

Powder Diffraction Applications

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Spallation Neutron Source

National School on Neutron and X-ray Scattering

June 23, 2010

- ❑ Phase ID and Quantitative analysis
- ❑ Structure and transport
- ❑ Neutron Powder Diffraction
- ❑ Combine X-rays and Neutrons
- ❑ Time resolved in-situ studies
- ❑ Ab-initio structure solution
- ❑ Proteins and Powder Diffraction
- ❑ Powder Diffraction at ORNL

Archeology and Powder Diffraction

Phase ID: "Finger Printing"

Huq et.al. Appl. Phys. A 83, 253 (2006)



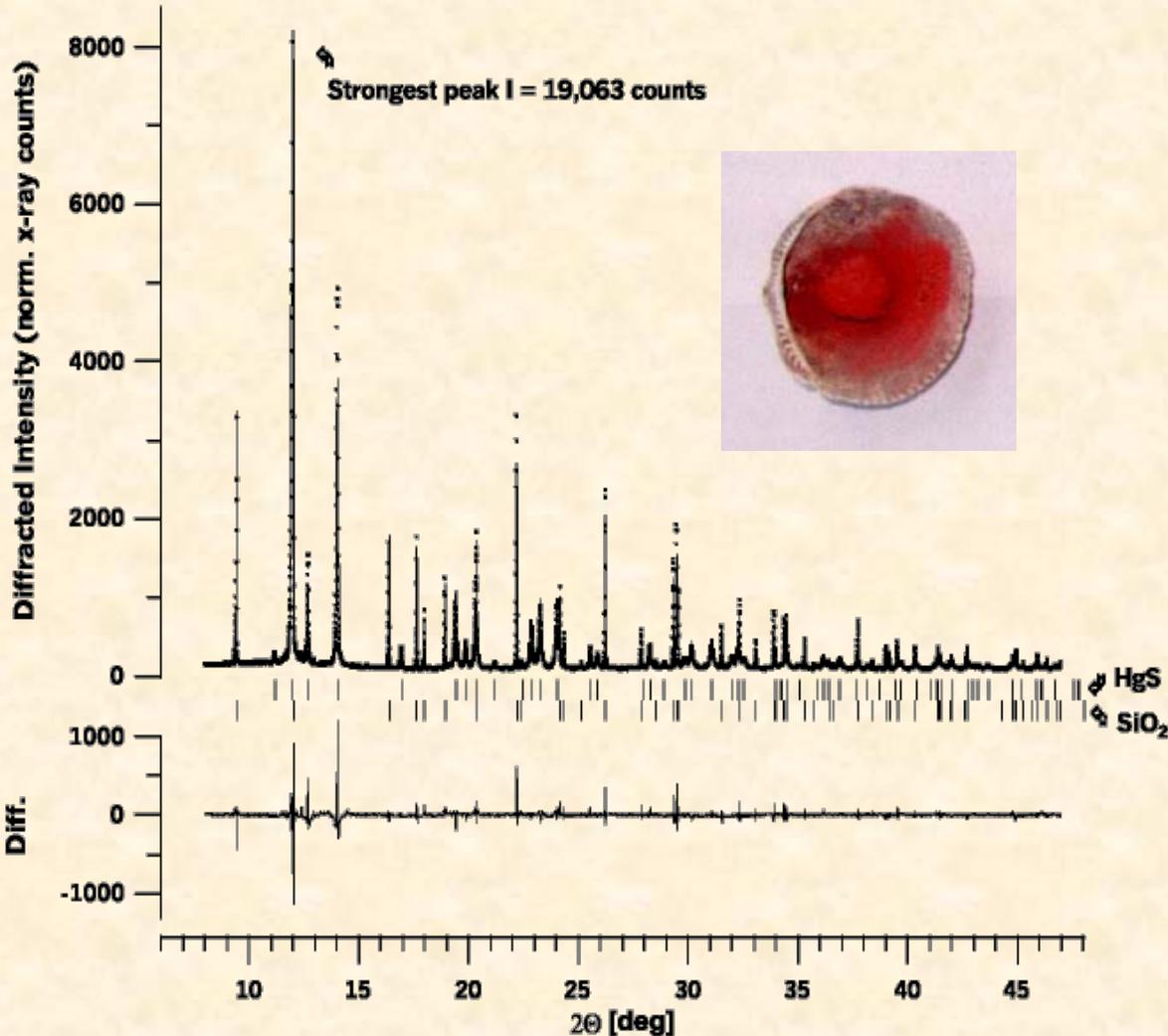
Natural antique colorants include red pigments such as cinnabar and ochre and pink pigments such as madder. These archaeological pigments have been used as ritual and cosmetic make-up and they are a material proof of handcraft activities and trade in the Mediterranean.

The pigments were discovered during different excavations in archaeological sites of Tunisia (Carthage, Kerkouane, Bekalta, Bouaarada and elsewhere).



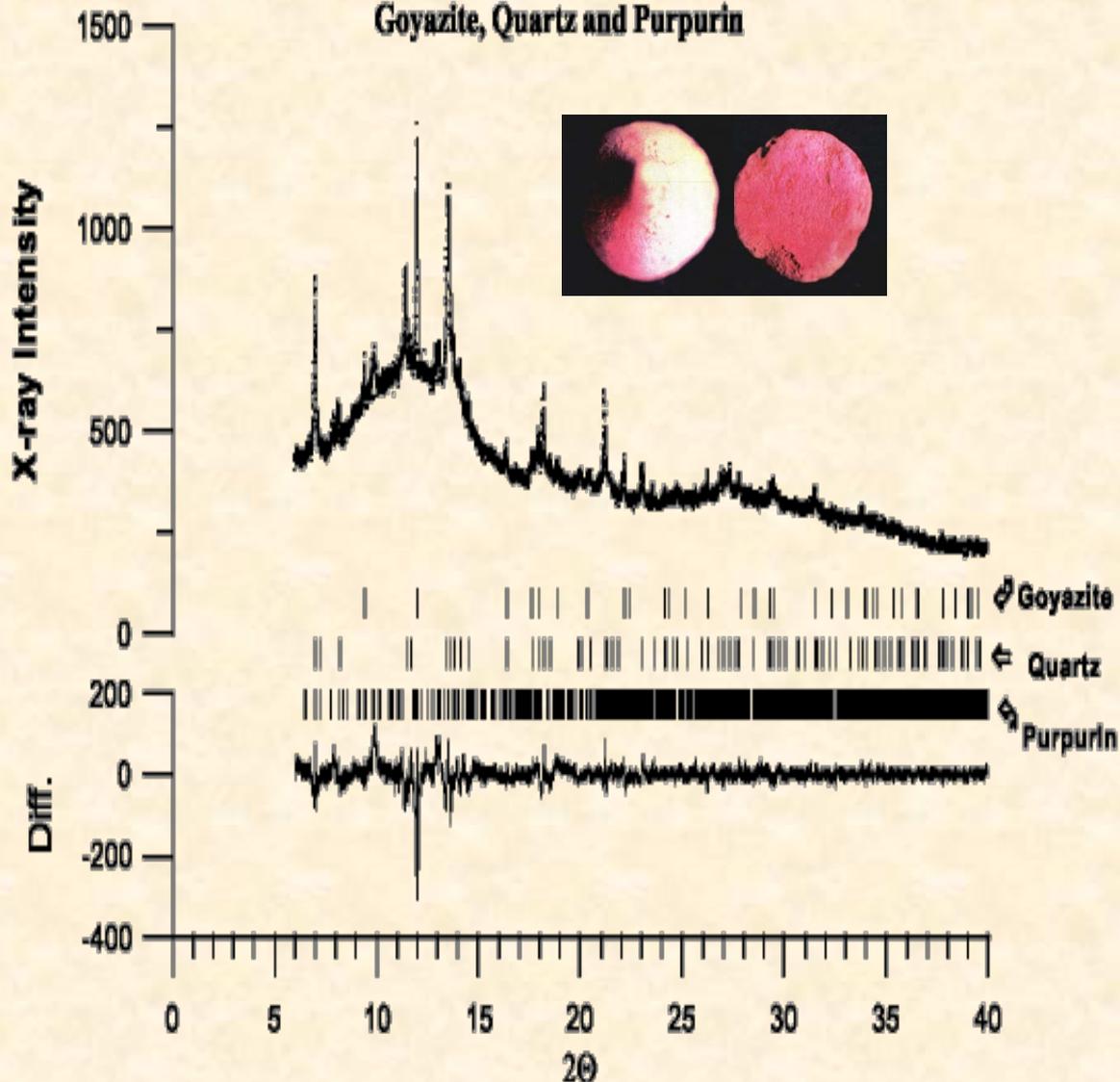
OAK RIDGE NATIONAL KERKOUANE ♥
U. S. DEPARTMENT OF ENERGY

Sample : FCC5
Cinnabar and Quartz



- ❖ fit peak: search database for matches.
- ❖ Look up structure.
- ❖ Rietveld refinement.
- ❖ For mixture quantitative phase analysis.

Sample : C41C
Goyazite, Quartz and Purpurin



Conclusions

Ten punic make-up samples were studied with SR-XRD using a 2D CCD detector and high angular resolution powder diffraction. Four samples (B1, B2, B3 and FCC5) contain quartz and cinnabar while four other samples (B10, FCC4, FCC6 and OCRB) contain quartz and hematite. The presence of quartz is probably due to sand/clay from the excavation area.

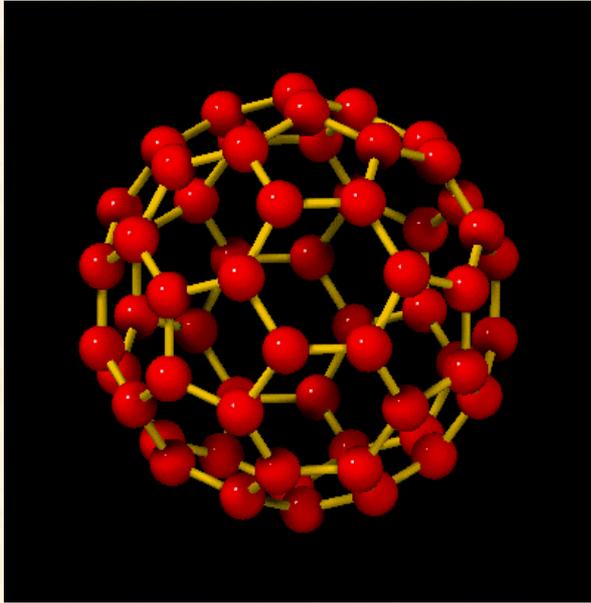
These results are similar to what would be obtained from raw materials indicating that these eight samples were not subject to any preparation by the Carthaginians. These eight samples were used as ritual make-up. However, the last two samples (FCC2 and C41C) showed an amorphous background, their preparation required sophisticated techniques corresponding to cosmetic make-up; they contain purpurin as major pigment which is formulated in a similar fashion as a lacquer.

