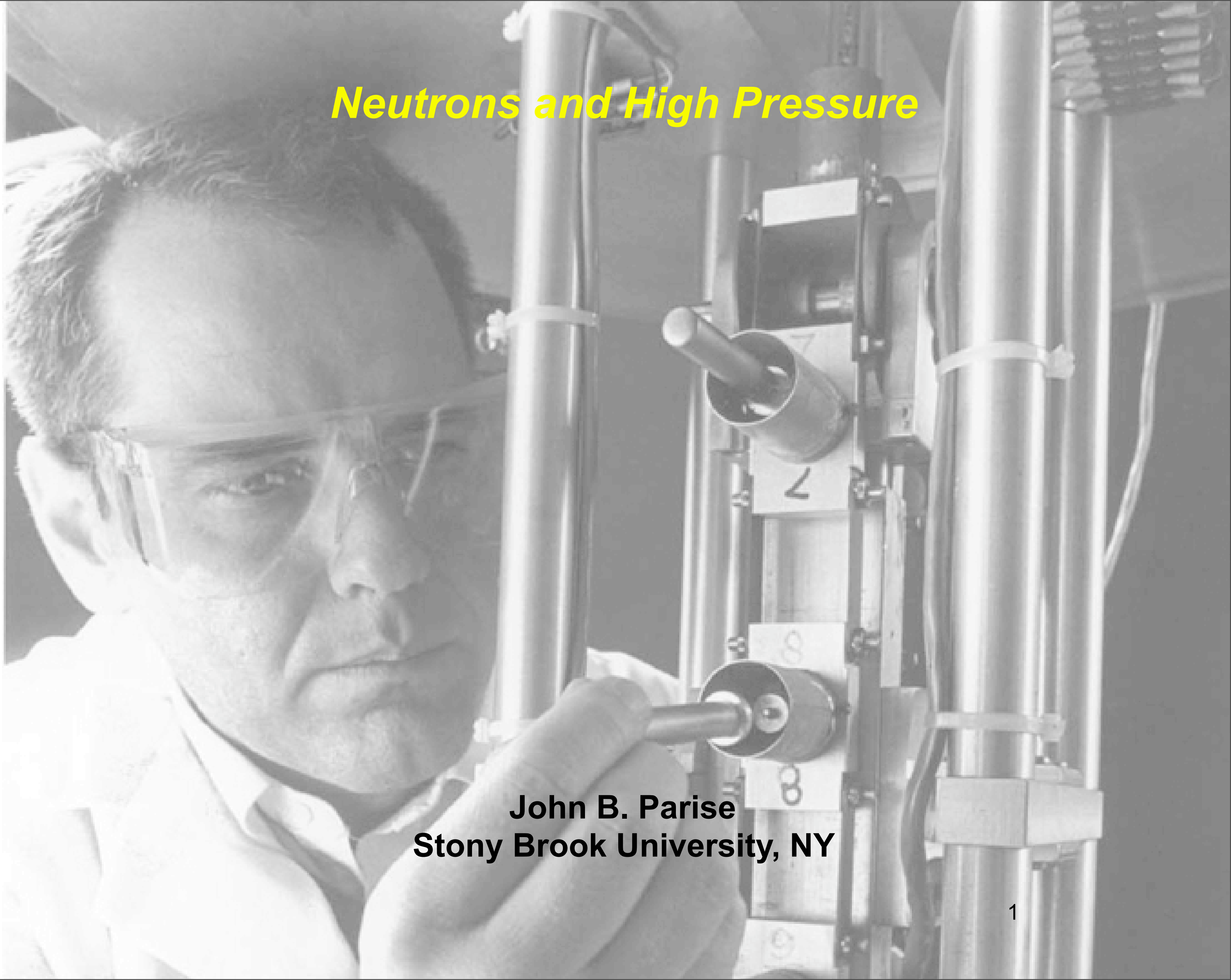
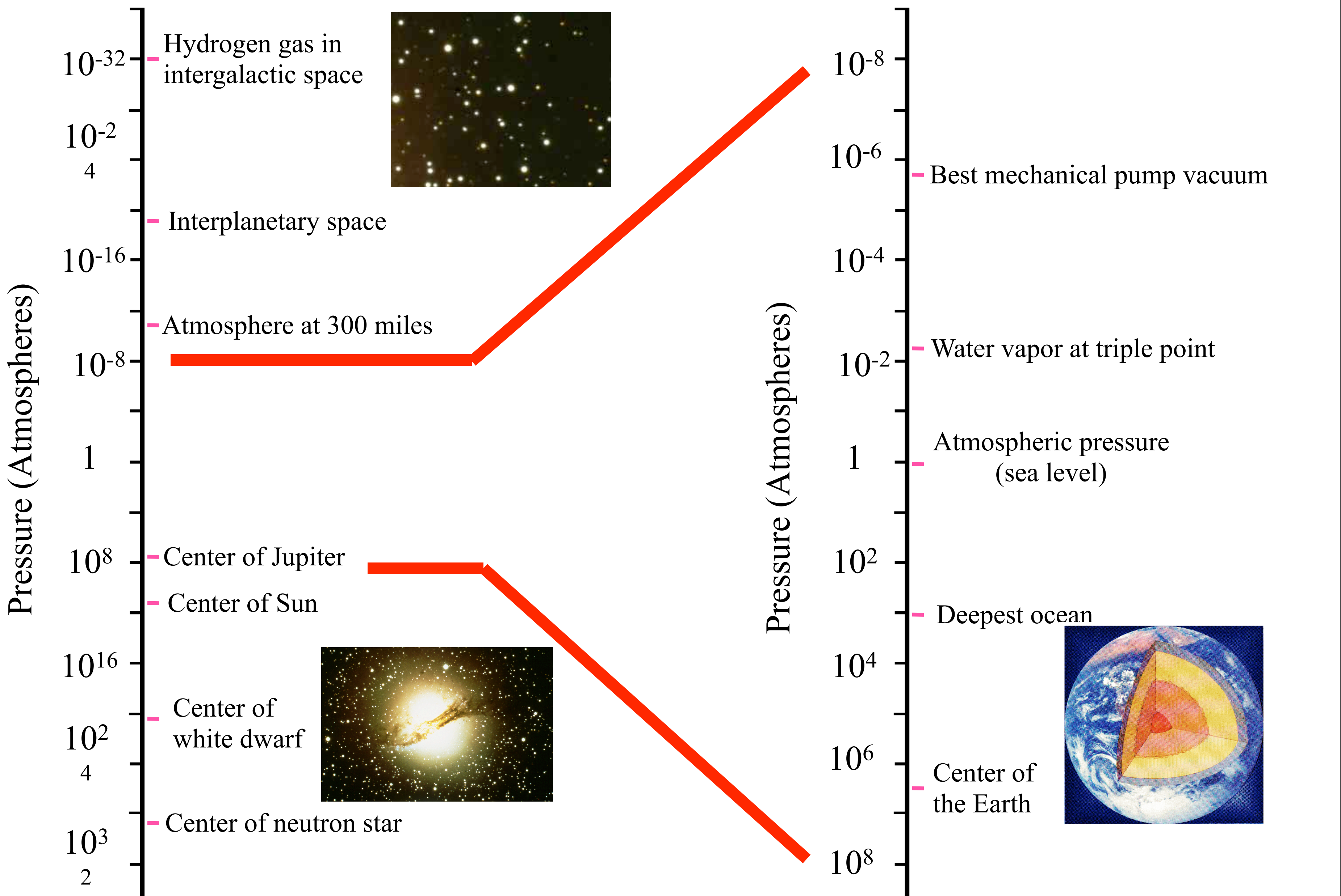


Neutrons and High Pressure

John B. Parise
Stony Brook University, NY

RANGE OF PRESSURES IN THE UNIVERSE



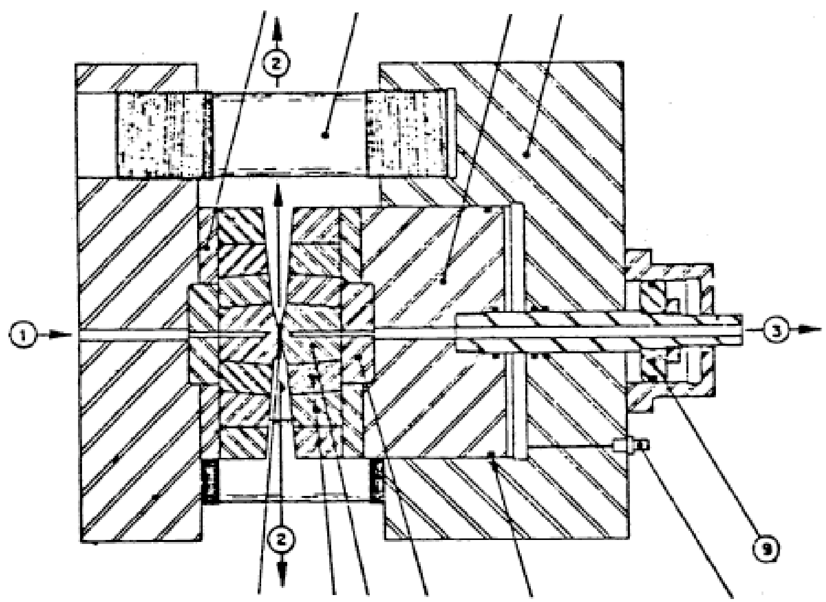
Why Pressure? Science Issues Addressed

- **Nature of dense hydrogen - *cryogenic to brown dwarf conditions***
- **Composition, elasticity, and thermal state of Earth's core**
- **Structures of complex hydrous phases**
  - ***Clathrates, molecular compounds, hydrous silicates, metal hydrides***
- **Hydrogen bonding - *Organic & inorganic systems, inc. liquids***
- **Supercritical fluids and liquids**
  - ***Structure and dynamics and effect on chemical reactions***
- **Structure and dynamics of silicate melts and glasses**
- **Planetary ices - *Structure, strength, and dynamics***
- **Influence of pressure and stress on magnetic properties**
- **Structure and dynamics of nanomaterials under pressure**
- **General phase transition studies**
- **Chemical kinetics and reaction mechanism - *inner vs. outer sphere***
- **Your favorite application here**

Early cells (courtesy JD Jorgensen)

R. M. Brugger, R. B. Bennion, T. G. Worlton, and W. R. Meyers, Trans. Amer. Crystallogr. Assn. 5, 141 (1969).

100 kbar opposed-anvil press for time-of-flight neutron powder diffraction



Al_2O_3 anvils with 1 inch diameter faces

Sample volume = 0.05 cm^3

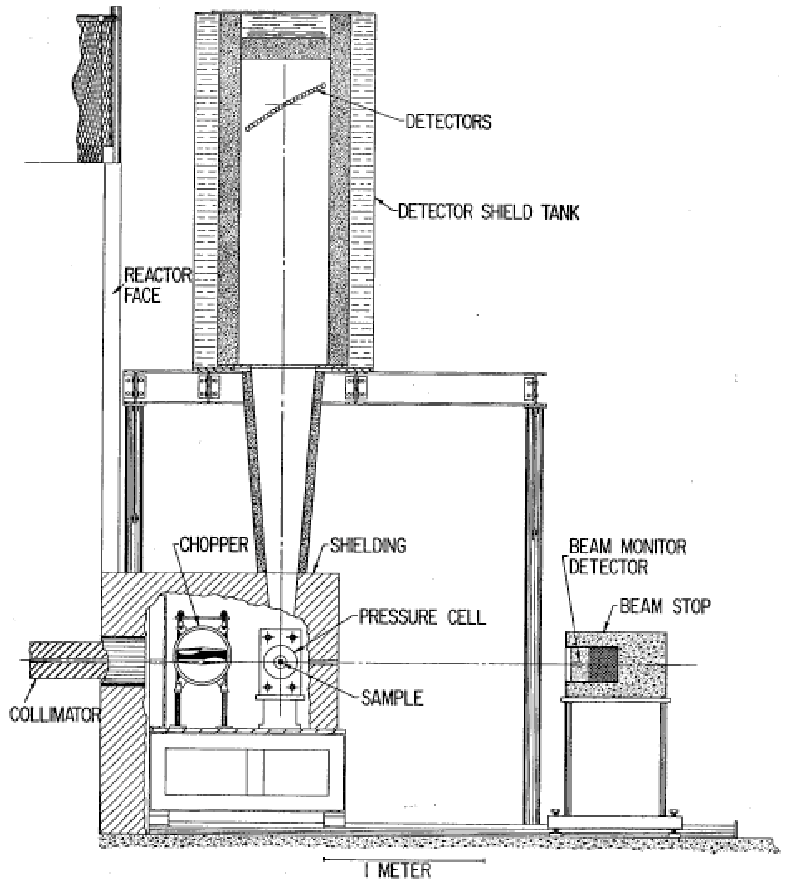
(0.6 cm diam. x 0.3 cm thick)

From early experiments at reactors

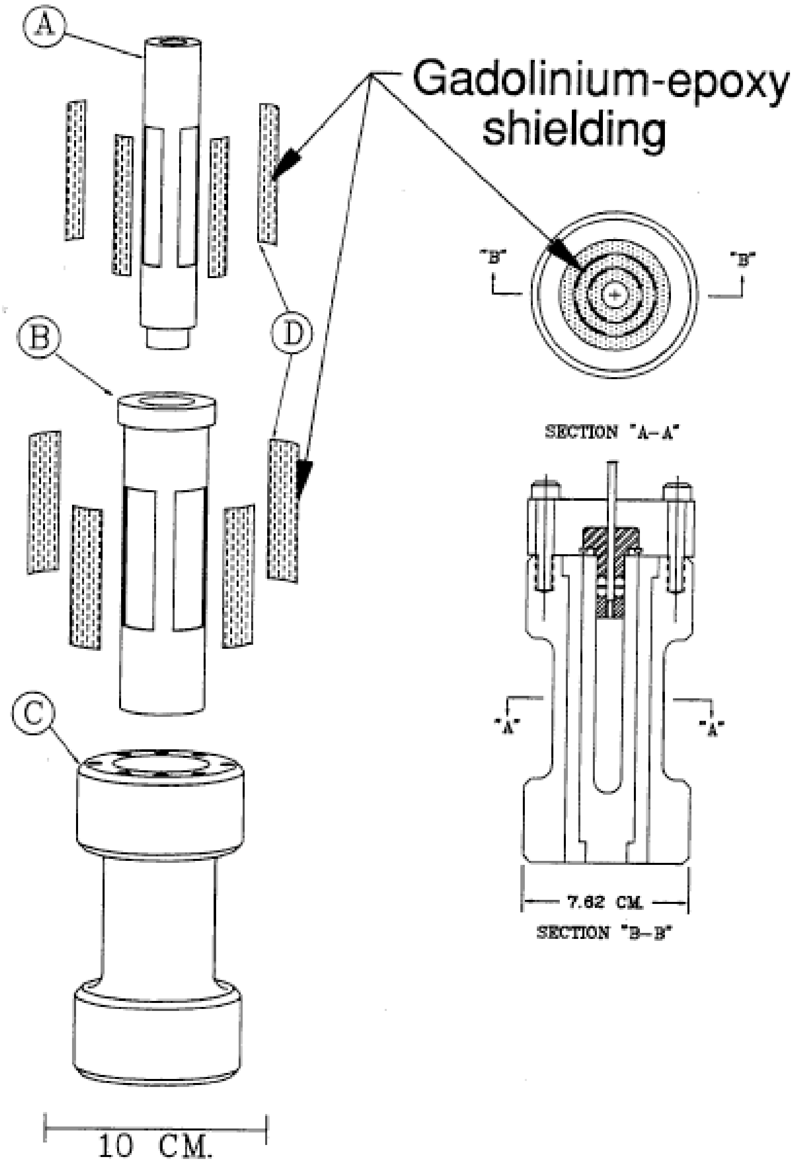
- Jim recognized early
  - Clear interference from cell a huge problem in angle dispersive mode
  - Solutions
    - fixed exit cells (spallation or ED rather than AD scattering)
    - either live with the interference or rethink alternatives
- Other considerations
  - Gas cells (low P but high precision)
  - Solid media cells (high P) but also high noise

Early fixed angle time of flight set up

High-Pressure TOF Powder Diffractometer
at Argonne's CP-5 Research Reactor



Gas Pressure Cell for Time-of-Flight Neutron Diffraction



Argonne High Pressure Neutron Diffraction Publications: (1966-2005, compiled by J. D. Jorgensen)

(Includes work done at the MTR Research Reactor at Argonne West in the late 1960's and later work at Argonne National Laboratory's CP-5 Reactor and Intense Pulsed Neutron Source)

- 1966: Neutron Diffraction Studies of Samples at High Pressures, R. M. Brugger, R. B. Bennion, E. R. Peterson, and T. G. Worlton, USAEC Report IDO-17170 (May 1966)
- 1967: Neutron Diffraction Studies of Samples at High Pressures, R. M. Brugger, R. B. Bennion, T. G. Worlton, and E. R. Peterson, in Research Applications of Nuclear Pulsed Systems, IAEA Vienna (1967)
- Pressure Dependence of the Morin Transition in a-Fe2O3 to 26 kbar, T. G. Worlton, R. B. Bennion, and R. M. Brugger, Phys. Lett. 24A, 653 (1967)
- The Crystal Structure of Bismuth-II at 26 kbar, R. M. Brugger, R. B. Bennion, and T. G. Worlton, Phys. Lett. 24A, 714 (1967)
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- Diffusion of Mercury and Cadmium in Lead at High Pressures, H. B. Vanfleet, J. D. Jorgensen, J. D. Schmutz, and D. L. Decker, Phys. Rev. B 15, 5545-5549 (1977)
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- Compression Mechanisms in [illegible]-Quartz Structures--SiO2 and GeO2, J. D. Jorgensen, J. Appl. Phys. 49, 5473-5478 (1978)
- **Perhaps the most highly cited paper from the early days.
- 1979
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- **A truly beautiful piece of work on the pressure-induced soft-mode transition in ReO3. This is one of the few cubic perovskites that does what perovskites are predicted to do at high presssure.

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- **A key paper for learning how pressure can increase Tc in a cuprate superconductor.
- Pressure-Induced Phase Transition and Pressure Dependence of Crystal Structure in Low (a) and Ca/Al-doped Cristobalite, J. B. Parise, A. Yeganeh-Haeri, D. J. Weidner, J. D. Jorgensen, and M. A. Saltzberg, J. Appl. Phys. 75, 1361-1367 (1994)
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- **A nice summary of how high-pressure work on cuprate superconductors leads to an understanding of how Tc is optimized in these materials.
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- **I think this is the first high-pressure diffraction paper on this classic negative thermal expansion material.
- Rare-Gas Intercalation into Fullerene Interstices, G. H. Kwei, J. D. Jorgensen, J. D. Schirber, B. Morosin, Fellerene Science and Technology 5, 243-256 (1997)
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- **1995 -** Room Temperature Compressibility of C_{60}: Intercalation Effects with He, Ne and Ar, J. E. Schirber, G. H. Kwei, J. D. Jorgensen, R. L. Hitterman, B. Morosin, Phys. Rev. B 51, 12014-12017 (1995)
- *****A very interesting paper about how rare gas intercalation affects the compressibility of C_{60}.***

- **1996 -** Structural Features that Optimize High Temperature Superconductivity, J. D. Jorgensen, D. G. Hinks, O. Chmaissem, D. N. Argyriou, J. F. Mitchell, B. Dabrowski, in Recent Developments in High Temperature Superconductivity, edited by J. Klamut, B. W. Veal, B. M. Dabrowski, P. W. Klamut, M. Kazimierski (Springer-Verlag, Berlin, 1996), p. 1-16
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- **2000 -** Key Insights from Structural Studies of High-Temperature Superconductors: Is There a Path to Higher T_c?, J. D. Jorgensen, in Advances in Superconductivity XII, edited by T. Yamashita and K. Tanabe (Springer-Verlag, Tokyo, 2000) pp.9-14.
- *****A nice summary of how high-pressure work on cuprate superconductors leads to an understanding of how T_c is optimized in these materials.***
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- *****A nice example of how pressure can drive dramatic changes in hydrogen bonding resulting in charge transfer and modification of transport and superconducting properties.***

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- **1994 -** Pressure-Induced Structural Changes in Superconducting $HgBa_2Ca_{n-1}Cu_nO_{2n+2+}$⊠ (n=1,2,3) Compounds, B. A. Hunter, J. D. Jorgensen, J. L. Wagner, P. G. Radaelli, D. G. Hinks, H. Shaked, R. L. Hitterman, and R. B. Von Dreele, Physica C 221, 1-10 (1994) *****A key paper for learning how pressure can increase T_c in a cuprate superconductor.***
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- Ne Intercalated C_{60}: Diffusion Kinetics, B. Morosin, Z. Hu, J. D. Jorgensen, S. Short, J. E. Schirber, G. H. Kwei, Phys. Rev. B 59, 6051-6057 (1999)
- *****A novel experiment exploring time-dependent intercalation effects in C_{60}.***
- **2000 -** Key Insights from Structural Studies of High-Temperature Superconductors: Is There a Path to Higher T_c?, J. D. Jorgensen, in Advances in Superconductivity XII, edited by T. Yamashita and K. Tanabe (Springer-Verlag, Tokyo, 2000) pp.9-14.
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- Thermal Expansion and Compressibility in Superconducting $Na_xCoO_2•4xD_2O$ (x≈1/3): Evidence for Pressure-Induced Charge Redistribution, J. D. Jorgensen, M. Avdeev, D. G. Hinks, P. W. Barnes, and S. Short, Phys. Rev. B 72, 224515 (21 December 2005)
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- **1994 -** Pressure-Induced Structural Changes in Superconducting $HgBa_2Ca_{n-1}Cu_nO_{2n+2+}$[x] (n=1,2,3) Compounds, B. A. Hunter, J. D. Jorgensen, J. L. Wagner, P. G. Radaelli, D. G. Hinks, H. Shaked, R. L. Hitterman, and R. B. Von Dreele, Physica C 221, 1-10 (1994) *****A key paper for learning how pressure can increase T_c in a cuprate superconductor.***
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- *****I think this is the first high-pressure diffraction paper on this classic negative thermal expansion material.***

1980 - Structural Parameters for the Suspected Pressure-Induced Electron Transition in InBi, J. D. Jorgensen and J. B. Clark, Phys. Rev. B 22, 6149-6154 (1980)***** first high-pressure study of an electronic topological (Lifshitz) transition.***

W. Sleight, Phys. Rev. B 59, 215-225 (1999)

- Ne Intercalated C_{60}: Diffusion Kinetics, B. Morosin, Z. Hu, J. D. Jorgensen, S. Short, J. E. Schirber, G. H. Kwei, Phys. Rev. B 59, 6051-6057 (1999)
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Argonne High Pressure Neutron Diffraction Publications: (1966-2006, compiled by J. D. Jorgensen)

- **1994 -** Pressure-Induced Structural Changes in Superconducting $HgBa_2Ca_{n-1}Cu_nO_{2n+2+\boxtimes}$ (n=1,2,3) Compounds, B. A. Hunter, J. D. Jorgensen, J. L. Wagner, P. G. Radaelli, D. G. Hinks, H. Shaked, R. L. Hitterman, and R. B. Von Dreele, Physica C 221, 1-10 (1994) *****A key paper for learning how pressure can increase T_c in a cuprate superconductor.***
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- Sign Reversal of the Mn-O Bond Compressibility in $La_{1.2}Sr_{1.8}Mn_2O_7$ Below T_C: Exchange Striction in the Ferromagnetic State, D. N. Argyriou, J. F. Mitchell, J. B. Goodenough, O. Chmaissem, S. M. Short, J. D. Jorgensen, Phys. Rev. Lett. 78, 1568-1572 (1997) *****A very interesting study of how pressure can strongly modify magnetic coupling, leading to unusual structural responses.***
- **1999 -** Pressure-Induced Cubic-to-0rthorhombic Phase Transition in ZrW_2O_8, J. D. Jorgensen, Z. Hu, S. Teslic, D. N. Argyriou, S. Short, J. S. O. Evans, A.

- **Order Parameter and Critical Exponent for the Pressure-induced Phase Transitions in ReO3, J.-E. Jørgensen, J. D. Jorgensen, B. Batlogg, J. P. Remeika, and J. D. Axe, Phys. Rev. B 33, 4793-4798 (1986)**
- ****A truly beautiful piece of work on the pressure-induced soft-mode transition in ReO3. This is one of the few cubic perovskites that does what perovskites are predicted to do at high presssure.**

Jorgensen, M. Avdeev, D. G. Hinks, P. W. Barnes, and S. Short, Phys. Rev. B 72, 224513 (21 December 2005)
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J. D. Jorgensen's High Pressure legacy

- There are important insights provided by studying materials @ HP
- Good signal-to-noise is essential
  - How to get it is the challenge
- Jim's solutions
  - spallation
  - quiet gas pressure cells
  - Unstinting support for instrument development (including SNAP at SNS)

J. D. Jorgensen's High Pressure legacy

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- **The HP community strives to match what Jim did - at higher pressures in solid media devices (contributors to this talk in parentheses)**

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  - area detectors and white beams (ILL, McIntyre)
- Bringing it all together - the SNAP beamline (Tulk)

HP Devices: the DIA and PE cells

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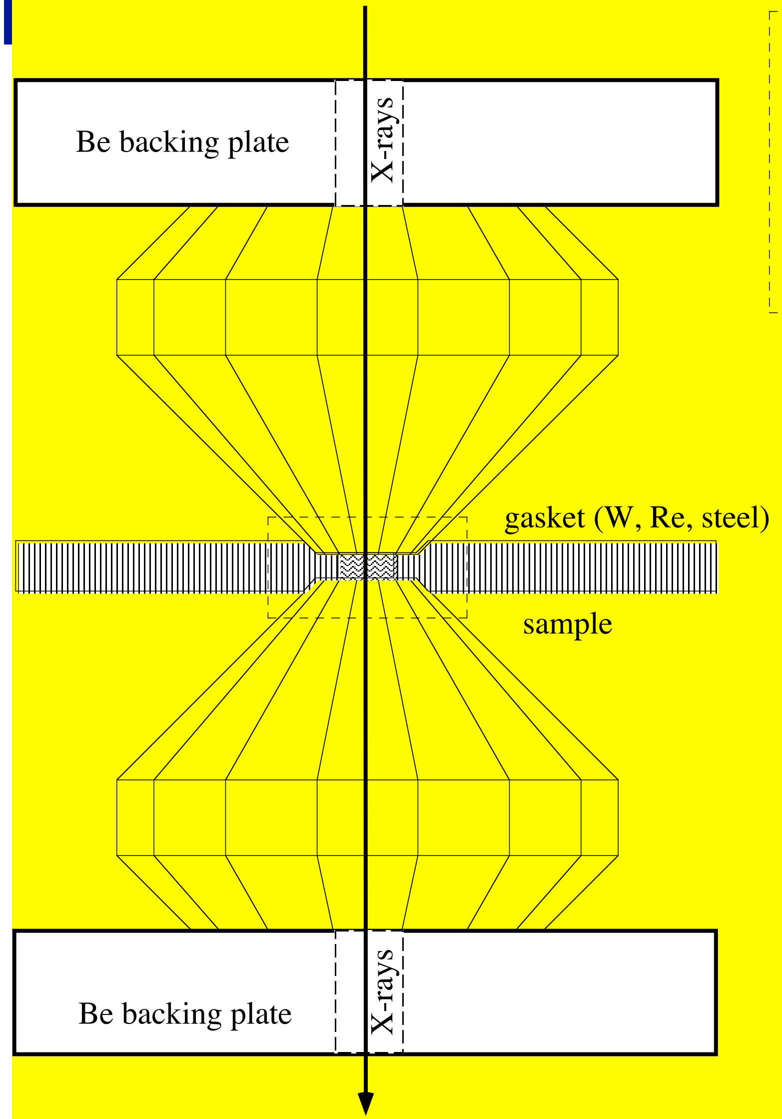
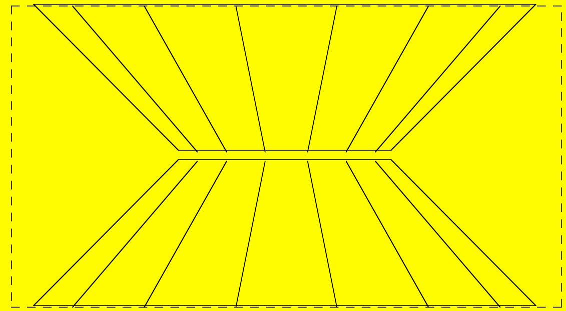
- Pressure generation
  - P = F/A -
    - Diamond anvils - small volume high P
    - Paris-Edinburgh cell
    - Large - volume (80 mm^3 +) at 10+ GPa
-

