Neutron scattering: basic concepts and high pressure examples

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Outline

- Introduction
- Basic concepts
- Instruments
- Examples
- How to apply for beam time

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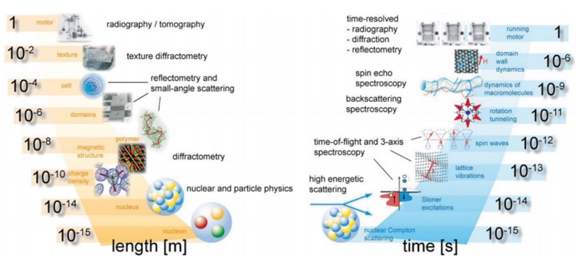
Introduction
Accesible Length and Energy scales



T. Brückel

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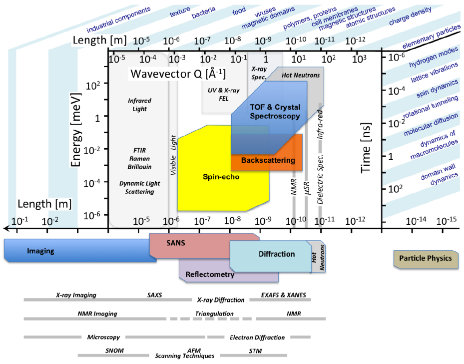
Complementarity



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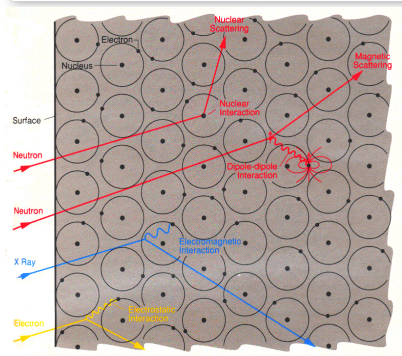
Probing the matter



R Pynn

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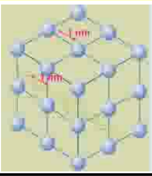
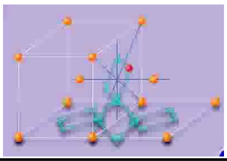
Basic concepts
Properties

- **Neutrons have similar wavelengths that the interatomic distances and therefore they can diffract**
  - Wide range of wavelengths (0.05 to 20 Å)
  - To observe different length scales



- **Same energy range that the atomic and electronic processes (meV a eV) with the possibility to detect changes less than µeV**
  - Tunneling,
  - Rotations
  - Vibrations
  - Electronic Transitions
  - Diffusion.



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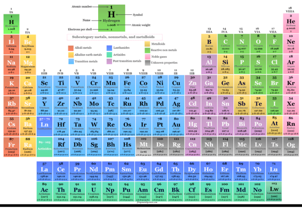
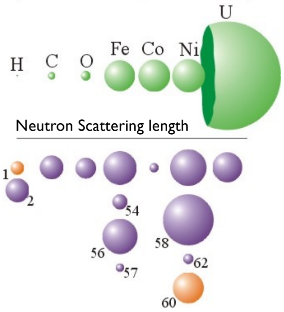
Basic concepts
Properties

- **The interaction neutron-matter is *via* nucleus (*strong interaction*) and not with the electronic cloud**
  - Scattering length for atoms does not depend on the atomic number Z
    - Easy to see light atoms (H, Li) in presence of heavy atoms
    - Easy to distinguish next neighbors atoms in the periodic table





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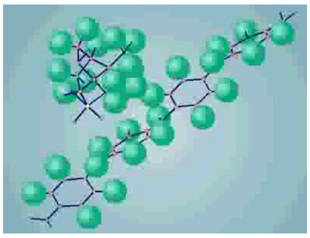
Basic concepts
Properties

The dependence of the scattering length with the nuclei do that different isotopes of the same element scatter in a very different way (isotopic substitution and contrast experiments)



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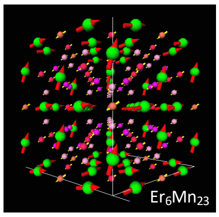
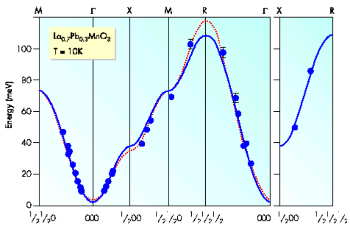
Basic concepts
Properties

Neutrons carry magnetic moment then they "see" (via the magnetic dipolar interaction) the magnetism present in the matter; unpaired electrons, nuclei

- To study magnetic structures
- Magnetic excitations





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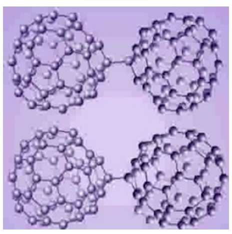
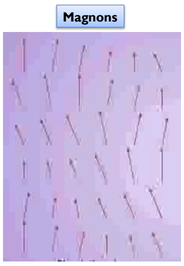
Basic concepts
Properties

- **With neutrons we can measure simultaneously the structure and the dynamics**

Phonons



Magnons



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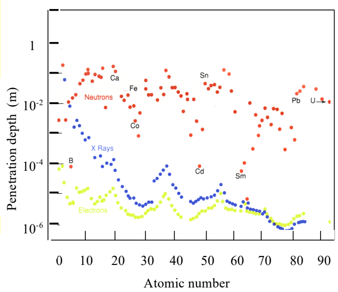
Basic concepts
Properties

Penetration. Neutron enters into materials without problems

- X Ray, EM , optical methods are surface probes.
- Hard exp. conditions
  - Cryostats
  - Furnaces
  - Pression clamps
  - Electrochemical cells
  - Etc...



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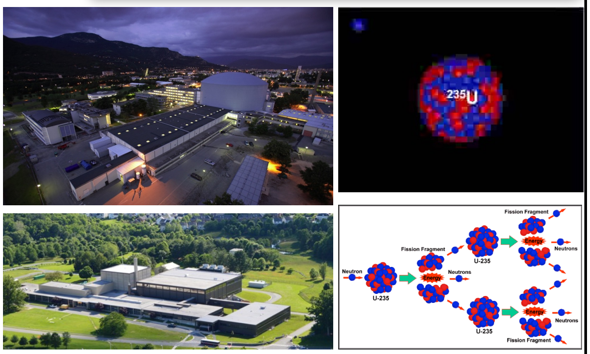
Basic Concepts
Neutron production: nuclear reactors



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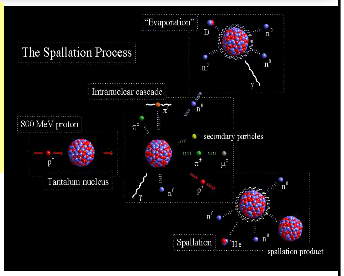
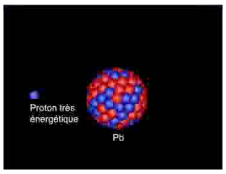
Basic Concepts
Neutron production: spallation sources