Resources (databases)

- **Powder diffraction file, maintained by ICDD:** Release 2008 of the Powder Diffraction File contains 622,117 unique material data sets. Each data set contains diffraction, crystallographic and bibliographic data, as well as experimental, instrument and sampling conditions and select physical properties in a common standardized format. <http://www.icdd.com/products/overview.htm>
 - * **CCDC (Cambridge Crystallographic database):** organic structures
 - * **ICSD (Inorganic crystal structure database):** FIZ
 - * **NIST & MPDS**

Superconductivity in Fullerenes and Scientific Ethics! (Publishing in high profile journal)

Buckminsterfullerene

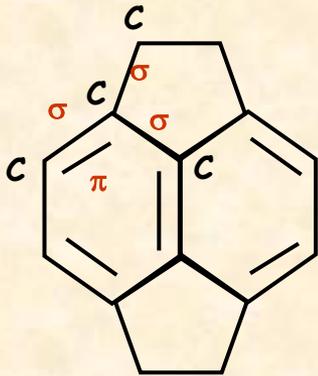


1985 : R.F. Curt, H.W. Croto & R.E. Smalley discover C_{60} . They are awarded the Nobel prize in Chemistry in 1996.

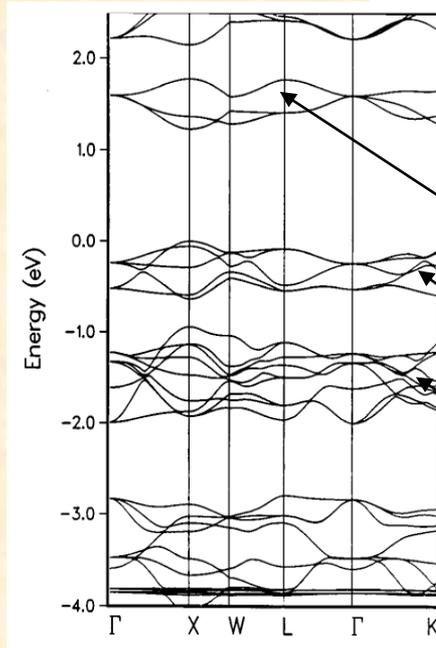
1990 : W. Kratchmer and D.R. Huffman produces isolable quantities of C_{60} .

1991 : A group at AT&T Bell labs, finds superconductivity in alkali doped C_{60} with $T_c=18K$ for K_3C_{60} . Later $T_c=28K$ is observed for Rb_3C_{60}

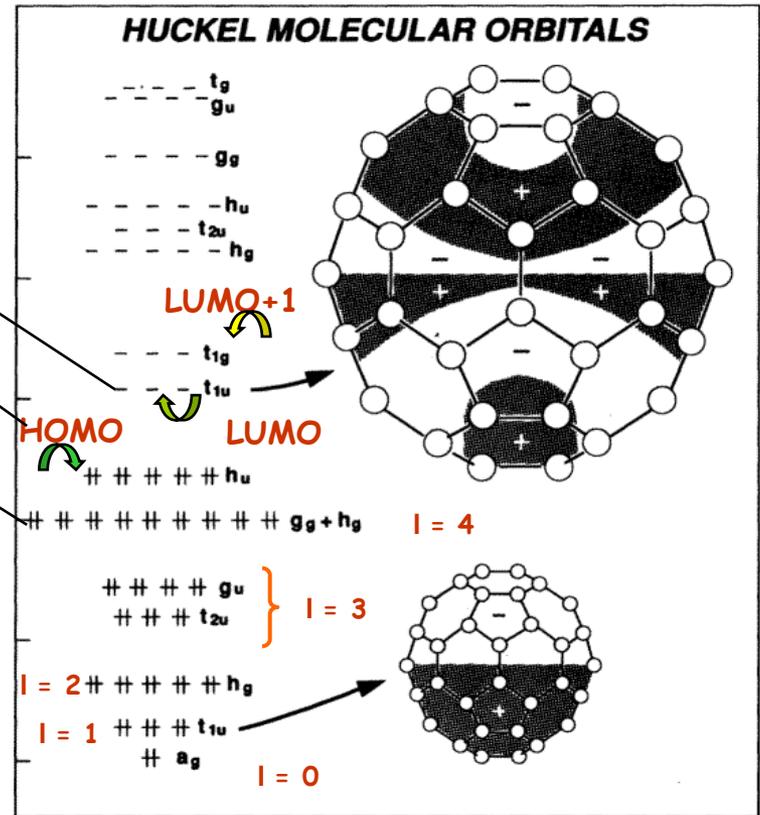
Diameter of molecule 10\AA . The atoms are positioned at the 60 vertices of a truncated icosahedron. 90 edges, 12 pentagons, 20 hexagons.



60 electrons
that take
part in
conduction



sub bands around
 E_F of solid C_{60} .
(Erwin 1993)



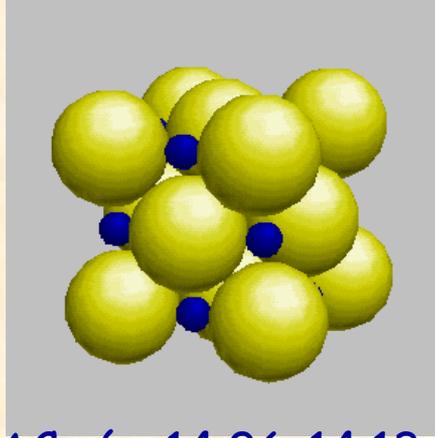
$n = 2(2l + 1)$
works
up to $l = 4$

HOMO - LUMO
~2eV

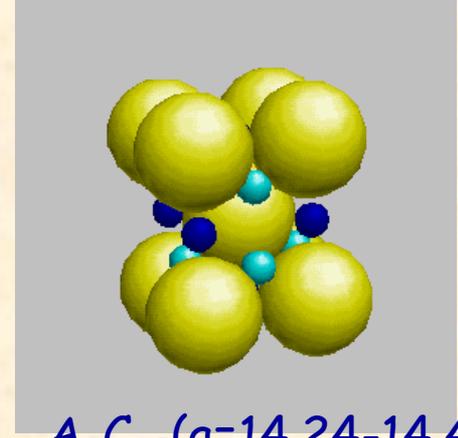
Alkali(K,Rb,Cs) doped C_{60}



fcc C_{60} ($a=14.17\text{\AA}$)



AC_{60} ($a=14.06-14.13\text{\AA}$)
A in octahedral site

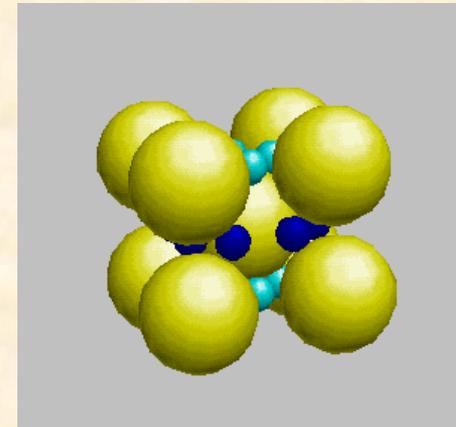


A_3C_{60} ($a=14.24-14.44$)
A in both tetrahedral
And octahedral site



A_4C_{60} bct (Cs_4C_{60} orthorhombic
& orientationally ordered.)

2/3 filling
but a band
insulator !



A_6C_{60} bcc ($a=11.39-11.84\text{\AA}$)

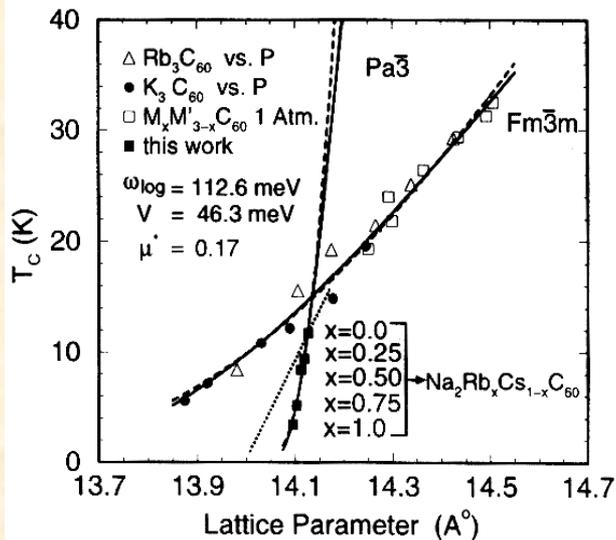
C₆₀ base Superconductors:

Changing the lattice parameter in Alkali doped fullerides, (either decreasing it with pressure or increasing it by substitution of a larger cation) increases the DOS $N(E_F)$.