HP Devices: the DIA and PE cells



(a)



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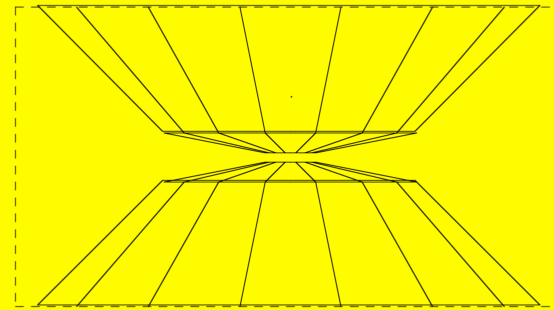

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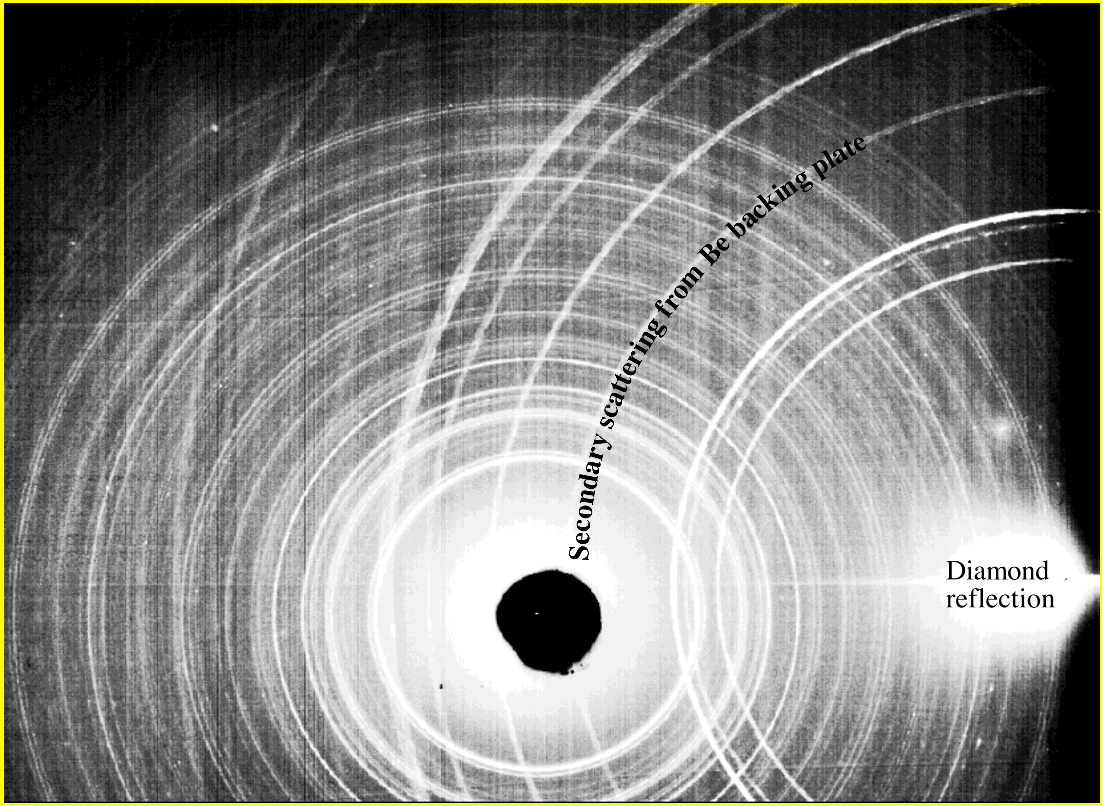
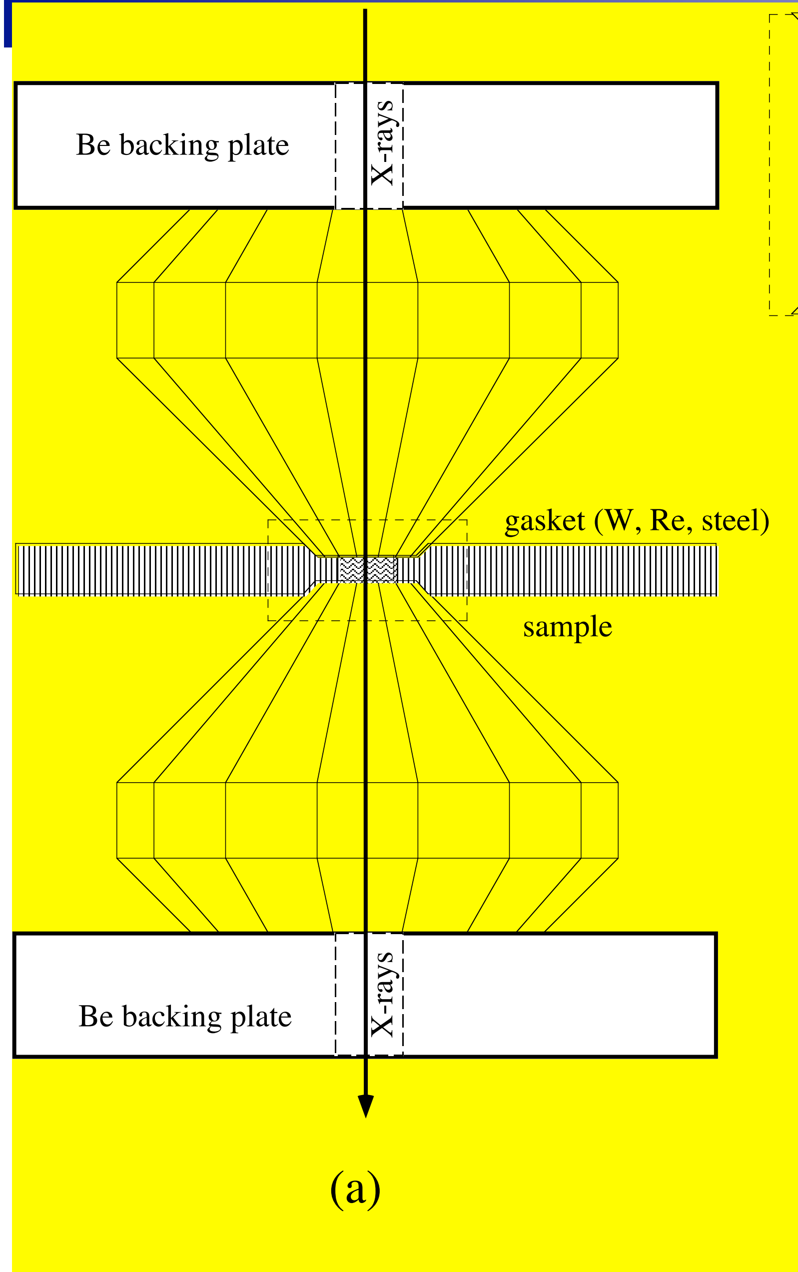
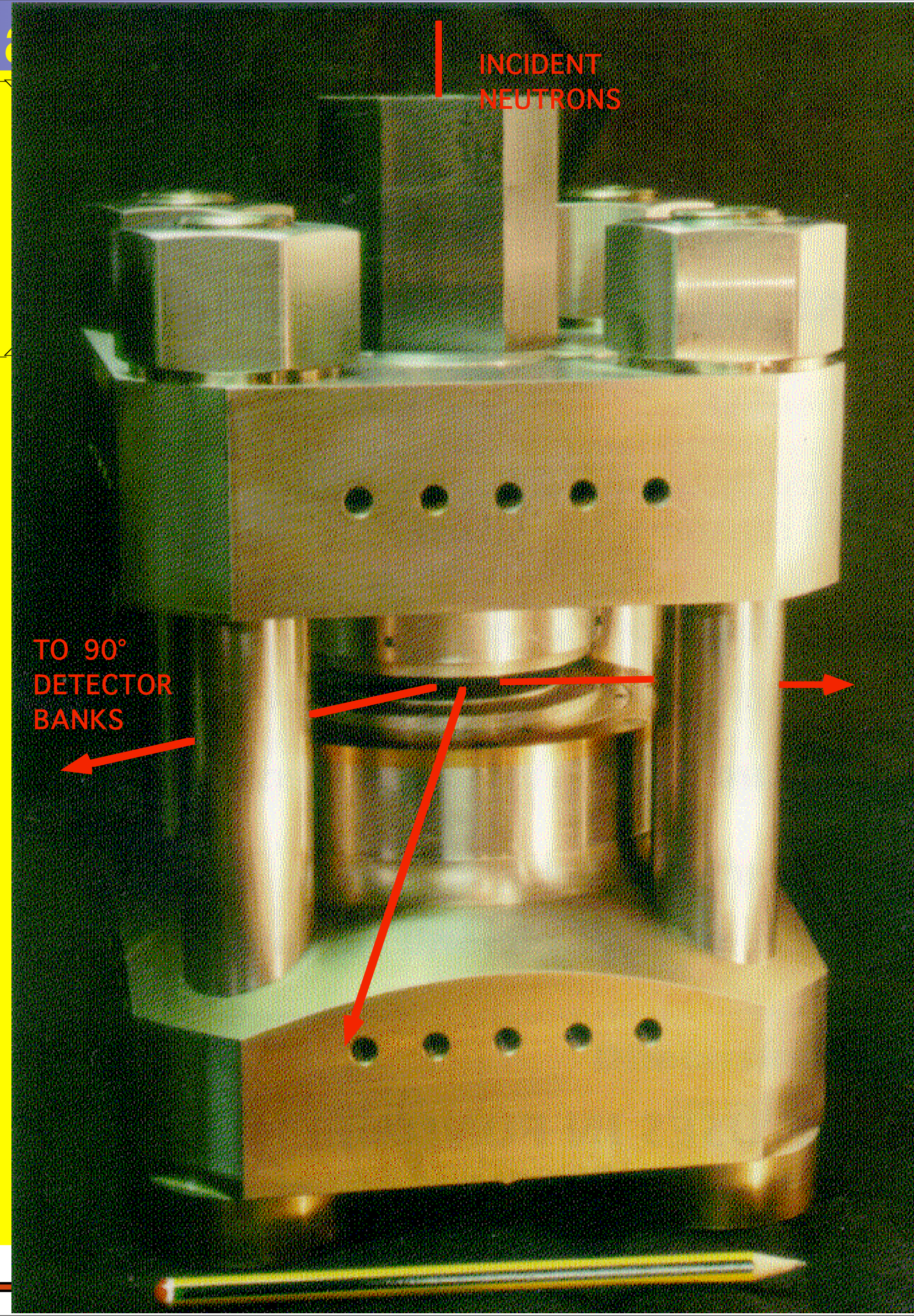
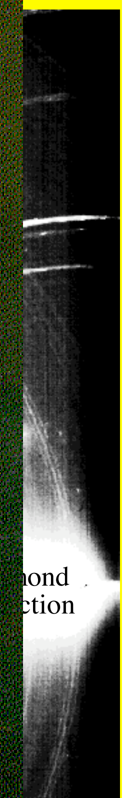


(d)

HP Devices: the DIA a







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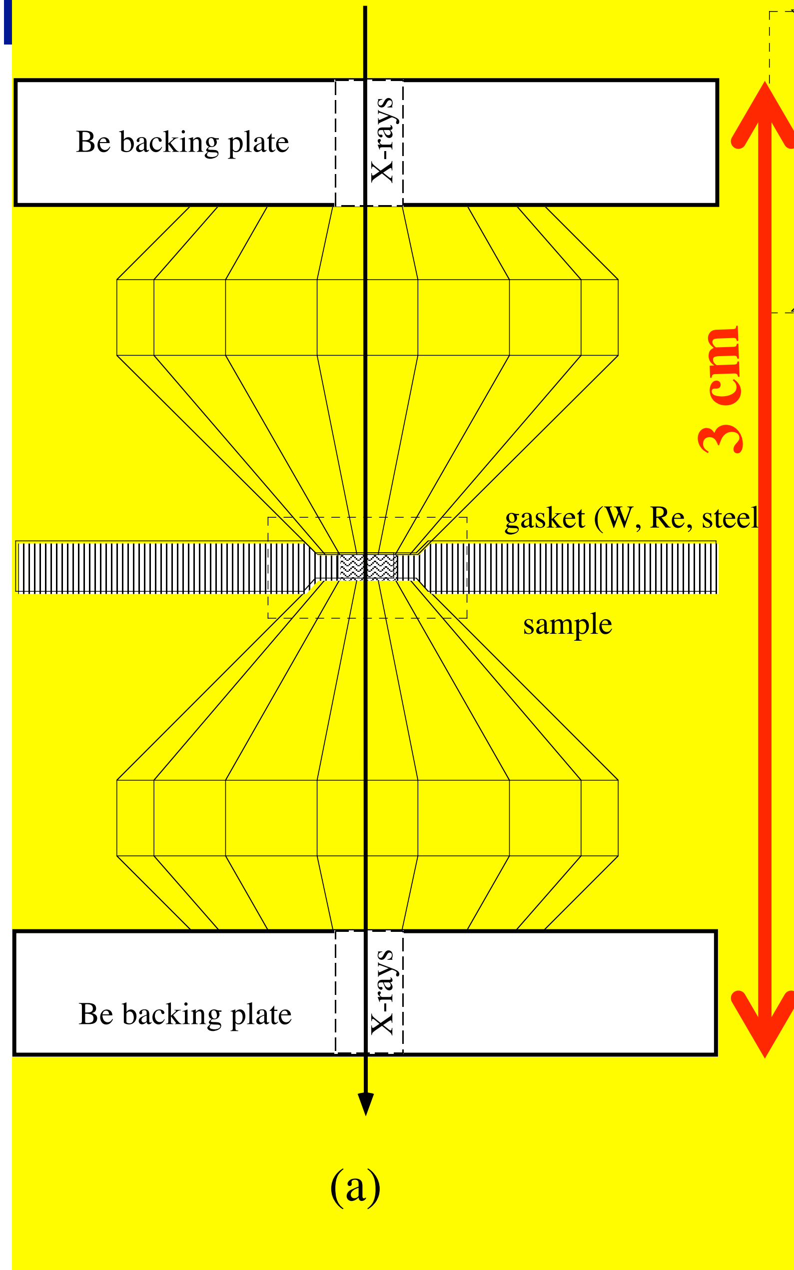

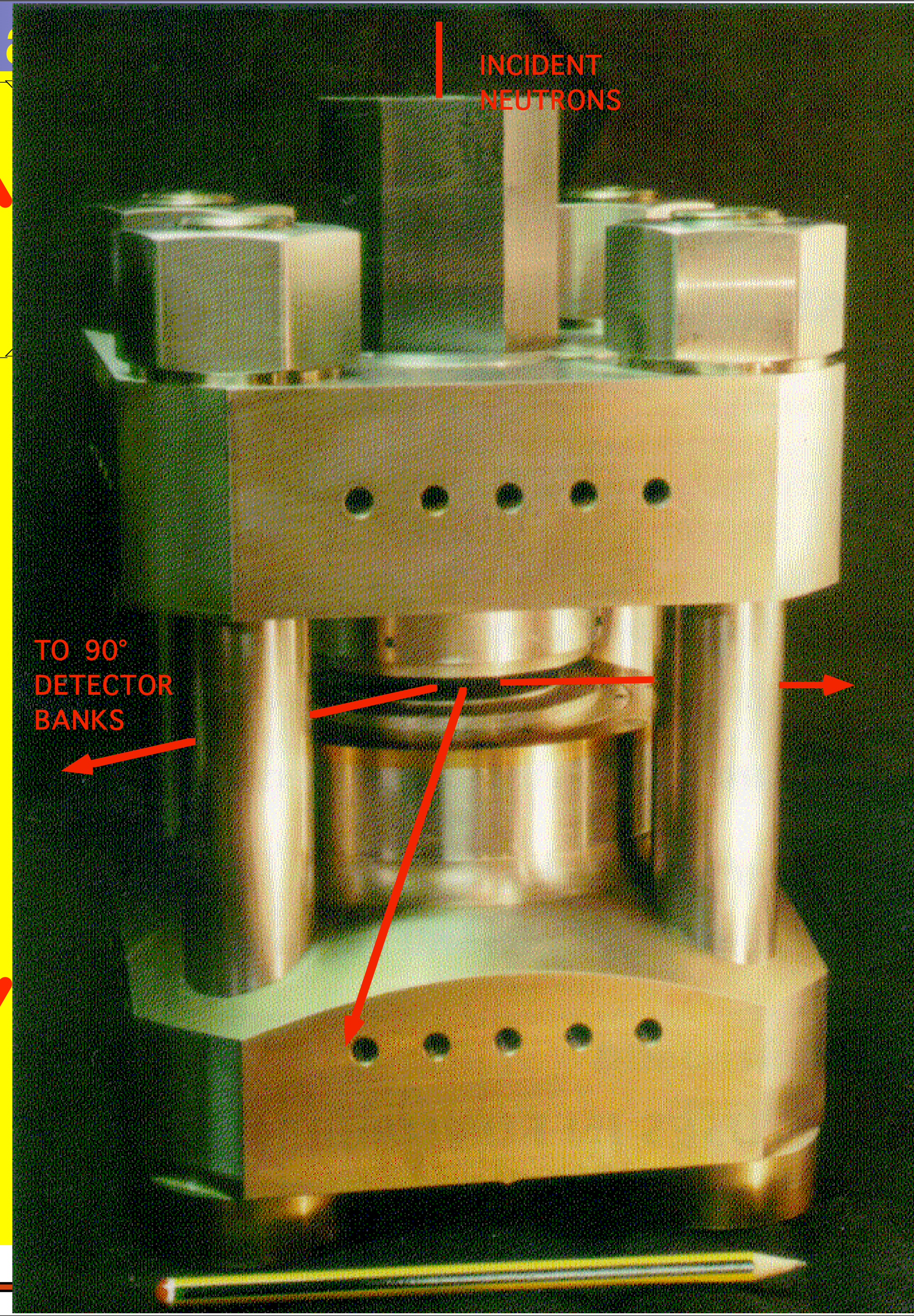

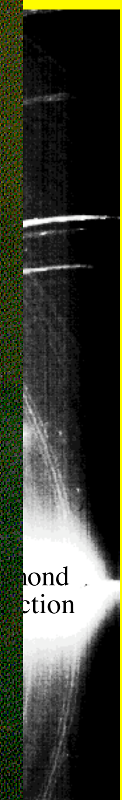

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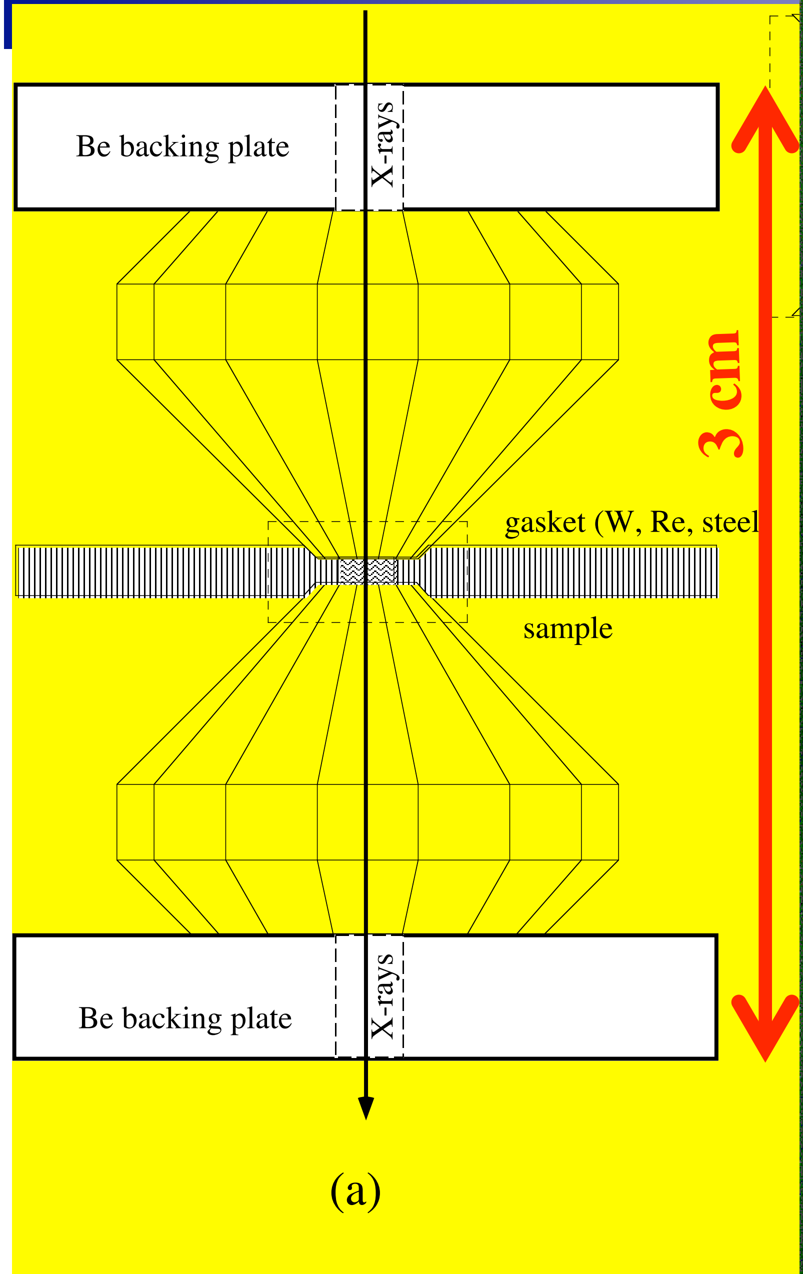
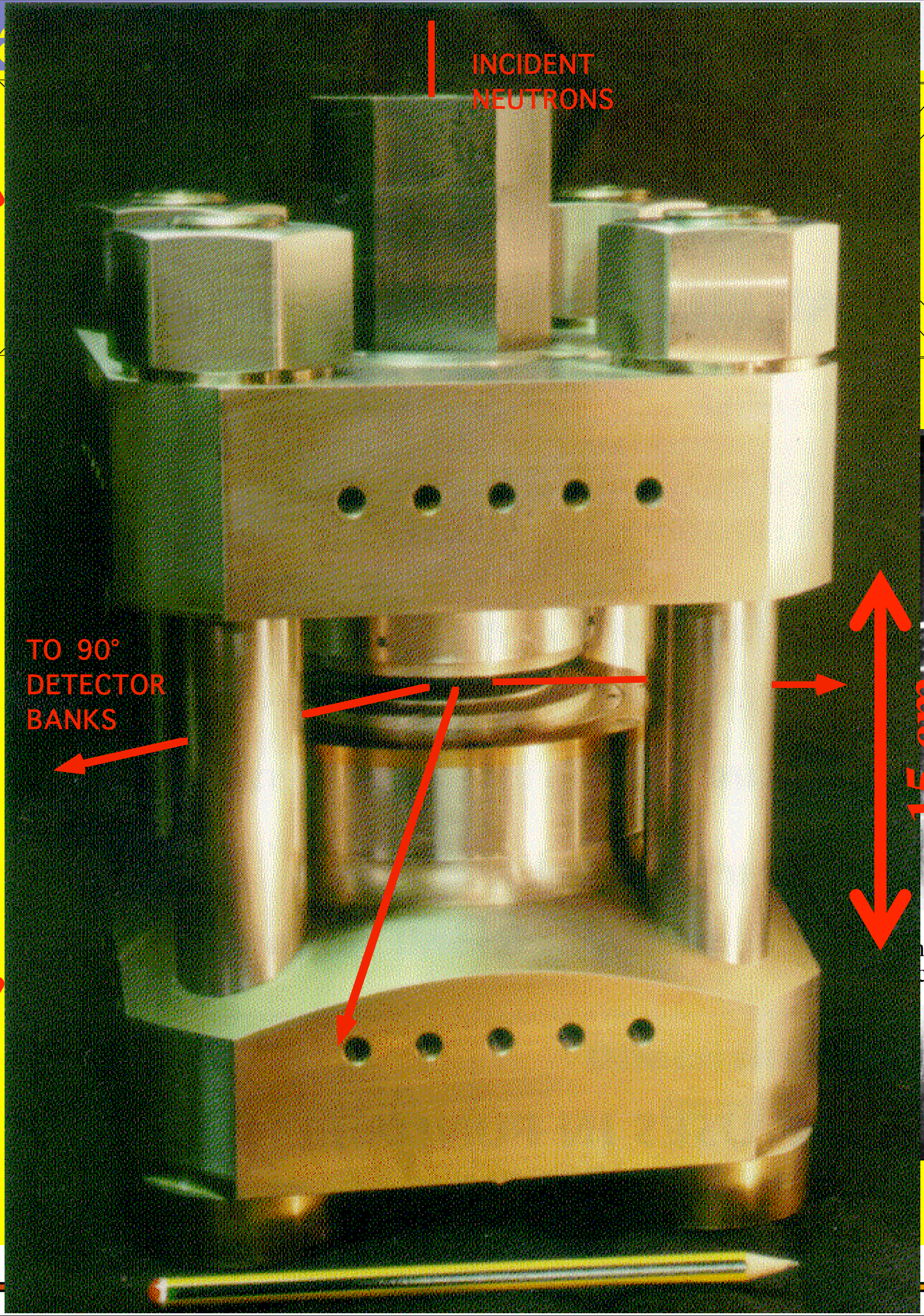
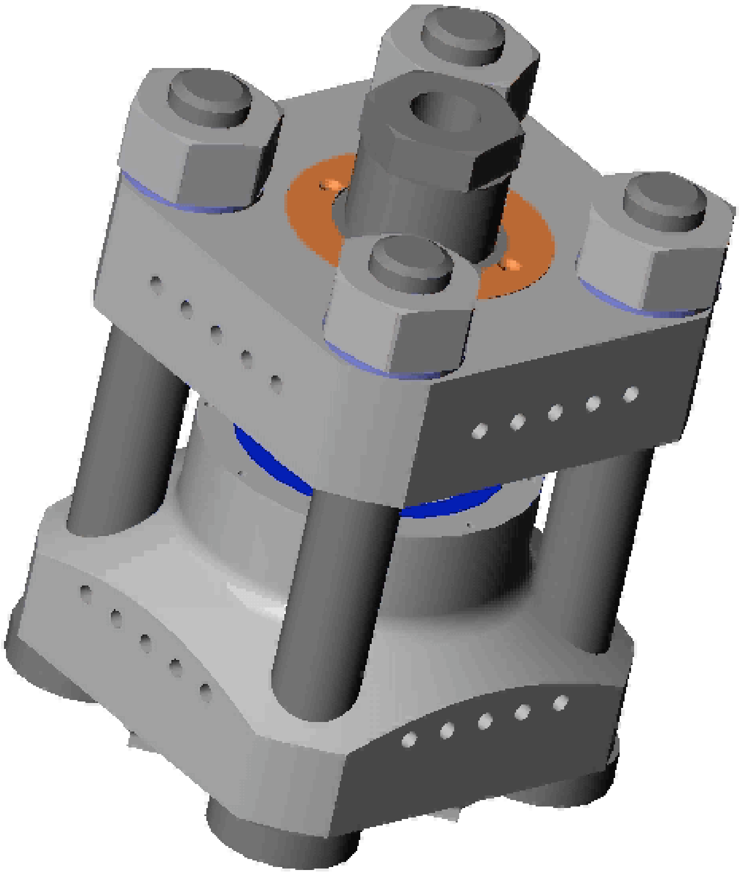
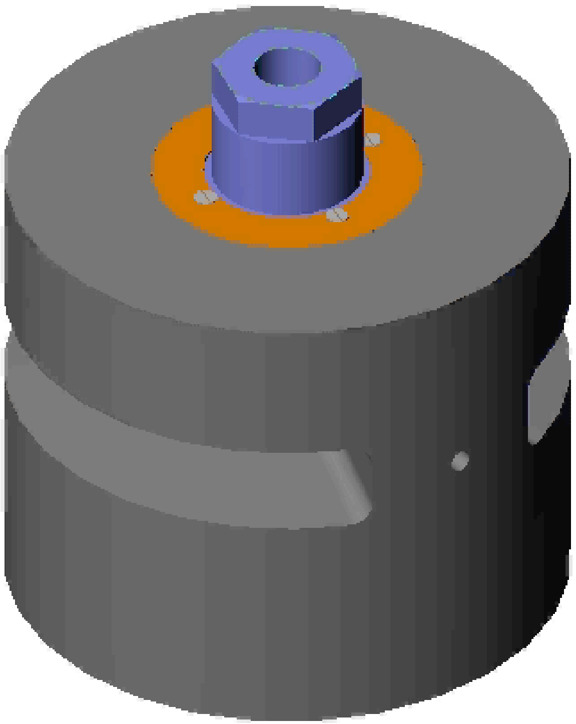


