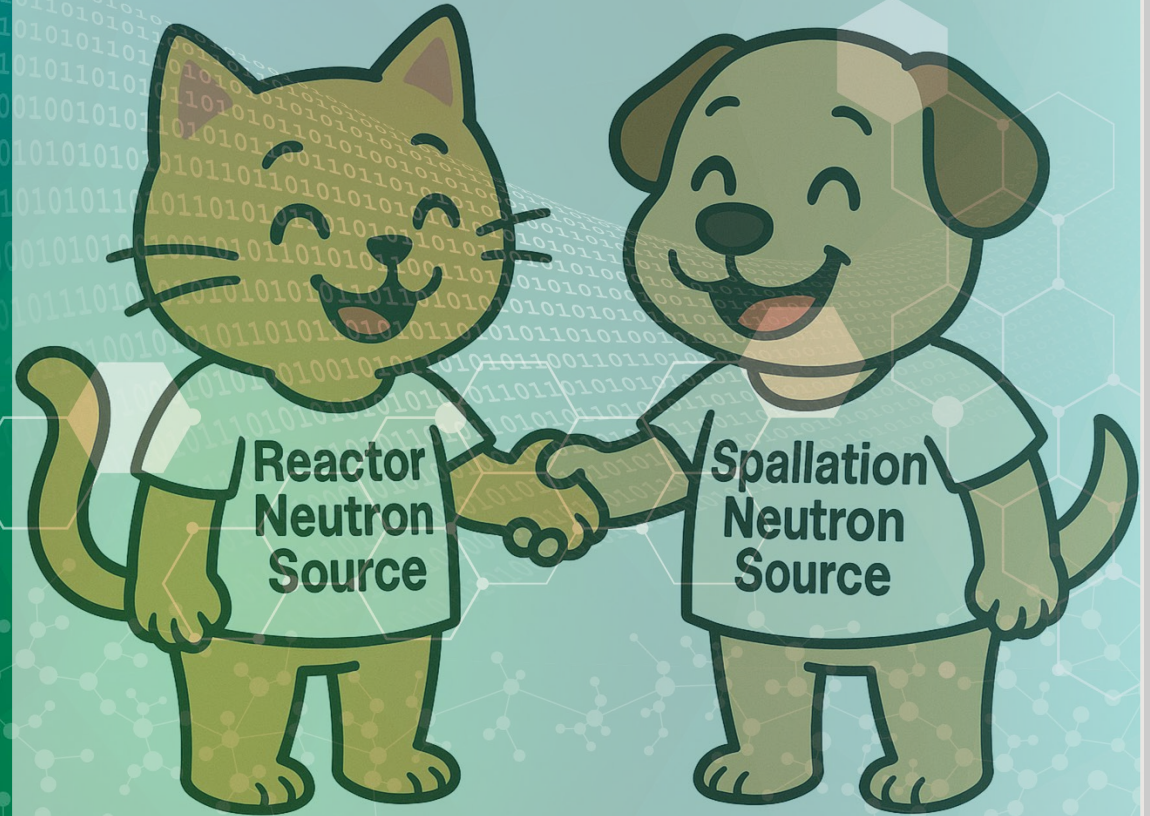


Diffraction at Time-of-Flight (TOF) ~~vs~~ *and* Continuous Sources (CS)

Stuart Calder

Neutron Scattering Division
Oak Ridge National Laboratory



ORNL is managed by UT-Battelle, LLC for the US Department of Energy



U.S. DEPARTMENT OF
ENERGY

How I got here?



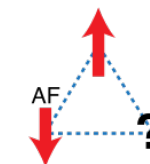
Undergraduate in Physics
University of Edinburgh



Postdoc at ORNL



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



Current role:

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



ISIS, UK (TOF)



ILL, France
(Continuous source)

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

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

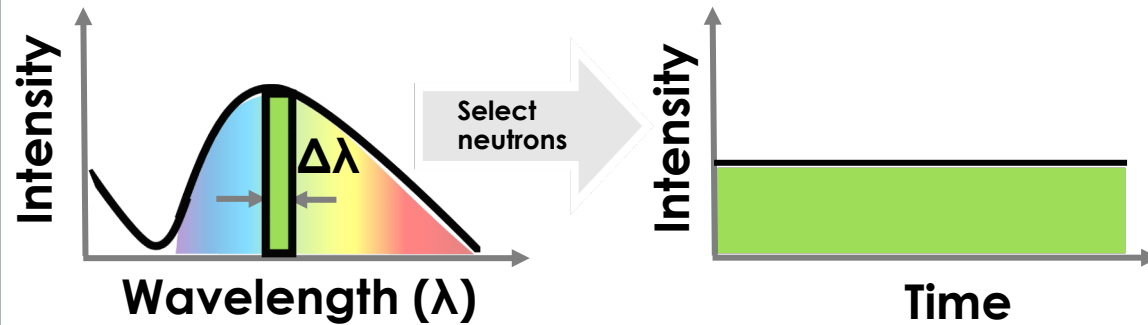
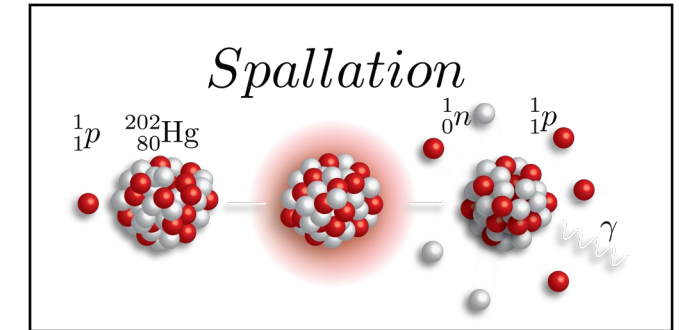
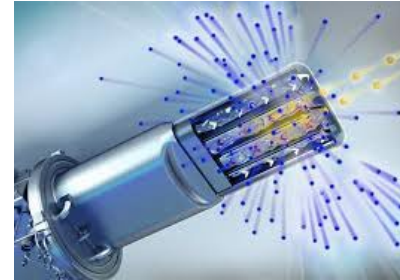
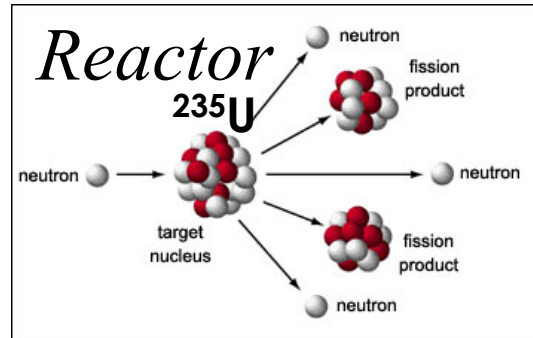
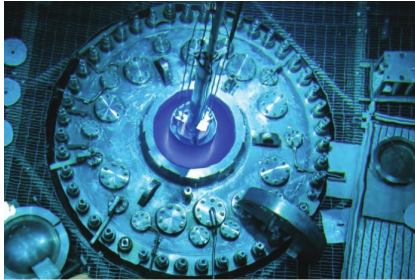
Always talk to an instrument scientist

Neutron Sources around the world

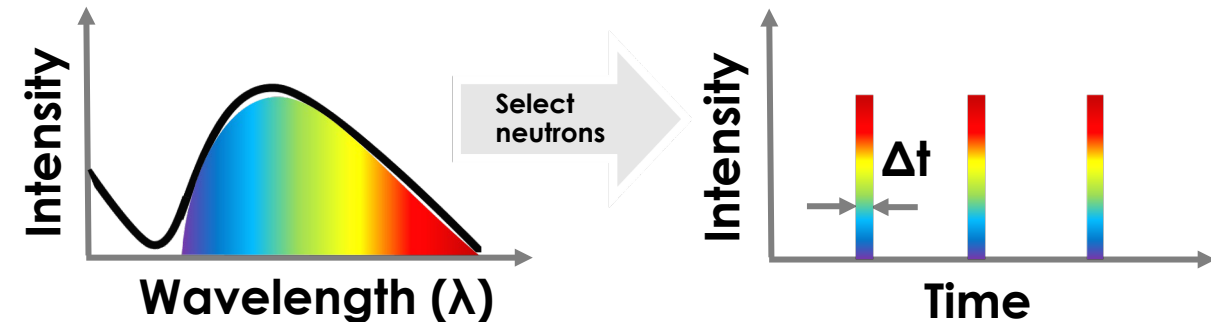
- Reactor
- Spallation



Neutron sources: Reactor (HFIR) and Spallation (SNS)