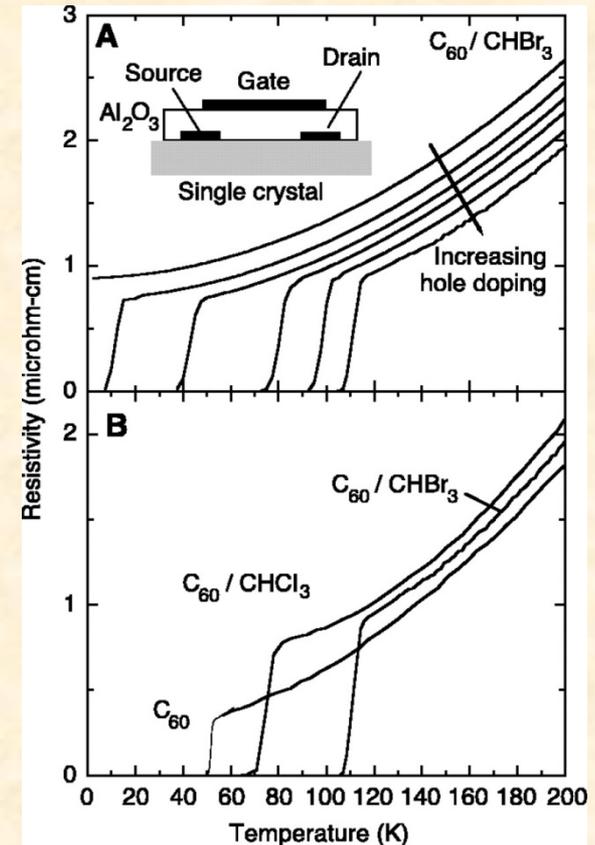
According to BCS theory

$$T_c = 1.13 \frac{\hbar \omega_{\log}}{k_B} \exp\left(\frac{-1}{N(E_F)V}\right)$$

Increase in $N(E_F)$ => Increase in T_c



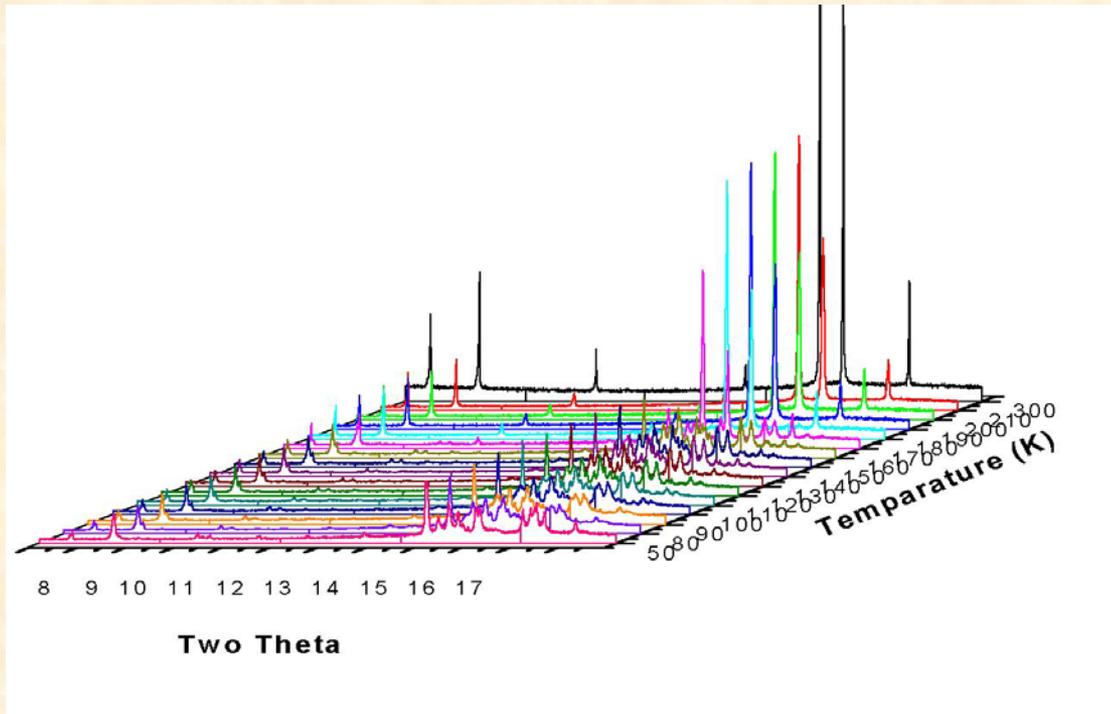
*Yildirim T et. al., 1995, Solid State Commun. 93 269-74



Interesting new superconductors, FET of organic materials (anthracene, pentacene, tetracene C₆₀. Record $T_c=117K$ for C₆₀ / CHBr₃. ($T_c=80K$ for C₆₀ / CHCl₃)

J.H. Schön, Ch. Kloc, B. Battlogg, *Science* 293, 2432-4 (2001).

What happens to the crystal structure as we decrease T?



Hexagonal

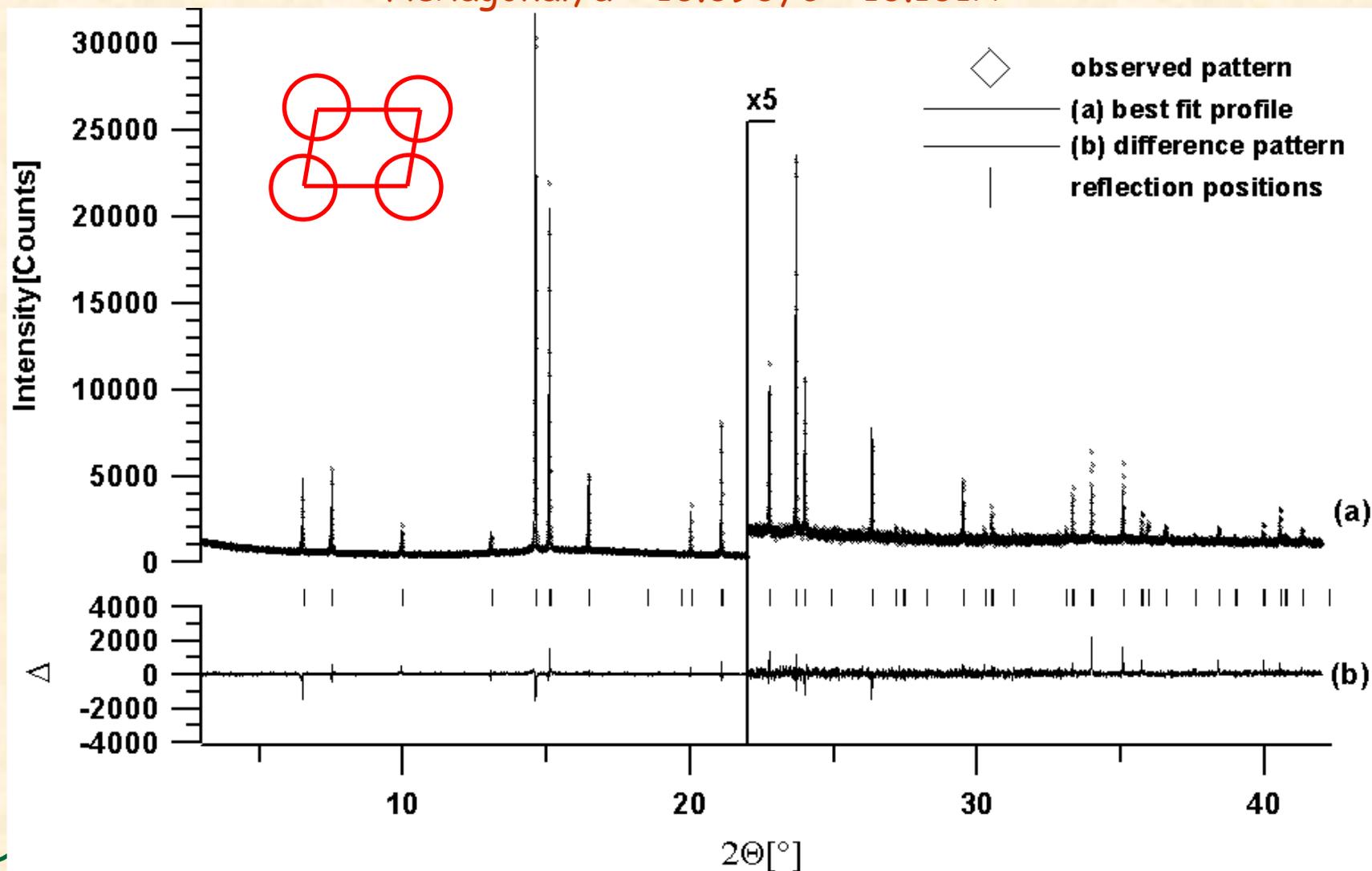
Monoclinic

Triclinic

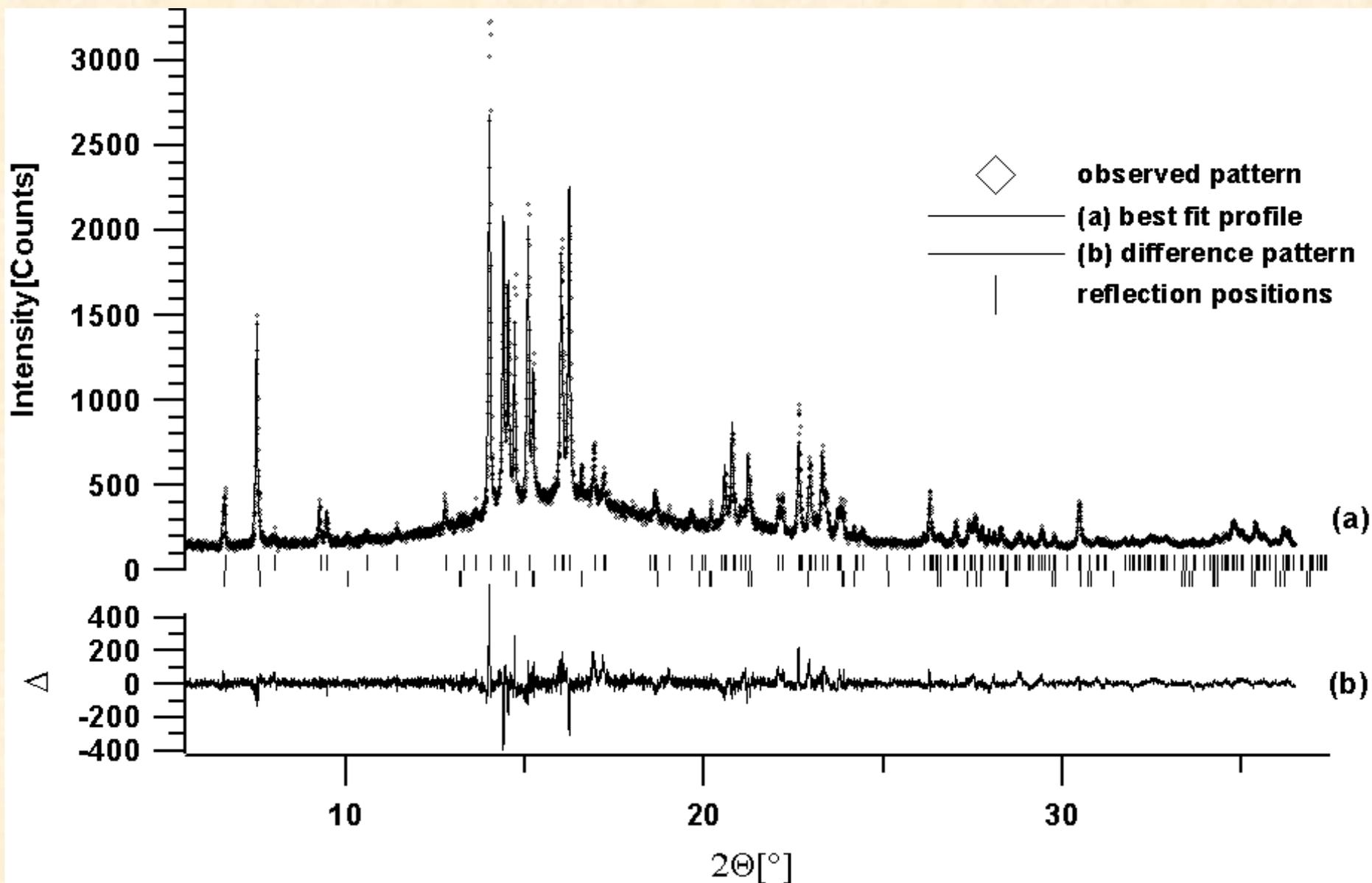
Heating-cooling cycles showed pronounced hysteresis and co-existence of the different phases over a large temperature range.