- H⁺ accelerated to 1~5 GeV
- Target W, Pb, Hg…
- Average 20 to 25 neutrons / H⁺
- 50 to 16 Hz Pulses
- Pulse time ~ μs to ms
- Pulsed Flux 3.7 10^{16} ns^{-1} (ISIS)





- SNS @ ORNL
- ISIS @ United Kingdom
- MLF @ JPARC

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Basic Concepts
Neutron production: spallation sources

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Basic Concepts
Neutron production: spallation sources

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Basic Concepts
Neutron production: spallation sources

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The European spallation source, ESS

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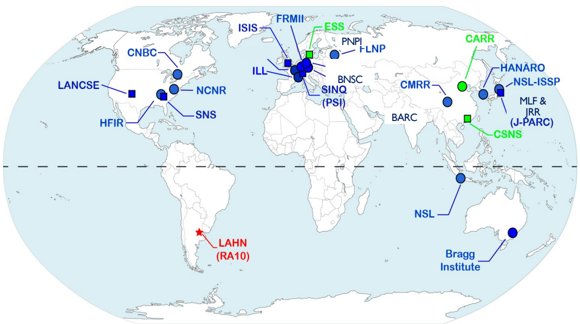
Some neutron sources



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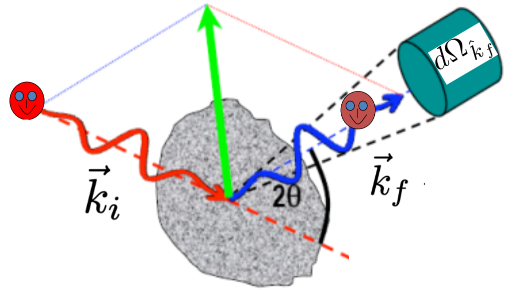
Exploring the matter with neutron scattering

$$\vec{q} = \vec{k}_f - \vec{k}_i$$



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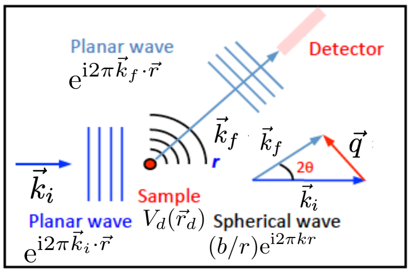
Fermi pseudo potential



$$V_d(\vec{r}_d) = \frac{2\pi\hbar^2}{m} b_d \delta(\vec{r}_d)$$

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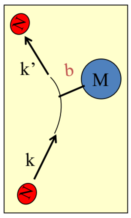
Scattering lenght

- The number b_j determine how the neutron is scattered by the nucleus, is called *scattering length* (is a distance) and

$$V_d(\vec{r}_d) = \frac{2\pi\hbar^2}{m} b_d \delta(\vec{r}_d)$$



- b_j does not depend nor the direction of the incoming neutron neither its wave vector
- b_j is a complex number which depends
  - The particular isotope (isotopic *substitution*)
  - The nuclear spin
  - The neutron energy
- The imaginary part is related with absortion of neutrons by the nuclei
- The real part determine the scattering amplitud and takes values positives and negatives (*contrast*)

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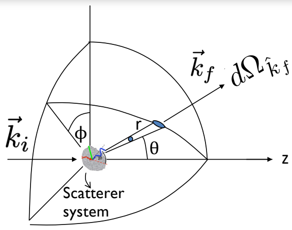
Scattering cross section

- **Double partial differential cross section**



$$\frac{d^2\sigma}{d\Omega_{\hat{k}_f} dE_f} = \frac{\text{Number of scattered neutrons per second in a solid angle } d\Omega_{\hat{k}_f} \text{ in the direction given by } \hat{k}_f \text{ with final energy between } E_f \text{ and } E_f + dE_f}{\varphi \, d\Omega_{\hat{k}_f} dE_f}$$

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Scattering cross section

- Differential cross section

$$\frac{d\sigma}{d\Omega_{\hat{k}_f}} = \frac{\text{Number of neutrons scattered per second in the solid angle } d\Omega_{\hat{k}_f} \text{ in the direction } \hat{k}_f}{\phi \, d\Omega_{\hat{k}_f}}$$

- Total cross section

$$\sigma_{\text{Tot}} = \frac{\text{Scattered neutrons per second}}{\phi}$$

$$\frac{d\sigma}{d\Omega_{\hat{k}_f}} = \int_0^\infty \left(\frac{d^2\sigma}{d\Omega_{\hat{k}_f} dE'}\right) dE' \qquad \sigma_{\text{Tot}} = \int_{\Omega_{\hat{k}_f}} \left(\frac{d\sigma}{d\Omega_{\hat{k}_f}}\right) d\Omega_{\hat{k}_f}$$

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Pair Correlation Function

- Pair Correlation Function depending on time

$$G(\vec{r},t)=\frac{1}{N}\sum_{i,j}\langle\delta\left(\vec{r}-\left[\vec{r}_i(0)-\vec{r}_j(t)\right]\right)\rangle$$

- It is equal to the probability to find two atoms at a distance $\vec{r}$ at a time t
- Gives how the two particles correlations depends on time

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Pair Correlation Function

$G(\vec{r},t)=\frac{1}{N}\sum_{i,j}\langle\delta\left(\vec{r}-\left[\vec{r}_i(0)-\vec{r}_j(t)\right]\right)\rangle$ If $\vec{r}=\vec{r}_2-\vec{r}_1=d_{(1,0)}$ then $G(\vec{r},t)\neq 0$

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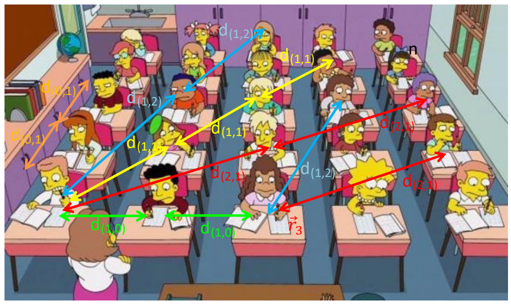
Pair Correlation Function



$G(\vec{r},t)=\frac{1}{N}\sum_{i,j}\langle\delta\left(\vec{r}-\left[\vec{r}_i(0)-\vec{r}_j(t)\right]\right)\rangle$ If $\vec{r}=d_{(1,0)}, d_{(1,1)}, d_{(1,2)}, d_{(2,1)}$... then $G(\vec{r},t)\neq 0$

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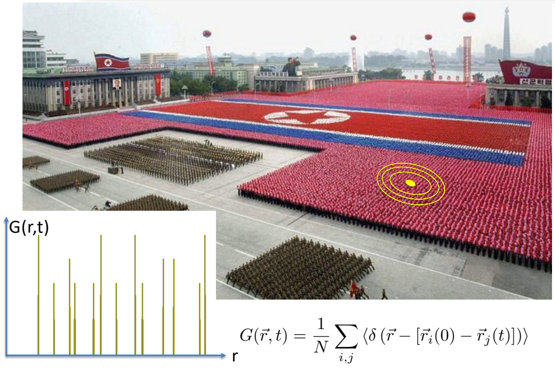
Pair Correlation Function