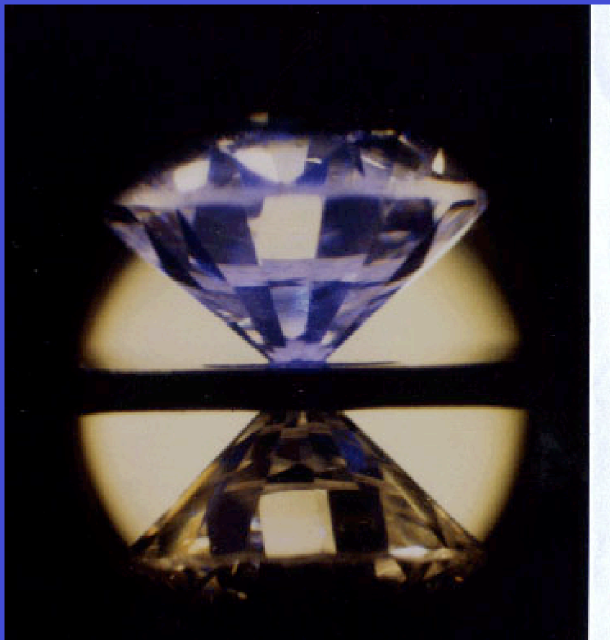


The PE cell first (V3) and later (V13) generations (Klotz, Hamel)

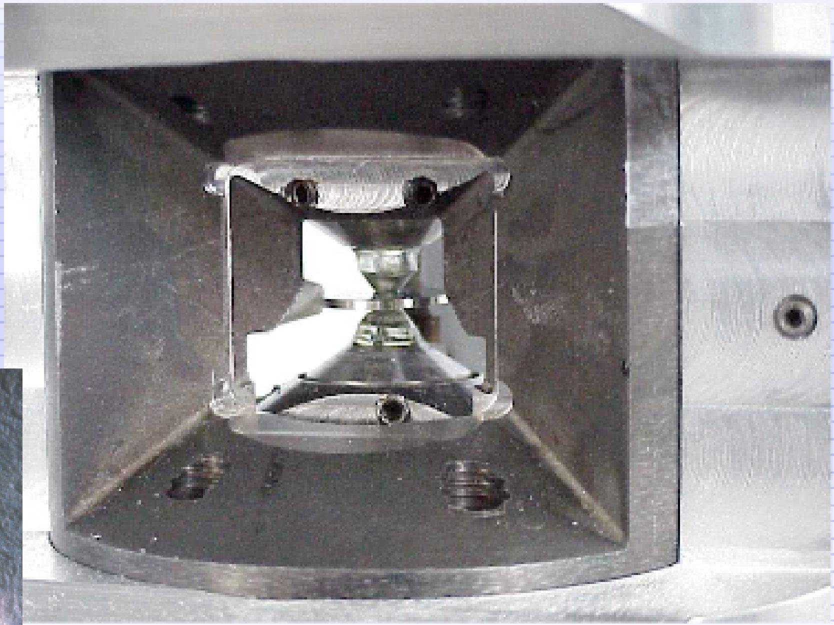


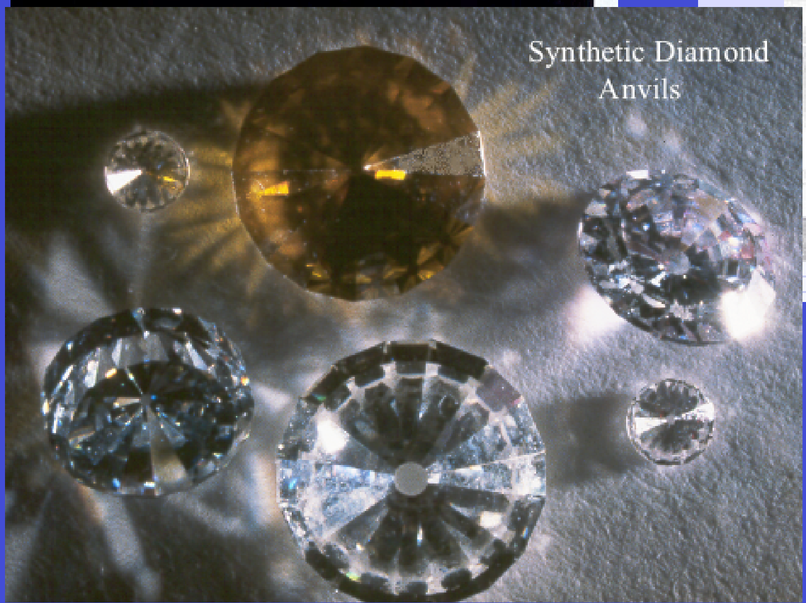


BEYOND THE SATE OF THE ART:300 - 500 GPa? NEW WINDOWS ON PLANETARY MATERIALS (Xu, Mao, Hemley) - GPL



New Diamond Anvil Cells:
LARGE VOLUME AND "3-D" ACCESS



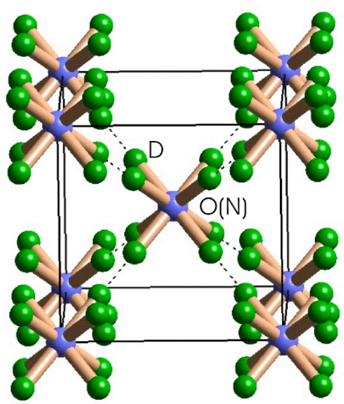


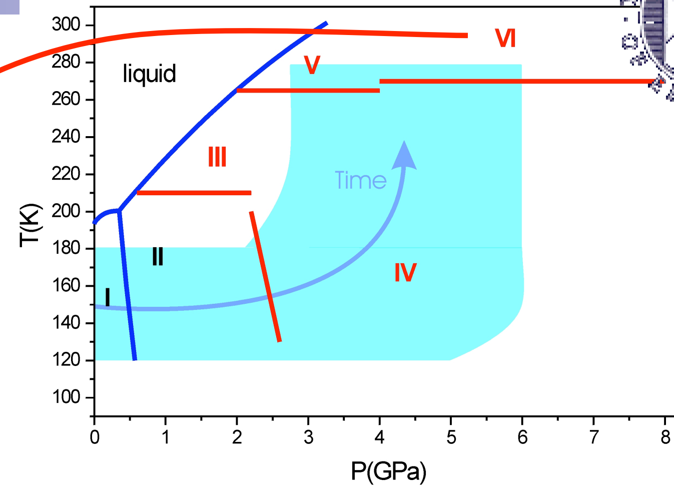
- New 'Transparent' Gaskets
- Direct Measure of Stress-Strain
- New High-Pressure Probes
- Transport Measurements

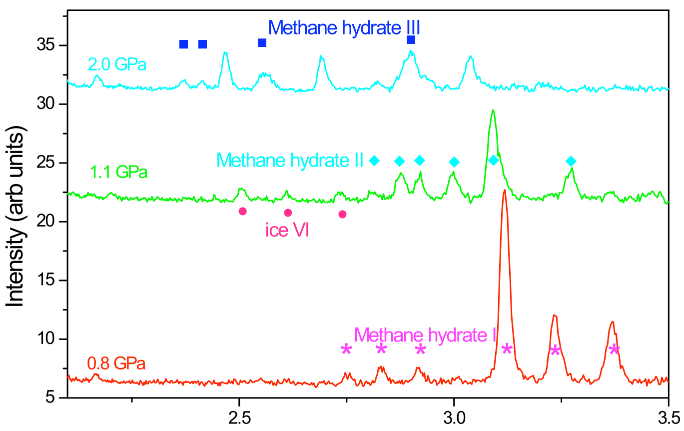
Ammonia monohydrate Loveday and Nelmes PRL 1999

Four new phases discovered up to 6 GPa. Titan models assume negligible compression and no phase transitions

Phase VI is a simple bcc structure with substitutional site disorder of water and ammonia

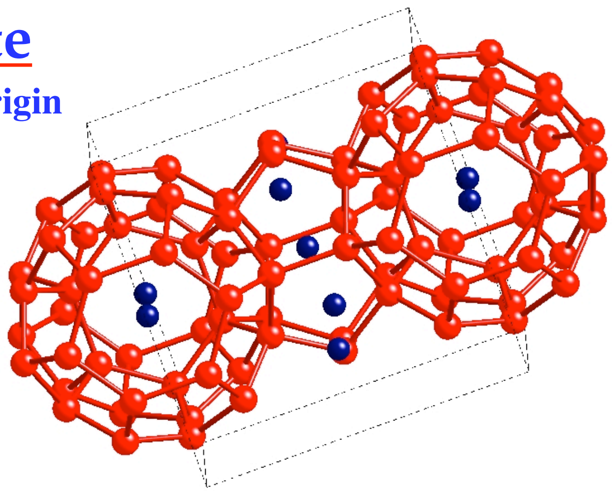






Methane hydrate

Central to models of the origin of Titan's atmosphere



Previously thought to decompose into ice and methane in the 1-2 GPa range

- Two new high pressure hydrates
  - phase II $(H_2O)_{3.5}(CH_4)$
  - phase III $(H_2O)_2(CH_4)$

stable to at least 10 GPa Collaboration between University of Edinburgh and NRC Ottawa

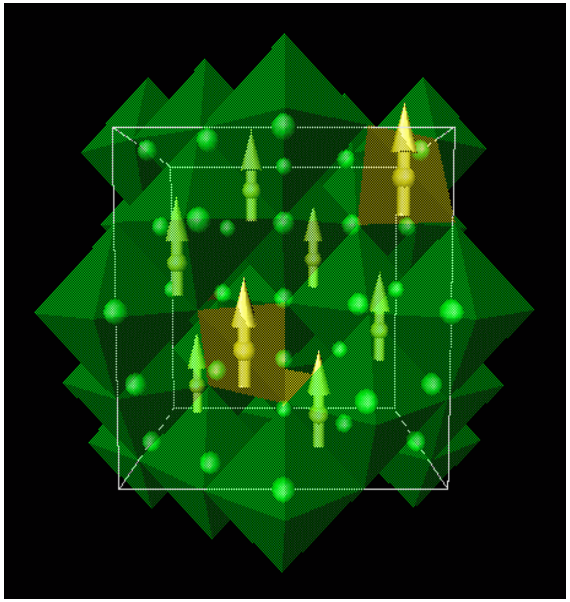
Next generation - HP at low T(< 20 K)

Next generation - HP at low T(< 20 K)

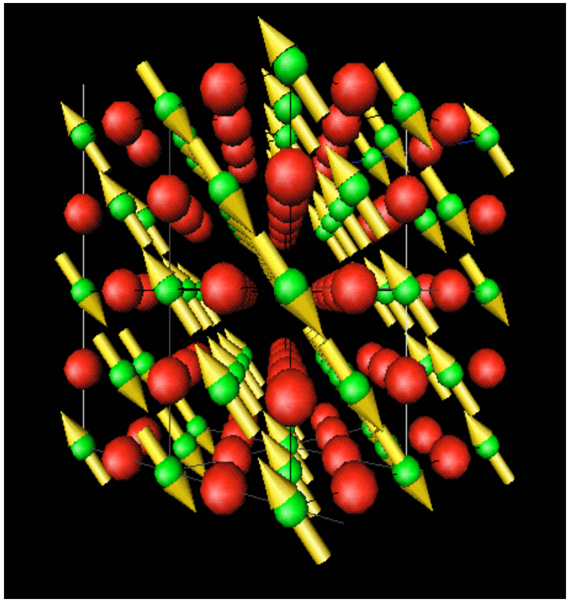
This is clearly NOT
a DOE facility

Properties - magnetic moment

Applications



Ferrimagnetic magnetite Fe_3O_4

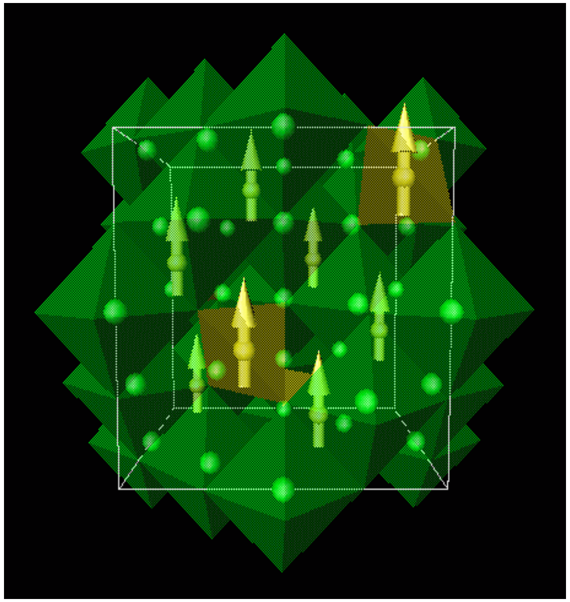


Antiferromagnetic manganese oxide MnO

Properties - magnetic moment

Applications

- Determine **magnetic structure** from powder and single crystal patterns



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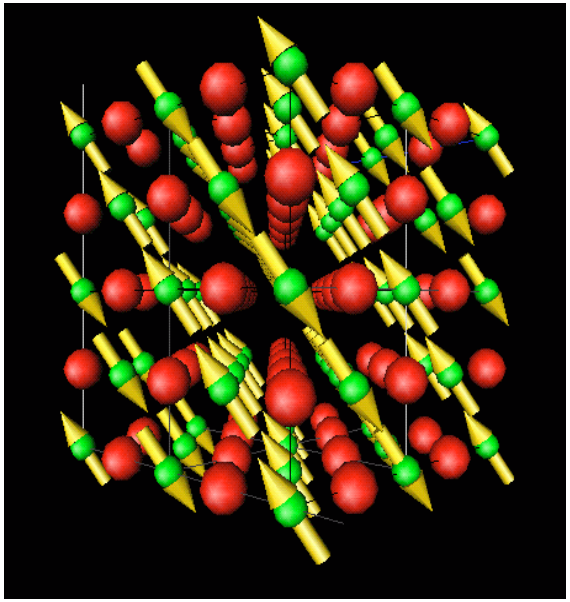

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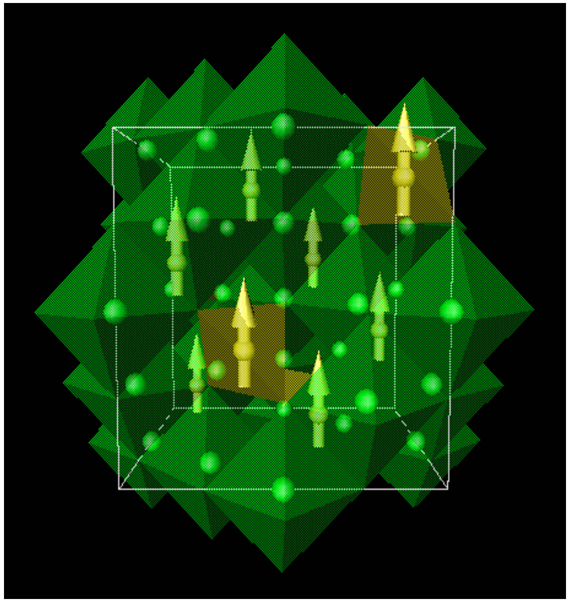

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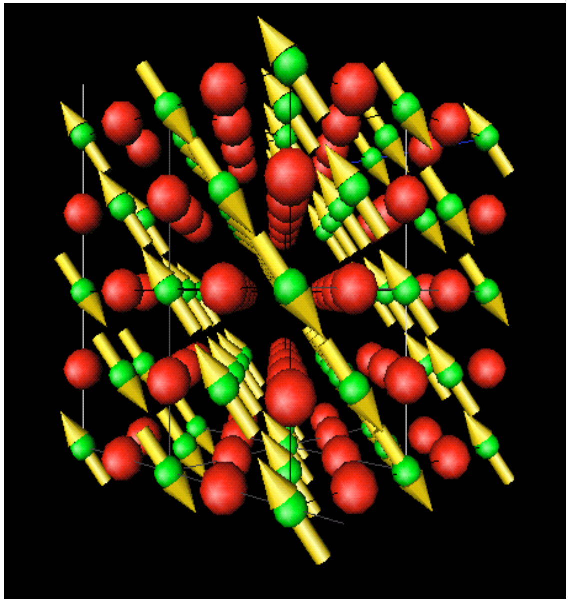

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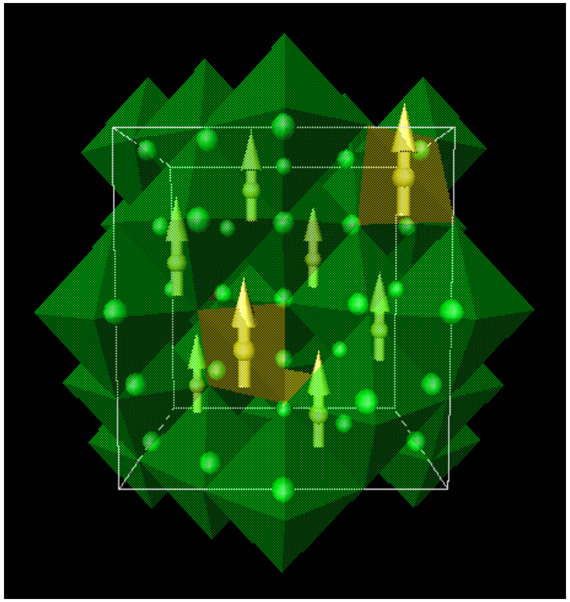
- Magnetism diagnostic for (nuclear) psuedo-symmetric phase transitions.



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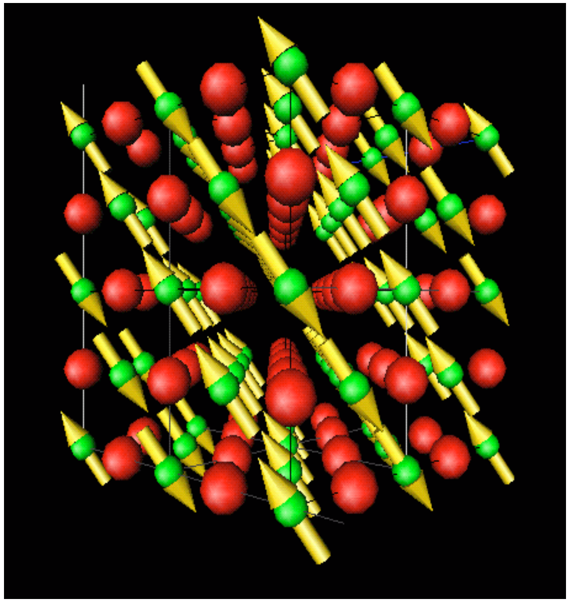

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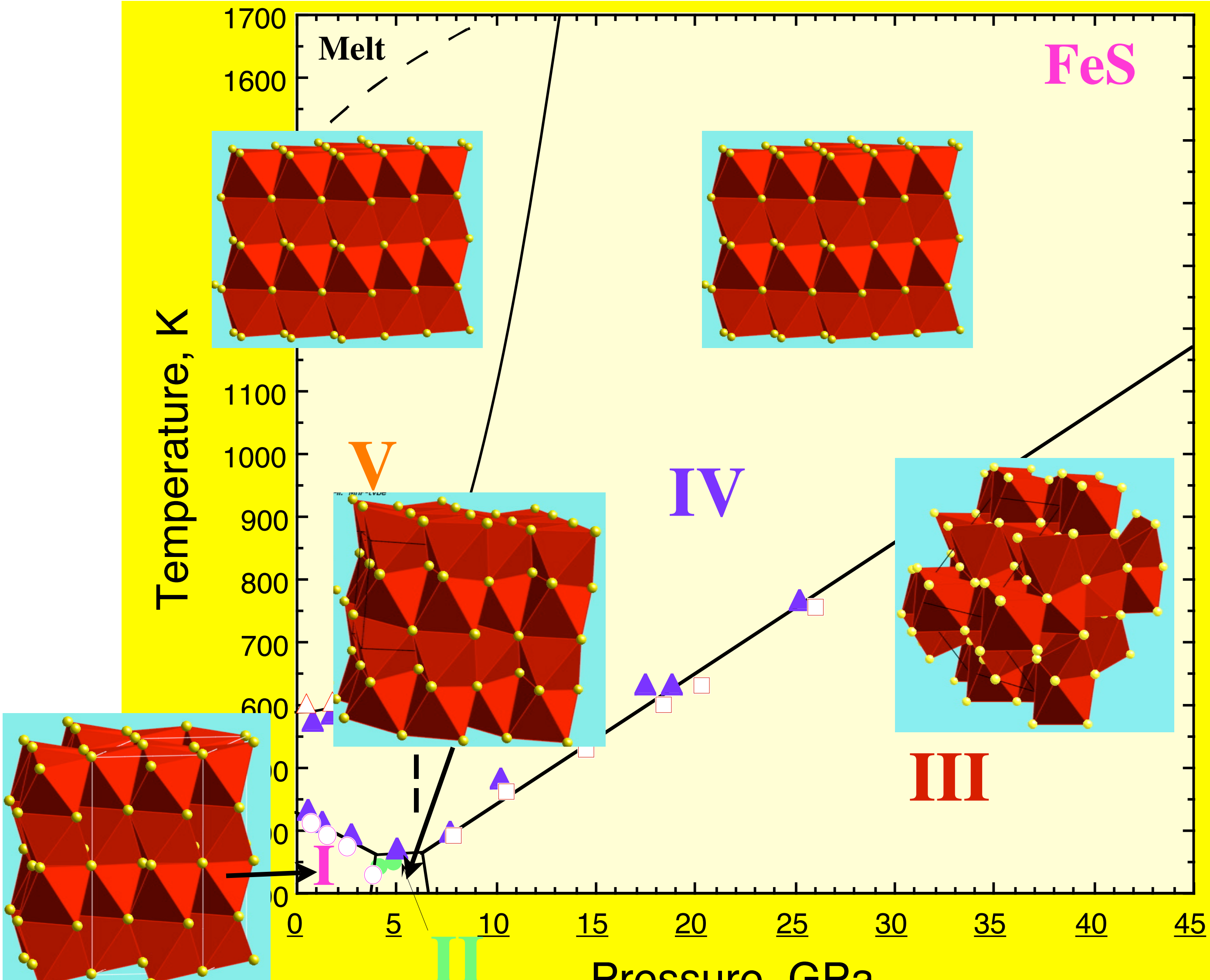
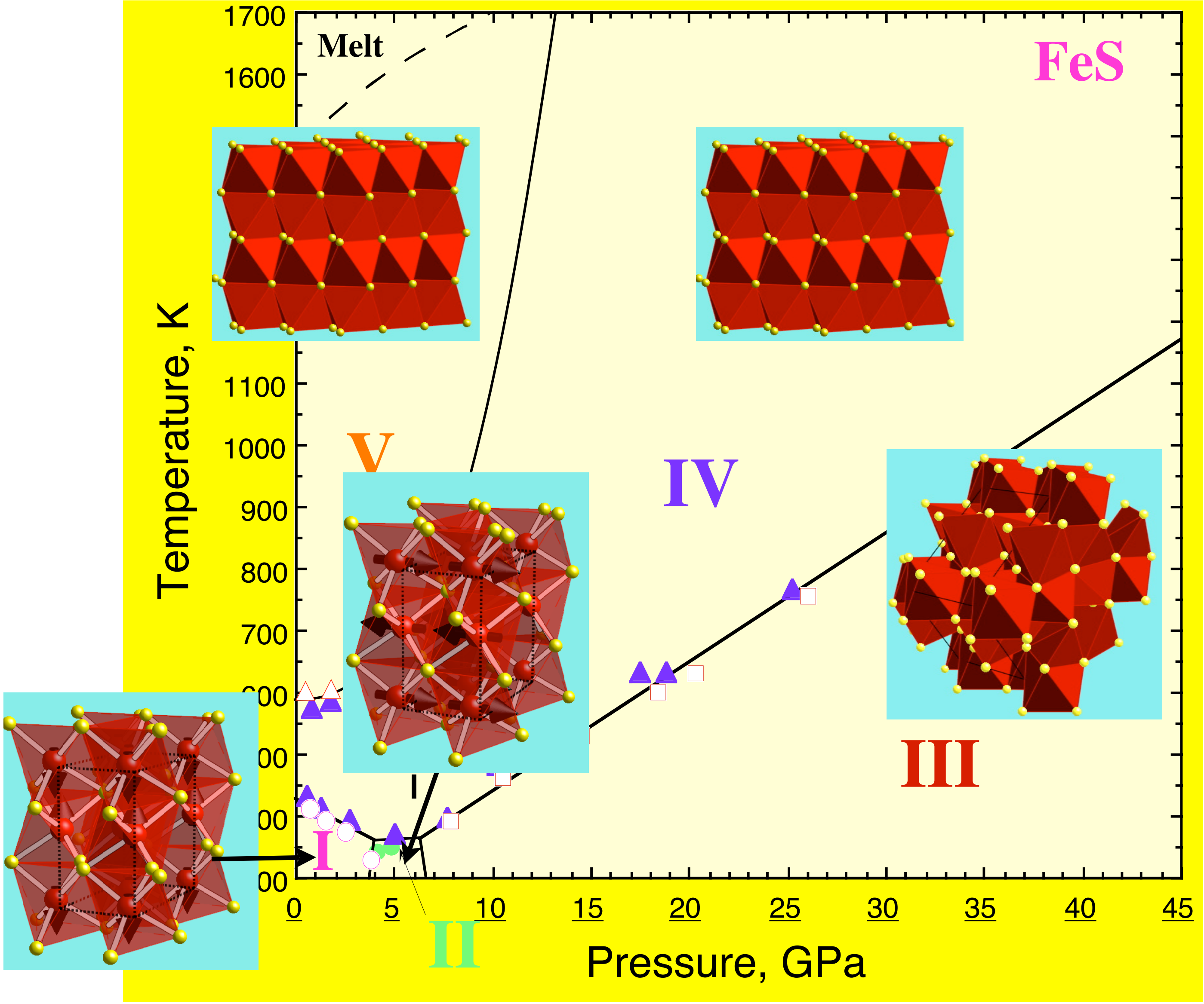
FeS phase diagram



Fei, Prewitt, Mao, and Bertka (1995), *Science*

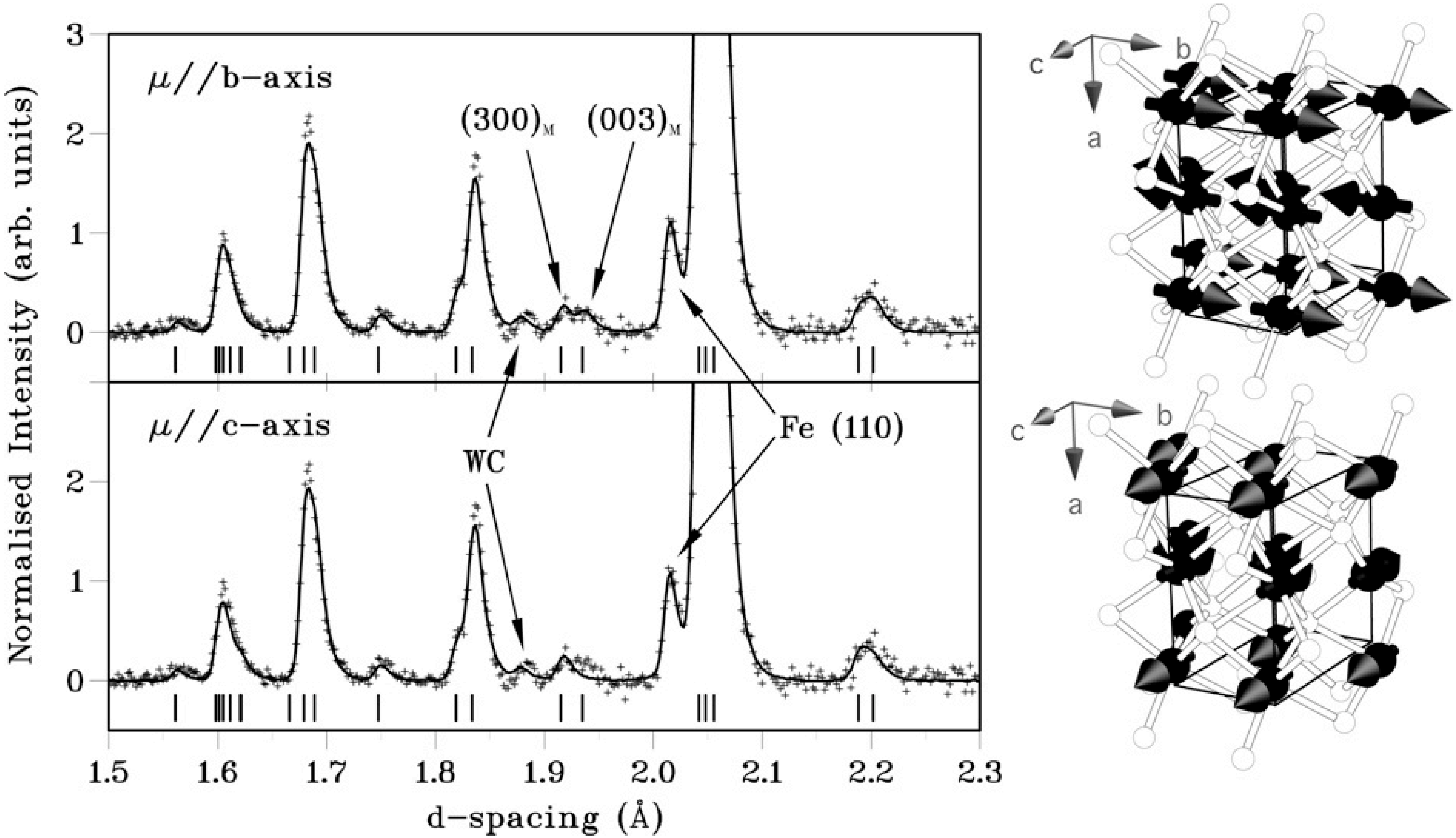
FeS phase diagram

MAGNETIC STRUCTURES



Marshall, Loveday, Nelmes, Klotz, Hamel, Besson and Parise (2000) Phys. Rev. B61, 11201