$C_{60} \cdot 2CHCl_3$ at room temp.

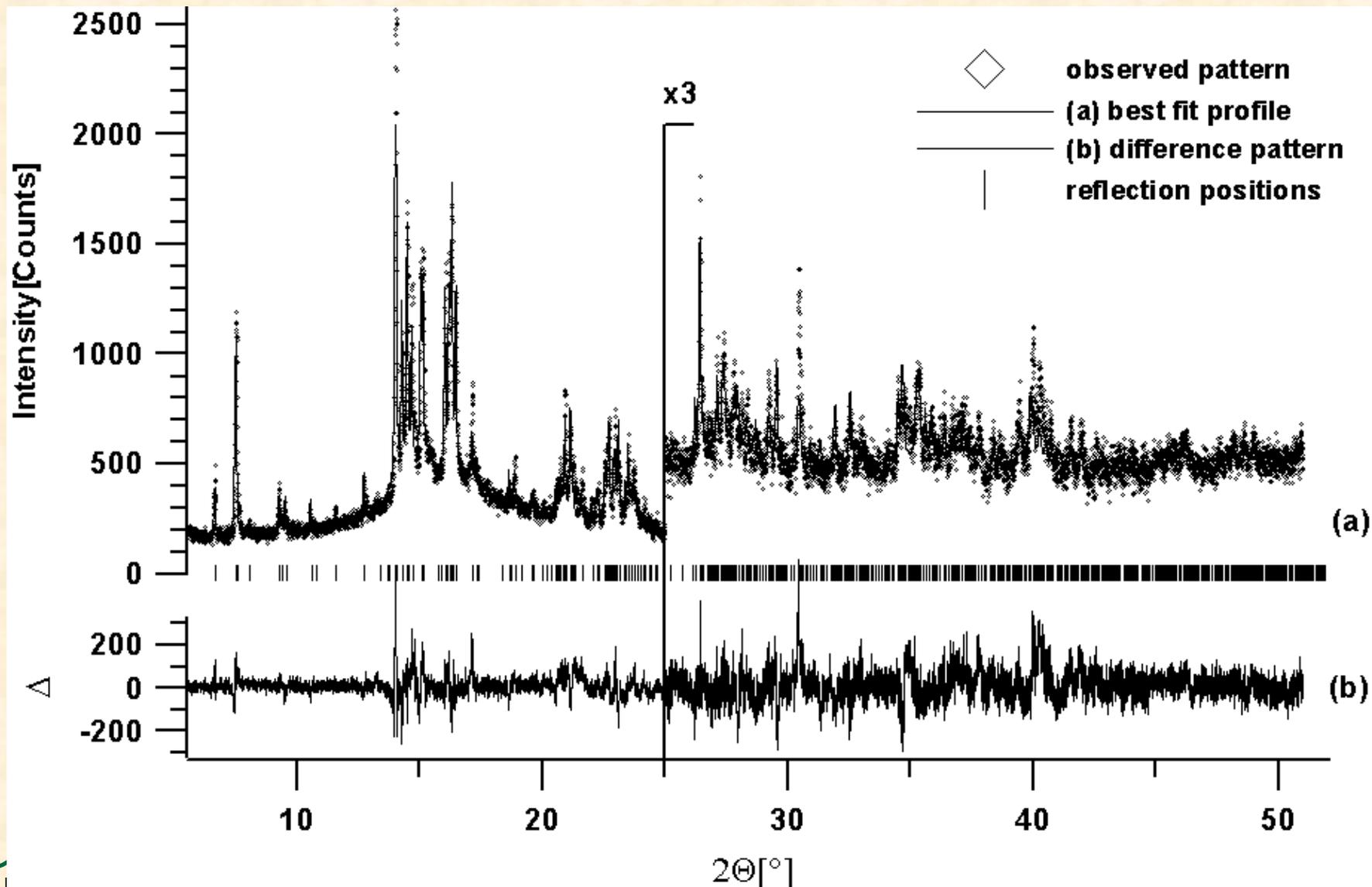
Hexagonal, $a = 10.096$, $c = 10.101 \text{ \AA}$



$C_{60} \cdot 2CHCl_3$ at 170K
monoclinic(16.821Å, 10.330Å, 10.159Å, 102.051°)



$C_{60} \cdot 2CHCl_3$ at 50K
(9.836Å, 10.091Å, 9.818Å, 101.36°, 116.46°, 79.78°)



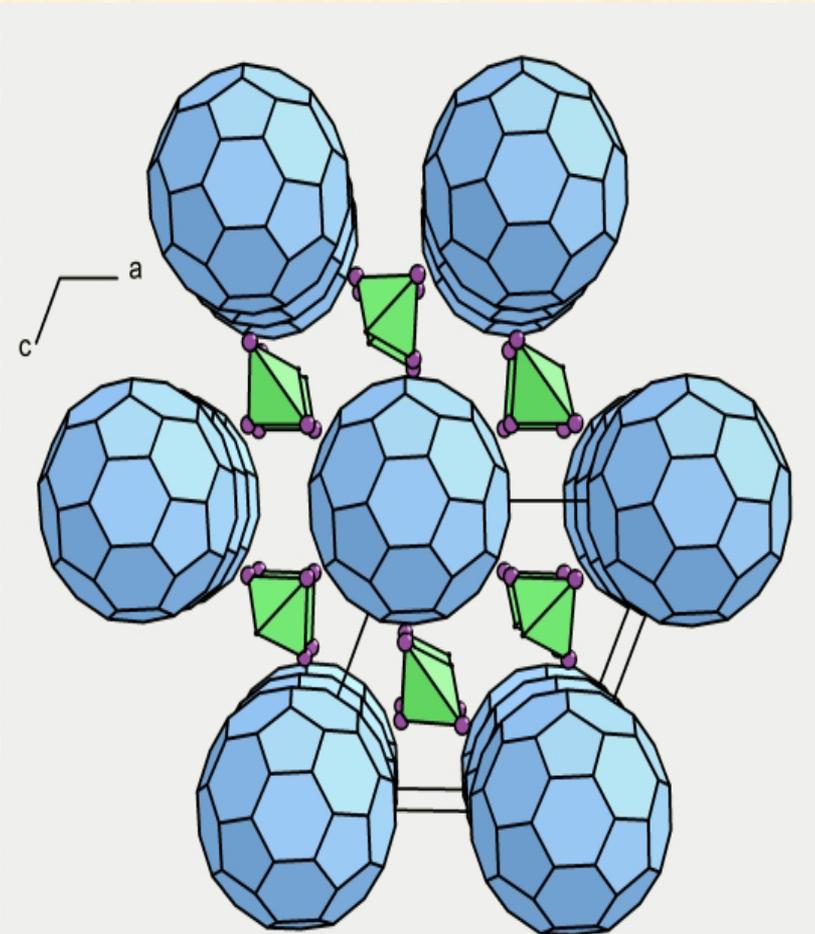
The crystal structure of C_{60} intercalated with $CHCl_3/CHBr_3$ is not fcc but hcp. More over when it is cooled it undergoes a phase transition and at $\sim 150K$ they are converted into a fully order triclinic phase.

	Sp Group	Lattice	T_c	d_{nn}
K_3C_{60}	Fm3m	14.24	18 (e-)	10.069
Rb_3C_{60}	Fm3m	14.44	28 (e-)	10.211
C_{60}	Fm3m, (Pa-3)	14.16, (14.04)	52	10.013
$C_{60} \cdot 2CHCl_3$	P 6/mmm	10.09, 10.095	80	10.09
$C_{60} \cdot 2CHBr_3$	P 6/mmm	10.211, 10.216	117	10.211

along	d_{nn} (Å)
001	9.8179
100	9.8361
010	10.091
101	10.348
011	12.6165
-110	12.781

$C_{60} \cdot 2CHCl_3$ (P-1)

Interfullerene distances



In plane:

9.82, 9.84, 10.35

Between plane: 10.09



In plane:

9.90, 9.90, 10.50

Between plane: 10.34

cf. C₆₀: 9.93 (5K)

K₃C₆₀: 10.07

Conclude: Strong increase of T_c from intercalations is not just an effect of simple lattice expansion.