Small $\Delta\lambda$ used, but source on all the time



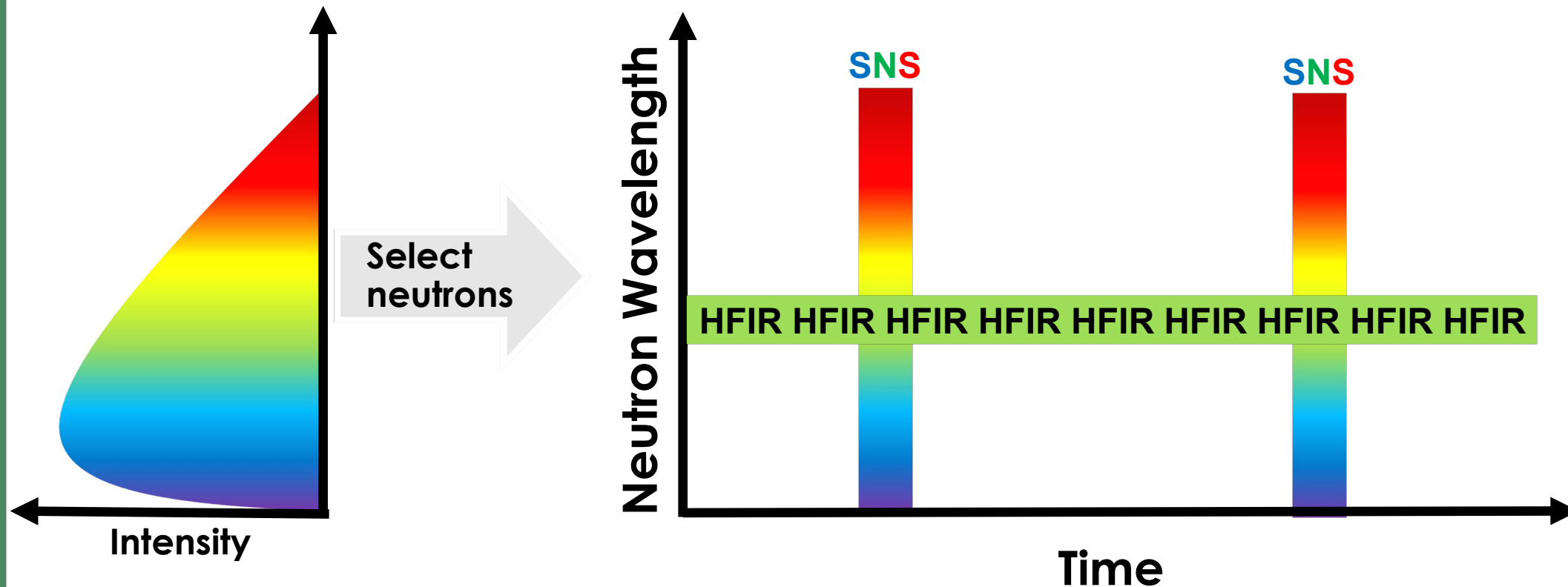
Each pulse of neutrons contains a broad spectrum of energy (λ)

Pulse of neutrons
~60 times per second

How do you like your neutrons?

HFIR: Some of the neutron wavelength all the time?

SNS: All the neutron wavelengths some of the time?

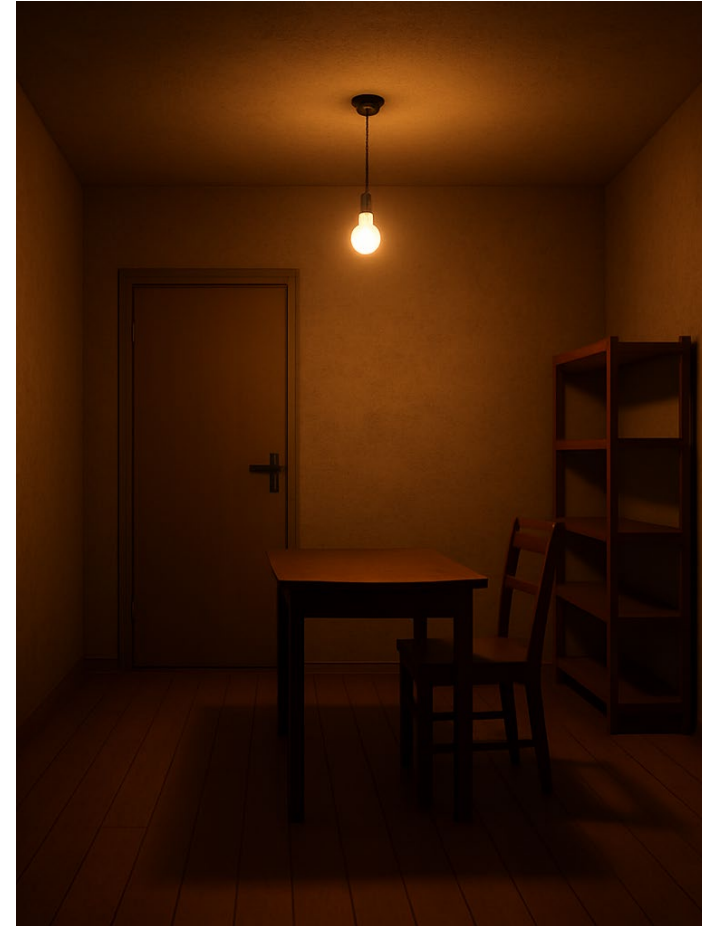


Neutron sources: Reactor and Spallation

HFIR:
High flux over a chosen region.

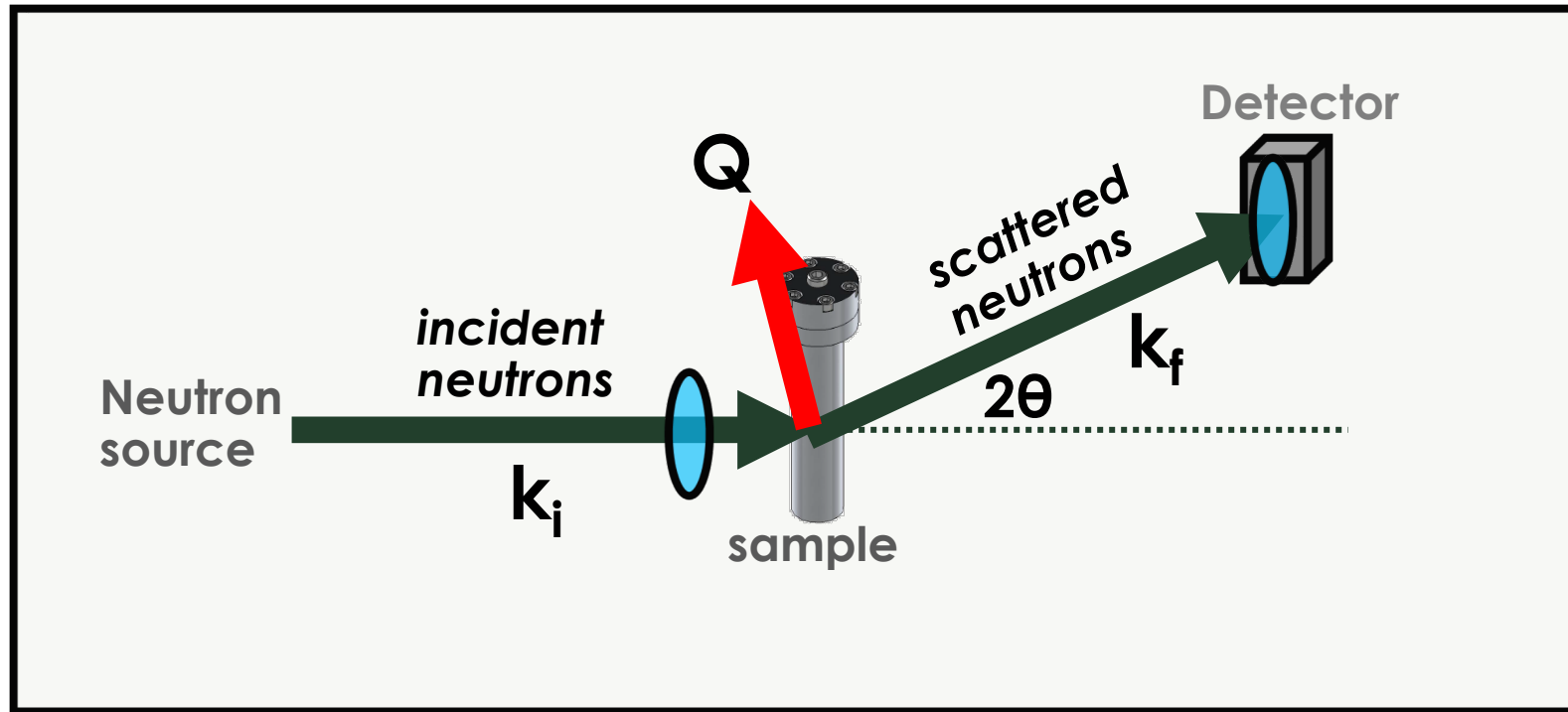


SNS:
Large coverage.