Magnetic Scattering for FeS - a lucky break
Marshall et al, ISIS



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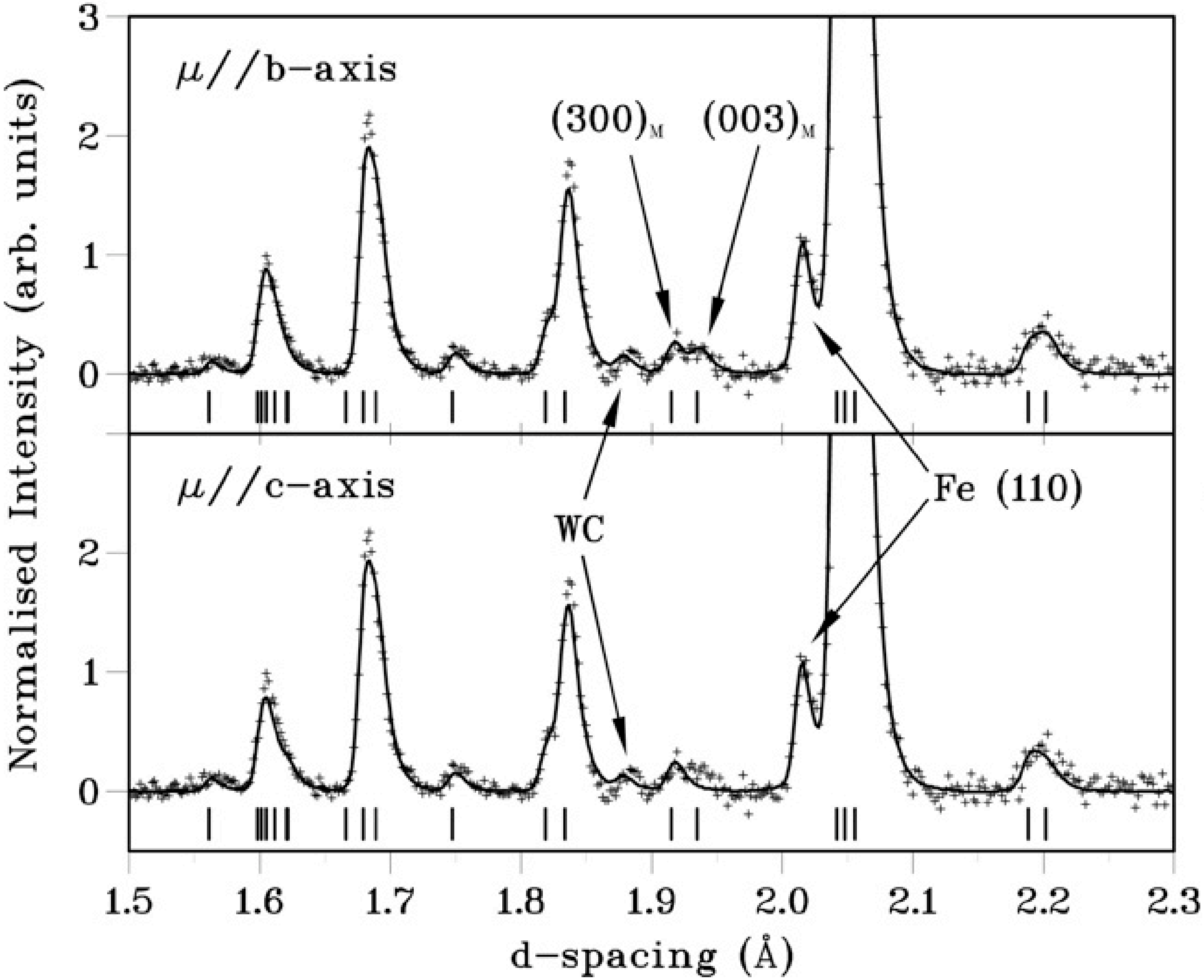
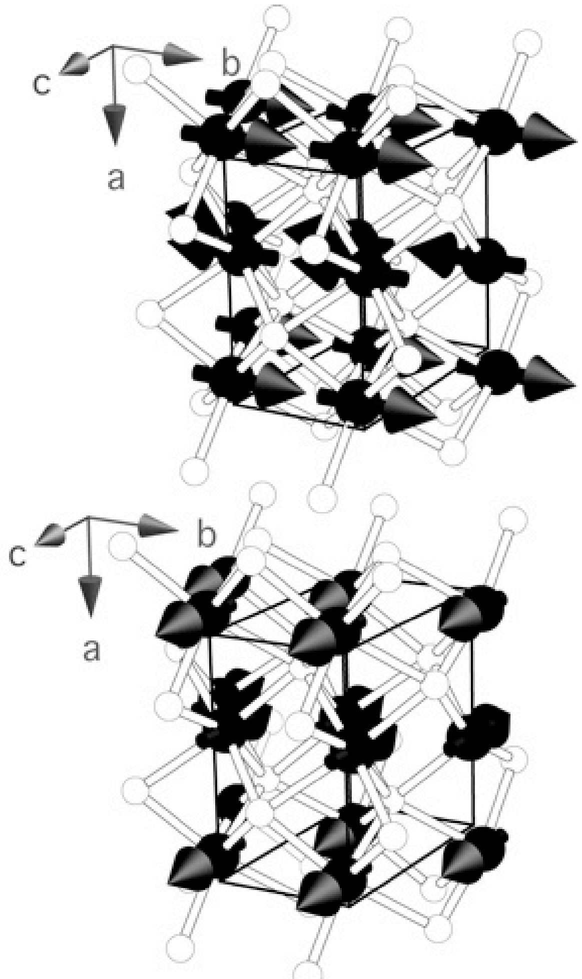
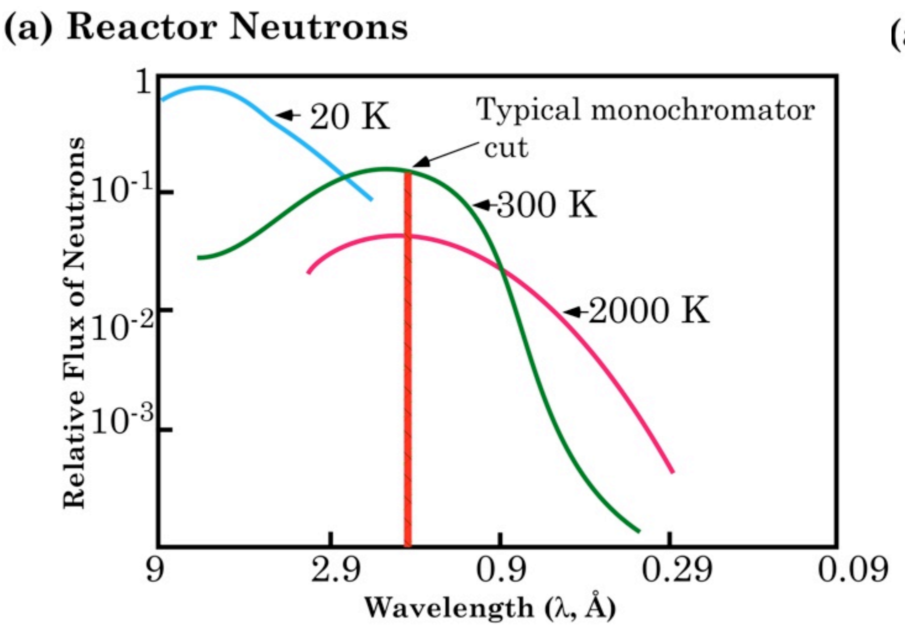


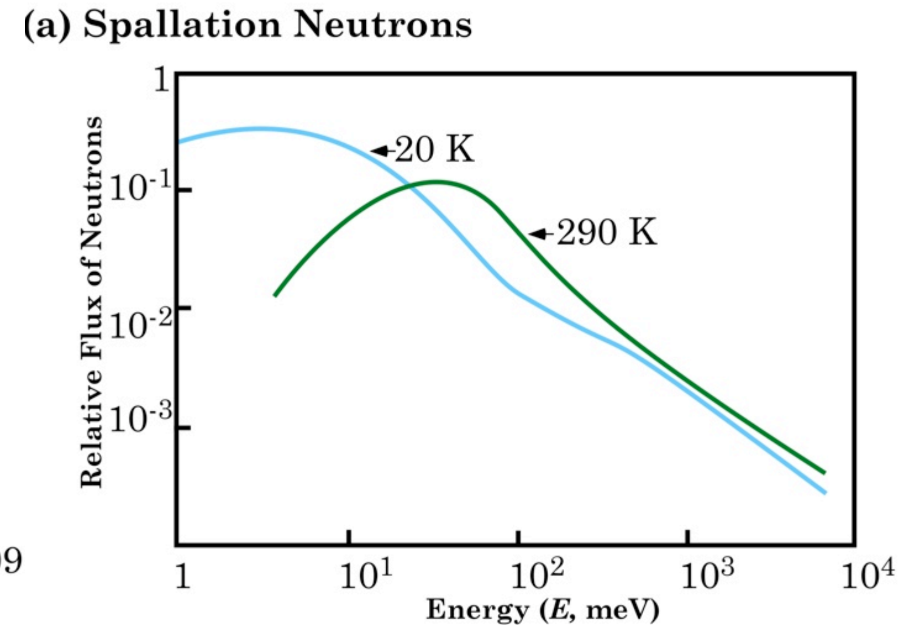


This is subtle! and most time, with powders, we only know the moment is either in the plane or not. Best to use single crystals

The PE cell at reactor sources - advantages

Neutron mass ~ proton mass: shift spectrum by allowing neutrons to come to thermal equilibrium with proton-rich (CH_4, H_2, H_2O, D_2O) condensed matter at different temperatures)





The PE cell at reactor sources - advantages

STONY BROOK
STATE UNIVERSITY OF NEW YORK

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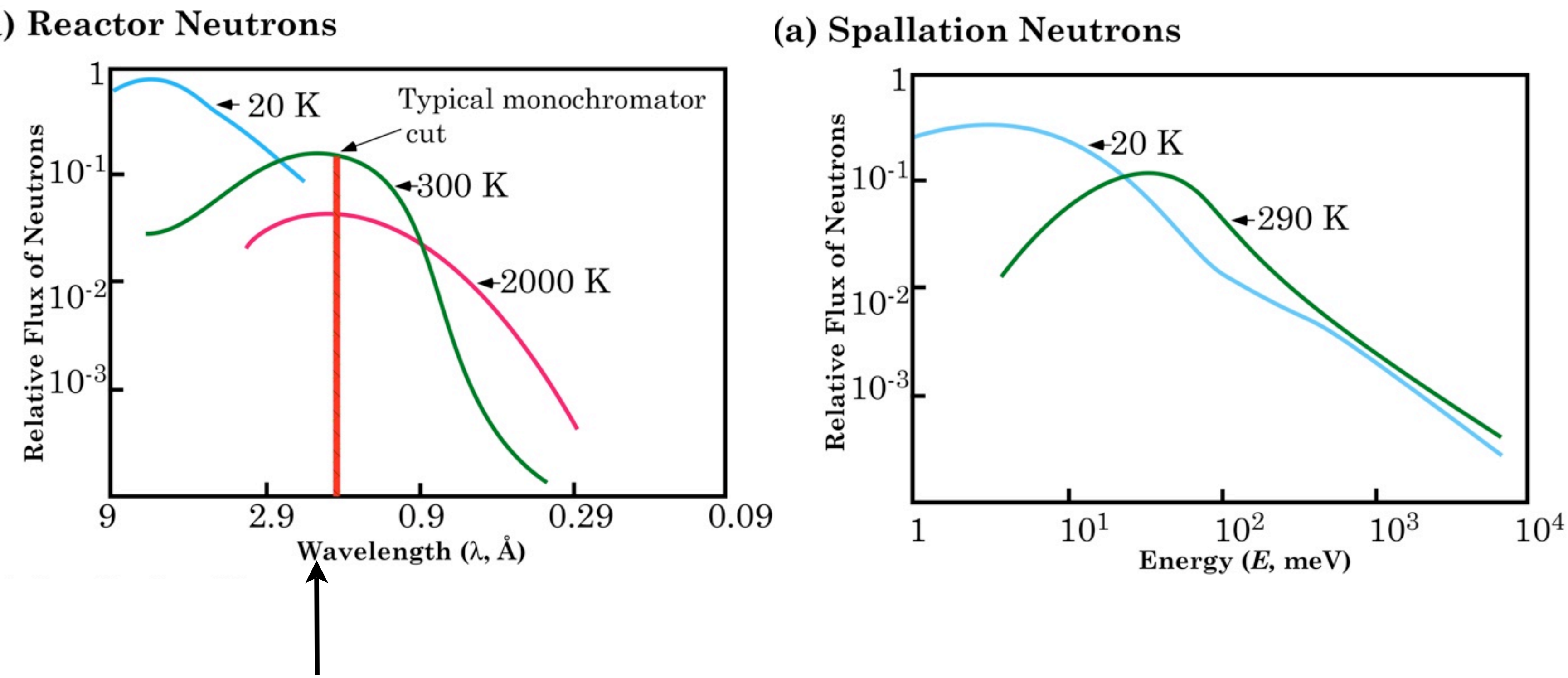

optimized to low E (high d-space) magnetism, phonons, INS

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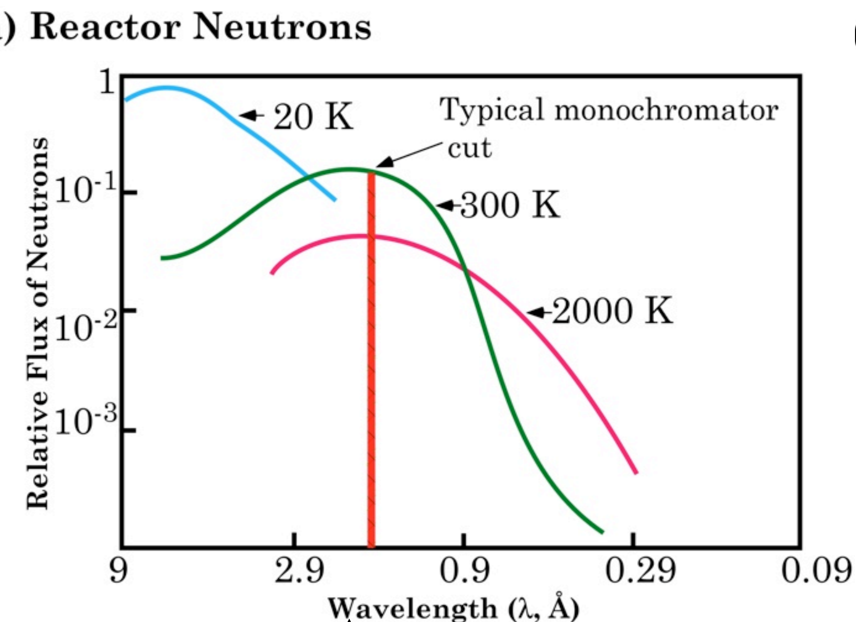
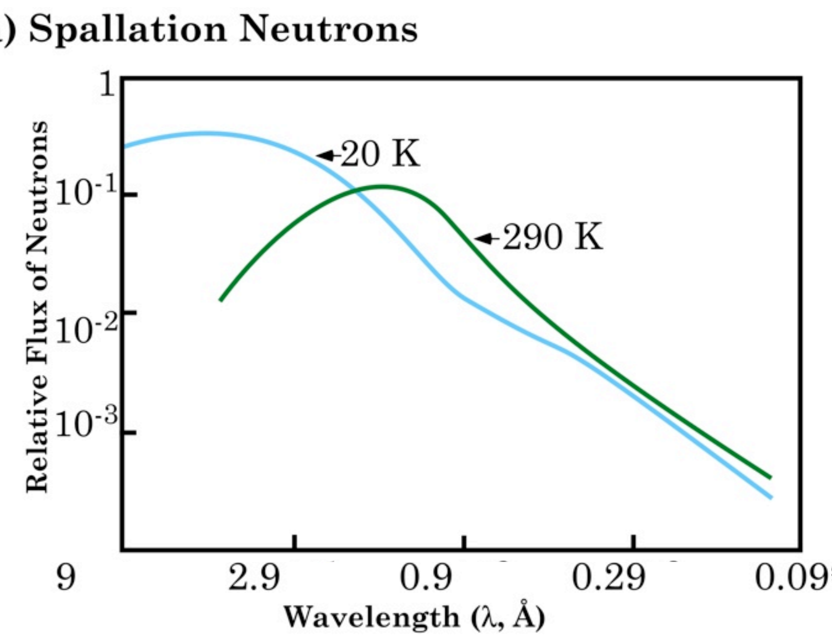
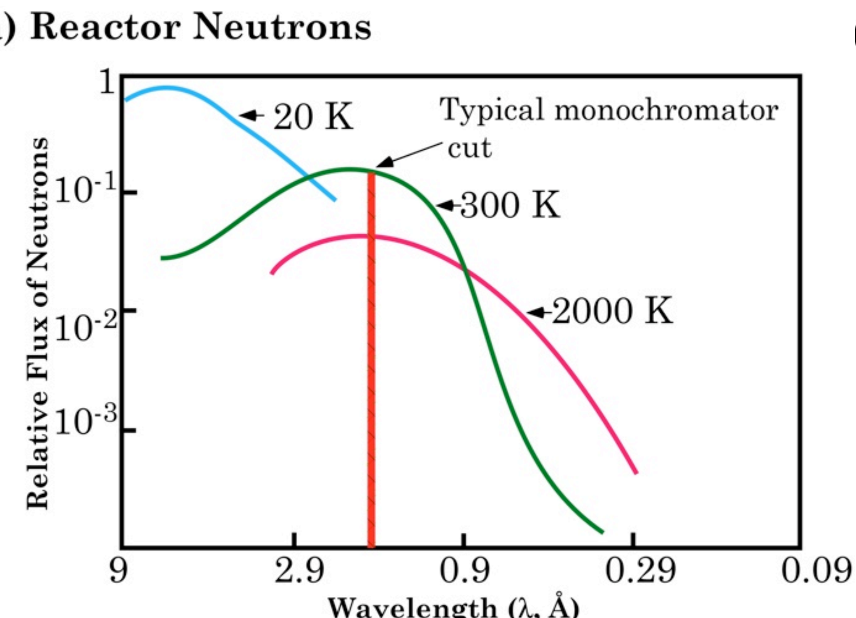


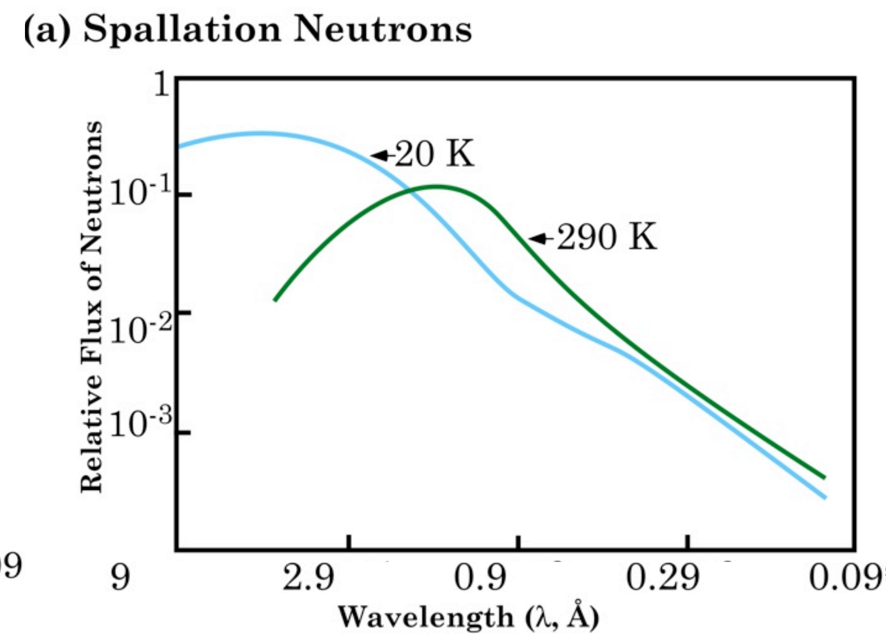


optimized to low E (high d-space) magnetism, phonons, INS

The PE cell at reactor sources - advantages

Neutron mass ~ proton mass: shift spectrum by allowing neutrons to come to thermal equilibrium with proton-rich (CH_4, H_2, H_2O, D_2O) condensed matter at different temperatures)



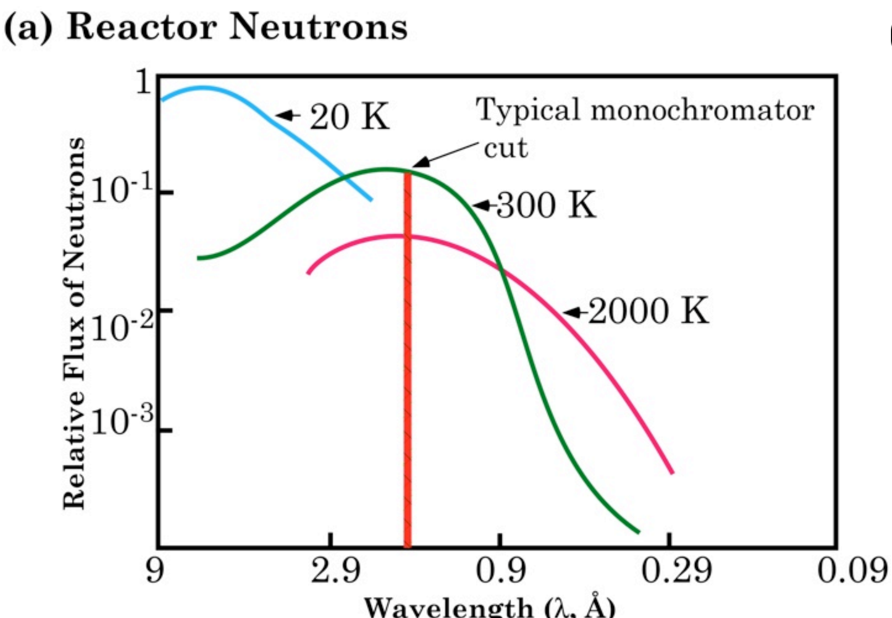


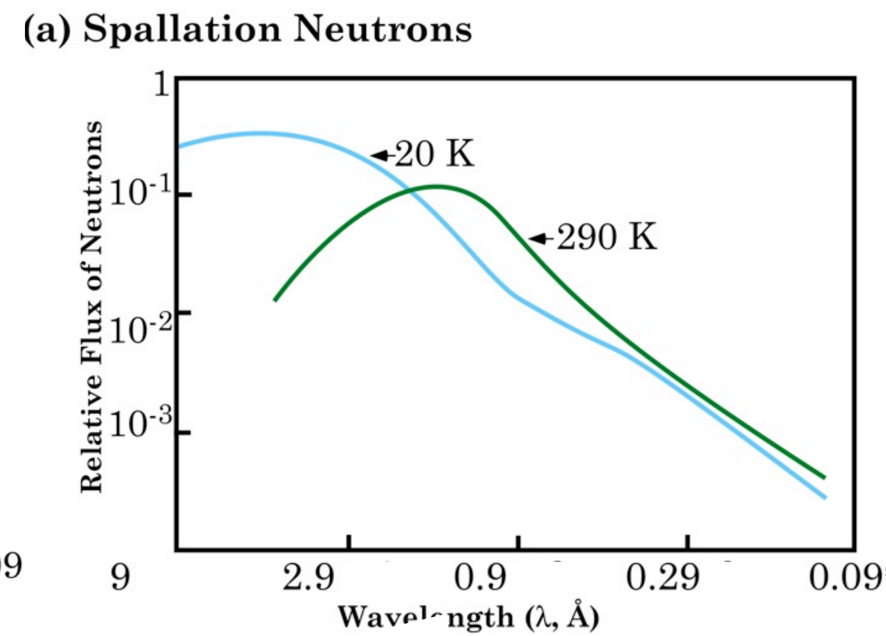
optimized to low E (high d-space) magnetism, phonons, INS

Optimized high E (low d-space) high precision structural studies (PDF for eg)

The PE cell at reactor sources - advantages

Neutron mass ~ proton mass: shift spectrum by allowing neutrons to come to thermal equilibrium with proton-rich (CH_4, H_2, H_2O, D_2O) condensed matter at different temperatures)



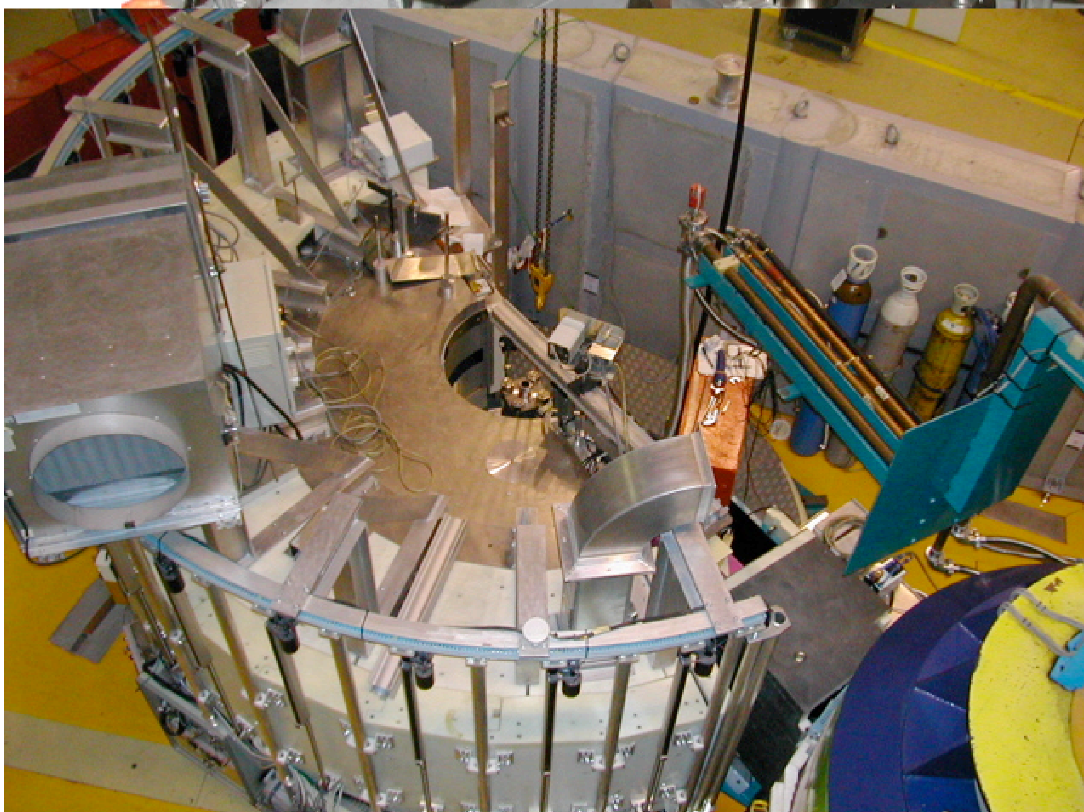


optimized to low E (high d-…
magnetism, pho…

These are generalizations

…ptimized high E (low d-space)
high precision structural studies
(PDF for eg)

The PE cell at reactor sources - advantages



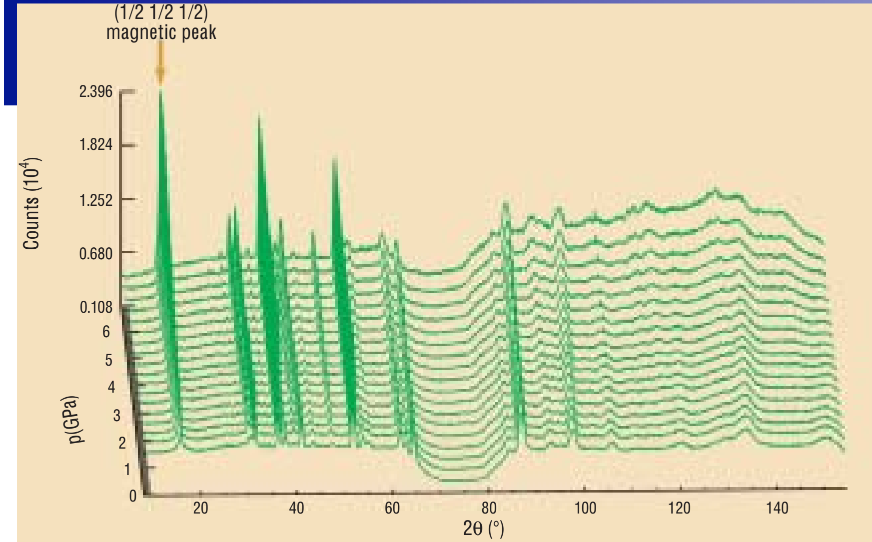


Figure 1: Pressure dependence of the CoO diffraction patterns measured at 300 K on the D20 diffractometer. The appearance and increase of the magnetic contribution is well evidenced with the (1/2,1/2,1/2) magnetic peak intensity variation. Th
mulation time for each pattern is 40 min. The data have
nuclear peak.