Evidence against lattice expansion as the sole explanation for T_c increase in chloroform- and bromoform-doped C_{60}

R. E. Dinnebier¹, O. Gunnarsson¹, H. Brumm¹, E. Koch¹, A. Huq²,

P. W. Stephens², M. Jansen^{1,*}

Structure of Haloform Intercalated C_{60} and Its Influence on Superconductive Properties

Robert E. Dinnebier,¹ Olle Gunnarsson,¹ Holger Brumm,¹ Erik Koch,¹ Peter W. Stephens,² Ashfia Huq,² Martin Jansen^{1,*}

www.sciencemag.org SCIENCE VOL 296 5 APRIL 2002

Similar Graphs Raised Suspicion on Bell Labs Research

http://www.sciencemag.org/doi/10.1126/science.1100000

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 SEARCH ARCHIVES
 20 May 2002

Technology
 Similar Graphs Raised Suspicion on Bell Labs Research
 By KENNETH CHANG
 What had been hailed a few months ago as a molecule-size electronics is now in doubt, a Bell Laboratories is under suspicion of improperly manipulating data in research papers published in prestigious scientific journals.

The accusations, by scientists not connected with it, came to light this week, when Bell Labs appointed a panel to look into them. Yesterday, the scientists' concerns focused on graphs that were nearly identical they appeared in different scientific papers and had different device. In some graphs, even the noise should arise from purely random fluctuations match.

Science
 A Sudden Host of Questions on Bell Labs Breakthroughs
 By KENNETH CHANG
 On a ski slope in Utah in March, Paul Grant and Rick Greene made a bet — about superconductors.

Dr. Grant and Dr. Greene, who had been longtime colleagues at the I.B.M. Almaden Research Center in San Jose, Calif., had debated all day a sensational scientific report that molecules of carbon shaped like soccer balls had been turned into superconductors — materials that carry electricity with virtually no resistance — at surprisingly warm temperatures.

Dr. Grant doubts the findings. Dr. Greene said he thought that they would be verified.

Last week, Dr. Grant sent an e-mail message reminding Dr. Greene of the wager, because the lead researcher of the experiment was Dr. J. Hendrik Schön, the Bell Labs scientist who is now the center of a scientific misconduct investigation. Nearly identical graphs appear in several of Dr. Schön's scientific papers, even though the graphs represent different data from different experiments. Bell Labs, part of Lucent Technologies, has convened an independent panel to investigate.

But even before the two main papers cited in the investigation were published, a debate had arisen over the superconductor claims.

"There's been a lot of buzz for well over a year," said Dr. Grant, now a science fellow at the Electric Power Research Institute in Palo Alto, Calif.

Dr. Schön and his collaborators have developed a revolutionary technique that allows them to explore systematically the electronic properties of various materials. Dr. Grant had called the team's "touchy-feel" work paper "a tour de force of physics" when it was announced. Other scientists said it might be worthy of a Nobel Prize.

197A



In 2001 he was listed as an author on an average of one research paper every eight days!

On October 31, 2002, Science withdrew eight papers written by Schön. On December 20, 2002, the Physical Review journals withdrew six papers written by Schön. On March 5, 2003, Nature withdrew seven papers written by Schön.

BORATORY NERGY

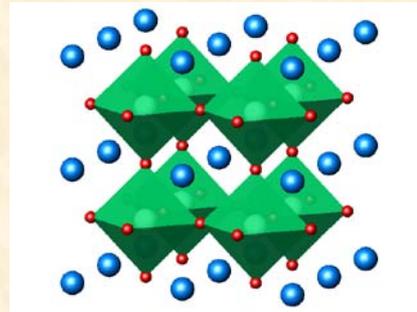


Neutron Powder Diffraction

Why Neutrons ?

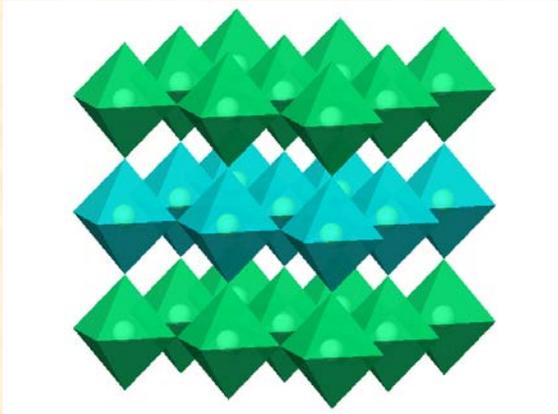
- Electrically neutral; penetrates centimeters of bulk material (allows non-destructive bulk analysis)
- Detects light atoms even in the presence of heavy atoms (organic crystallography) - H is special!
- Distinguishes atoms adjacent in Periodic table and even isotopes of the same element (changing scattering picture without changing chemistry)
- Magnetic moment (magnetic structure)
- Ease of *in-situ* experiments, e.g. variable temperature, pressure, magnetic field, chemical reaction etc.

Ba₂CuWO₆: An Ordered Tetragonal Perovskite

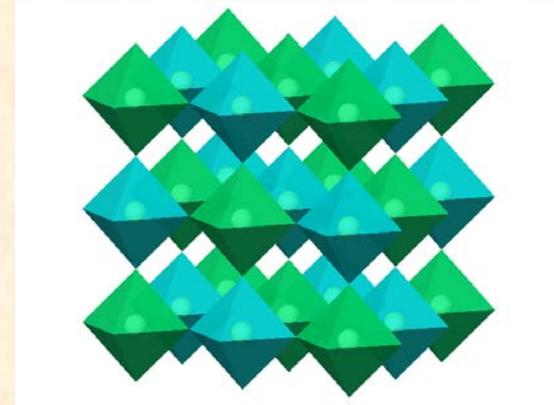


Simple cubic AMX₃
perovskite: $a = 3.8045$.

Double Perovskites A₂MM'O₆: Out of 3 possible ordering only 2 observed



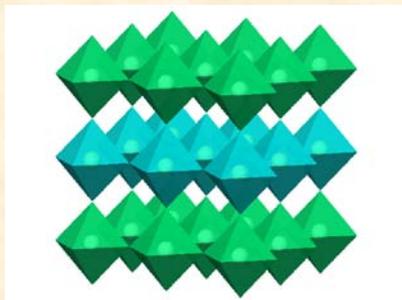
Model #1: Ordered alternation of MO₆ and M'O₆ octahedra in one direction, leading to formation of layered perovskite.



Model #2: Ordered alternation in the three directions of space, resulting in rock-salt ordered superstructure.

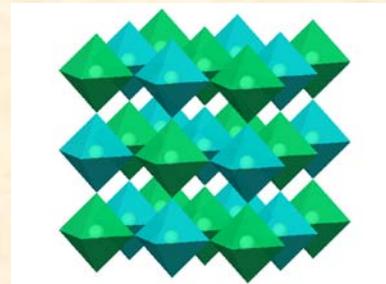
Model #1 – Layered Ordering:

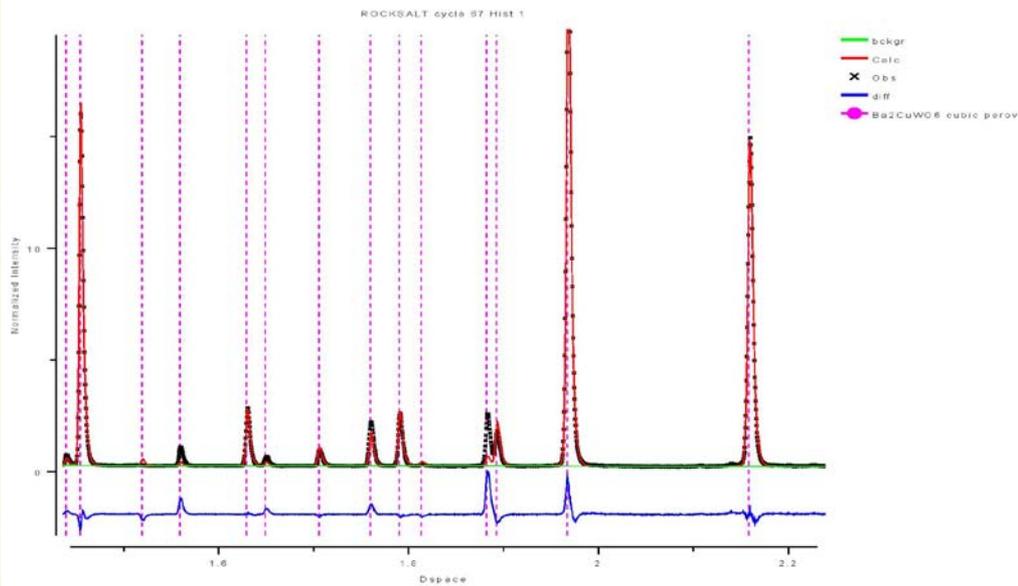
<u>Space Group</u>	<i>P4/mmm</i>			
<u>Lattice</u>	$a = 3.94 \text{ \AA}; c = 8.64 \text{ \AA}$			
<u>Atom</u>	<u>x</u>	<u>y</u>	<u>z</u>	<u>Occupancy</u>
Ba	1/4	1/4	1/2	1
Cu	0	0	0	1
W	0	0	0	1
O(1)	0	0	1/4	1
O(2)	1/2	0	0	1
O(3)	1/2	0	1/2	1



Model #2 – Rock Salt Type Ordering:

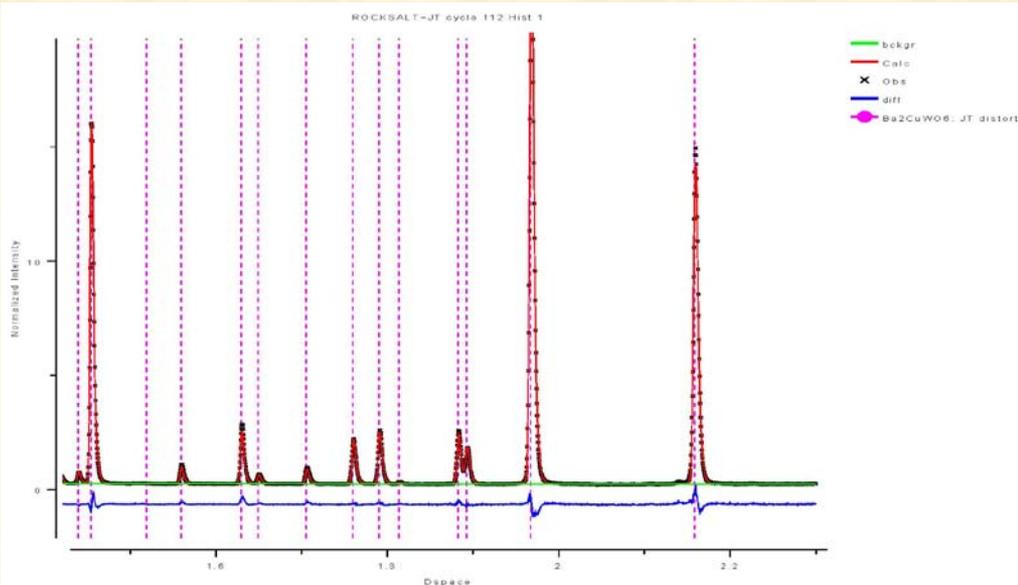
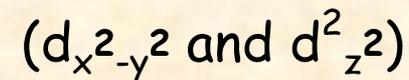
<u>Space Group</u>	<i>I4/m</i>			
<u>Lattice</u>	$a = 5.57 \text{ \AA}; c = 8.64 \text{ \AA}$			
<u>Atom</u>	<u>x</u>	<u>y</u>	<u>z</u>	<u>Occupancy</u>
Ba	0	1/2	1/4	1
Cu	0	0	0	1
W	0	0	0	1
O(1)	0	0	0.25	1
O(2)	0.25	0.25	0	1





Recall Cu^{2+} electronic configuration $(t_{2g})^6(e_g)^3$: Jahn Teller Distortion?