$G(\vec{r},t)=\frac{1}{N}\sum_{i,j}\langle\delta\left(\vec{r}-\left[\vec{r}_i(0)-\vec{r}_j(t)\right]\right)\rangle$

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Pair Correlation Function

$G(\vec{r},t)=\frac{1}{N}\sum_{i,j}\langle\delta\left(\vec{r}-\left[\vec{r}_i(0)-\vec{r}_j(t)\right]\right)\rangle$

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Dynamic Structure Factor

- Dynamic Structure Factor

$$S(\vec{q},\nu)=\int\frac{dt}{h}\mathrm{e}^{-\mathrm{i}2\pi\nu t}\int d^3r\,\mathrm{e}^{\mathrm{i}2\pi\vec{q}\cdot\vec{r}}\,G(\vec{r},t)$$

- Scattering Law

Experiment, Probe, Scatterer

$$\boxed{I(\vec{q},\nu)=\frac{d^2\sigma}{d\Omega_{\hat{k}_f}dE_f}}=\boxed{\frac{\sigma}{4\pi}\frac{k_f}{k_i}}\boxed{N_n\,S(\vec{q},\nu)}$$

- The number of scattered neutrons is proportional to the Fourier Transform in time and space of the pair correlation function $G(\vec{r},t)$

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Coherent and incoherent scattering

Coherent — Incoherent

$$I(\vec{q},\nu) = \frac{d^2\sigma}{d\Omega_{\hat{k}_f} dE_f} = \frac{k_f}{k_i} N_n \left[\frac{\sigma_{\rm coh}}{4\pi} S_{\rm coh}(\vec{q},\nu) + \frac{\sigma_{\rm inc}}{4\pi} S_{\rm inc}(\vec{q},\nu)\right]$$

$$\sigma = \sigma_{\rm coh} + \sigma_{\rm inc}$$

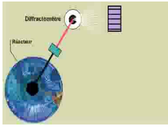
Squared mean

$$\sigma_{\rm coh} = 4\pi\,|\langle b\rangle|^2$$

Structural Information

Collective dynamics

Correlations two particles <i,j>



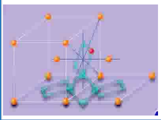
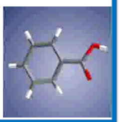
variance

$$\sigma_{\rm inc} = 4\pi\left(\left\langle |b|^2\right\rangle - |\langle b\rangle|^2\right)$$

Diffusive motions

Internal dynamics

Self Correlation <i, i(t)>

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Coherent and incoherent scattering

- **Coherent involves correlations to two nuclei (j,l)**
  - Describe interference phenomena produced by scattering of a neutron by all nuclei in the sample.
  - Depends on the scattering angle
    - Elastic → Structure
    - Inelastic → Collective modes
- **Incoherent involves correlations between the position of atom *j* at zero time and the same atom at t time.**
  - Not interference between scattering waves coming from differents nuclei.
  - Incoherent scattering gives acces to study diffusion phenomena
  - It is an isotropic scattering

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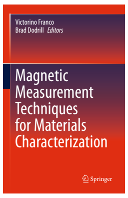
Bibliography

- G. L. Squires, "*Thermal neutron scattering*", Cambridge Universtity Press, 1978
- S.W. Lovesey, "*Theory of neutron scattering from Condensed Matter*", Vol I y II Oxford University Press, 1986
- Javier Campo and Víctor Laliena "*Neutron Scattering in Magnetism: Fundamentals and Examples*" https://doi.org/10.1007/978-3-030-70443-8_14



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Diffraction
Structure factors

$$I(\vec{q}) = \frac{(2\pi)^3}{V_0}\sum_{\vec{H}} \delta(\vec{H}-\vec{q})|N_{\vec{q}}|^2$$ Diffraction

$$N_{\vec{q}} = \sum_d \Psi_d\, e^{i2\pi\vec{q}\cdot\vec{r}_d} e^{-W_d(\vec{q})}$$ Nuclear Structure factor

$$\Psi_d(\vec{q}) = b_d$$ Neutrons *Scattering lenght* $$V_d(\vec{r}_d) = \frac{2\pi\hbar^2}{m} b_d \delta(\vec{r}_d)$$

$$\Psi_d(\vec{q}) = f_d(\vec{q})$$ XR *Form factors* $$V_d(\vec{r}_d) \propto \rho_d(\vec{r}_d)$$

Debye-Waller factor $$W_d(\vec{q}) = \langle\{\vec{q}\cdot\vec{u}_d(0)\}^2\rangle$$

$$T_d(\vec{q}) = e^{-W_d(\vec{q})}$$

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General expression for NON polarised neutrons

$$\frac{d\sigma^{(n,e)}_{\rm coh}}{d\Omega_{\hat{k}_f}} = \frac{N_c}{V}\sum_{\vec{H}} \delta(\vec{q}-\vec{H})|N_{\vec{q}}|^2.$$

Nuclear structure factor

$$N_{\vec{q}} = \sum_d^{N_{uc}} b_d\, T_d(\vec{q})\, e^{i2\pi\vec{q}\cdot\vec{r}_d}$$

$$\frac{d\sigma^{(m,e)}_{\rm coh}}{d\Omega_{\hat{k}_f}} = \frac{N_c}{V}\sum_{\vec{H}\vec{K}} \delta(\vec{q}-\vec{H}-\vec{K})\,|\vec{M}_{\perp\vec{q}}|^2$$

Magnetic Interaction Vector

$$\vec{M}_{\perp\vec{q}} = \hat{q}\times(\vec{M}_{\vec{q}\vec{K}}\times\hat{q})$$

Magnetic Structure factor

$$\vec{M}_{\vec{q}\vec{K}} = p\sum_{d_m}^{N_{uc}} F_{d_m}(\vec{q}) T_{d_m}(\vec{q})\, e^{i2\pi\vec{q}\cdot\vec{r}_{d_m}} \vec{S}_{\vec{K}d_m}.$$

$$\vec{q} = \vec{H} \pm \vec{K}$$

Reciprocal Lattice Vector

Propagation vector

Fourier Coefficients

$$I_{\vec{q}} = |N_{\vec{q}}|^2 + |\vec{M}_{\perp\vec{q}}|^2$$

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Blume-Maleyev equations

Cross-section (Equation 1):

$$I_{\vec{q}} = |N_{\vec{q}}|^2 + |\vec{M}_{\perp\vec{q}}|^2 + \underbrace{(N_{\vec{q}}\vec{M}^*_{\perp\vec{q}} + N^*_{\vec{q}}\vec{M}_{\perp\vec{q}})}_{\text{Nuclear-Magnetic Interference Vector}}\cdot\vec{P}_i + {\rm i}\underbrace{(\vec{M}^*_{\perp\vec{q}}\times\vec{M}_{\perp\vec{q}})}_{\text{Chiral Vector}}\cdot\vec{P}_i$$