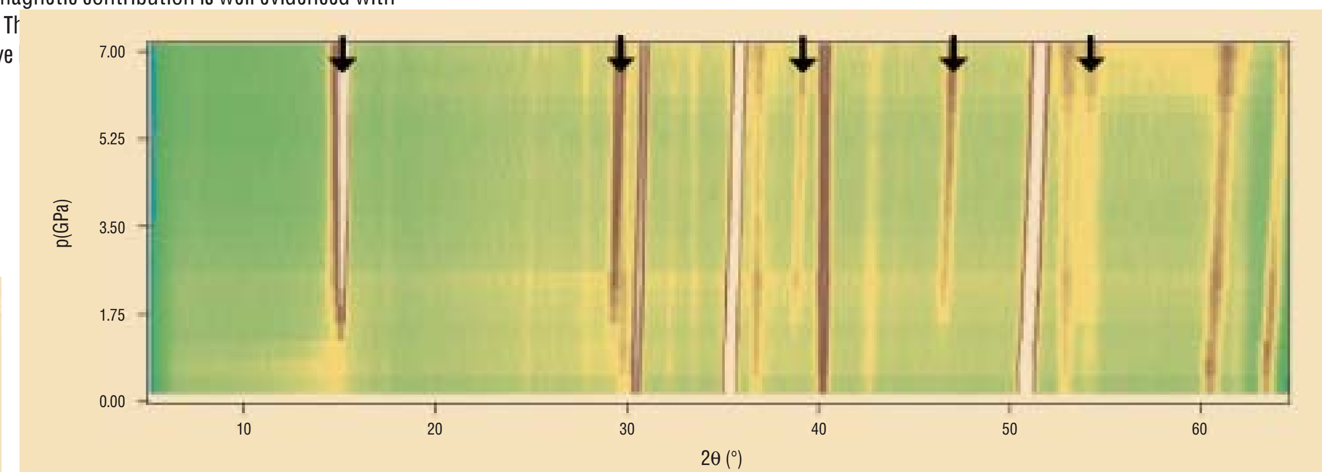


Figure 2: Low angular range zoom of the CoO diffraction pattern measured at 300 K under pressure. The magnetic contributions are marked with arrows.

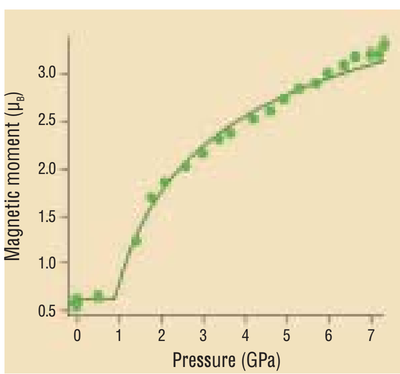
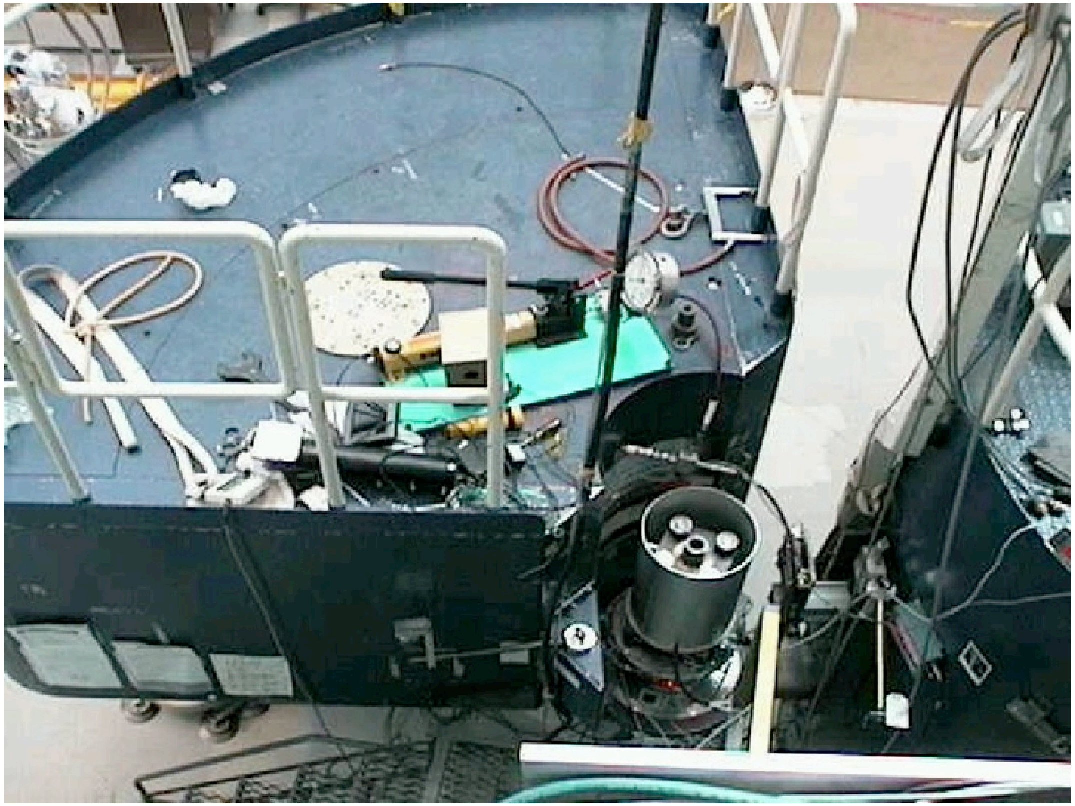
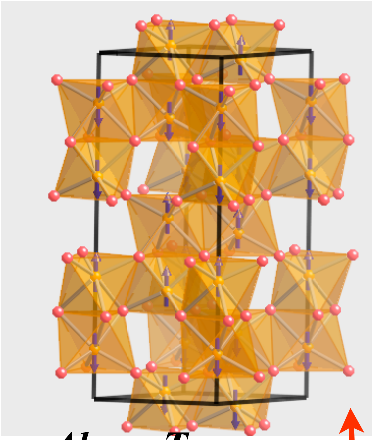


Figure 3: Pressure dependence of the CoO magnetization at 300 K, derived from Rietveld fits to the patterns shown in figure 2. The line is a guide for the eye.

Chalk River and ORNL

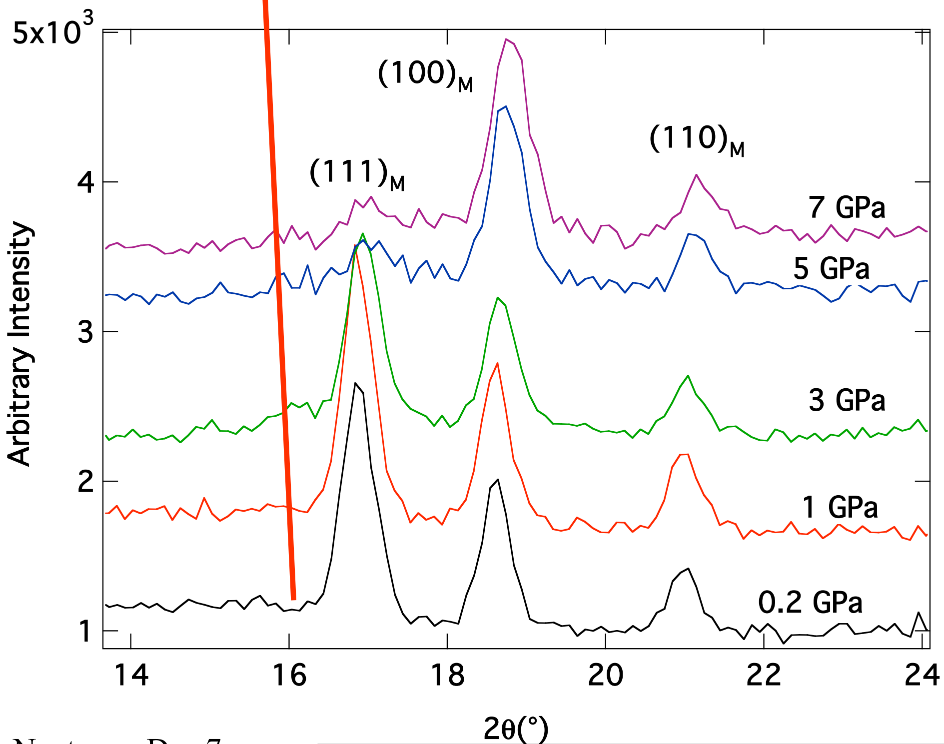


Other ambiguous magnetic structure



Above T_M

T > 250 K; low P



Parise, Locke, Tulk, Swainson, Cranswick, Physica B (2006)

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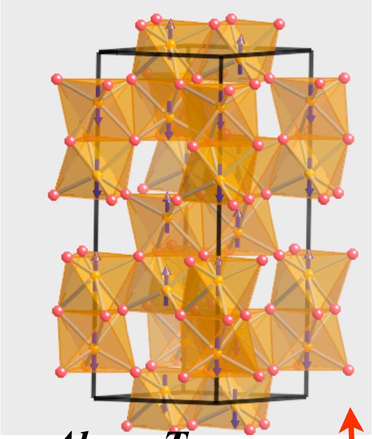

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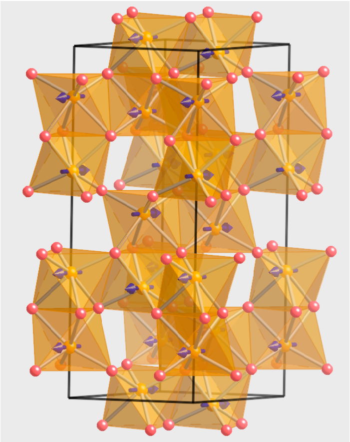
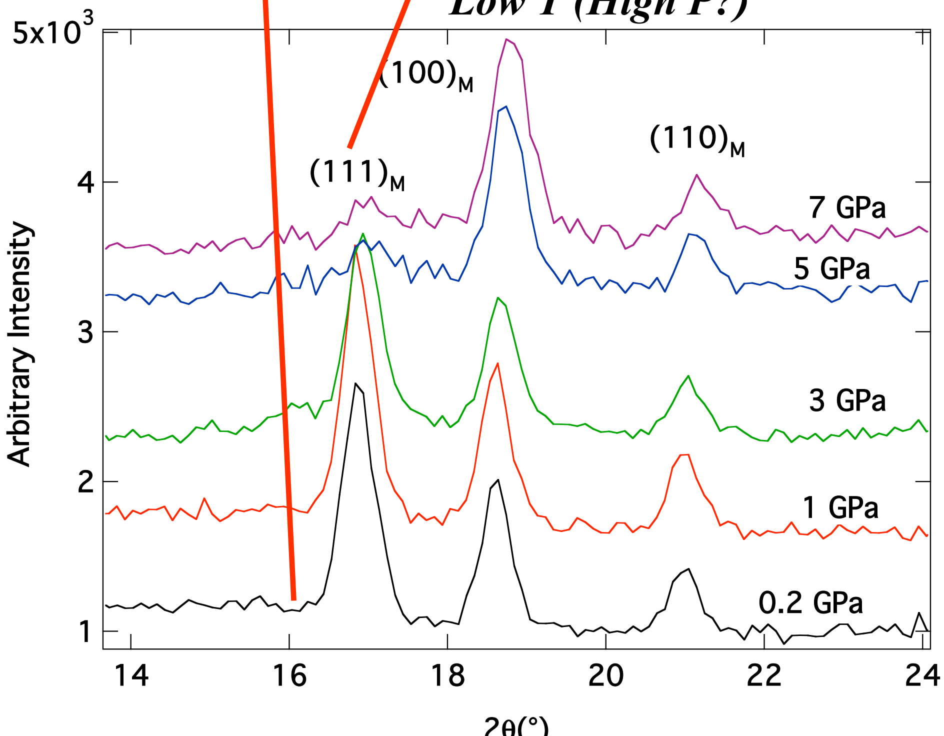


Morin transition
Low T (High P?)



Parise, Locke, Tulk, Swainson, Cranswick, Physica B (2006)

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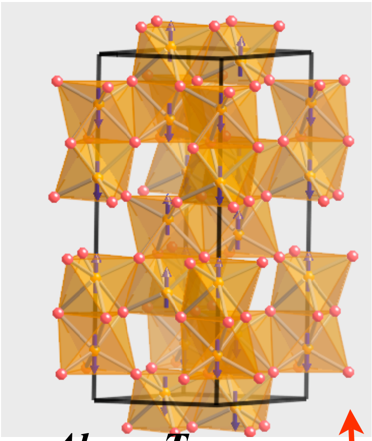

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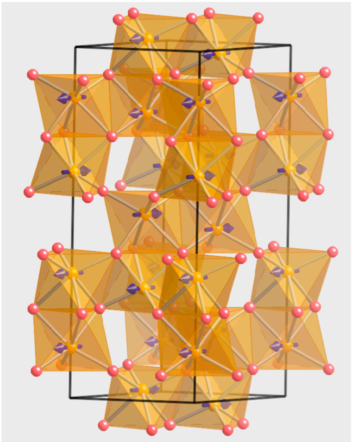

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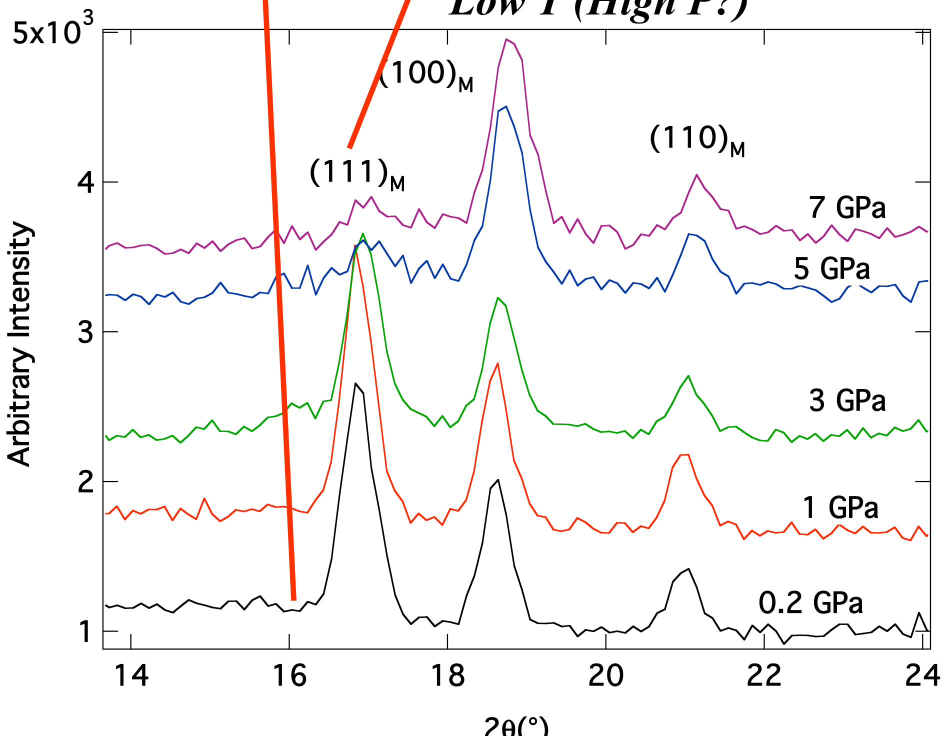

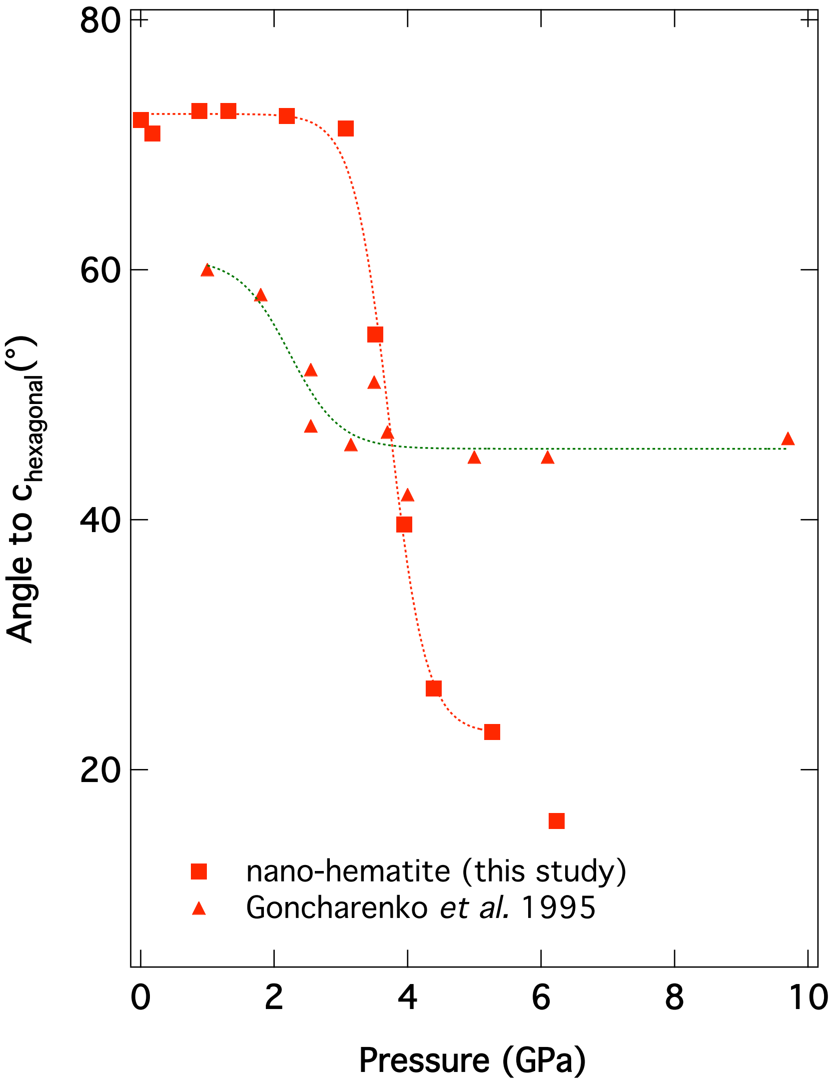

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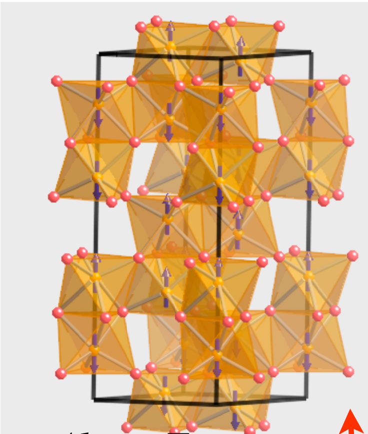

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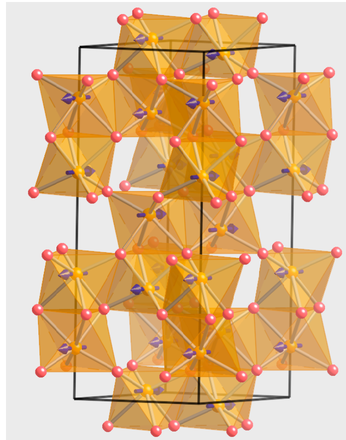

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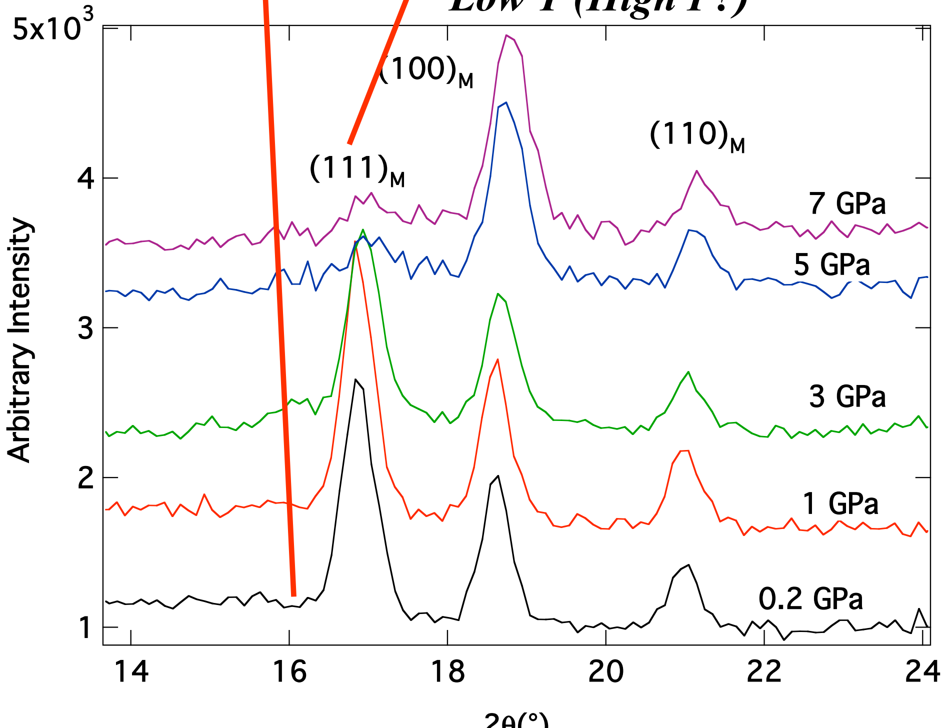

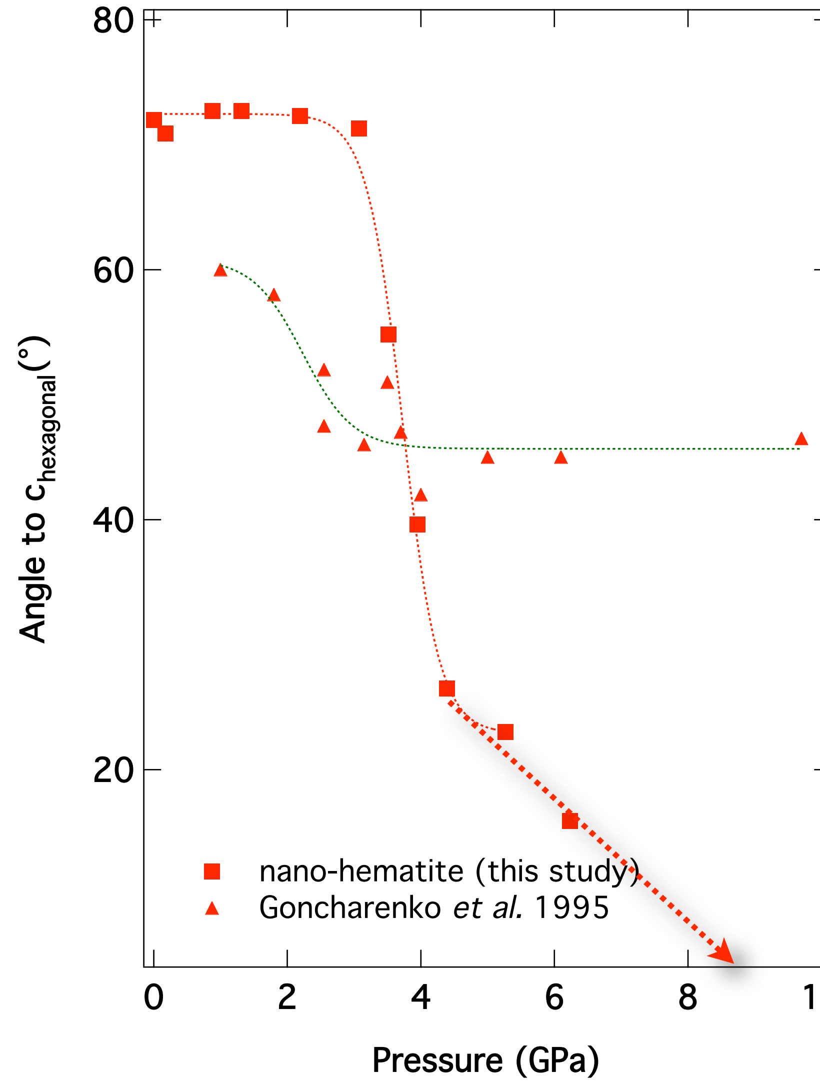

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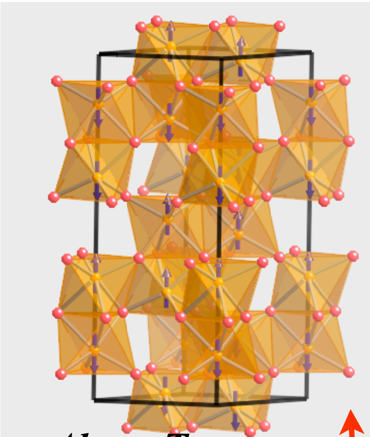

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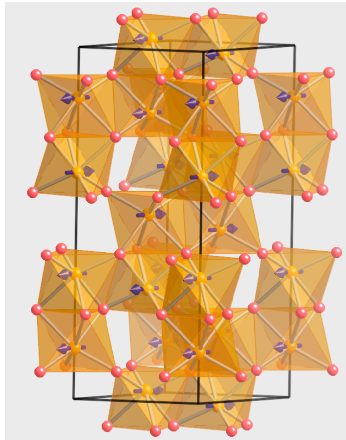
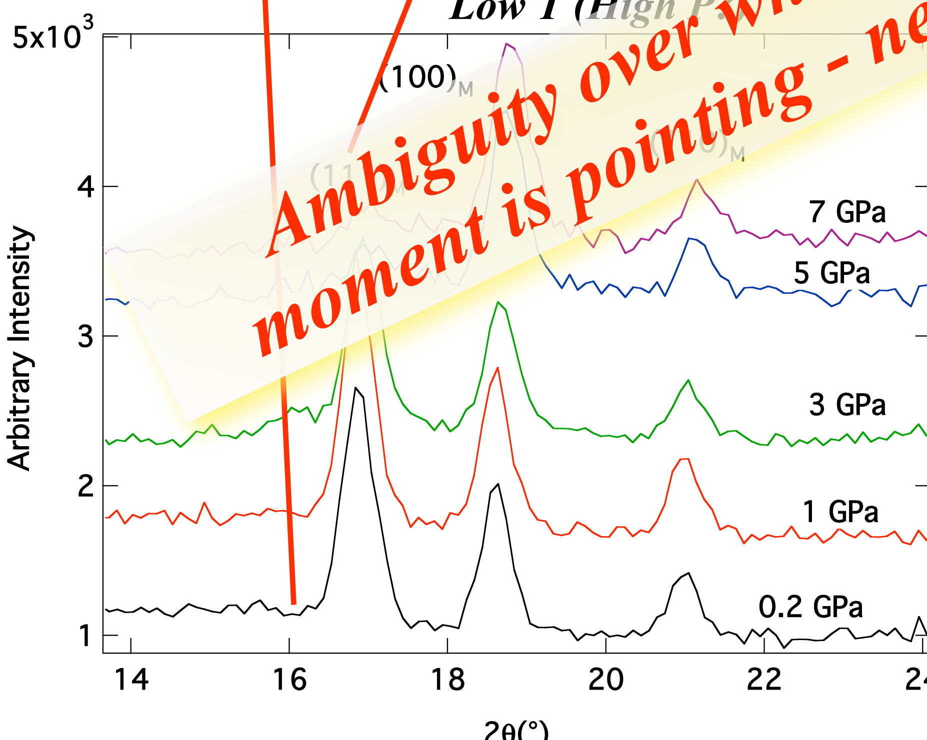
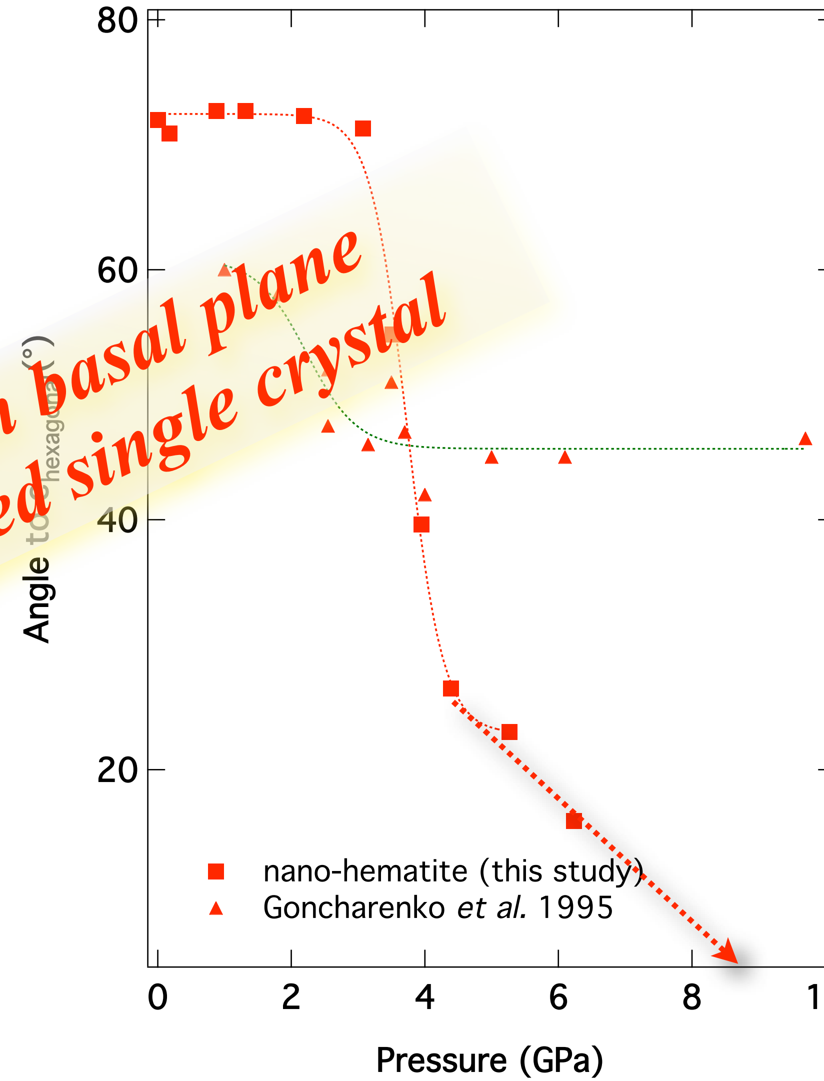


Morin transition
Low T (High P?)