So in fact CuO_6 octahedra are elongated along the c axis. The e_g orbital is split into

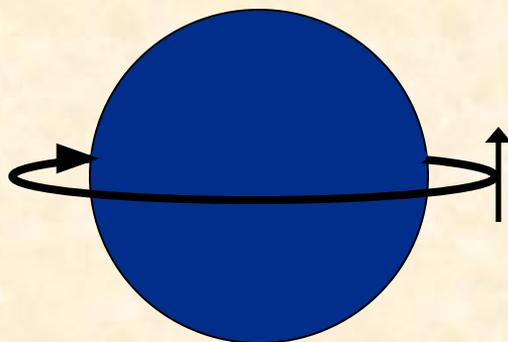


Iwanaga et. al. J. Solid State. Chem. 147, 291(1999)

Magnetism & powder diffraction

Introduction to Magnetism

- Origin of magnetism - electrons.
 - Electrons have a magnetic moment (dipole; μ_S).
Magnetic moments arise from two properties of an electron:
 1. Motion around the nucleus (gyromagnetic ratio)
 2. Total spin quantum number ($S = \Sigma s$; $s = \pm\frac{1}{2}$)



- Dipole unit - Bohr magnetons (μ_B). $1 \mu_B = 9.2742 \times 10^{-24} \text{ J/T}$

- Ions with magnetic properties have unpaired electrons. Materials that contain magnetic ions have magnetic properties.
- Examples - Cu^{2+} and low spin Co^{3+} in an octahedral ligand field:



Cu^{2+}

d^9 ion

$S = 1/2$

$\mu = 1.9\text{-}2.1 \mu_B$

Paramagnetic ion

Co^{3+} (low spin)

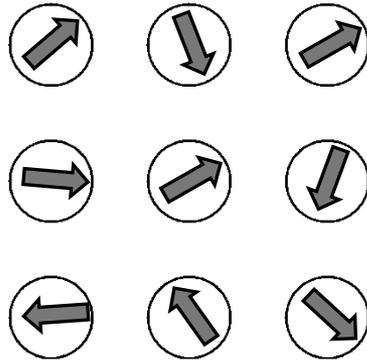
d^6 ion

$S = 0$

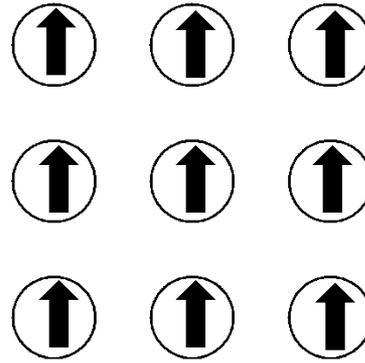
No magnetic moment

Diamagnetic ion

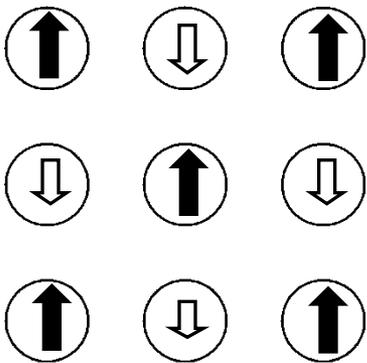
Magnetic Ordering Types



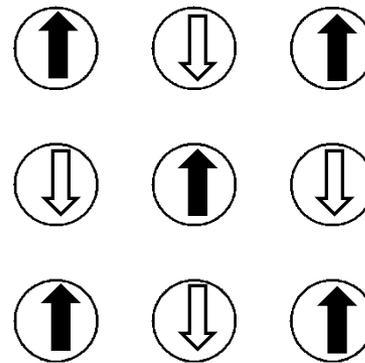
Paramagnetic (PM)



Ferromagnetic (FM)



Ferrimagnetic

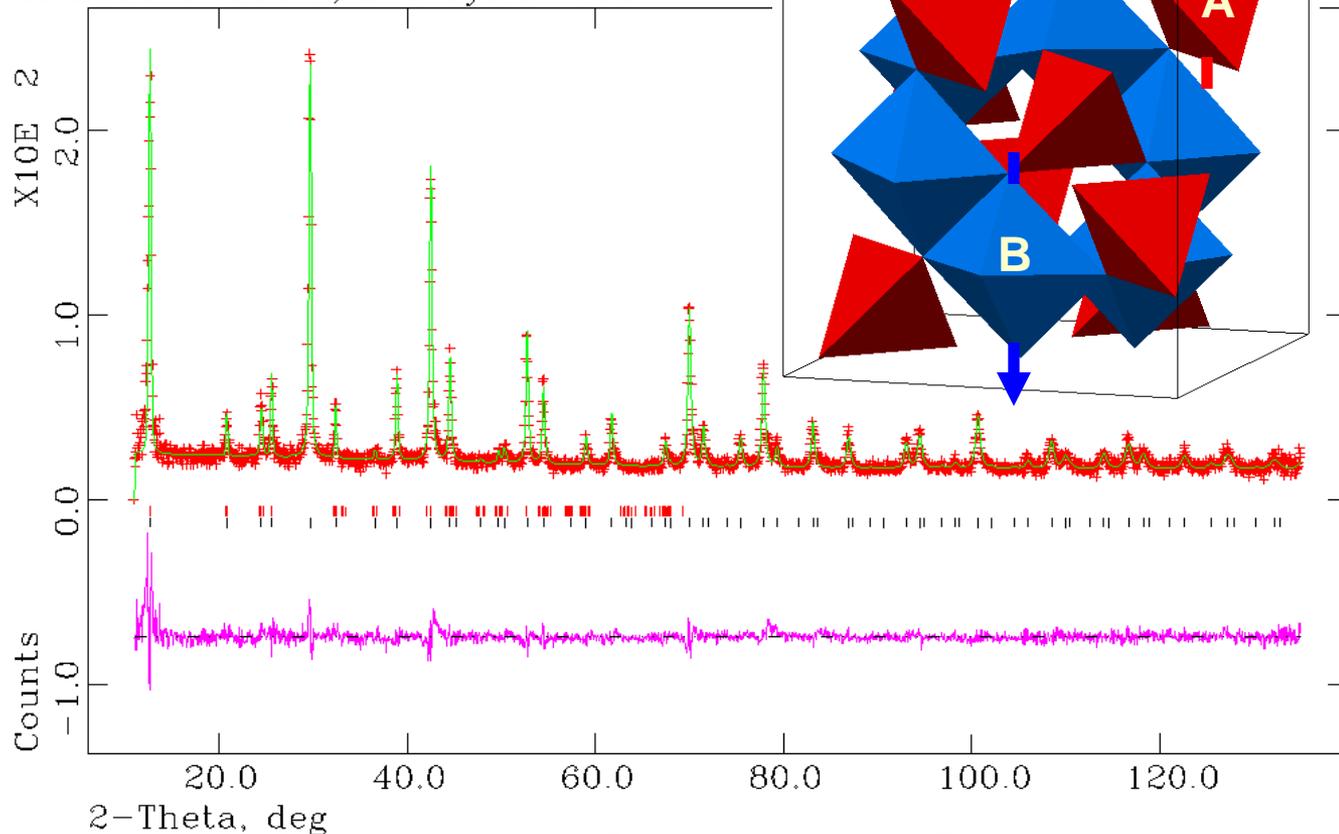


Antiferromagnetic (AFM)

FERRIMAGNETIC AB₂O₄ SPINEL STRUCTURE



Lambda 1.0910 Å, L-S cycle 202



2-phase refinement
nuclear + magnetic
structure

Results give

- lattice parameter
- oxygen position
- distribution of Mn/Fe on T and O sites
- atomic displacement parameters
- magnetic moments on the T and O sites (e.g., -2.9 and 2.0 μ_B)