$$\vec{q} = \vec{H} \pm \vec{K}$$

Initial Polarization

Final polarisation (Equation 2):

$$I_{\vec{q}}\vec{P}_f = \left(|N_{\vec{q}}|^2 - |\vec{M}_{\perp\vec{q}}|^2\right)\vec{P}_i + \left(\vec{P}_i\cdot\vec{M}^*_{\perp\vec{q}}\right)\vec{M}_{\perp\vec{q}} + \left(\vec{P}_i\cdot\vec{M}_{\perp\vec{q}}\right)\vec{M}^*_{\perp\vec{q}}$$
$$-{\rm i}\left(N_{\vec{q}}\vec{M}^*_{\perp\vec{q}} - N^*_{\vec{q}}\vec{M}_{\perp\vec{q}}\right)\times\vec{P}_i + N_{\vec{q}}\vec{M}^*_{\perp\vec{q}} + N^*_{\vec{q}}\vec{M}_{\perp\vec{q}}$$
$$-{\rm i}\left(\vec{M}^*_{\perp\vec{q}}\times\vec{M}_{\perp\vec{q}}\right)$$

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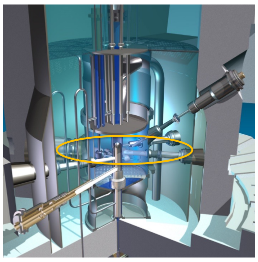
ILL reactor



- compact design
- heavy-water cooling
- single control rod

- 57 MW thermal power
- cold, thermal, hot sources
- neutron flux 5×10^{18} n/cm²/s

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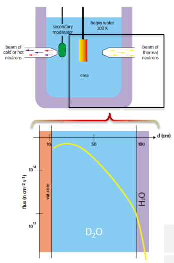
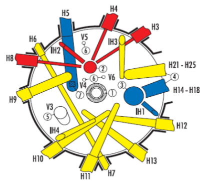
ILL reactor





- fast neutrons need to be slowed down
- energy dissipated through collisions
- light non-absorbing elements work best
- neutrons extracted close to the core → maximum flux

	cold	thermal	hot
moderator	liquid D_2	liquid D_2O	graphite
moderator T	20K	300K	2000K
neutron λ	3-20 Å	1-3 Å	0.3-1

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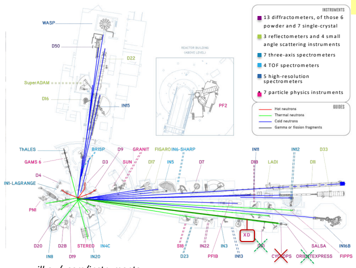
ILL instrument suit

29 instruments
9 CRG instruments



www.ill.eu/users/instruments

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Spanish instruments at the ILL: XtremeD and D1B

Two Spanish instruments at the ILL (XtremeD and D1B) where we could perform "*high pressure*" experiments

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In brief

In brief

A **dedicated** diffractometer optimized for extreme conditions (**high P, high H**, low and high T) but with **flexibility** to accommodate a broad range of studies

Technically:
A flexible powder diffractometer with single crystal capabilities, with a variable focused beam on the sample, optimized signal/background ratio, big solid angle position-sensitive detector, and dedicated sample environment

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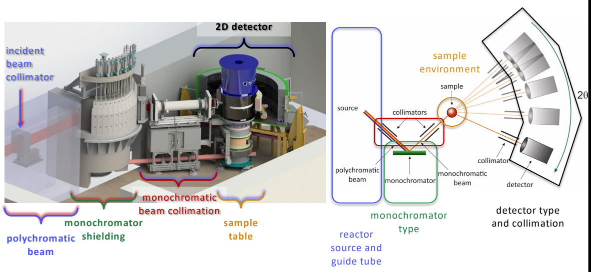
XtremeD



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Construction works H24

Floor @ ILL7

Marble tanzboden

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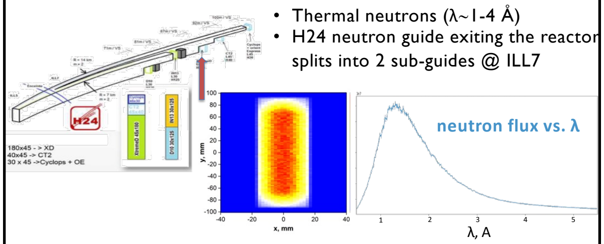
Neutron guide H24

- Curved guide, m=3, L=87m, H×W=180×45 mm^2
- Thermal neutrons (λ~1-4 Å)
- H24 neutron guide exiting the reactor splits into 2 sub-guides @ ILL7



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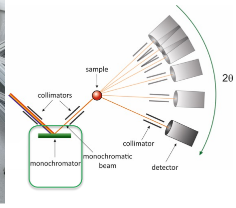
Monochromators

Shielding

Goniometer



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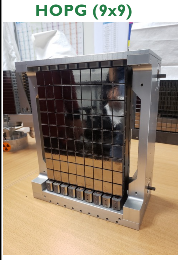
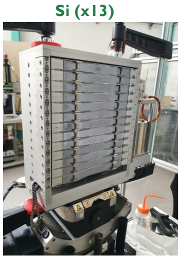
Monochromators

HOPG (9x9)



Si (x13)



Characteristics

	HOPG	Si
Number SCs	9x9	13
Reflecting plane	(002)	(111), (113), (331), (533)
λ range	2.29 – 3.5Å	1.0 – 3.5Å
Take-off	38 – 62°	38 – 140°
λ/2, λ/3	Yes	No
Filter	Yes	No
Focusing	H + V	H + V

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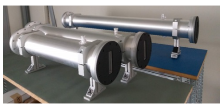
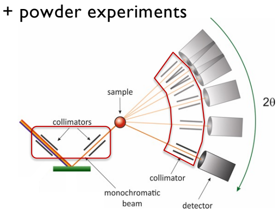
Collimation

Diaphragms & vacuum tubes



SC + powder experiments



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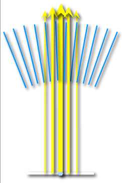
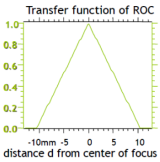
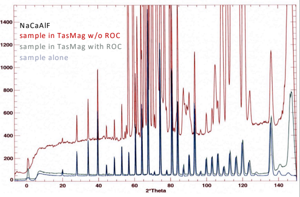
Collimation

Radial oscillating collimator





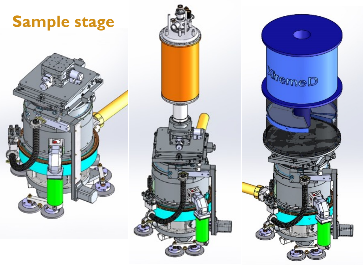
ROC & data quality



50

Sample environment

Sample stage



Characteristics

XY translation	± 100 mm
Z translation	± 50 mm
linear accuracy	± 0.05 mm
tilting range	± 20°
ω rotation	+180° to -180°
angular accuracy	± 0.005°
maximum load	15000 N