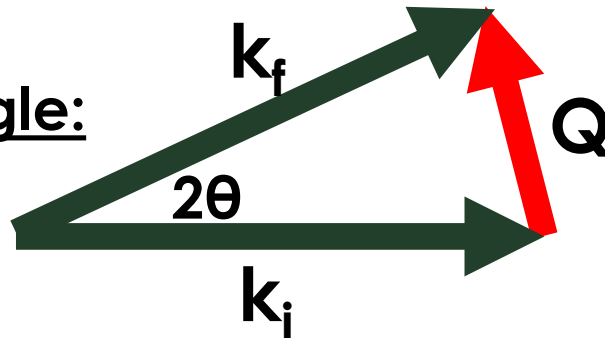


Diffraction experiments

Neutron Diffraction: No change in energy of neutron



Scattering triangle:

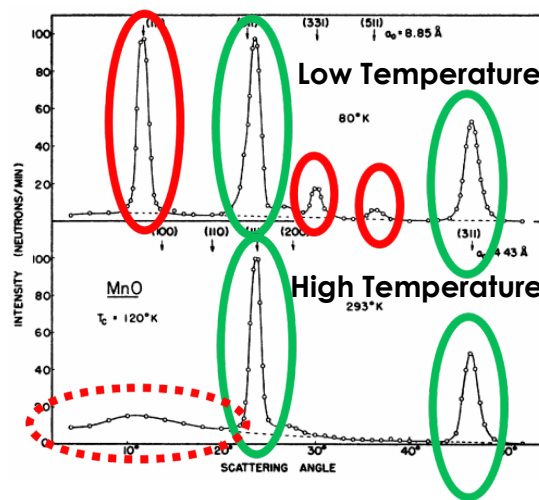
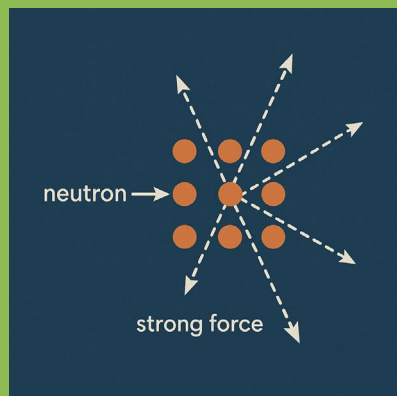


Bragg's law: $\lambda = 2d \sin \theta$

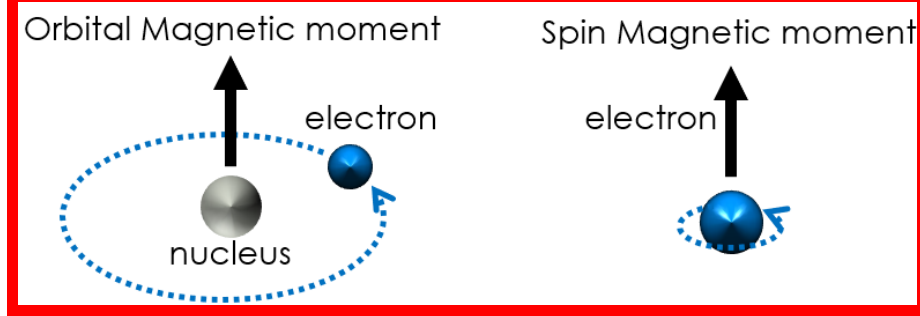
$$Q = \frac{4\pi \sin \theta}{\lambda} = \frac{2\pi}{d}$$

Nuclear and Magnetic scattering

- Scattering of neutrons from nucleus via strong force is used to determine crystal structures



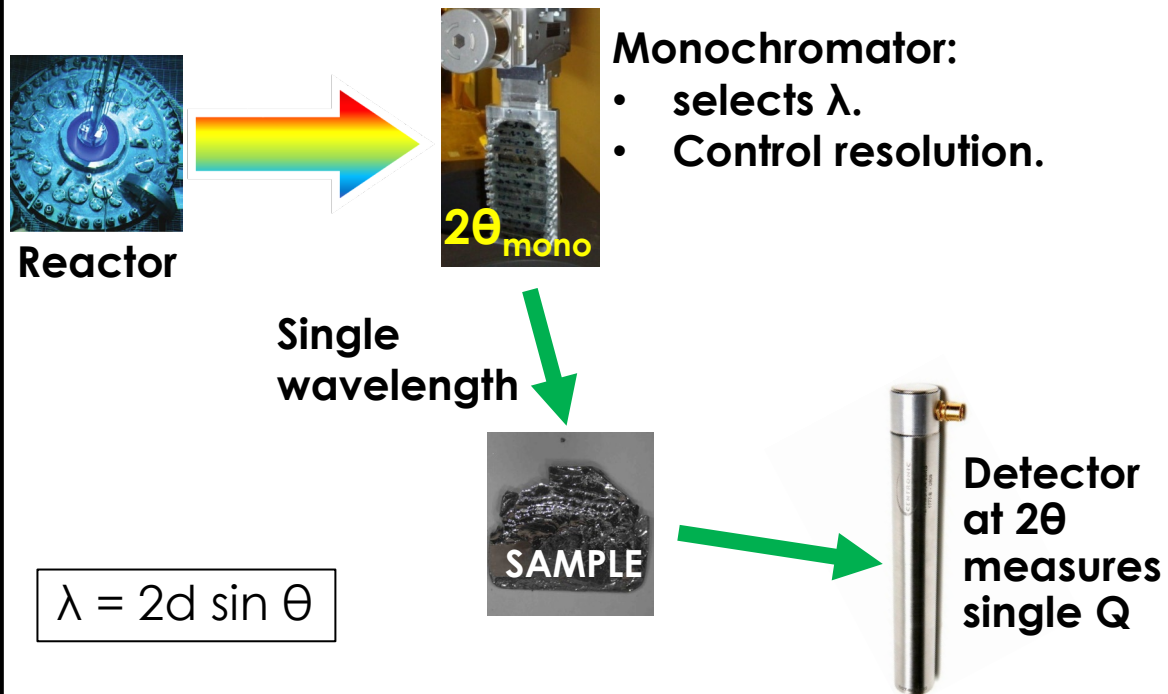
- Scattering of neutrons from magnetic potential of unpaired electrons is used to determine magnetic structures



- Magnetic/Nuclear scattering:
 - Different processes, but fortunately have similar intensities.
 - Can be distinguished in experiments

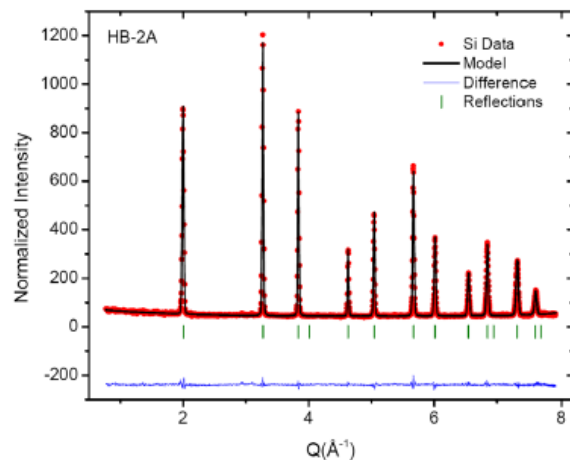
What do the different
sources mean for
diffraction experiments?

A typical diffractometer at HFIR



$$\lambda = 2d \sin \theta$$

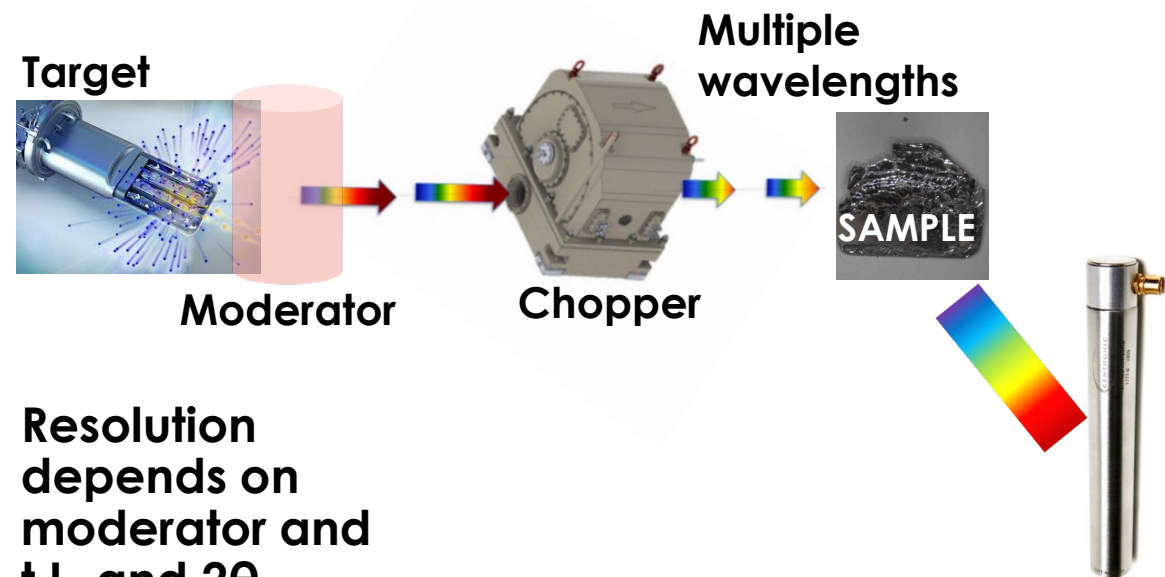
Q range limit is $4\pi/\lambda$



A typical diffractometer at SNS

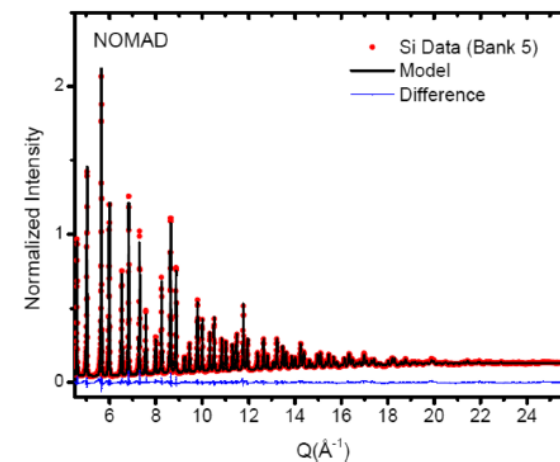
De Broglie wavelength: $\lambda = h/mv = ht/mL$