nuclear
phase

magnetic
phase

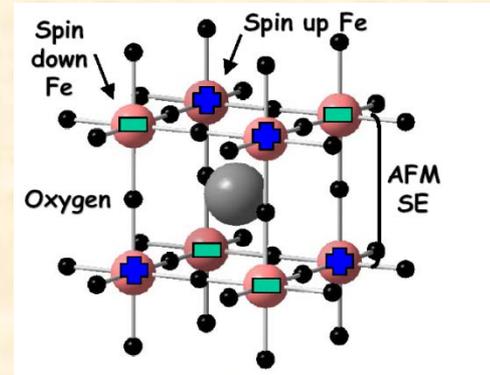
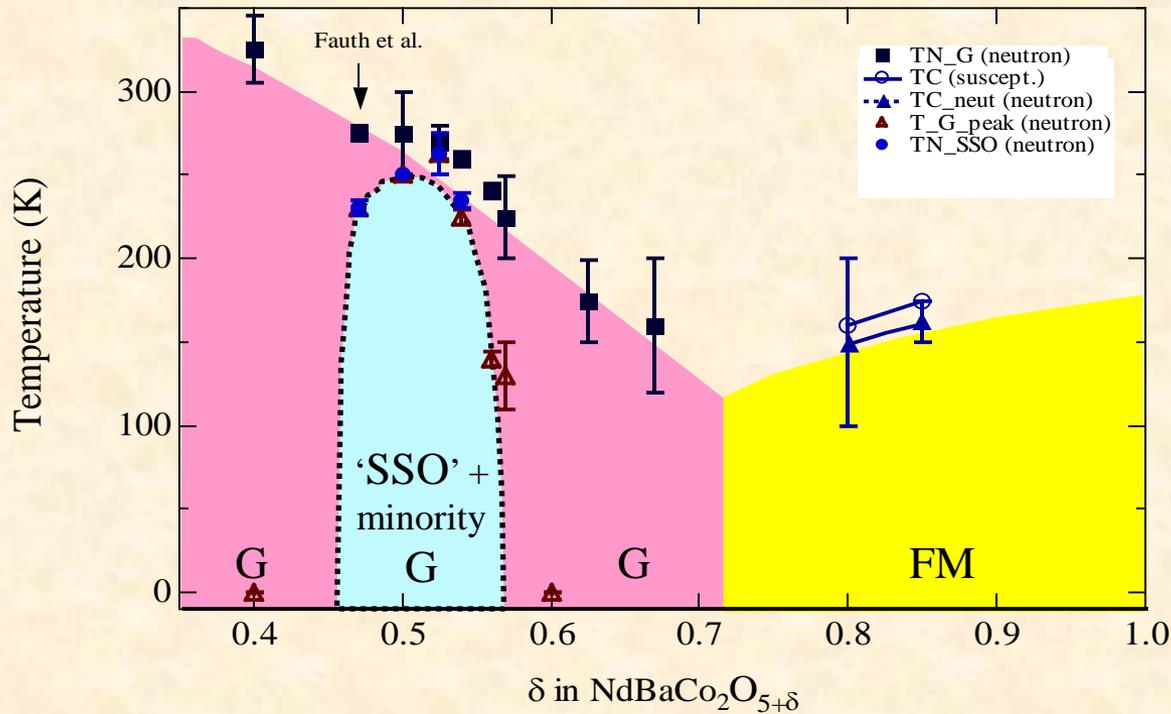
Zhang ZJ et. al. JACS, 120 1800 (1998)

name	type	ref/damp	fractional coordinates			Mlt*	Occupancy	Uiso
5 O	O	X0 U0 0	0.261035	0.261035	0.261035	32	1.0000	0.01250
3 B	MN	0 U0 F0	0.500000	0.500000	0.500000	16	0.1978	0.00788
4 B	FE	0 U0 F0	0.500000	0.500000	0.500000	16	0.8022	0.00788
1 A	MN	0 U0 F0	0.125000	0.125000	0.125000	8	0.8101	0.01850
2 A	FE	0 U0 F0	0.125000	0.125000	0.125000	8	0.1899	0.01850

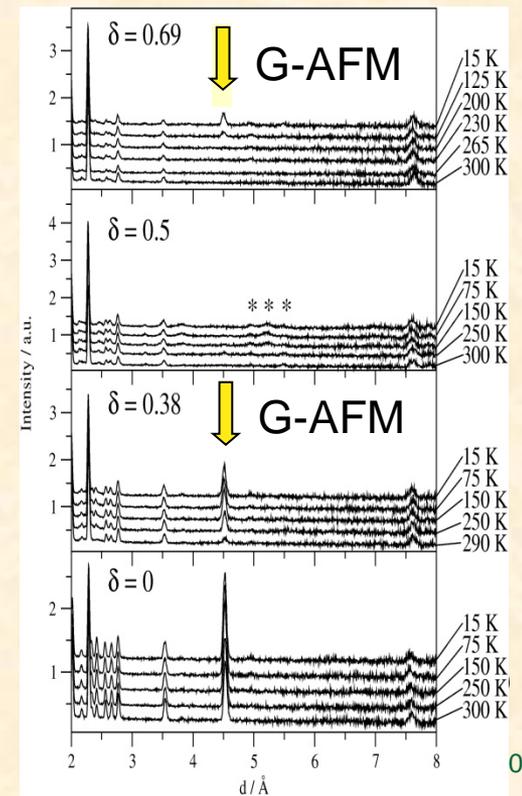
name	type	ref/damp	fractional coordinates			Mlt*	Occupancy	Uiso
2 B	FE	0 0 0	0.500000	0.500000	0.500000	16	1.0000	0.00700
1 A	MN	0 0 0	0.125000	0.125000	0.125000	8	1.0000	0.01800

Magnetic Ordering: Oxygen-deficient A-site Layered Perovskite $\text{NdBaCo}_2\text{O}_{5+\delta}$

Burley et. al. *J. Solid State. Chem.* 170, 339 (2003)



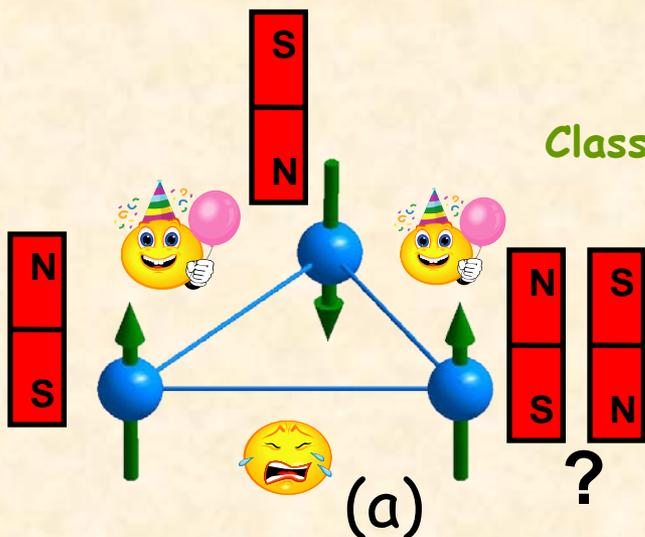
G type AF ordering in a simple perovskite



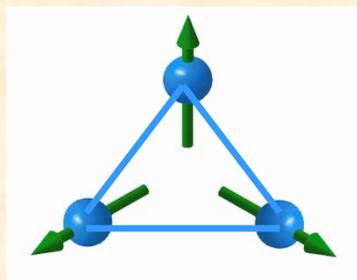
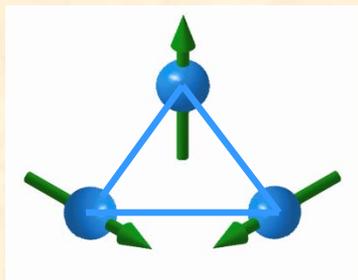
Very often life is not so simple and one has to use both X-rays and Neutrons to get to the right picture

Frustrated Lattice:

Classic example of geometric frustration (Triangular lattice)



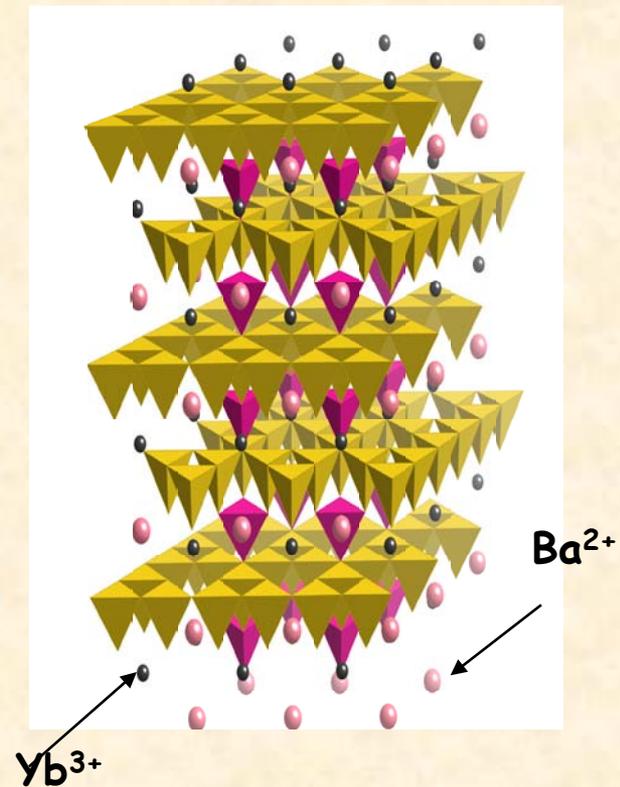
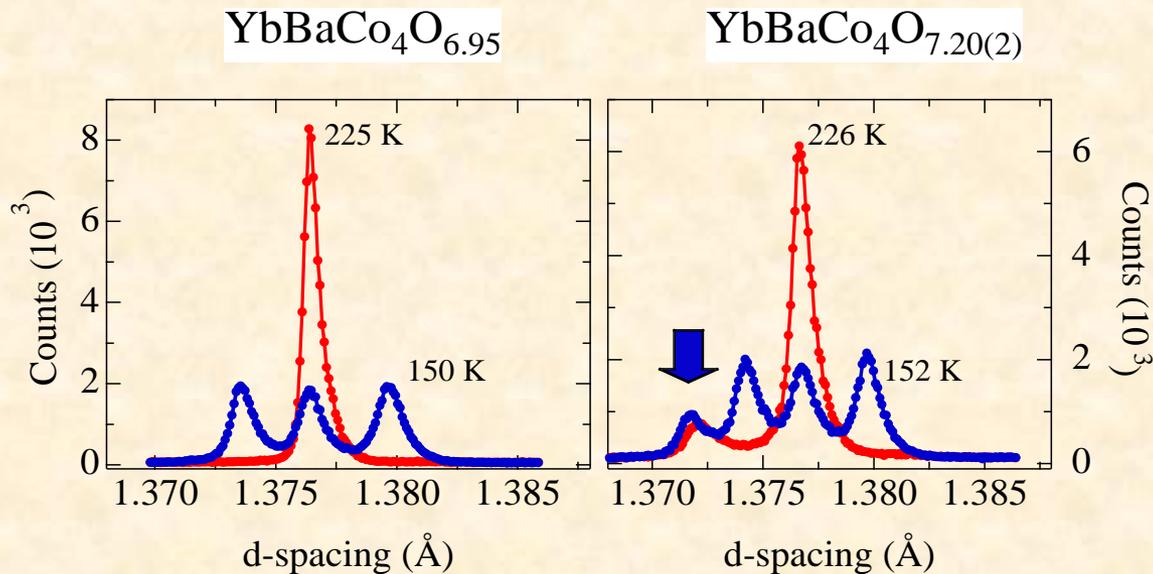
(a) Frustration for three equidistant Ising spins coupled via Antiferromagnetic exchange.