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Sample environment

cryostat **PE press** **cryomagnet** **HT furnace** **pressure cell**

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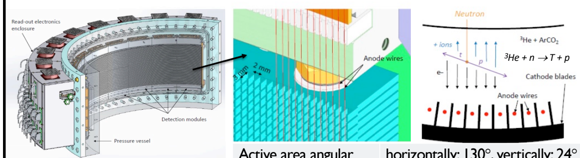
Detector: trench multi-wire proportional



- High efficiency and angular resolution
- Excellent γ-event rejection ⇒ low background
- Cathode trench configuration ⇒ uniform gain along the wire
- Spatial confinement of neutron events

Active area angular coverage	horizontally: 130°, vertically: 24°
Angular resolution	Horizontally: 0.15°, vertically: 0.2°
Height	32 cm
Radius of curvature	76 cm
Efficiency @ 2.5 Å	75%
Local counting rate	50 kHz per channel
Global counting rate	2 MHz

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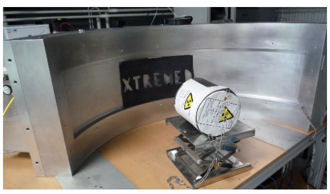
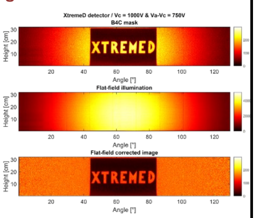
Detector

B_4C mask in front of the detector



Images taken with an AmBe source



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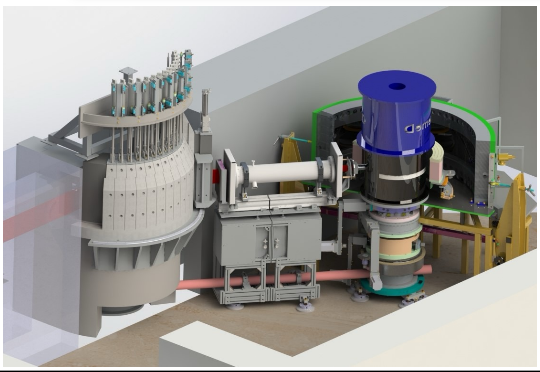
XtremeD



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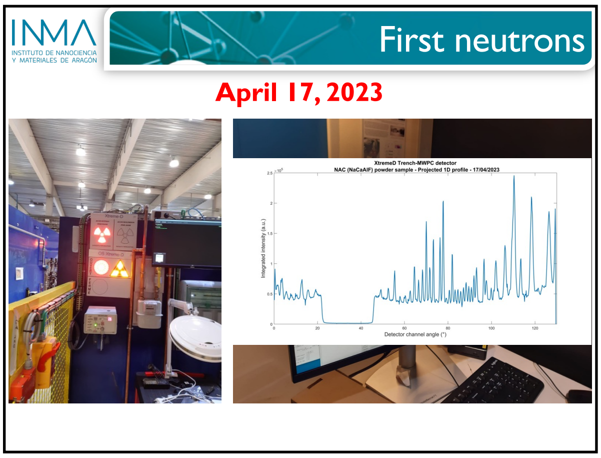
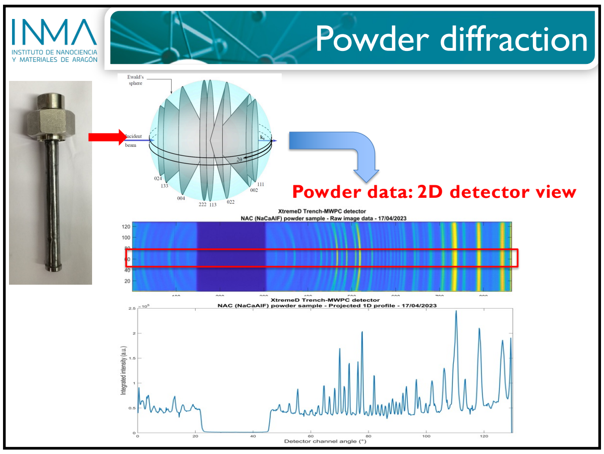


57



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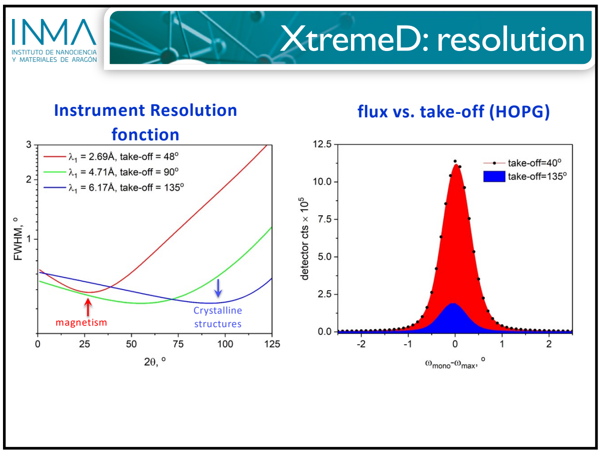

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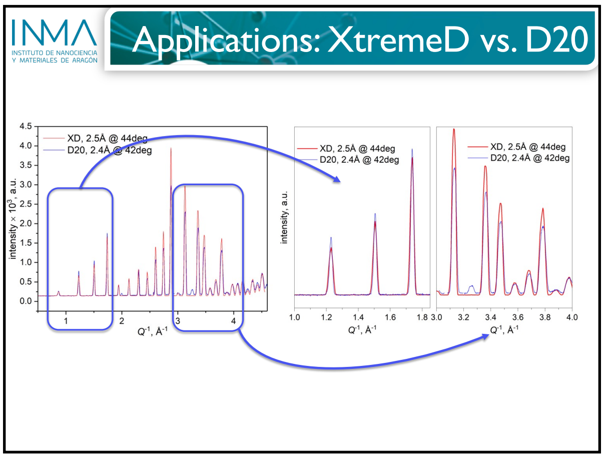
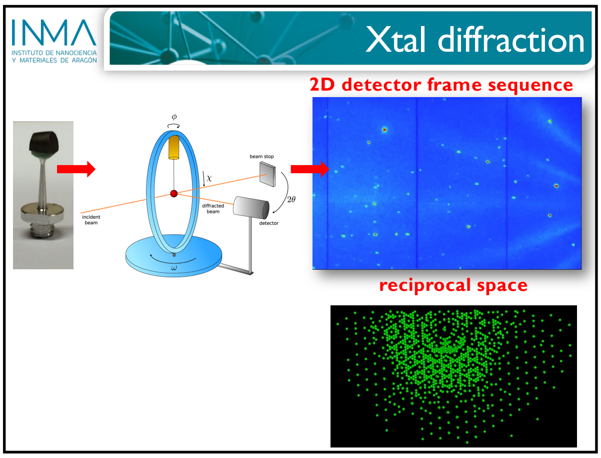


60



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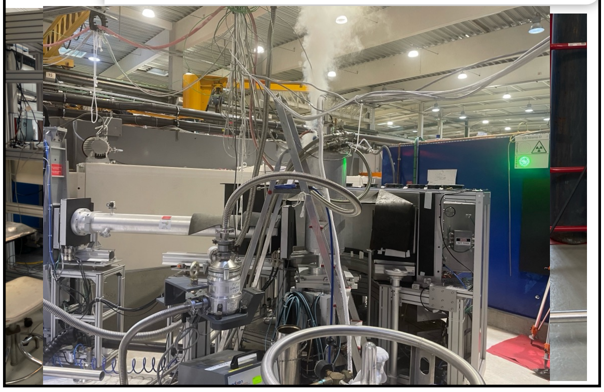
XtremeD instrument



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D1B instrument

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What is around?