Ambiguity over where in basal plane moment is pointing - need single crystal

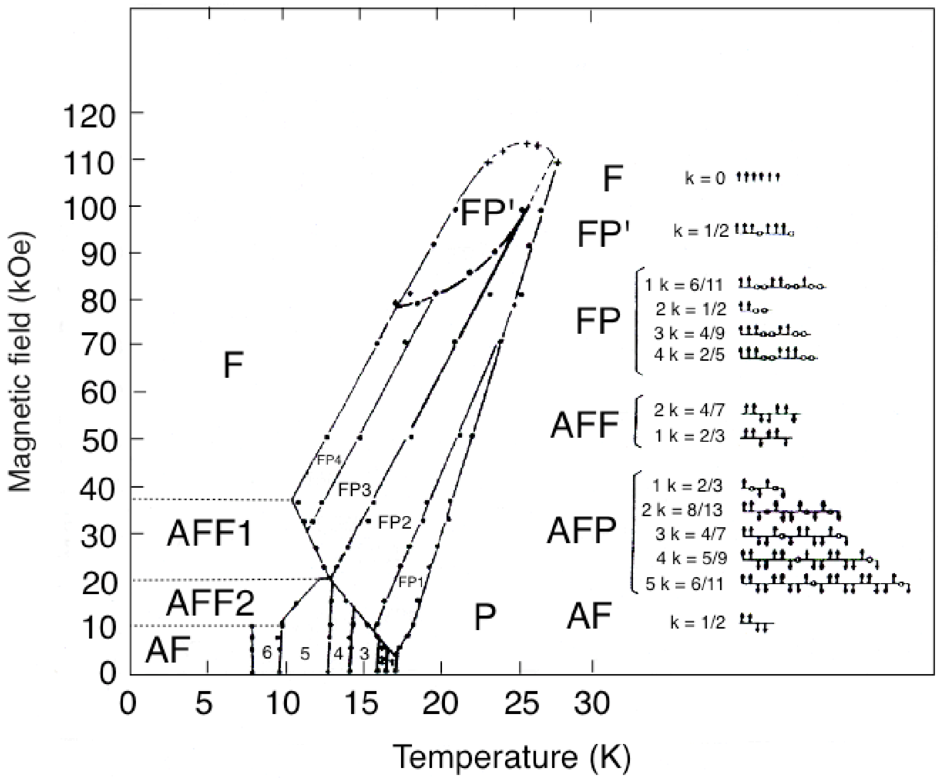




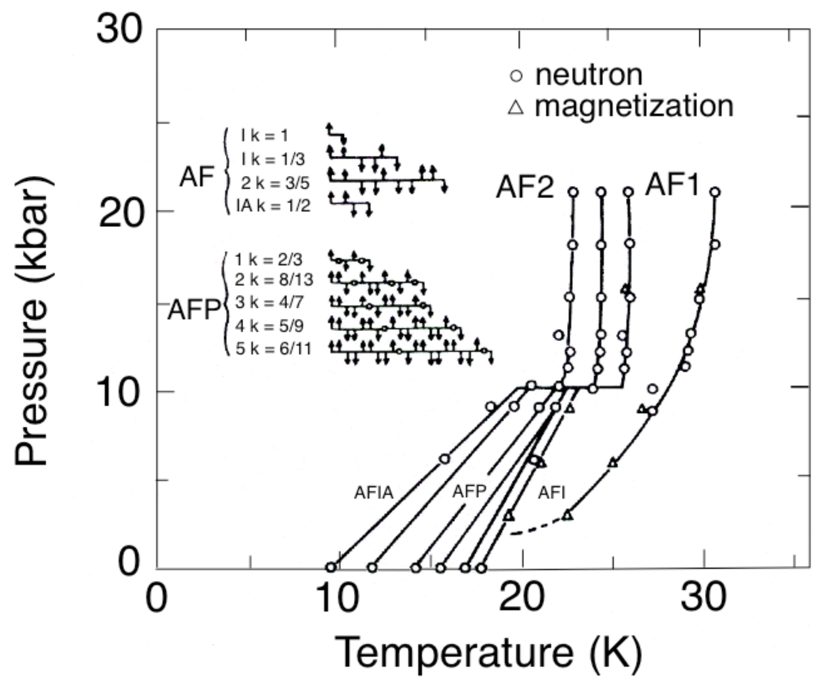
Parise, Locke, Tulk, Swainson, Cranswick, Physica B (2006)

Science opportunities: Kondo-lattice system CeSb: Rossat-Mignod and coworkers at ILL, Grenoble

- ***Most complex magnetic phase diagram known so far:***
- ***consists of sixteen phases.***
- ***At H = 0 the low temperature Phase is the type-IA phase.***
- ***The rest are modulated AFP phases containing paramagnetic planes.***



- ***high P studies to 2 GPa***
- ***Coworkers:P. Burlet, J. Rossat-Mignod, C. Vettier, in ILL clamp cell***
- ***Important results:***
- ***Stabilization of type I AF phase.***
- ***Disappearance of AFP phases.***
- ***Type I and type IA at high pressure.***
- ***CeSb behaves like CeBi at high P, containing paramagnetic planes.***



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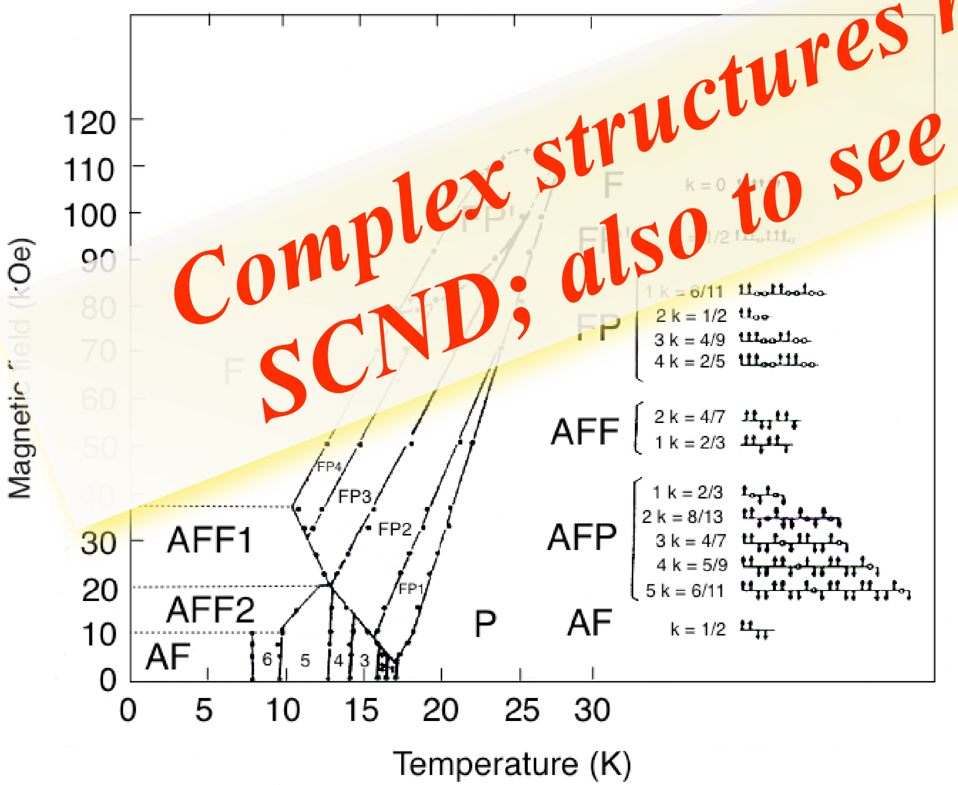
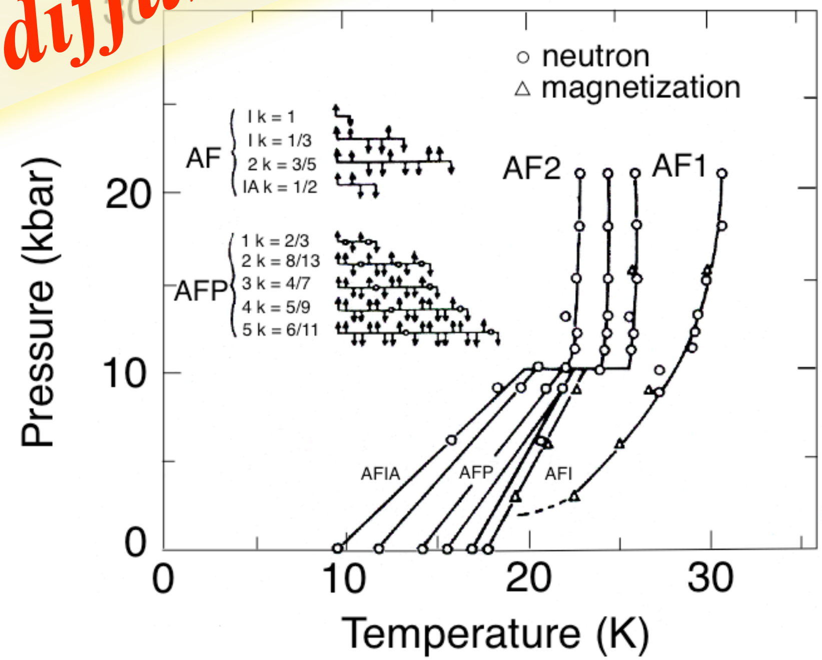
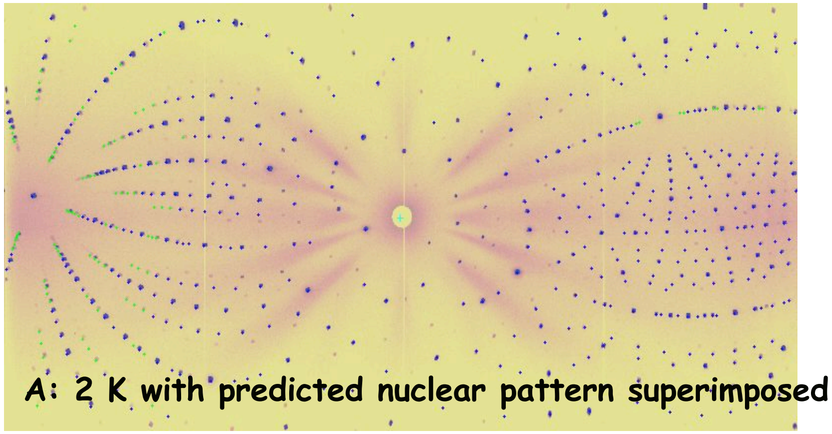
Complex structures need resolution by SCND; also to see diffuse scattering

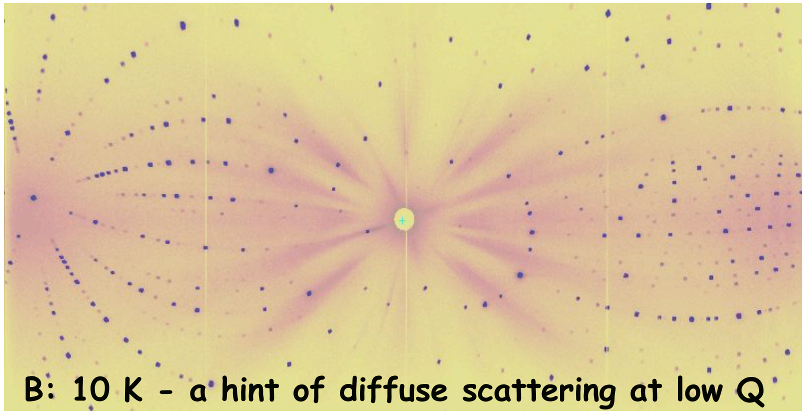




VIVALDI - magnetic diffuse scattering (thanks to G. McIntyre)

- $FeTaO_6$, Chung, Balakrishnan, Visser & Paul (Warwick), McIntyre (ILL)
- 3-D antiferromagnetic order at 8 K, 2-D order above 8 K (ALL room pressure).
- Difficult to imagine interpretation of complex magnetic structure from powders





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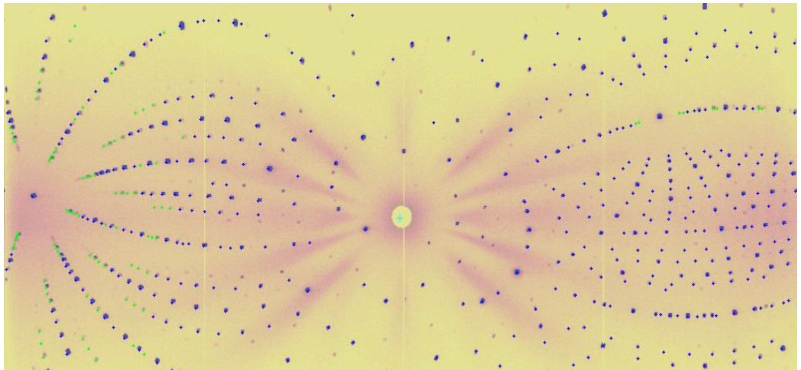
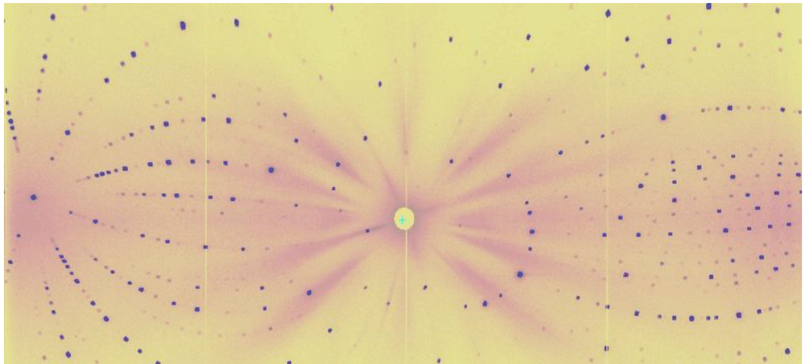
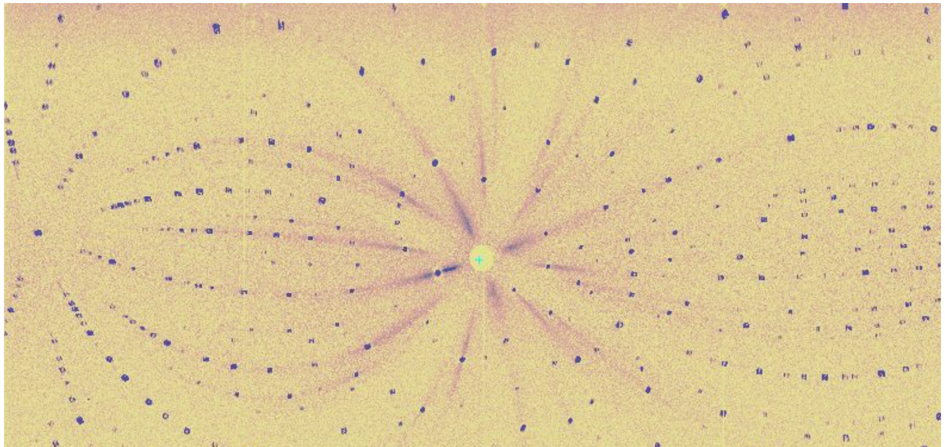


A: 2 K with predicted nuclear pattern superimposed



B: 10 K - a hint of diffuse scattering at low Q



C: 10 K minus 2 K -> rods of magnetic scattering

High pressure single crystal neutron scattering: The way forward for complex hydrous minerals and magnetism

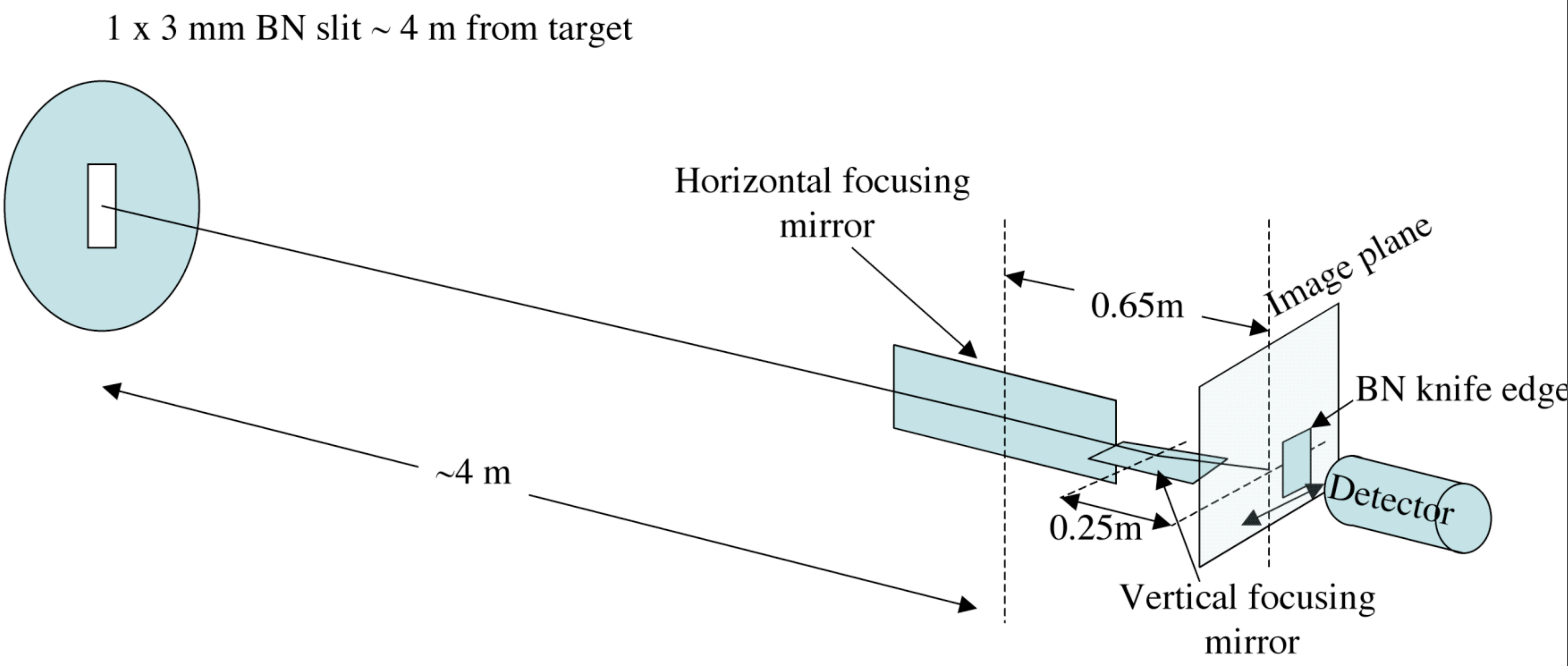
- Smaller samples
- Brighter beams
  - More flux (SNS)
  - Smaller beams (focusing)
- More sensitive, larger area, lower noise, detectors
- Laue technique

(1) More Flux - A good reason to go to Oak Ridge Tennessee

The Spallation Neutron Source (SNS) construction project will conclude in 2006

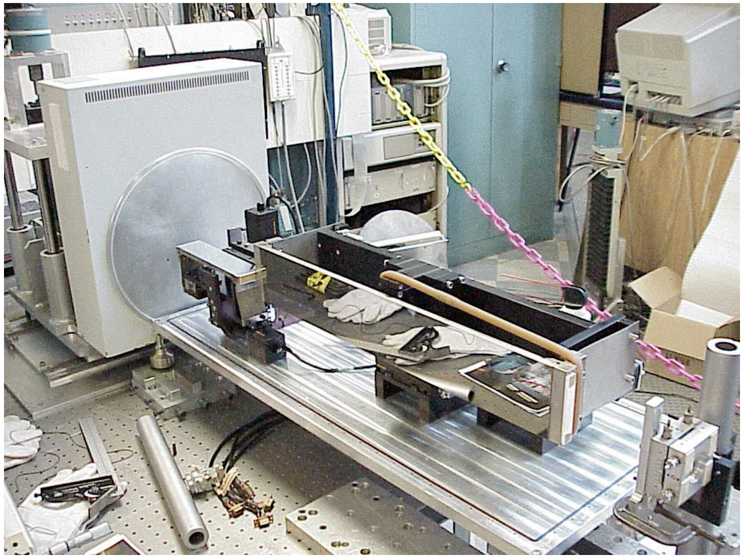
At 1.4 MW it will be the world's most powerful source of neutrons and the world's leading facility for neutron scattering. For what science will it be used?

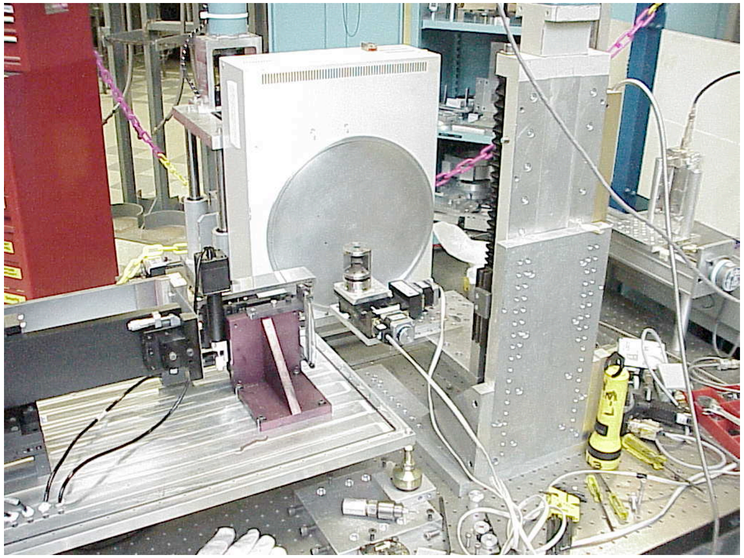
(2) Greater brightness: Neutron Focusing



Testing and Optimization of neutron mirrors

- *A new prototype microfocusing system tested.*
- *Measured spot size (90 x 90 um) with roughly 30 mrad on sample.*
- *Diffraction data from free standing 300x300 µm samples and from 200 um FeO single crystal sample in a panoramic cell--under pressure*





Tulk, Locke, Xu, Ice, Parise, et al. (2004), ORNL, NRCC, Stony Brook and Carnegie Institution.