Bragg's law: $\lambda = 2d \sin \theta = \text{constant} * t$

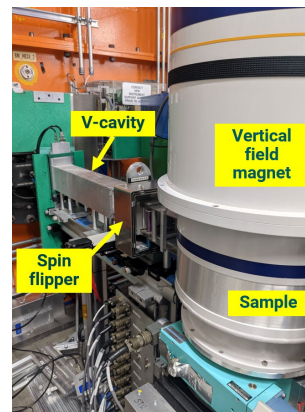
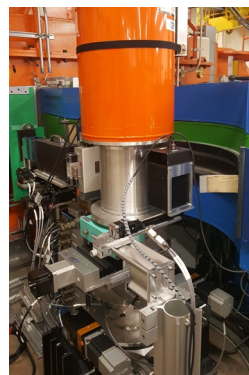
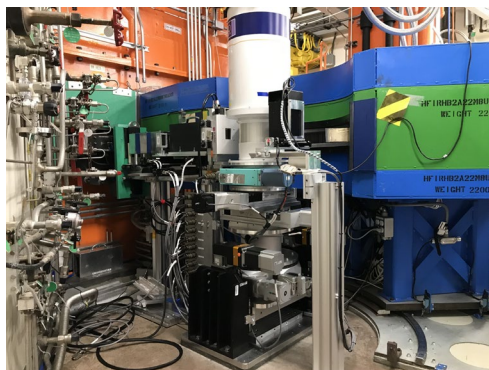


Resolution depends on moderator and t, L, and 2θ

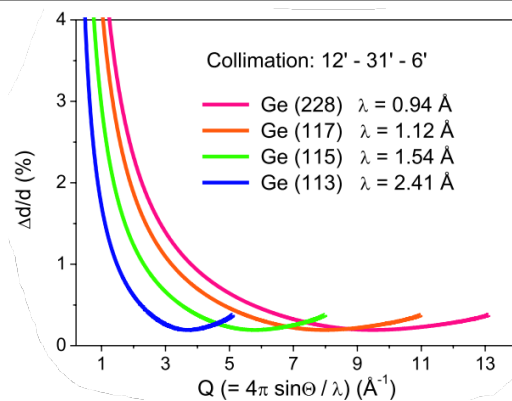
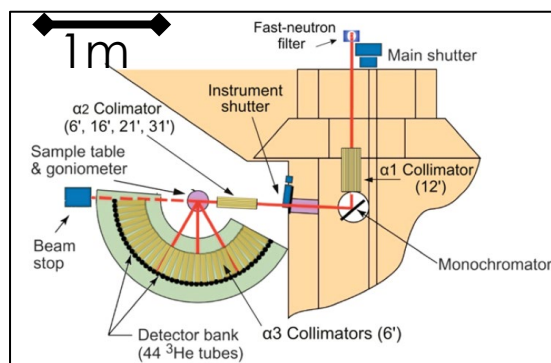
Q range determined by $\lambda_{\min}$, $\lambda_{\max}$ and 2θ



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

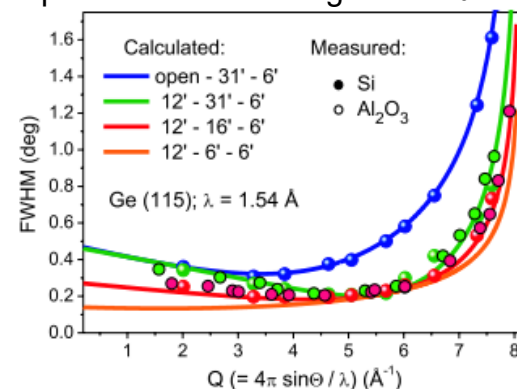


Versatile: open sample space and compact

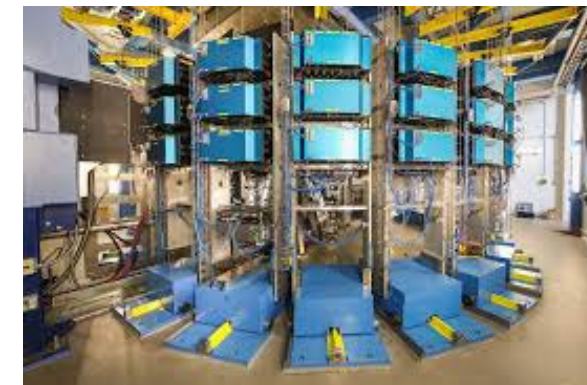
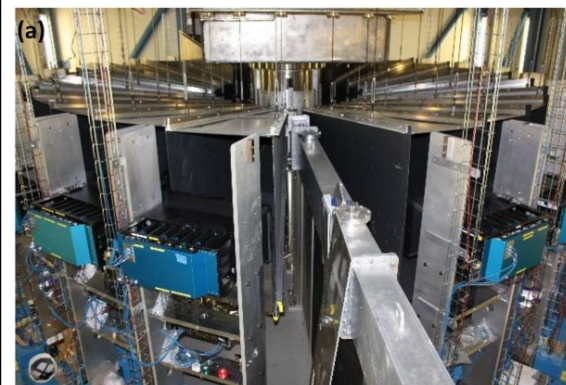


Ge(hkl)	λ (Å)	Q (Å ⁻¹)	Flux at sample (n/cm ² s)
(113)	2.41	0.2 - 5.1	5×10^6
(115)	1.54	0.35 - 7.9	1×10^7
(117)	1.12	0.5 - 10.9	4×10^6

Resolution:
best resolution $\Delta d/d$: 1×10^{-3}
Dependent on wavelengths and d



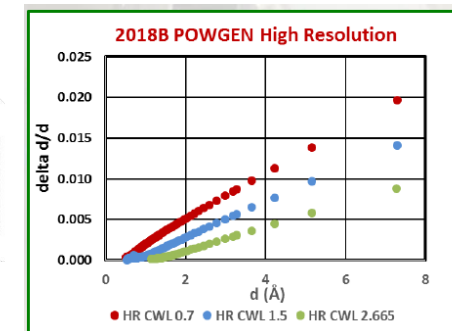
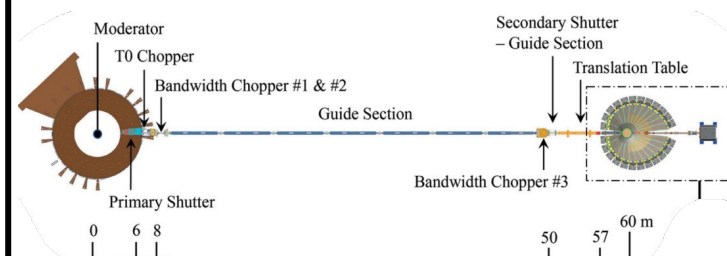
A typical TOF diffractometer (POWGEN, SNS)