How XtremeD compares?

In the general diffraction context:

- Less P than X-ray but essential for **light elements**, contrast, 'high quality' crystallography and **magnetism**

In the neutron facilities context:

- High intensity at low Q (w.r.t. most short pulse spallation instruments)
- Extended H & P range and **combination** of both parameters
- Both powders & single crystals
- Flexibility

In ILL's context:

- **Dedicated**
- Flux ~D20 but better signal to noise ratio and bigger solid angle
- Only single Xtal diffractometer at ILL with large 2D detector + high H (D19: space restrictions for high magnetic fields)

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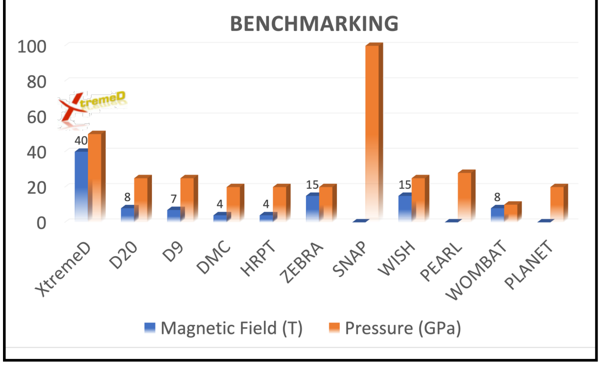
What is around?



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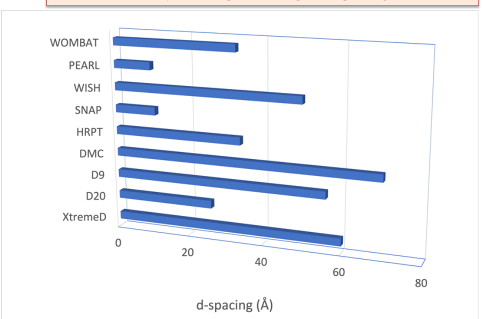
What is around?

Short Pulse Spallation source = Inaccessibility to low Q-reflections
VERY IMPORTANT FOR MAGNETISM



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Outline

- Introduction
- Basic concepts
- Instruments
- Examples
- How to apply for beam time

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Scientific case

Study of matter under extreme conditions

Necessary for progress in Condensed Matter Physics, Solid State Chemistry and Geosciences, and even in Molecular Biology

3 representative topics:

Structure

- Water-related systems
- Molecular chemistry

Magnetism

- Magnetic materials under extreme conditions

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Scientific case

Water-related systems

What would be the *XtremeD* contribution?

The study of these systems implies systematic studies of **hydrogen bonding and clustering, host-guest interactions in clathrate hydrates and the role of water in crystals.**

Far ranging implications in physics, chemistry, planetary sciences and geochemistry

- Planetary ices (water, methane, ammonia) and clathrates
- Segregation and related phenomena in water solutions at extreme conditions
- Mineral hydration: implications for the water cycle in Earth crust and mantle

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Scientific case

Molecular chemistry

high social contribution for an extreme conditions diffractometer

- Simple molecular systems
- Energy materials
- Production and characterization of new phases: pharmaceutical applications / molecular materials
- Extreme conditions studies on established phases: material applications, phase transformations, chemical reactions (green chemistry)
- "in situ" and "in operando"

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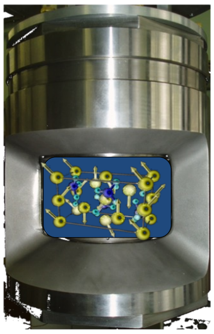
Scientific case

Magnetism & superconductivity under extreme conditions

- Transition metal oxides
- Multiferroics and magneto-electric materials
- Exotic magnetic states in magnetically frustrated materials (Spin liquid, etc…)
- Skyrmions
- Magnetic molecular materials (Spin crossover, etc…)
- Hydrides (superconductivity)



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Scientific case

PNAS

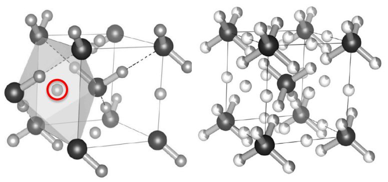
Neutron diffraction observations of interstitial protons in dense ice

Malcolm Guthrie[a,1], Reinhard Boehler[a], Christopher A. Tulk[b], Jamie J. Molaison[b], António M. dos Santos[b], Kuo Li[a], and Russell J. Hemley[a,1]

location of hydrogen atoms

25-50 GPa

RT



Dense ices:

- Starting of the destabilisation of water molecule.
- This phase, precursor of superionic behaviour (ice X)?
- Intermediate dissociation, with **H occupying of interstitial sites**

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Scientific case

Magnetism: quantum materials

Skyrmions, magnetic monopoles, frustrated magnetism…

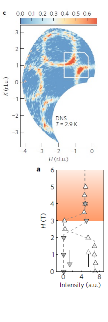
nature physics ARTICLES

PUBLISHED ONLINE: 24 OCTOBER 2016 | DOI: 10.1038/NPHYS3914

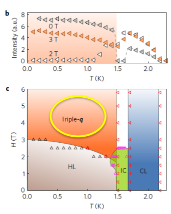
Spiral spin-liquid and the emergence of a vortex-like state in $MnSc_2S_4$

Shang Gao[1,2], Oksana Zaharko[1*], Vladimir Tsurkan[3,4], Yixi Su[5], Jonathan S. White[1], Gregory S. Tucker[1,6], Bertrand Roessli[1], Frederic Bourdarot[7], Romain Sibille[1,8], Dmitry Chernyshov[9], Tom Fennell[1], Alois Loidl[3] and Christian Rüegg[1,2]

Tuning the ground state by pressure/magnetic field



- A new type of spiral state: spins fluctuate collectively as spirals
- Vortex-like triple-q phase under magnetic field.