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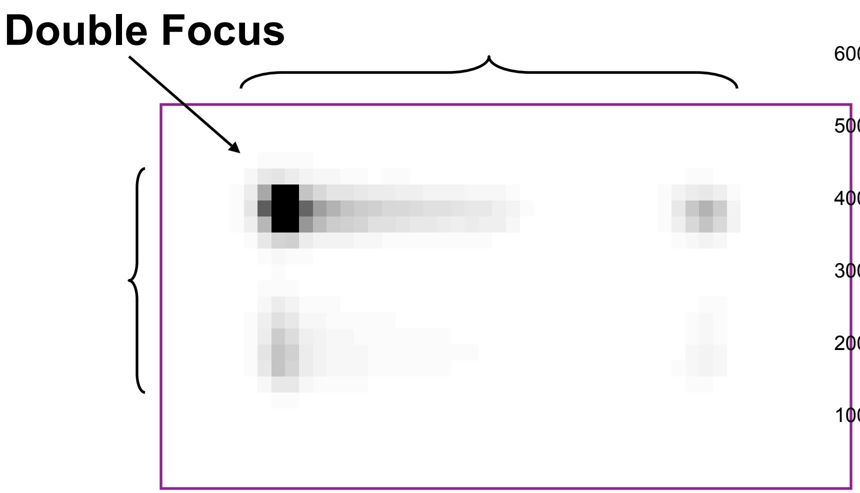

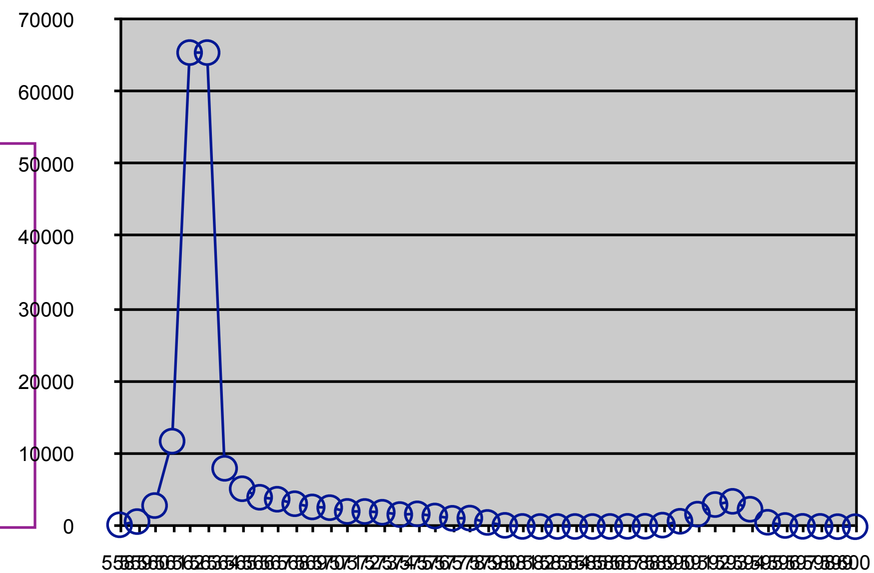

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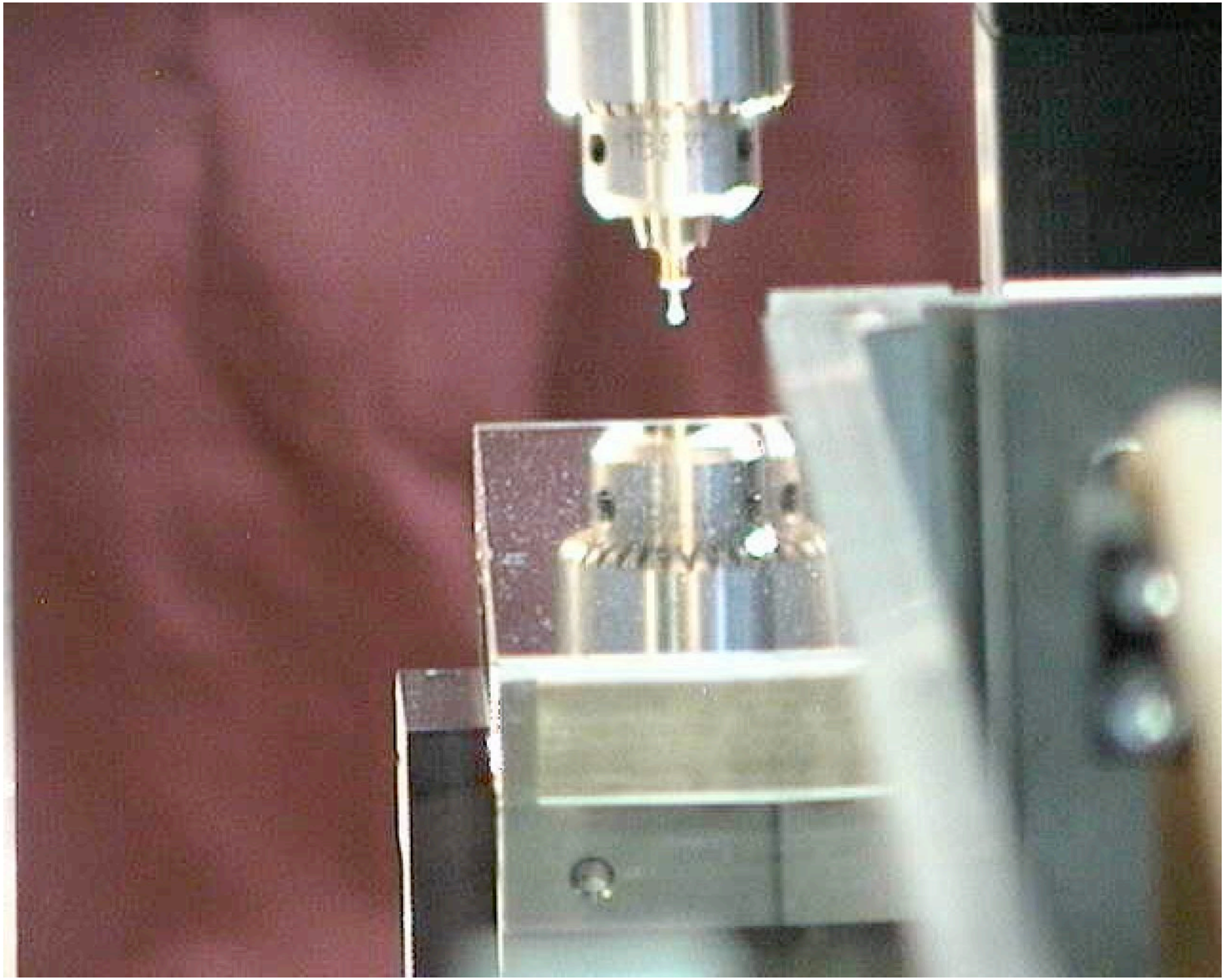
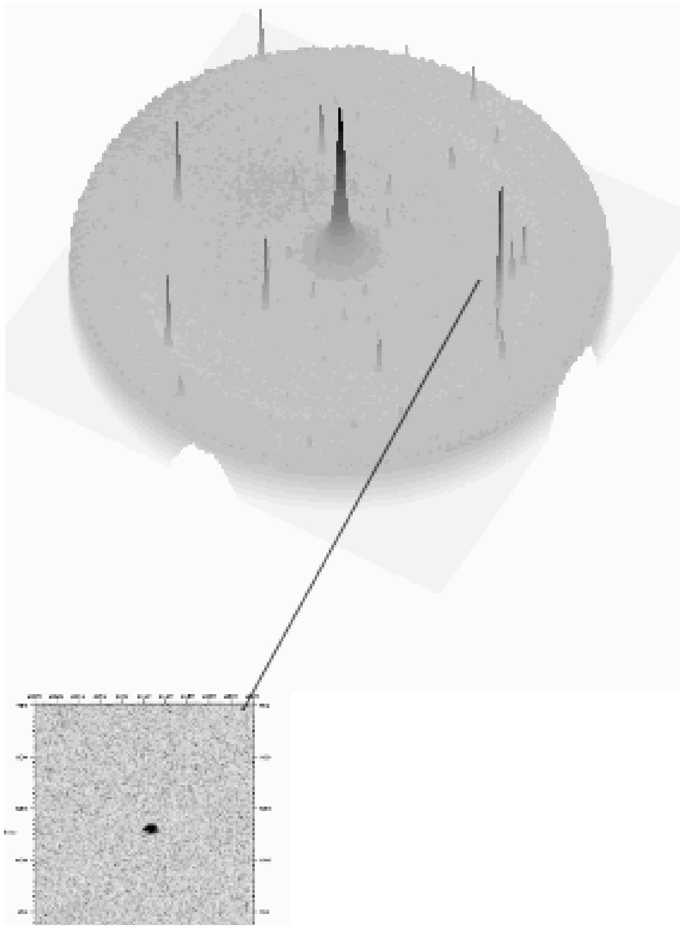
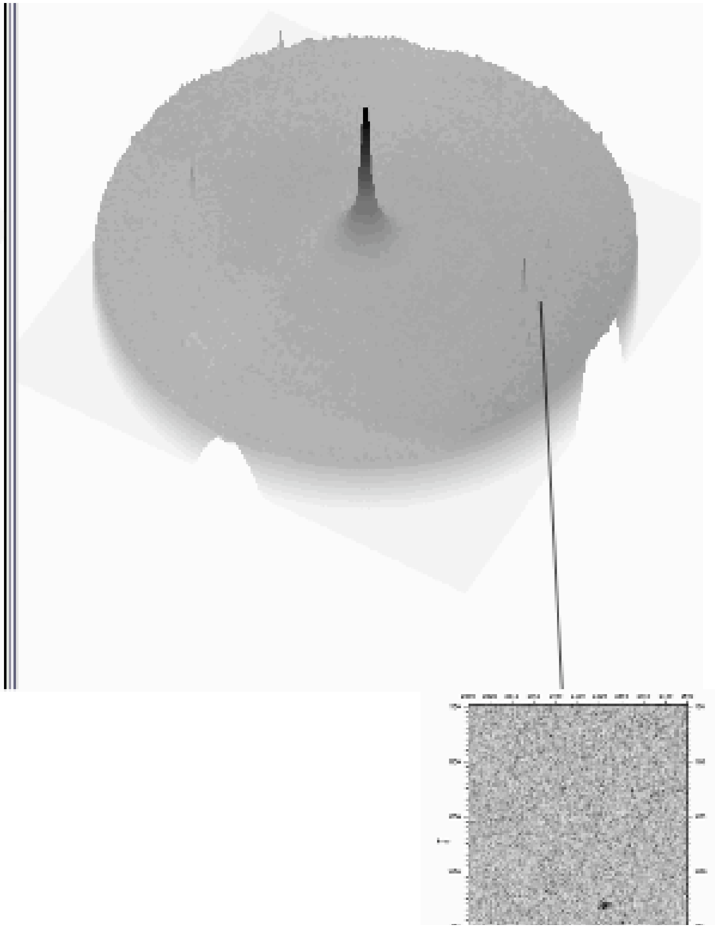




<u>With KB mirrors in place</u>

90 x 90 micron beam

SCATTERING VOLUME ~ 0.0045 mm^3
Forserite - Mg_2SiO_4
$\Phi = 170^\circ$

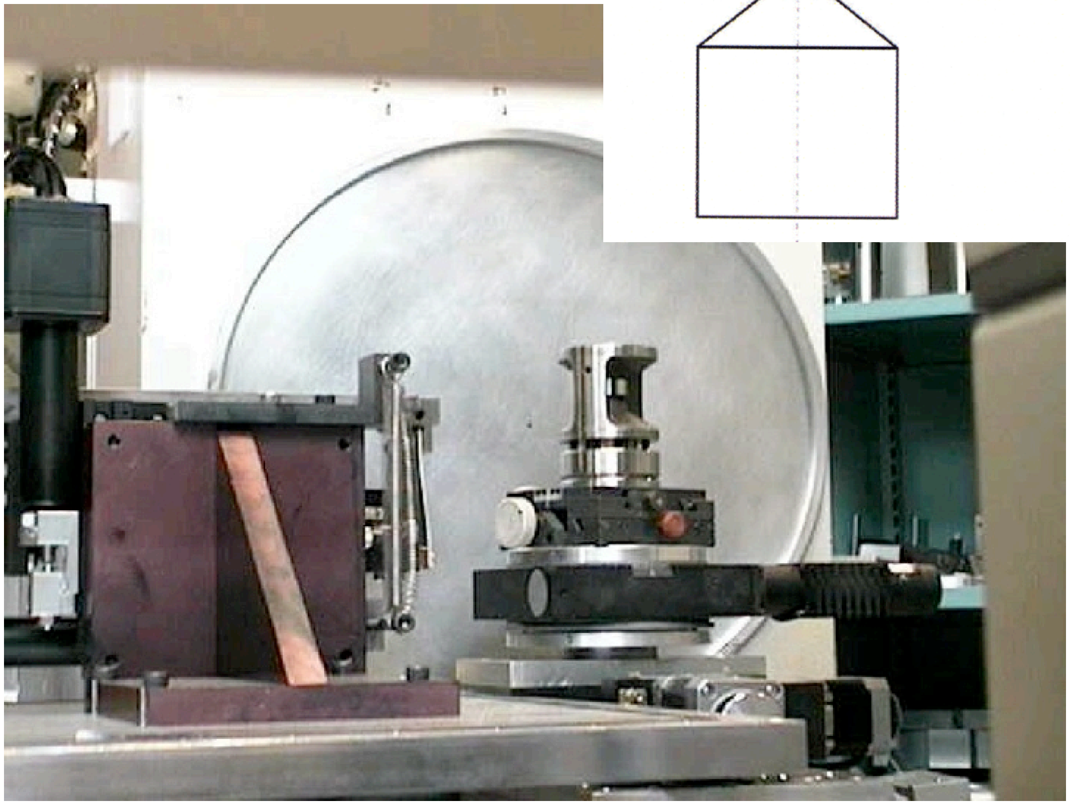


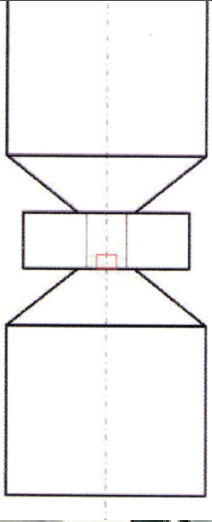
<u>Without KB mirrors in place</u>

Beam much larger than sample

SCATTERING VOLUME ~ 0.05 mm^3
Forserite - Mg_2SiO_4
$\Phi = 170^\circ$

Mirrors + cell





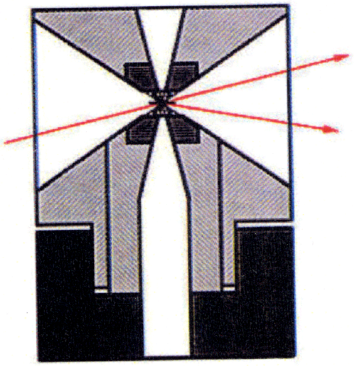
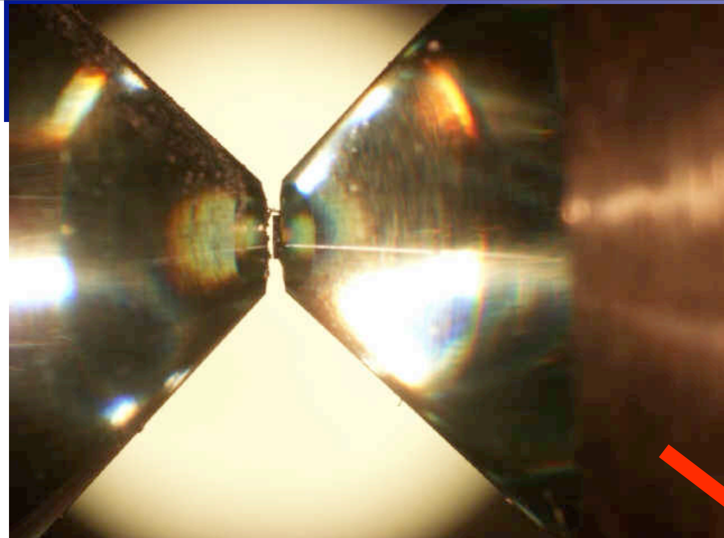
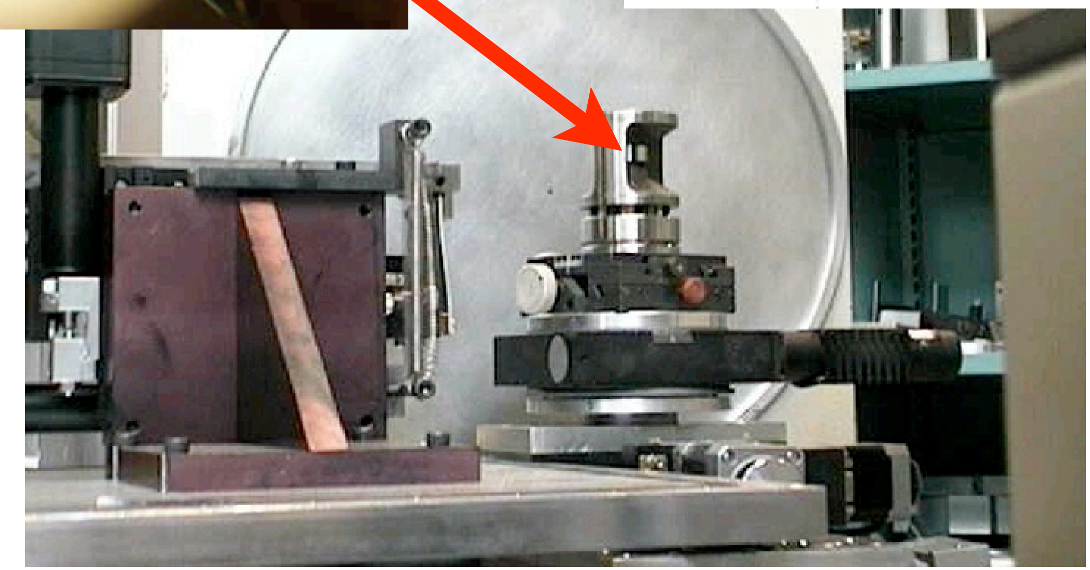
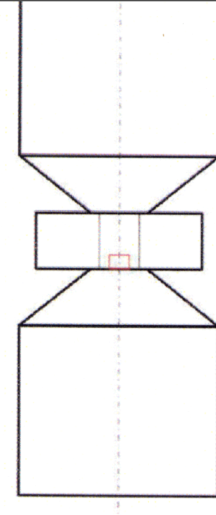


Figure 2 Panoramic cell







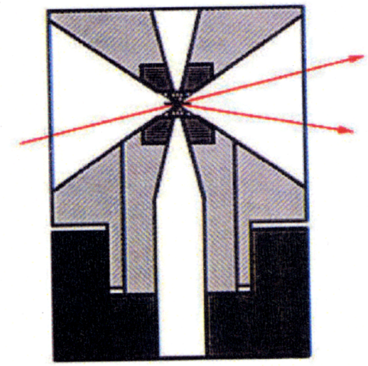
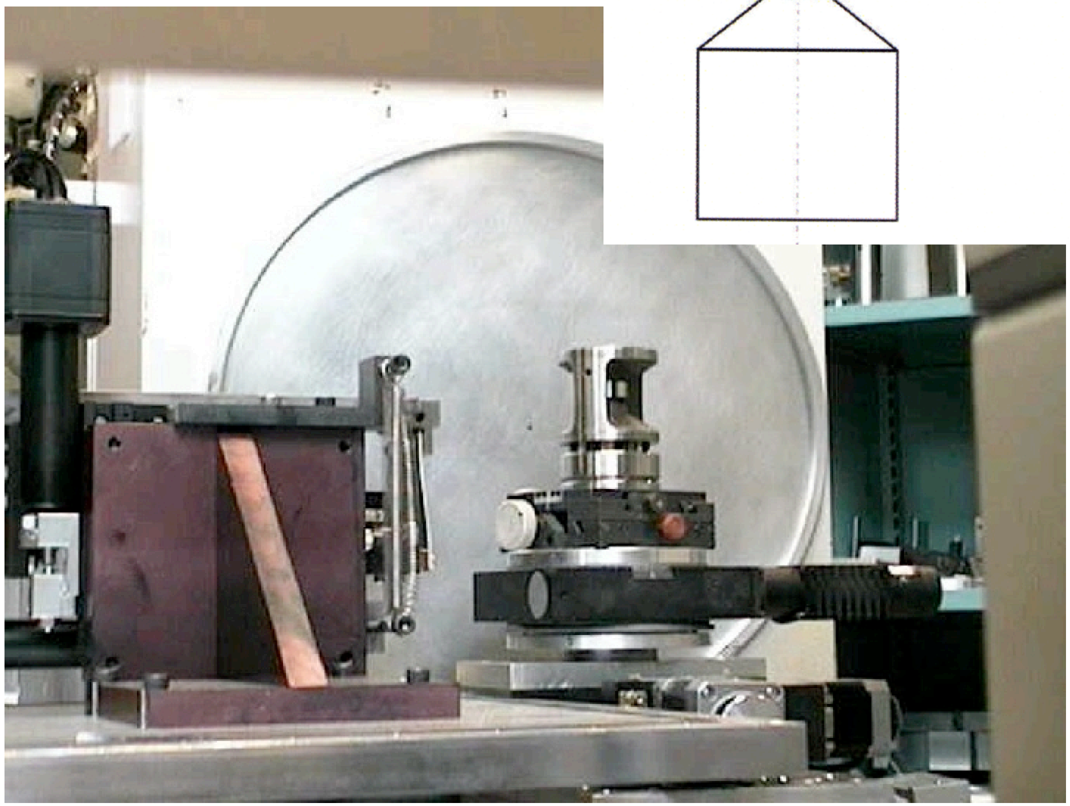
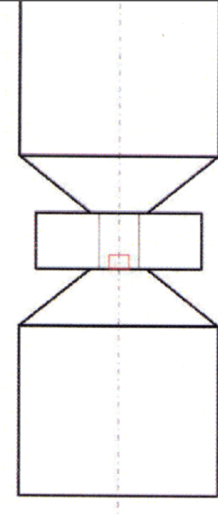
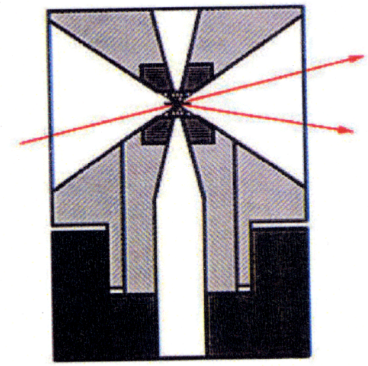


Figure 2 Panoramic cell

Mirrors + cell







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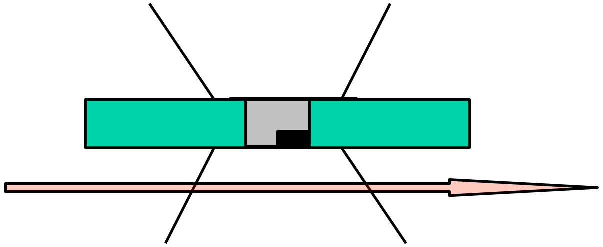
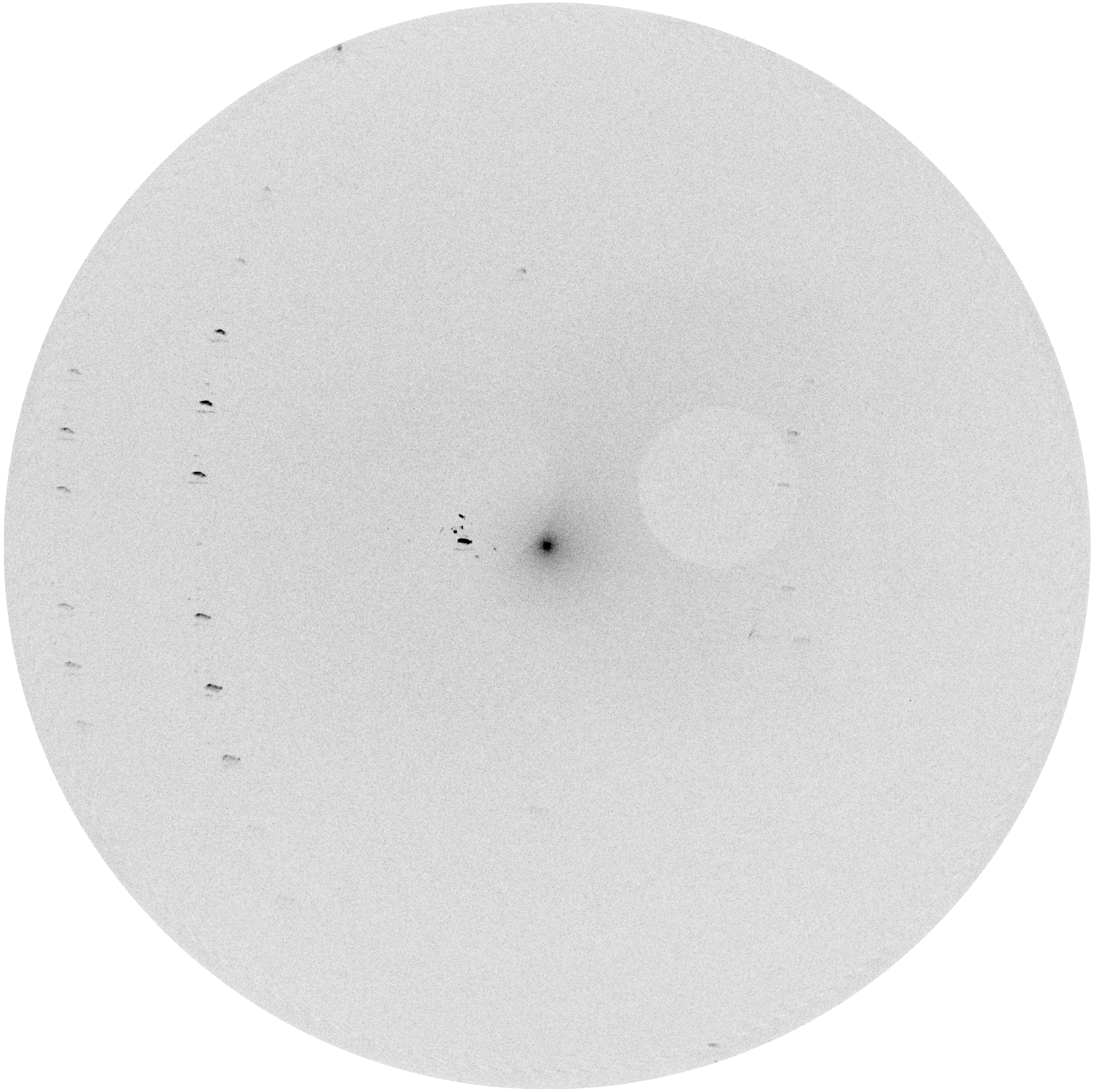
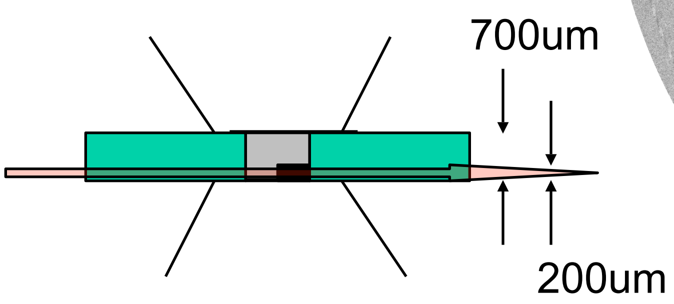
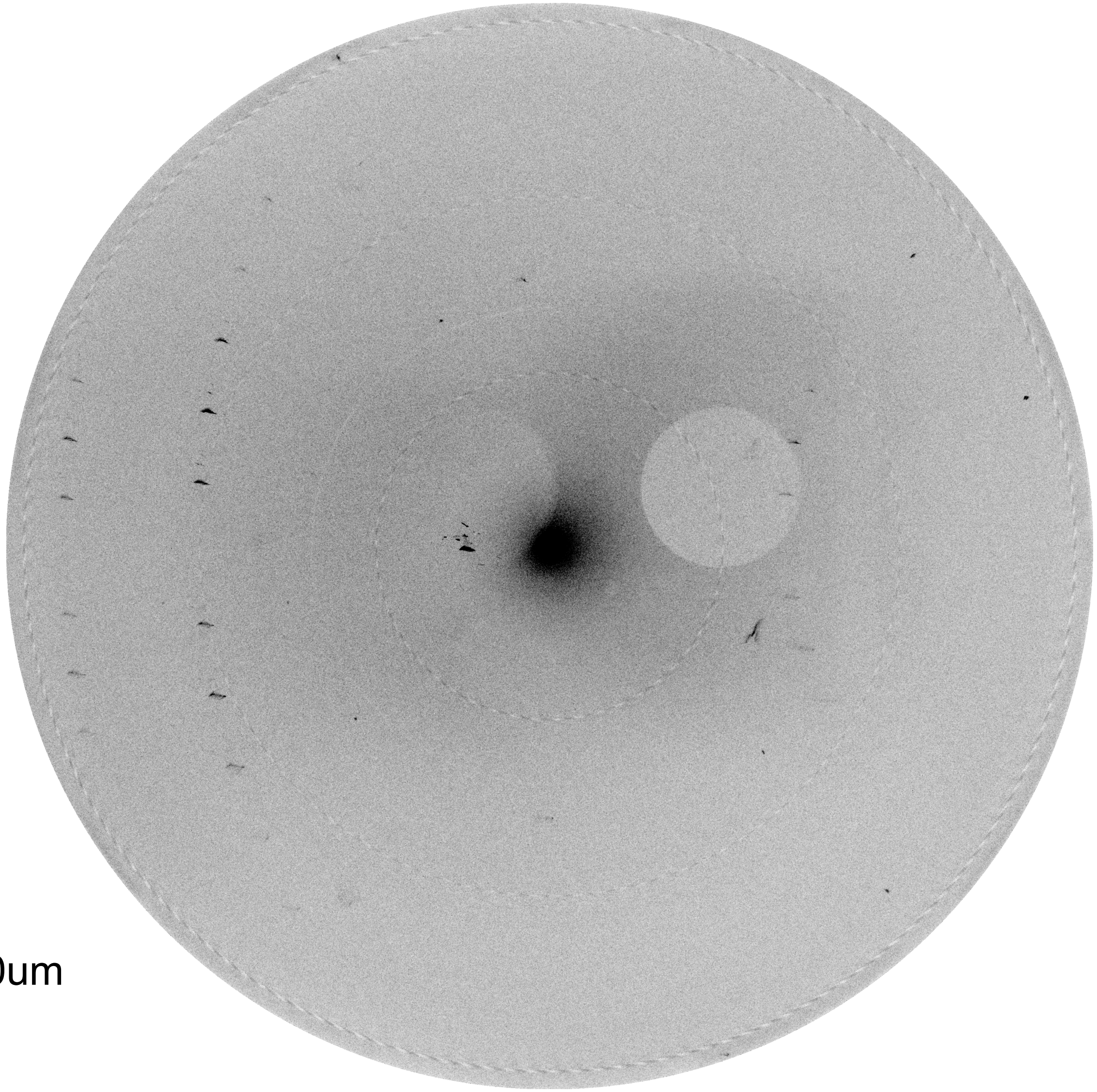