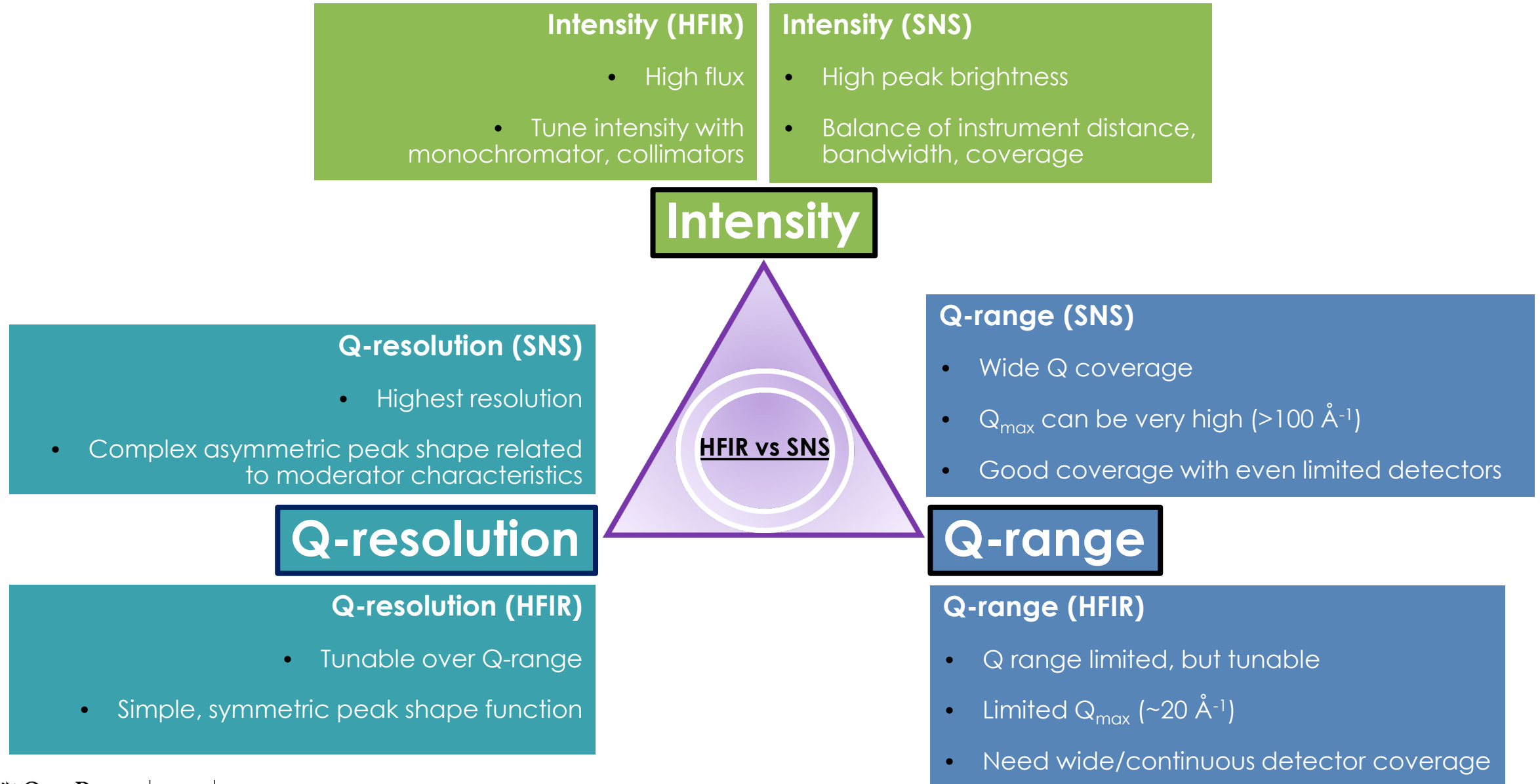
Larger, more detector coverage

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 $\Delta d/d$: 1×10^{-4}
Dependent on wavelengths and d



HFIR and SNS: Factors to consider for a diffraction experiment



Do simulations. Lots of tools!



Experiment Simulator

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: MnO_Fm-3m.cif

Select an instrument config:

Container Inner Diameter (cm): [Show/hide notes](#)

Container Material:

Sample Mass (g):

Counting Time (hour):

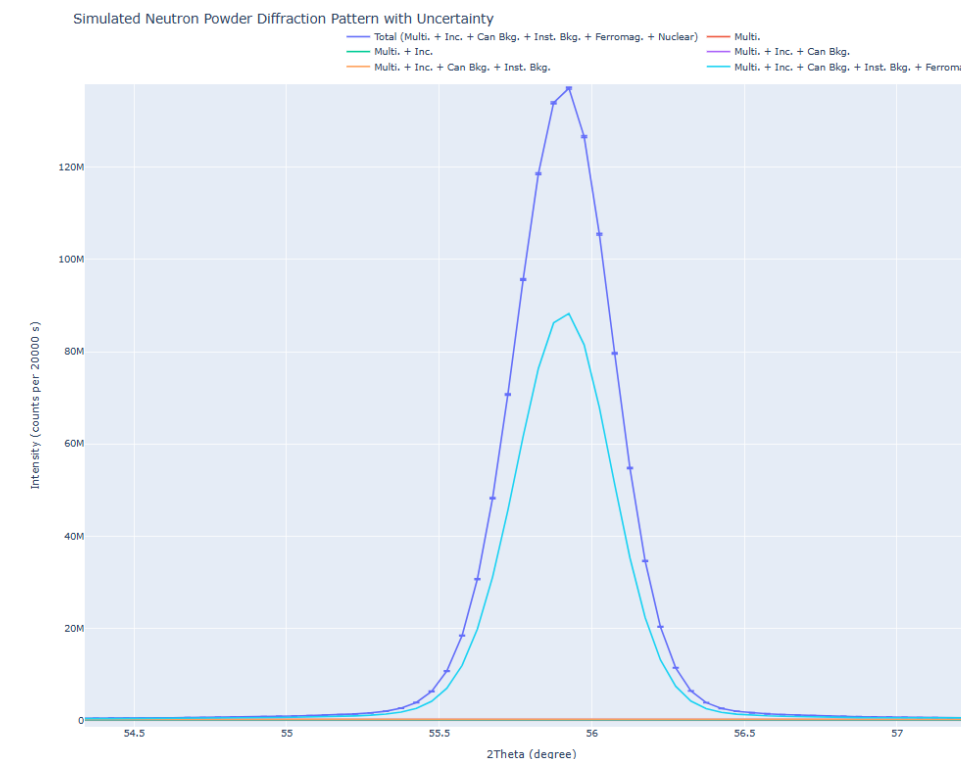
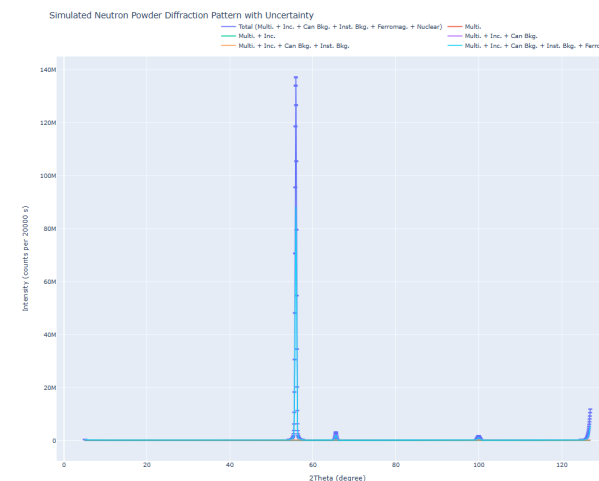
Number of Magnetic Atoms (per unit cell): [Show/hide notes](#)

Magnetic Moment (per atom, in μ_B):

*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

https://addie.ornl.gov/data_viz_hfirestimate



Some other strengths for diffraction experiments?

Diffractometer at HFIR

Main: High Flux beam, versatility

Simple beam profile corrections
- Absorbing, attenuating samples and holders

Open instruments with variable sample environments

Beam is always on
- Time dependent measurements

Polarization is easier

Diffractometer at SNS

Main: Resolution and wide Q-range

High peak brightness

Count rate
- Typically large detector coverage, optimized beams, lots of wavelengths

Pump probe measurements

Newer facility, STS

Sample Environment considerations

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



<https://neutrons.ornl.gov/sample>

Equipment

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

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	

Ask an instrument scientist what is available

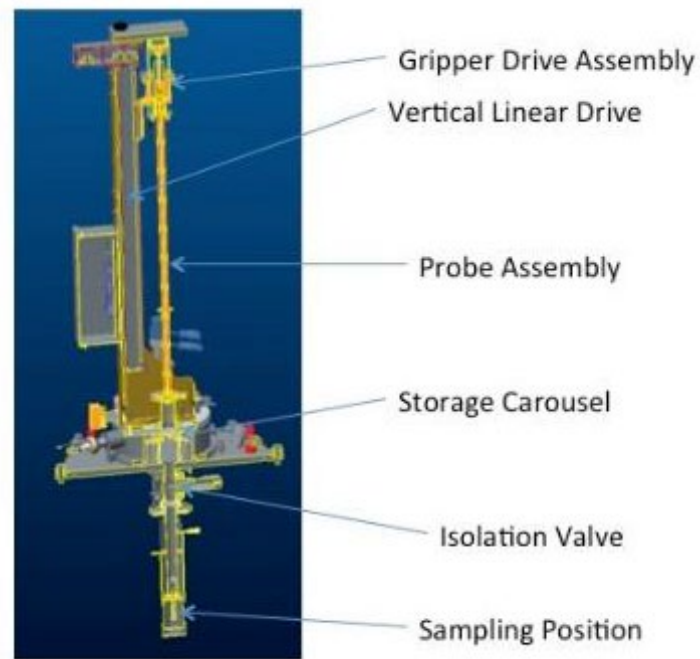
Types of Sample Changers

External Sample – External Manipulation



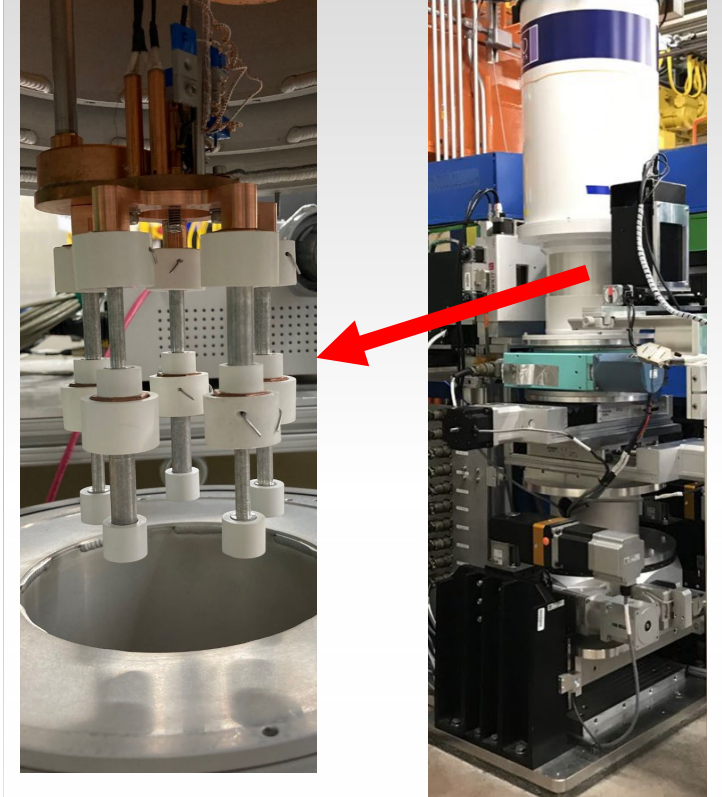
- 90 K to 573 K
- Open air environments
- Fast to load