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Scientific case

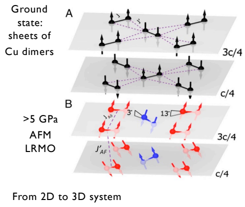
Magnetism: quantum materials

Skyrmions, magnetic monopoles, frustrated magnetism…

PNAS

Emergence of long-range order in sheets of magnetic dimers

Tuning the magnetic ground state of $SrCu_2(BO_3)_2$ by pressure and magnetic field



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Scientific case

Structure (& magnetism) Superconductivity

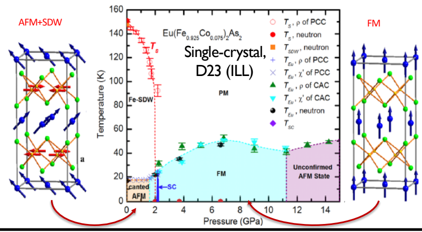
Fe-based superconductors

Coexistence of< superconductivity and LRMO?

SCIENTIFIC REPORTS

OPEN **Hydrostatic pressure effects on the static magnetism in $Eu(Fe_{0.925}Co_{0.075})_2As_2$**

2017



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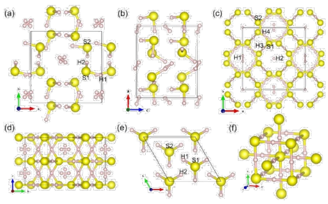
Scientific case

Structure (& magnetism) Superconductivity

LETTER

Conventional superconductivity at 203 kelvin at high pressures in the sulfur hydride system



Hydrides

- P > 150 Gpa → beyond the limits (for the moment)
- Location of hydrogen
- Crucial role of neutrons in superconductivity

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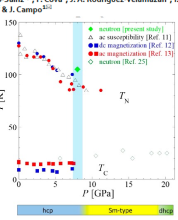
Magnetism of rare earths at high pressure

scientific reports

OPEN **Revisiting the magnetic structure of Holmium at high pressure by using neutron diffraction**

M. Pardo-Sainz, F. Cova, J. A. Rodríguez-Velamazán, I. Puente-Orench, Y. Kousaka, M. Mito & J. Campo



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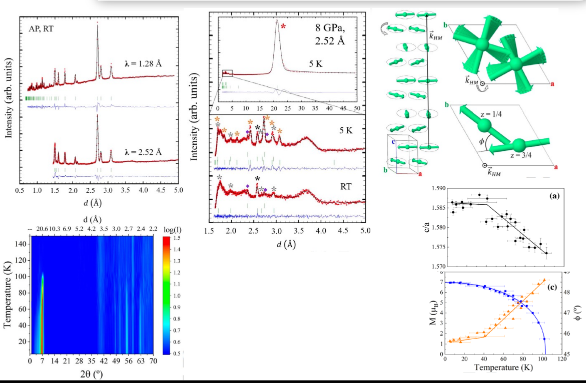
Magnetism of rare earths at high pressure



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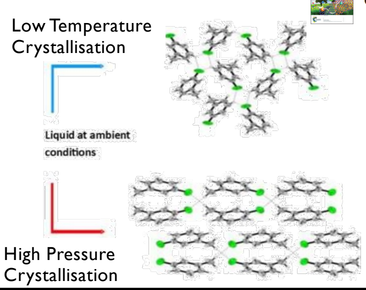
Scientific case

Molecular chemistry under extreme conditions

Structure of organic solids at low temperature and high pressure

Rachael Lee, Judith A. K. Howard, Michael R. Probert and Jonathan W. Steed

From the journal: **Chemical Society Reviews**



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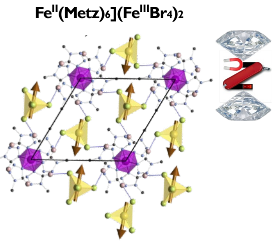
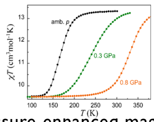
Scientific case

Materials responsive to external stimuli: Molecular materials

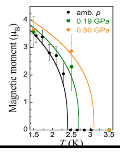
- Combination of spin-crossover and long range magnetic order
- Pressure-induced SCO

$Fe^{II}(Metz)_6](Fe^{III}Br_4)_2$





- Pressure-enhanced magnetism



J.A. Rodríguez-Velamazán et al. Chem.Eur. J. (2014)

80

Ringwoodite $[(Mg,Fe^{2+})_2SiO_4$ spinel]

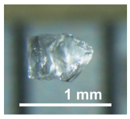
research papers

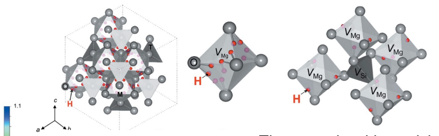
STRUCTURAL SCIENCE CRYSTAL ENGINEERING MATERIALS

ISSN 2052-5206

Determination of hydrogen site and occupancy in hydrous Mg_2SiO_4 spinel by single-crystal neutron diffraction

Narangoo Purevjav, Takuo Okuchi, Xiaoping Wang, Christina Hoffmann and Naotaka Tomioka





Refined crystal structure of hydrous ringwoodite along [111] and hydrogen sites in a vacant Mg site.

The most plausible model (3H+ at 192*i* sites within an Mg-vacant octahedron)

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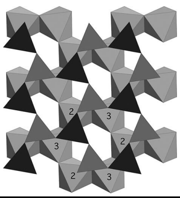
Cation order-disorder

- **Cation order-disorder** in Olivines and Spinels at high temperature
  - Fe-Ni, Mn-Fe, Mg-Al, Ni-Mn, Ni-Mg, etc…
- Mg/Al **Ordering** in Dioctahedral Micas
  - $K_{0.9}[Mg_{0.58}Al_{1.43}][Si_{3.57}Al_{0.43}]O_{10}(OH)_2$
- Al/Si and Mg/Al **ordering** in high-temperature amphibole
  - $NaCa_2[Mg_4Al]\ [Si_6Al_2]O_{22}(OH)_2$,



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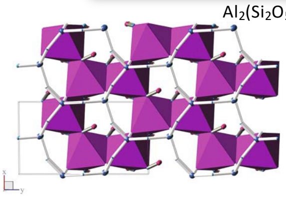
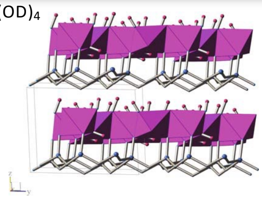
Kaolinite $[Al_2(Si_2O_5)(OD)_4]$

$Al_2(Si_2O_5)(OD)_4$





Crystal structure of kaolinite as refined from neutron diffraction. Layer slab with the $[AlO_6]$-octahedra in dark shading showing hydroxyl terminations between layers (left) and the excess hydroxyl between tetrahedral silicate layer and those oxygens that are not charge balanced (right) are shown.