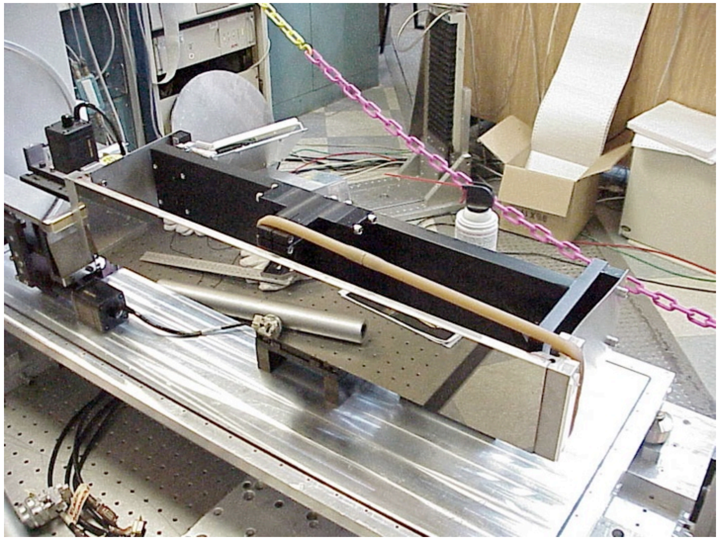



700um
200um



J. D. Jorgensen's High Pressure legacy

- Focusing (Gene Ice ORNL)



  - From this

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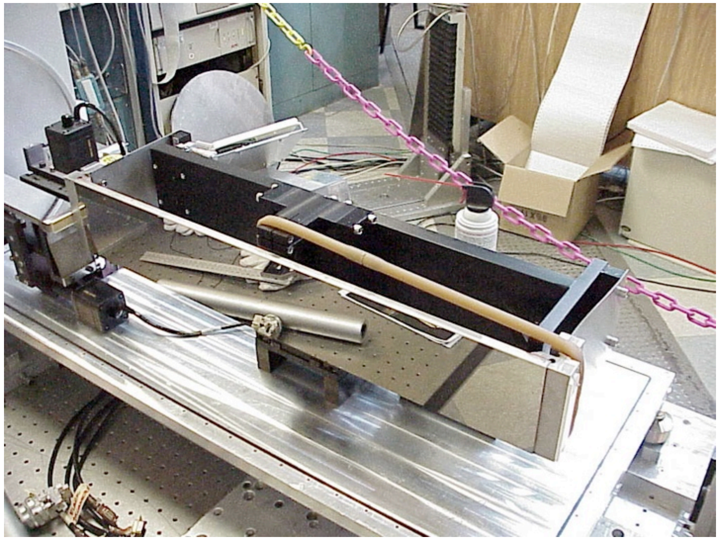

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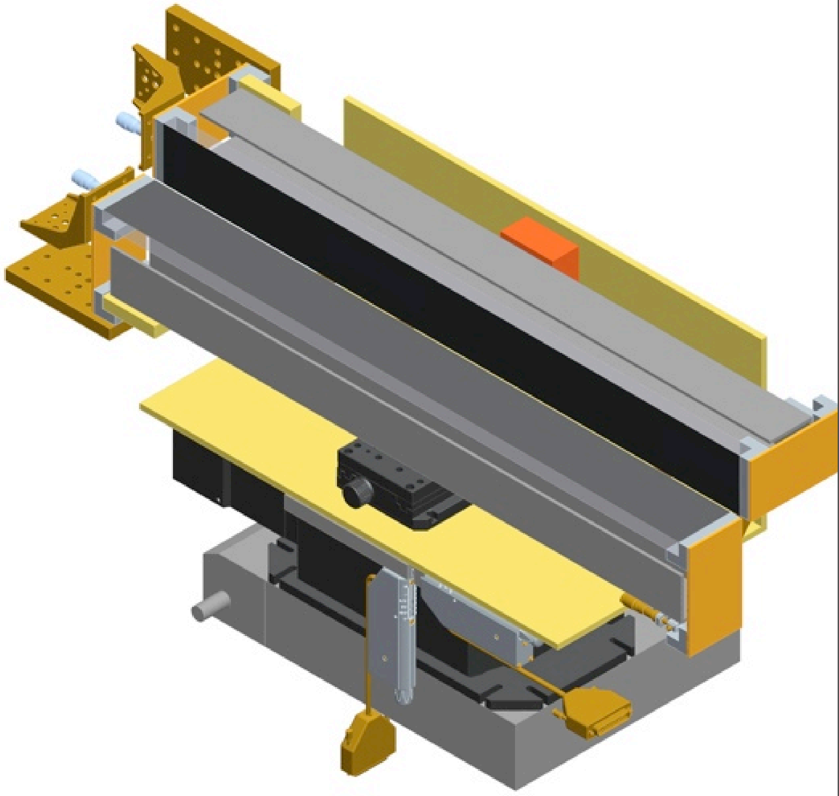


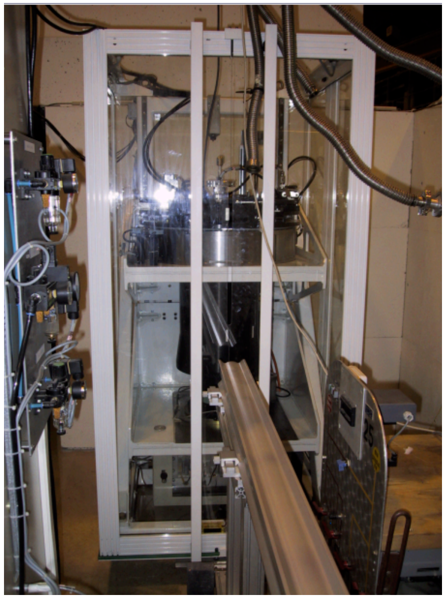
– to this

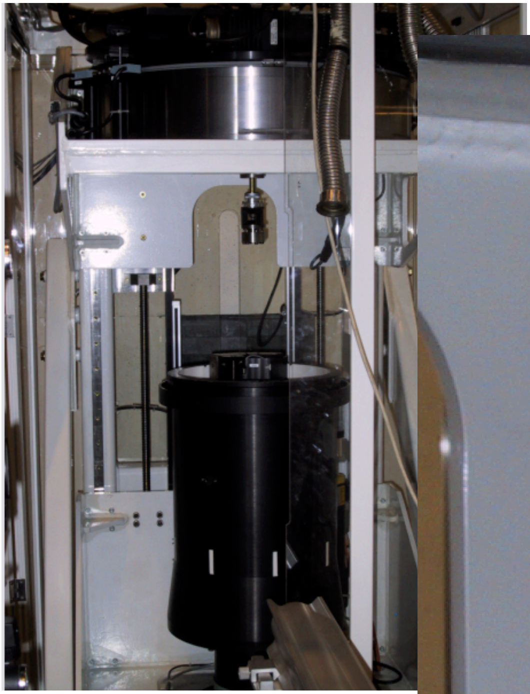
The way forward

- Smaller samples
- Brighter beams
  - More flux (SNS)
  - Smaller beams (focusing)
- More sensitive, larger area, lower noise, detectors
- Laue technique

Area Detectors and white beams (VIVALDI, McIntyre ILL)

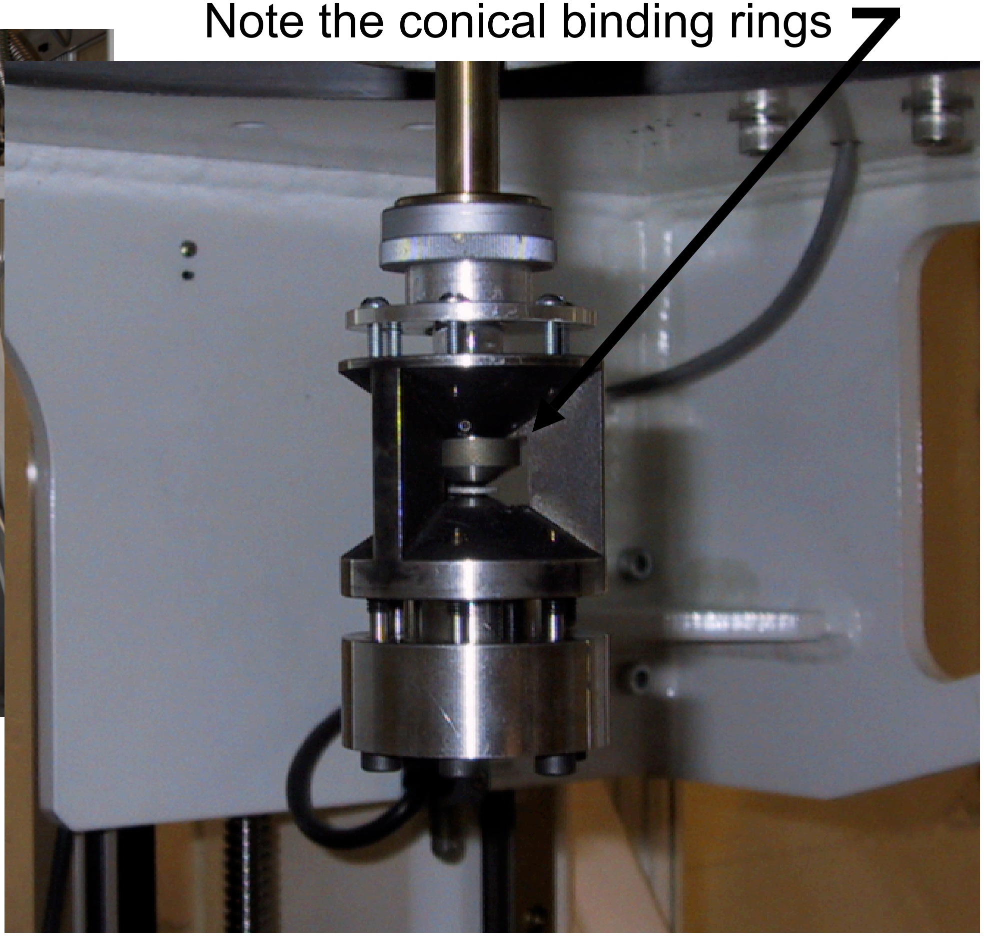
Closed Instrument





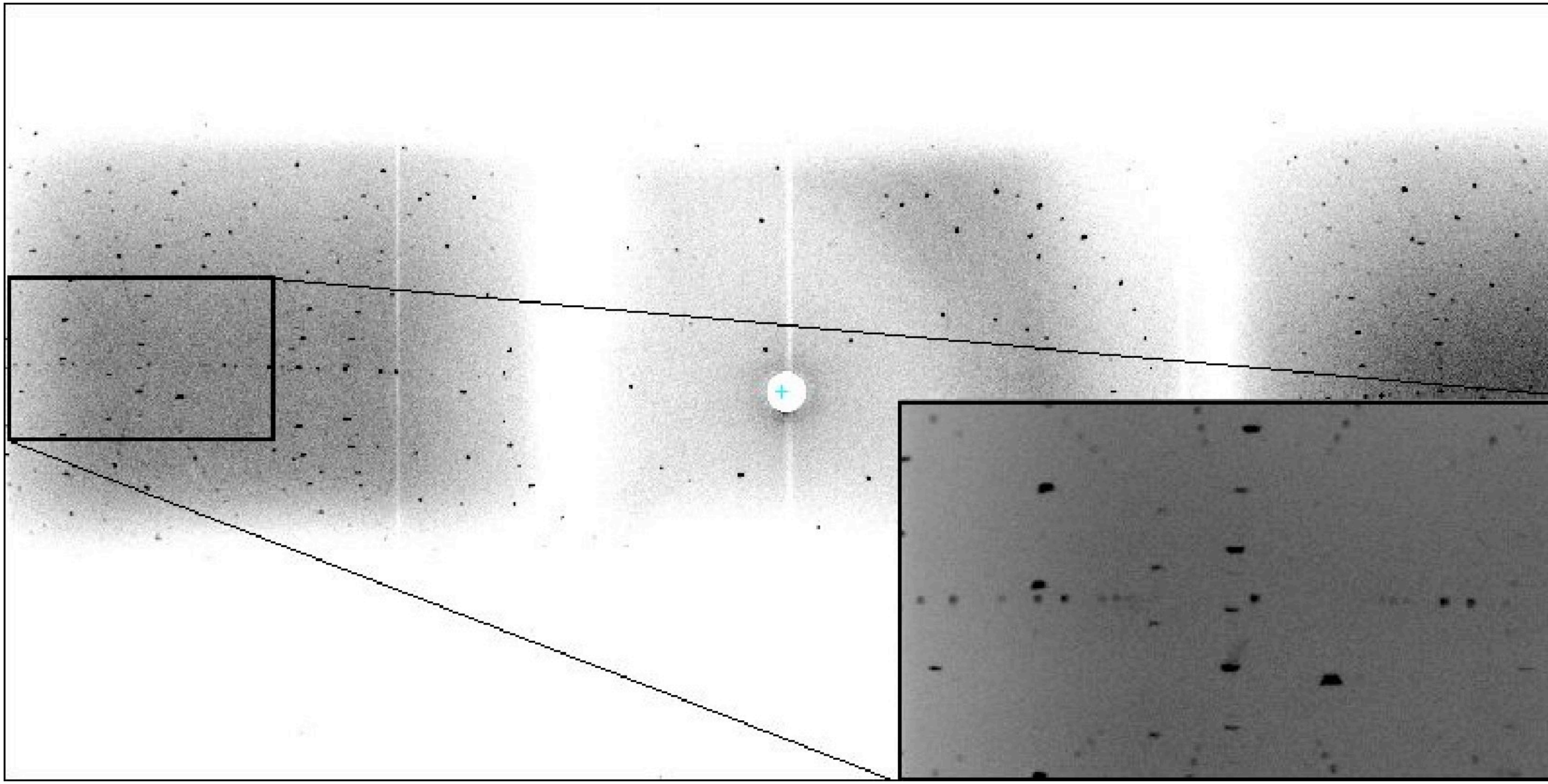
Mounting pressure cell in instrument

Note the conical binding rings



One picture says it all (McIntyre et al, 2005)

Natrolite single crystal in cell (no pressure)



Laue diffraction pattern from a 0.5 mm^3 natrolite sample in a moissanite-anvil cell: a) φ = 65°, exposure time 1 hr

CCDs at neutron (reactor) source

- First test last week

Bringing it all together - Spallation Neutrons And Pressure (SNAP) Instrument Components - synchrotron like

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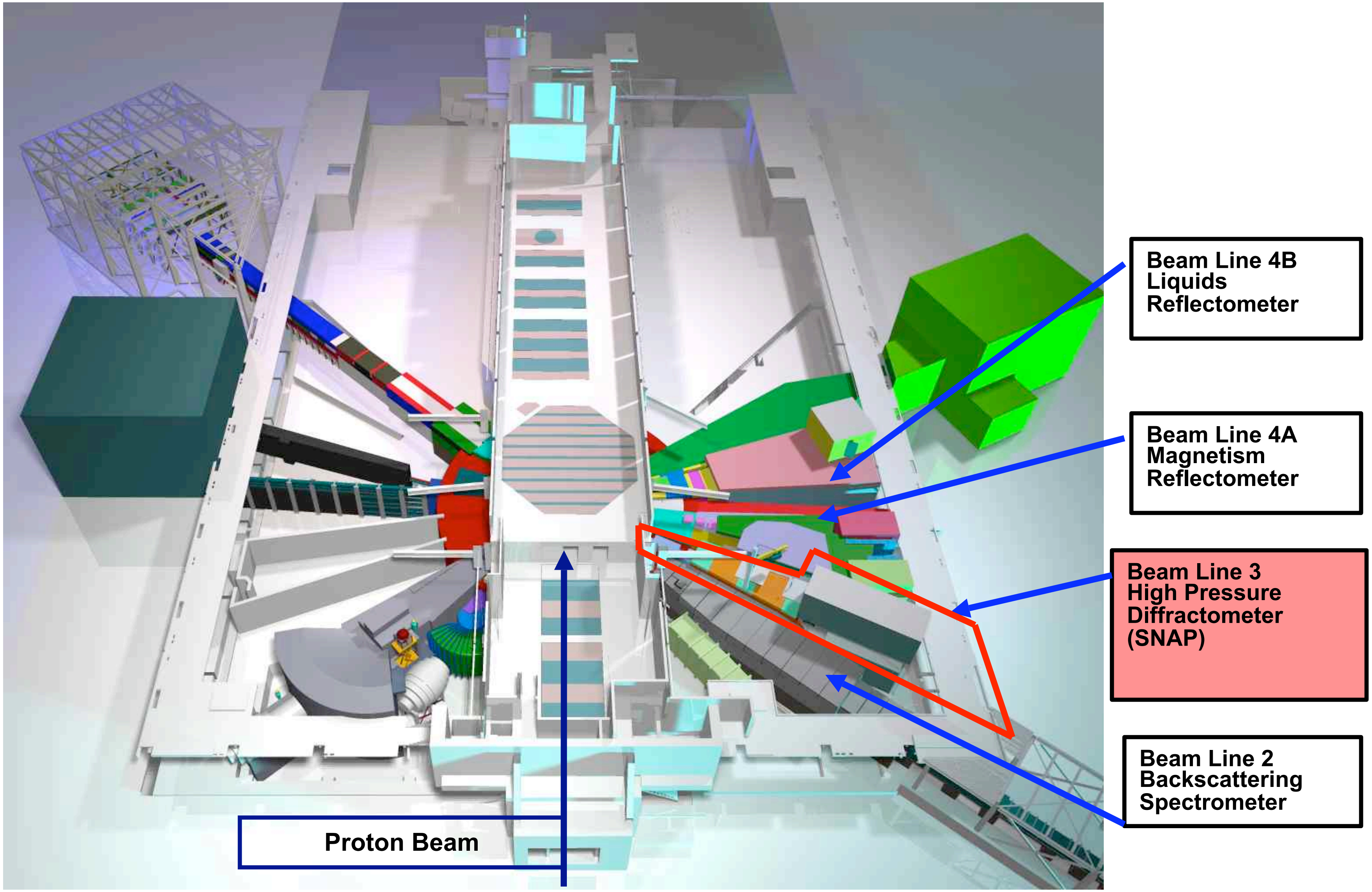
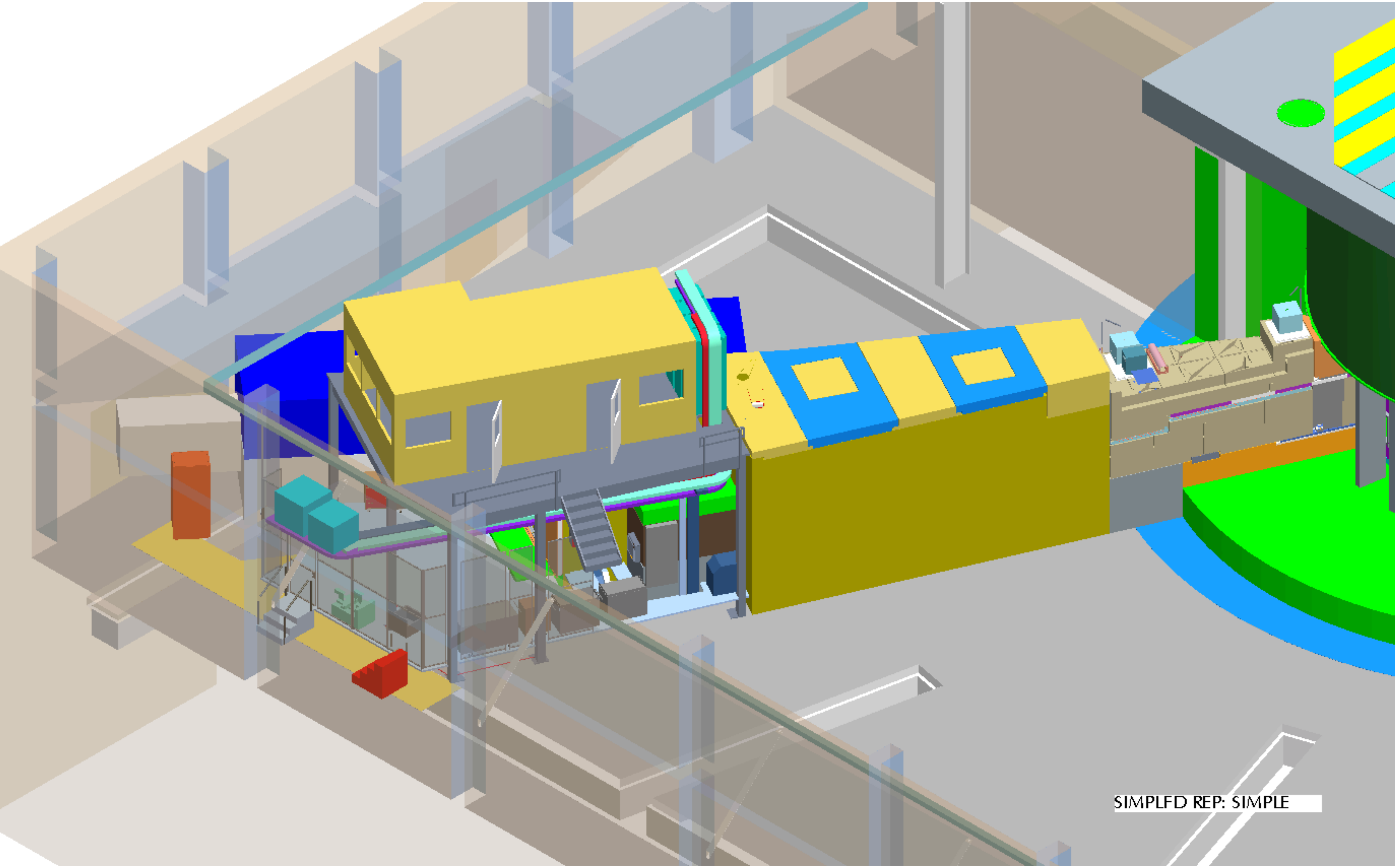
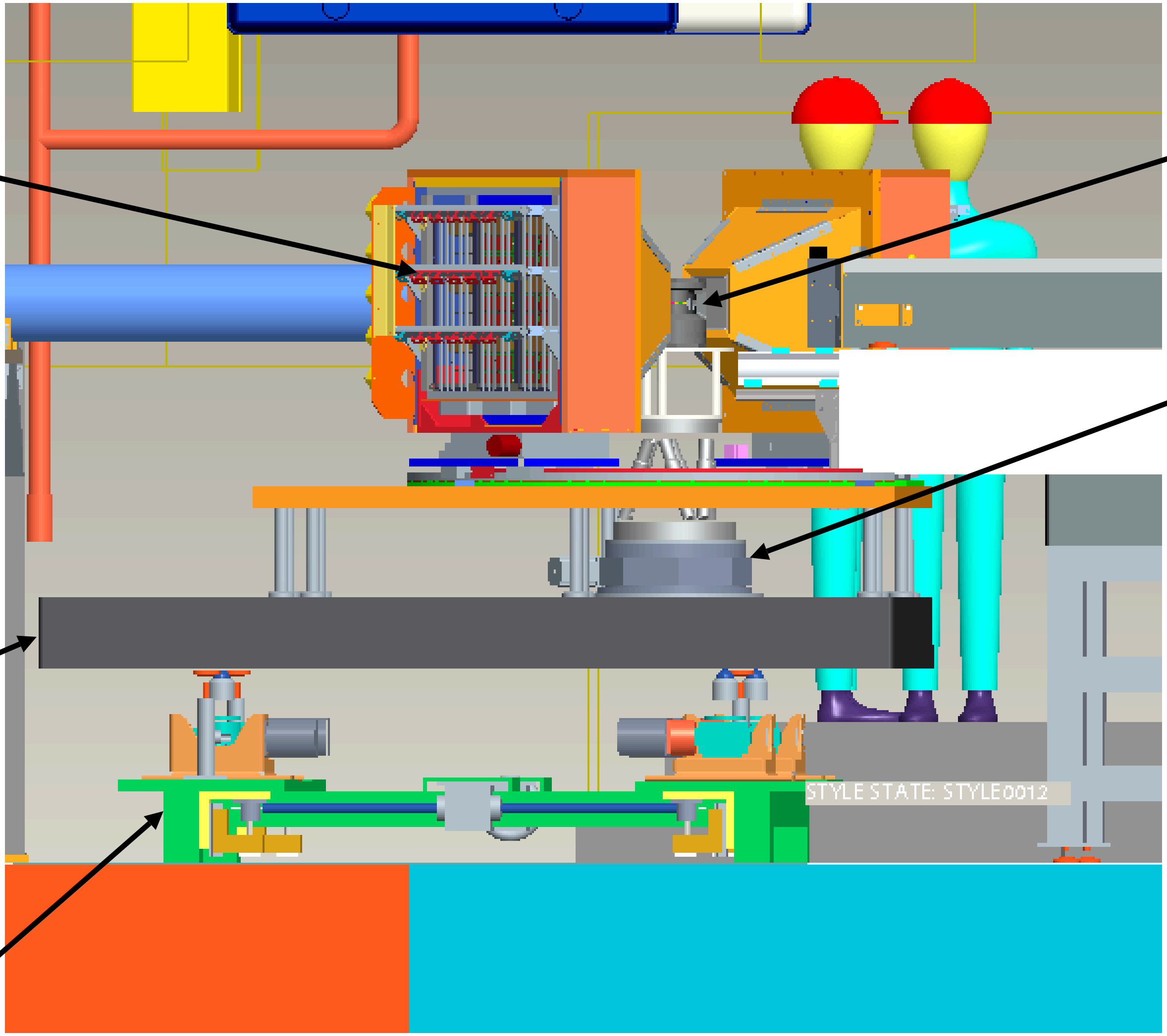
Target Building and Instrument Layout




SIMPLFD REP: SIMPLE

OVERVIEW (ELEVATION)



DETECTOR BANK

SAMPLE HOLDER

HEXAPOD

SAMPLE TABLE

TABLE LEVEL AND LIFT MECHANISM

SNAP on schedule to take beam in 2008

Bright future for HP neutrons

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- Unique properties of neutron plus:
  - Potentially revolutionary developments in
    - Sources - ISIS 2nd target, SNS, ILL upgrade
    - Detectors - esp ILL
    - Focusing - ORNL
    - HP cells - PE group, Saclay

↓

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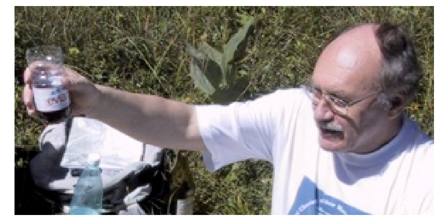
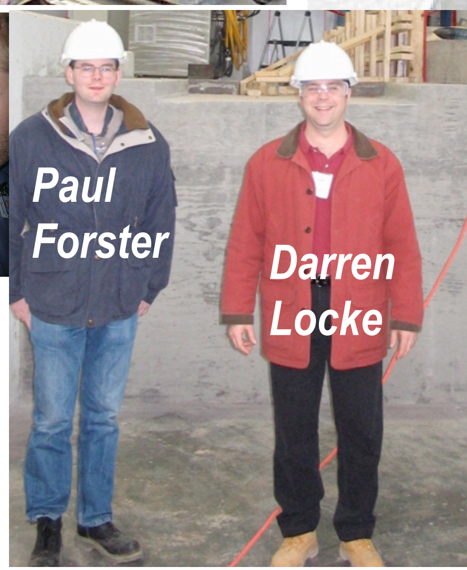
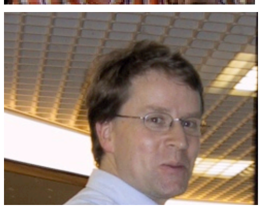
↓

- A bright future especially in complementary studies of
  - Structure
    - light elements
    - Isotope derived partials (glasses and melts)
    - Magnetic materials
  - Those momentum transfers not accessible by inelastic X-ray scattering

Acknowledgements







SNAP-IDT

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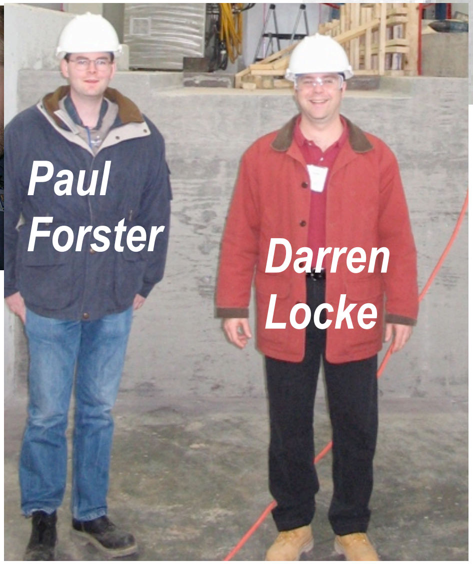

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