Internal Sample – Internal Manipulation



- 10 K – 300 K
- Only CCR range (not below 1 K)
- Slower to load

Internal Sample – External Manipulation

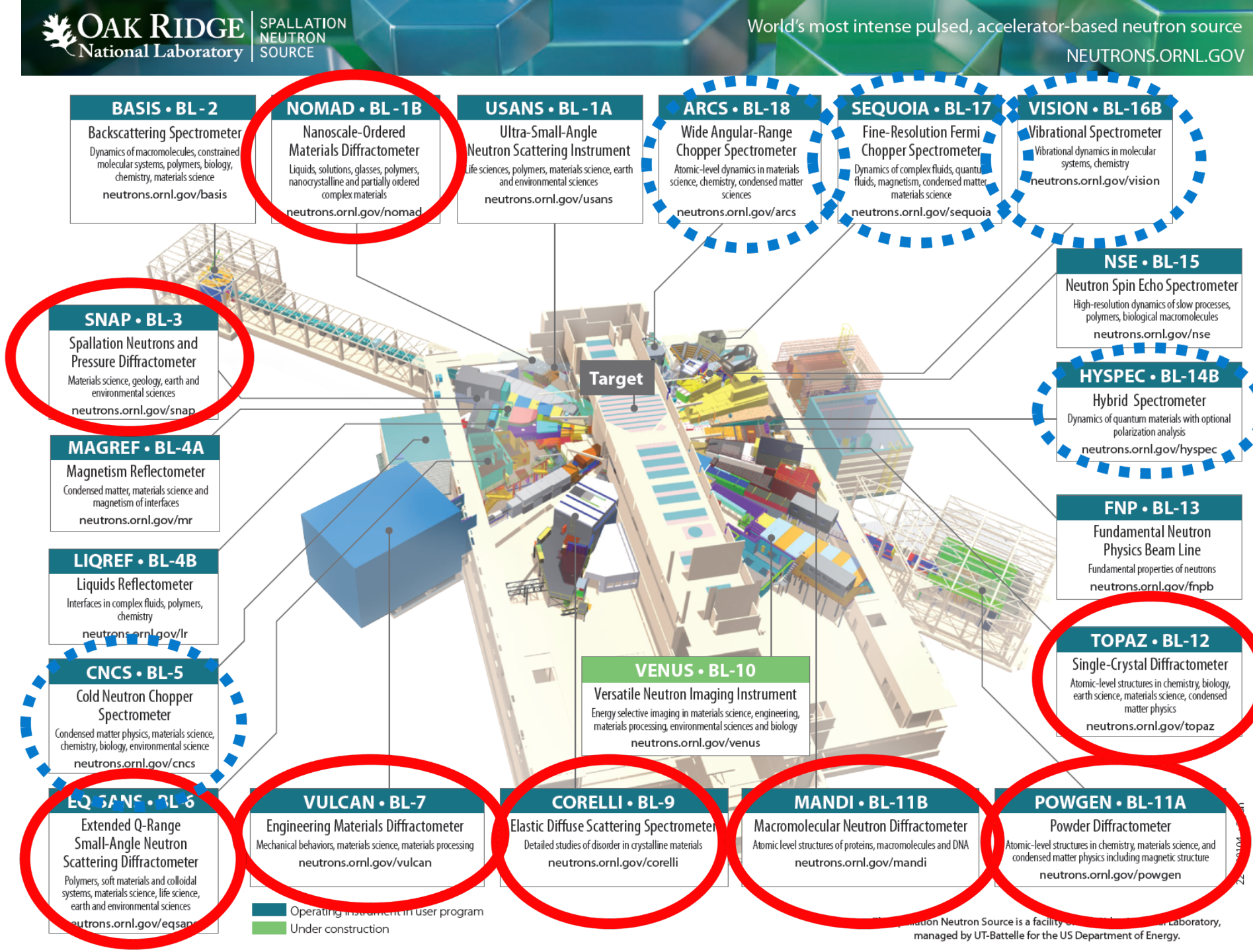


- 0.04 K to 700 K
- Full range plus magnets
- Slowest to load, do it offline

Lots of diffraction
instruments at HFIR/SNS
(almost any instrument is
partly a diffractometer...)

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

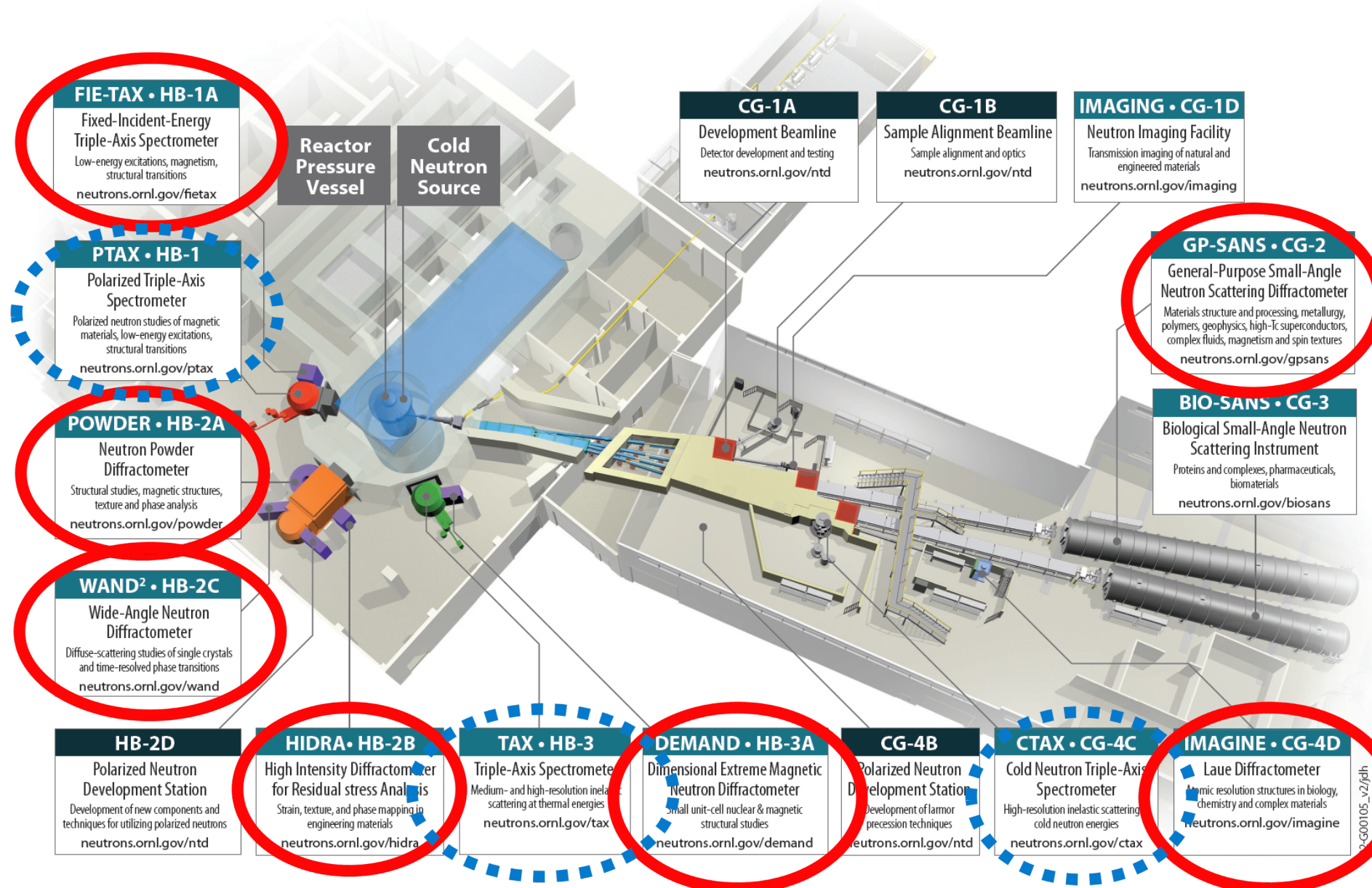
- HYSPEC
- ARCS
- SEQUOIA
- CNCS



HFIR (Continuous Source)