Neutrons clearly identify the location of the protons

E. Akiba, et al. Clays Clay Miner. 45, 781 (1997)

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Phase transformations

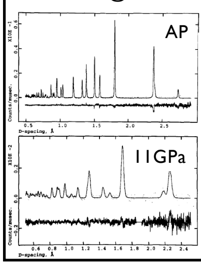
Static Compression and H Disorder in Brucite, $Mg(OH)_2$, to 11 GPa: a Powder Neutron Diffraction Study

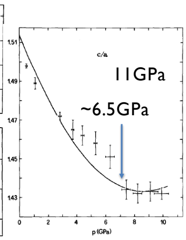
M. Catti, G. Ferraris, S. Hull, A. Pavese

PHYSICS AND CHEMISTRY OF MINERALS

© Springer-Verlag 1995

Polaris @ISIS





The onset of H disorder, and a jump of the c/a ratio vs. pressure at ~6.5 GPa, may be related to a second-order phase transition consistent with Raman results.

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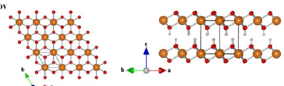
Effect of the H-D isotopes

Phys Chem Minerals (2010) 37:741–749
DOI 10.1007/s00269-010-0372-5

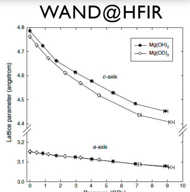
ORIGINAL PAPER

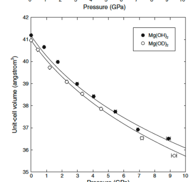
High-pressure neutron diffraction study on H–D isotope effects in brucite

Juske Horita · António M. dos Santos · Christopher A. Tulk · Bryan C. Chakoumakos · Veniamin B. Polyakov



WAND@HFIR





- a axis, parallel to the sheets of Mg–O octahedra, decrease only slightly with pressure with no effect of H–D substitution.
- c axis of $Mg(OD)_2$ is shorter and may exhibit greater compressibility with pressure than that of $Mg(OH)_2$.
- brucite preferentially incorporate deuterium over hydrogen under pressure

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Symmetrization O-H···O

SCIENTIFIC REPORTS

OPEN **Direct observation of symmetrization of hydrogen bond in δ-AlOOH under mantle conditions using neutron diffraction**

Received: 18 June 2018
Accepted: 1 October 2018
Published online: 19 October 2018

Asami Sano-Furukawa[1], Takanori Hattori[1], Kazuki Komatsu[2], Hiroyuki Kagi[2], Takaya Nagai[3], Jamie J. Molaison[4], António M. dos Santos[4] & Christopher A. Tulk[4]

Theoretical studies have indicated that symmetrisation triggers an increase in the bulk modulus and that it modifies the seismic wave velocity

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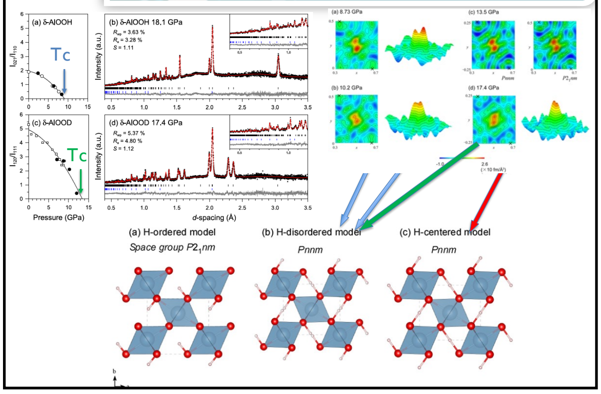
Symmetrization O - H ··· O



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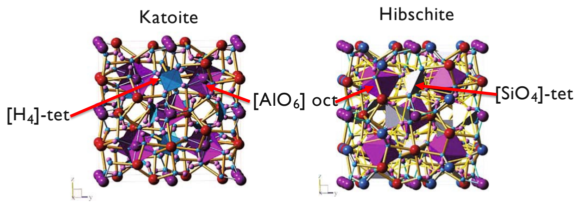
Hydrogarnets

- Hydrogarnet katoite, $Ca_3Al_2(O_4D_4)_3$, is used as a model for incorporation of (OH) into garnets and has been proposed as water carriers



in katoite the $[SiO_4]$-tet is almost completely replaced by the $[H_4O_4]$-tet, in hibschite, $Ca_3Al_2(SiO_4)_{2.30}(OD)_{2.8}$, still contains considerable amounts of residual $[SiO_4]$

G. A. Lager et al

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High Pressure and Temperature

Physics and Chemistry of Minerals (2019) 46:459–469
https://doi.org/10.1007/s00269-018-1016-4

ORIGINAL PAPER

Crystal structure change of katoite, $Ca_3Al_2(O_4D_4)_3$, with temperature at high pressure

Atsushi Kyono[1] · Masato Kato[1] · Asami Sano-Furukawa[2] · Shin-Ichi Machida[3] · Takanori Hattori[2]

effect of hydrogen on the katoite's phase transition under high-pressure and high-temperature conditions,

→ → high-temperature neutron diffraction measurements at about 8 GPa where it is expected that the phase transition from *Ia3d* to another one is observed

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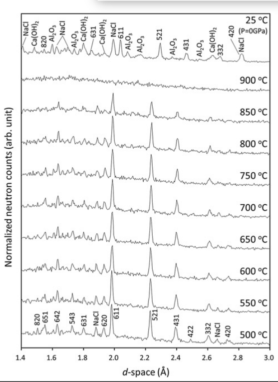
Thermal decomposition of katoite at high pressure



At 850 °C, all peaks corresponding to katoite start to decrease in intensity

the peaks from NaCl also disappear

katoite virtually unchanged during heating from 25 to 800 °C,

The sample pressure increased gradually from 7.1 to 7.8 GPa on heating up to 850 °C.

After cooling to RT and subsequent decompression to ambient pressure, the peaks of katoite reappear, together with those of corundum Al_2O_3 and portlandite $Ca(OH)_2$.

high pressure strongly suppresses the dehydration of katoite.

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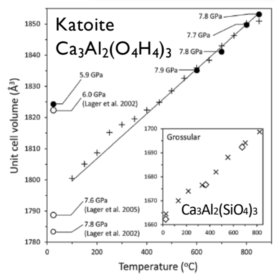
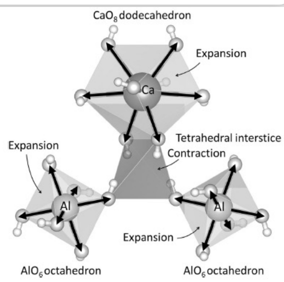
P-T phase diagram





By a further increase in pressure, hydrous silicates expand their stable temperature region to the higher temperatures. This study revealed that katoite remains fundamentally stable over a wide P–T area, at least up to 8 GPa and 850 °C.

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Outline

- Introduction
- Basic concepts
- Instruments
- Examples
- How to apply for beam time

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How to apply for beamtime

- Do you have a good idea for neutron scattering?
- Is neutron scattering the only technique to solve the problem?
- Do you have any previous experiments relevant for the proposal?
- Did you contact any scientist specialist in NS to help you with the more technical questions? (Local Contact…)
- Did you think in the expected results of the experiment?
- How will you analyze your data?
- How did you estimate the requested beamtime?
- Which is the best instrument and neutron source?
- Etc…

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Thanks

Thank you very much for your attention !!

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