,[1] M. D. Frontzek,[1] C. Hoffmann,[1] A. Y. Kovalevsky,[1] Y. Liu,[1] F. Meilleur,[1,2] A. M. dos Santos,[1] D. A. A. Myles,[1] X. P. Wang,[1] and F. Ye[1]

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(Received 26 March 2018; accepted 9 July 2018; published online 24 September 2018)

The nascent suite of single-crystal neutron diffractometers at the Oak Ridge National Laboratory has no equal at any other neutron scattering facility worldwide and offers the potential to re-assert single-crystal diffraction using neutrons as a significant tool to study nuclear and magnetic structures of small unit cell crystals, nuclear structures of macromolecules, and diffuse scattering. Signature applications and features of single-crystal neutron diffraction are high resolution nuclear structure analysis, magnetic structure and spin density determinations, contrast variation (particularly D_2O/H_2O) for nuclear structural studies, lack of radiation damage when using crystals of biological molecules such as proteins, and the fidelity to measure nuclear and magnetic diffuse scattering with elastic discrimination.
https://doi.org/10.1063/1.5030896

REVIEW OF SCIENTIFIC INSTRUMENTS **89**, 092701 (2018)

A suite-level review of the neutron powder diffraction instruments at Oak Ridge National Laboratory

S. Calder,[1,a)] K. An,[1] R. Boehler,[1,2] C. R. Dela Cruz,[1] M. D. Frontzek,[1] M. Guthrie,[3,4] B. Haberl,[1] A. Huq,[1] S. A. J. Kimber,[1] J. Liu,[1] J. J. Molaison,[1] J. Neuefeind,[1] K. Page,[1] A. M. dos Santos,[1] K. M. Taddei,[1] C. Tulk,[1] and M. G. Tucker[1]

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(Received 6 April 2018; accepted 19 July 2018; published online 28 September 2018)

The suite of neutron powder diffractometers at Oak Ridge National Laboratory (ORNL) utilizes the distinct characteristics of the Spallation Neutron Source and High Flux Isotope Reactor to enable the measurements of powder samples over an unparalleled regime at a single laboratory. Full refinements over large Q ranges, total scattering methods, fast measurements under changing conditions, and a wide array of sample environments are available. This article provides a brief overview of each powder instrument at ORNL and details the complementarity across the suite. Future directions for the powder suite, including upgrades and new instruments, are also discussed. https://doi.org/10.1063/1.5033906

ASK AN INSTRUMENT SCIENTIST!!!

https://neutrons.ornl.gov/suites/diffraction


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Stuart Calder
POWDER


Clarina dela Cruz
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Alicia Manjón Sanz
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Ke An
VULCAN


Xiaoping Wang
TOPAZ


Thomas Proffen
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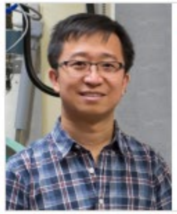
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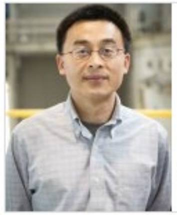
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Flora Meilleur
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Matthias Frontzek
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Jeffrey Bunn
HIDRA


Andrew Payzant
HIDRA


Chris Fancher
HIDRA


Si Athena Chen
WAND²


Christina Hoffmann
CORELLI, TOPAZ

Conclusions

ASK AN INSTRUMENT SCIENTIST!!!

https://neutrons.ornl.gov/suites/diffraction

- ORNL is unique in having world class CS and TOF instruments with different strengths.
- Choose the best instrument for your experiment based on your science.

Lots of good options for diffraction at HFIR/SNS.

Questions?