- HB-2A
- DEMAND
- IMAGINE
- WAND²
- VERITAS (HB-1A)
- HIDRA
- GP-SANS

- HB-1
- HB-3
- CTAX



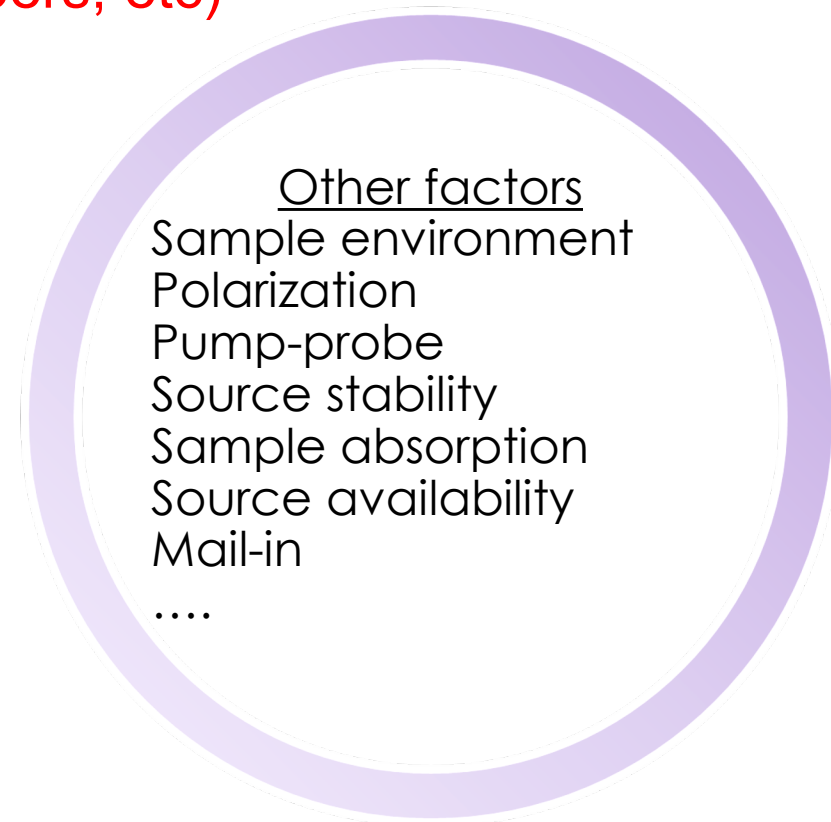
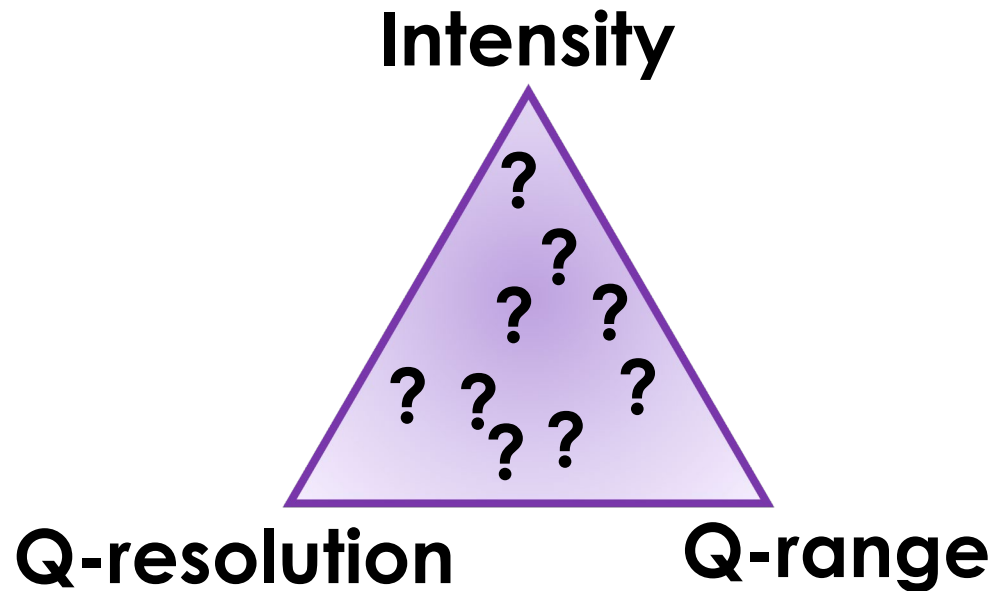
Instrument	Type	Methods	Applications	T (K)	P (GPa)	H (T)	Special	Resolution ($\Delta d/d$)	Q-range ($\text{\AA}^{-1}$)	Beam size H x V (mm)
HFIR HB-2A POWDER	constant wavelength	Rietveld	Magnetic structures	0.05 - 1800	< 0.6 gas cell < 2 clamp cell	< 7	Electric field	2 x 10-3 (variable)	0.2 - 5.1 and 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 $2\theta = 90^\circ$)	0.8-30	Typical, 1 cm2 < 1 mm2 for DACs

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? SNS or HFIR 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)



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 (**HFIR/SNS**)
- Large Q-range (**SNS**)
- High Q (**SNS**)

- SNS is typically best, especially for powders.
- Single crystal: SNS for largest unit cells, but HFIR can work well.

Specialized SE

HFIR/SNS

Absorbing, H

- HFIR easier
- SNS feasible

Specific Q region

HFIR, unless very high Q

Small sample

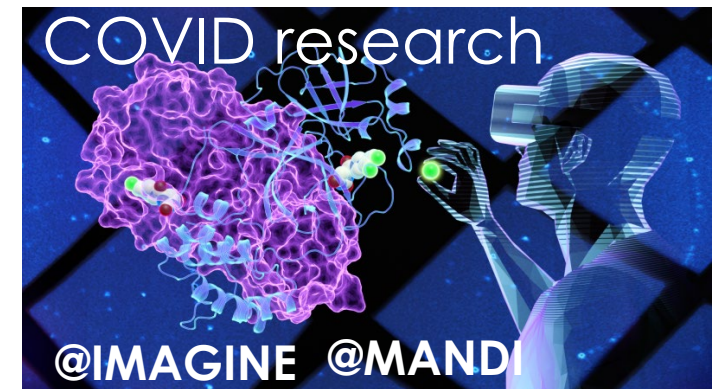
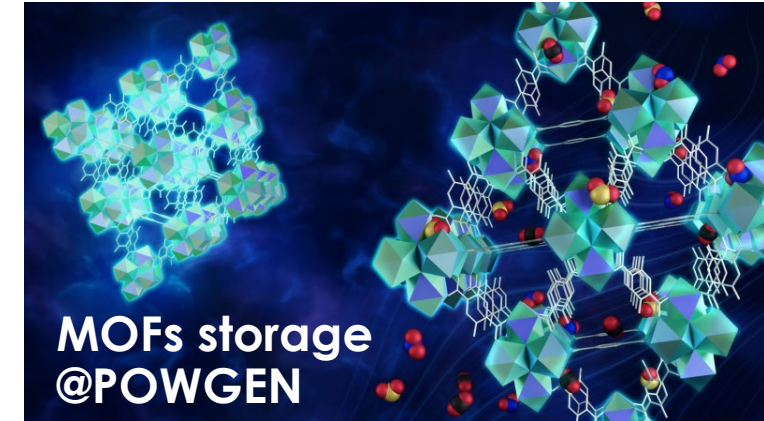
HFIR/SNS

Other factors?

Measure the crystal structure

Instruments at HFIR/SNS

- Powder samples
 - **POWGEN (SNS)**: High resolution and Q coverage
 - **NOMAD (SNS)**: High flux and Q coverage
 - **HB-2A (HFIR)**: Smaller unit cell, complex SE, absorbing samples.
- Single crystals
 - **DEMAND (HFIR)**: Smaller unit cell inorganic materials
 - **TOPAZ (SNS)**: High resolution and coverage for inorganic/organic and larger structures
 - **IMAGINE (HFIR)**: Quasi-Laue for macromolecules
 - **MANDI (SNS)**: Protein crystallography



DEMAND ($10^4 \text{ \AA}^3$)
TOPAZ ($10^5 \text{ \AA}^3$)
IMAGINE ($10^6 \text{ \AA}^3$)
MaNDi ($10^7 \text{ \AA}^3$)

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 (**HFIR/SNS**)
- High intensity (**HFIR**)
- Low background (**HFIR/SNS**)
- Low T and magnets (**HFIR**)

- **HFIR is typically best, but SNS has ever increasing options.**

Short-range order

Specialized SE

Absorbing, H

Small sample

Specific Q region

Polarization

HFIR/SNS

HFIR/SNS
Consider low Q or mPDF options

HFIR /SNS

HFIR

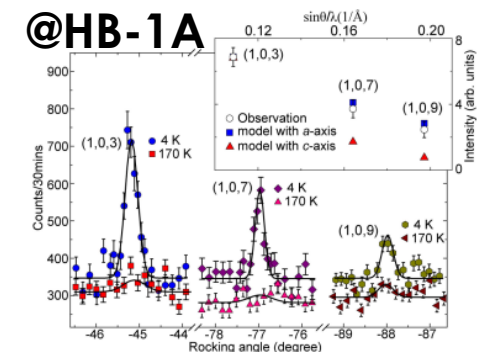
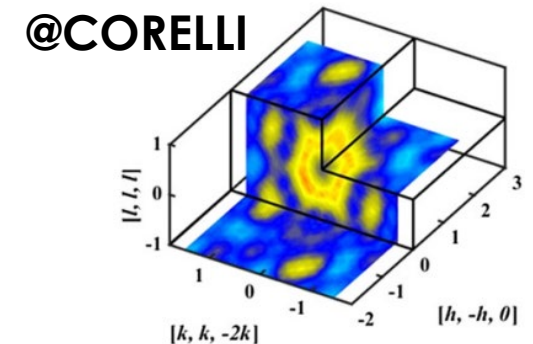
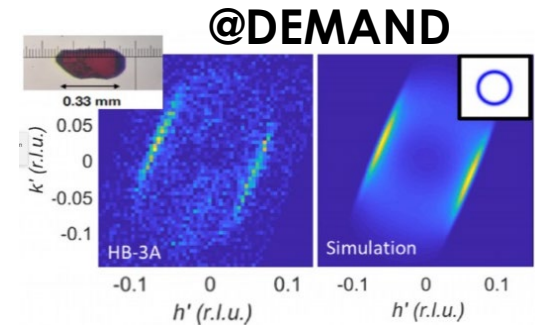
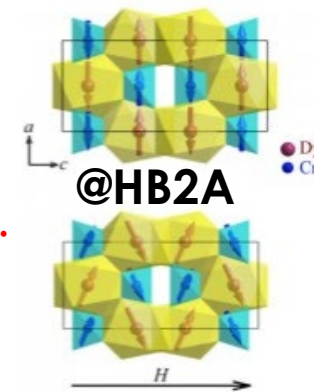
- **HFIR easier.**
- **Consider TAS.**
- **SNS feasible**

- **HFIR/SNS**
SNS: If need both high/low HFIR:
 - **One region**

Measure the magnetic structure

Instruments at HFIR/SNS

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



Disordered materials (PDF)

Need

- To access high Q and broad scattering

Instrument should have

- Wide Q-range (**SNS**)
- High Q_{max} (**SNS**)
- High intensity (**HFIR/SNS**)

- SNS is typically best, especially for powders.
- But HFIR can work well.

Other factors?

Specialized SE

Absorbing, H

Small sample

HFIR/SNS

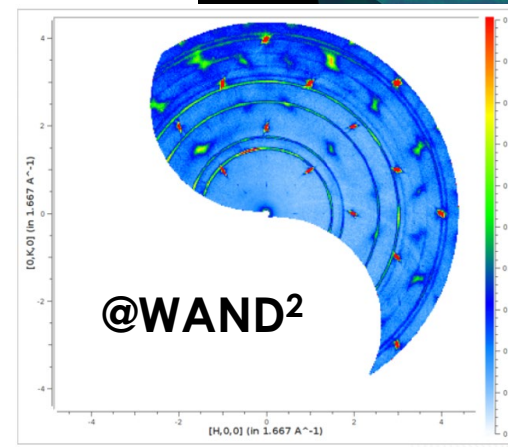
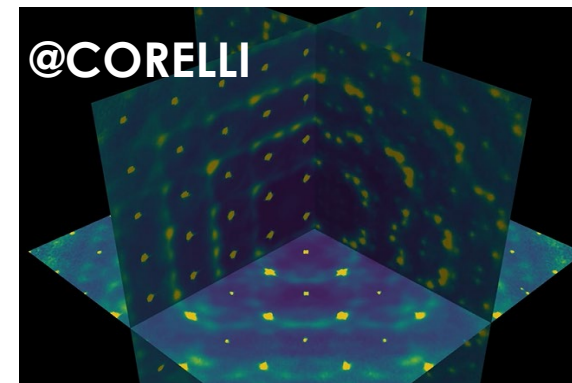
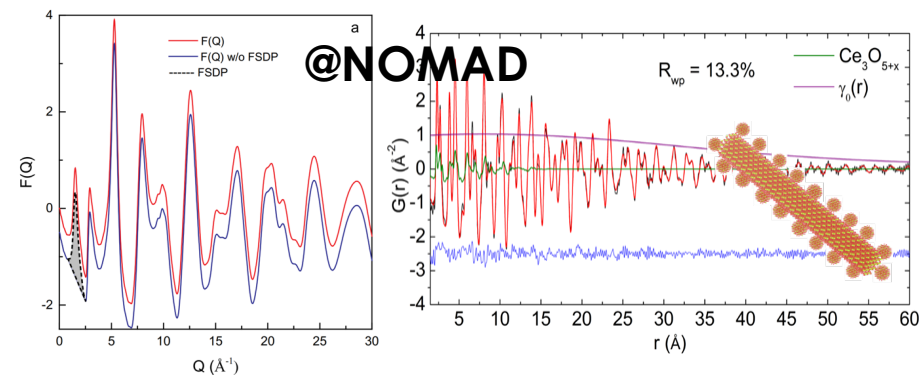
- Hard for powders

HFIR/SNS

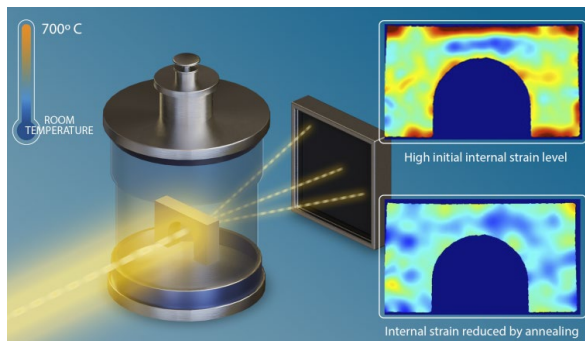
Disordered material (PDF)

Instruments at HFIR/SNS

- Powder
 - **NOMAD (SNS)**: Dedicated total scattering beamline.
 - **POWGEN (SNS)**: Longer counting, but better resolution if needed.
 - **HB2A and WAND² (HFIR)**: Magnetic PDF (mPDF)
- Single crystal
 - **CORELLI (SNS)**: Dedicated diffuse scattering beamline
 - **TOPAZ (SNS)**: Large reciprocal space coverage
 - **WAND² (HFIR)**: High flux and variety of sample environments



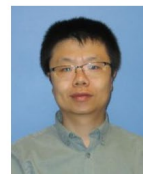
Many more diffraction experiments



Instrument Scientist



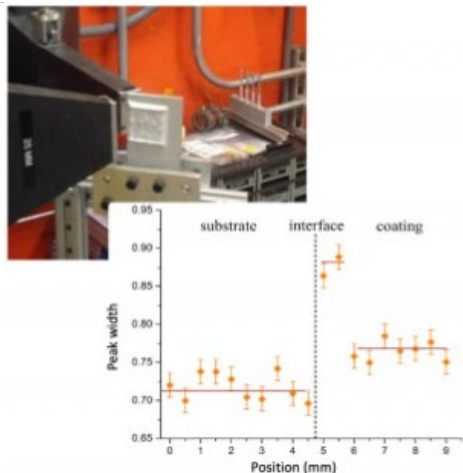
Instrument Scientist



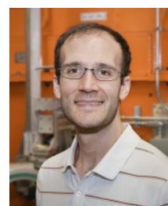
Instrument Scientist

**VULCAN
(SNS)**

Engineering diffraction
Measure strain/stress



Instrument Scientist



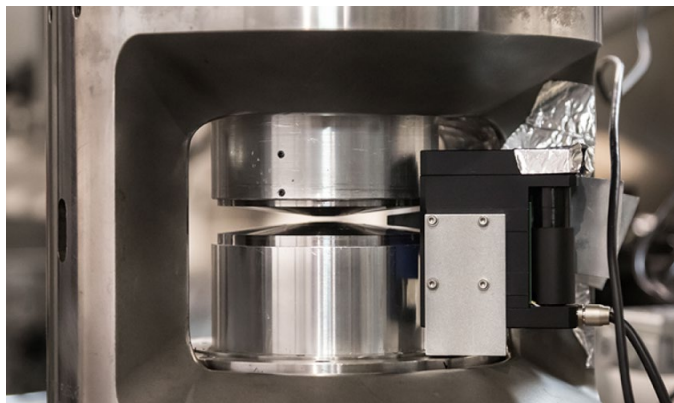
Instrument Scientist

**HIDRA
(HFIR)**

- 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.

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

Many more diffraction experiments



António M. dos Santos
Instrument Scientist



Chris Ridley
Instrument Scientist

**SNAP
(SNS)**

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

**High pressure
diffraction**



**WAND²,
DEMAND,
HB-2A,
TAS
(HFIR)
CORELLI
(SNS)**

- 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. Ye¹

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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 D₂O/H₂O) 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. © 2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>). <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. Neufeind,¹ K. Page,¹ A. M. dos Santos,¹ K. M. Taddei,¹ C. Tulk,¹ and M. G. Tucker¹

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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. © 2018 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>). <https://doi.org/10.1063/1.5033906>



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Computational Instrument Scientist



Andrey
Kovalevsky
IMAGINE, MANDI

ASK AN INSTRUMENT SCIENTIST!!!



Stuart Calder
POWDER



Clarina dela Cruz
POWDER



Danielle Yahne
POWDER



Joerg C.
Neufeld
NOMAD



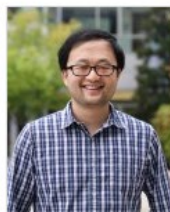
Dean Myles
IMAGINE, MANDI



Yan Chen
VULCAN



Cheng Li
NOMAD,
POWGEN



Jue Liu
NOMAD



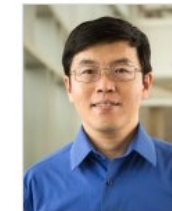
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POWGEN



Alicia Manjón
Sanz
POWGEN



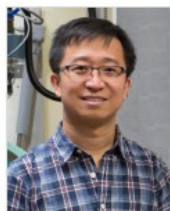
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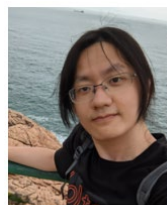
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Matthias Frontzek
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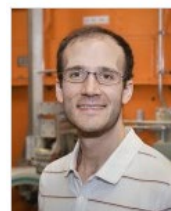


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Christina
Hoffmann
CORELLI, TOPAZ

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

Conclusions

ASK AN INSTRUMENT SCIENTIST!!!

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

- ORNL is unique in having world class instruments using two different sources.
- Choose the best instrument for your experiment based on your science.

Questions?

Lots of good options for diffraction at HFIR/SNS.