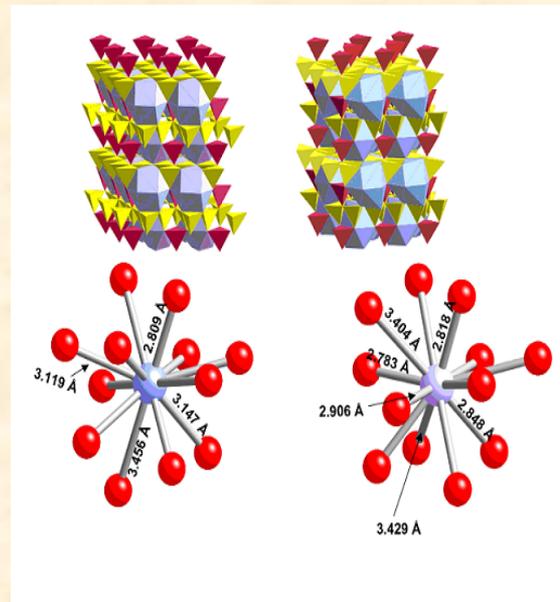
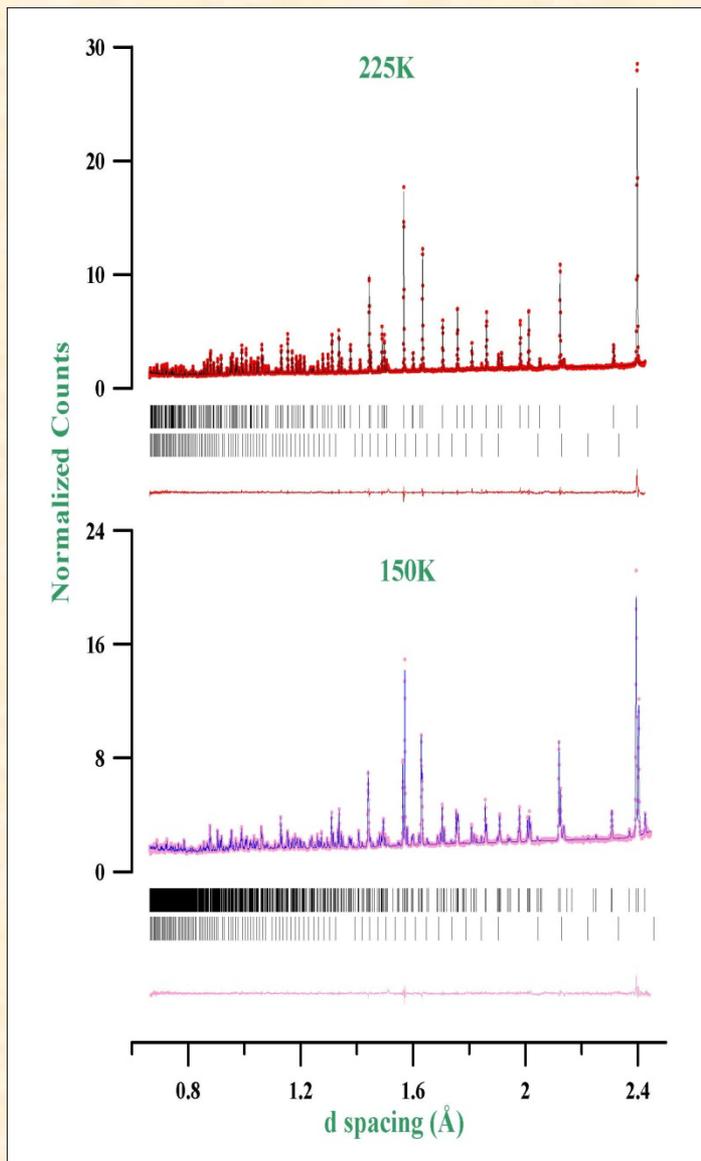
(b,c) For Heisenberg spins with a given spin orientation of the upper atom, two degenerate arrangements with +ve and -ve chirality are possible.

Mechanisms lifting the degeneracy often determine the phase diagrams and other physical properties of these systems.

$\text{RBaCo}_4\text{O}_{7\pm\delta}$: Phase transition



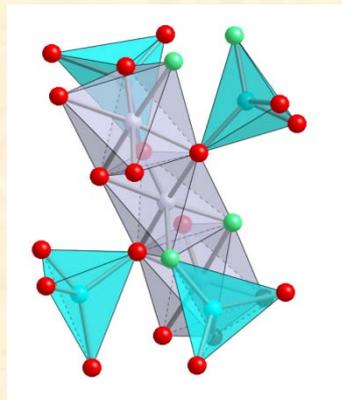
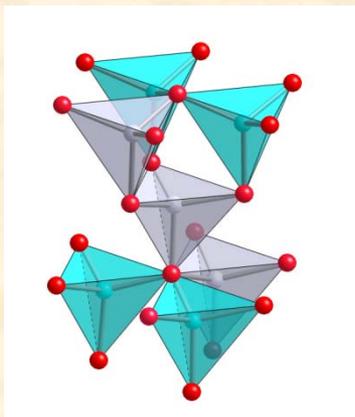
Comparison of synchrotron data collected above and below structural phase transition in a single phase $\text{YbBaCo}_4\text{O}_{6.95}$ and biphasic $\text{YbBaCo}_4\text{O}_{7.2}$ samples. Biphasic phase is formed in the presence of excess oxygen. Note that the minority phase does not transform upon cooling down to 10K. It is suspected that the excess O may enhance Ba-O bonding to the extent that the $P 3 1c$ to $P b n 2_1$ transition is no longer favorable.



Above the transition temperature Ba^{2+} is severely under bonded, giving rise to a structural instability. Bond Valence Calculation show convincingly that the O environment does not satisfy the bonding requirements of the Ba^{2+} ion. To compensate for this under bonding Yb 114 undergoes a structural phase transition. No compelling structural evidence of Co charge ordering.

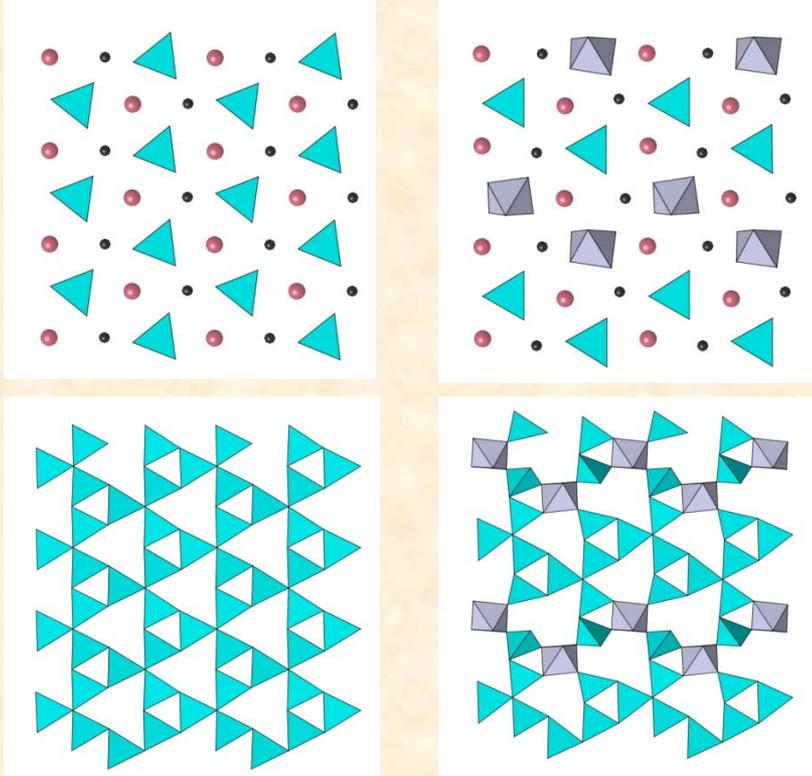
When R = Y and x=1 (YBaCo₄O₈)

We find that a large orthorhombic $Pbc2_1$ superstructure is formed with lattice parameters $2a_0 \times b_0 \times c_0$. Charge ordered Co ions form long range tetrahedral and octahedral zigzag patterns or chains of edge-sharing Co octahedra. No evidence for any structural phase transition or antiferromagnetic ordering observed at 10 K.



Structural building block of YBaCo₄O₇(left) & YBaCo₄O₈ (right) showing drastic displacement of some oxygen atoms and the location of two new ones.

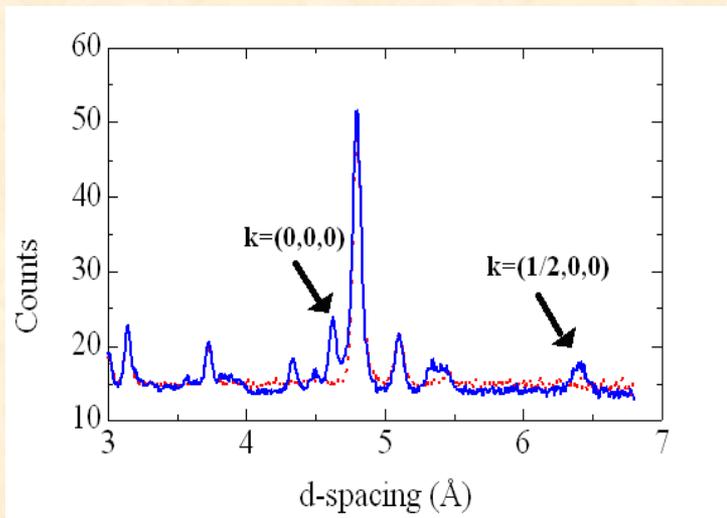
Charge ordering base on BVS



Bond Valence sum calculations show that Y and Ba have expected valences 3^+ and 2^+ in both their sites. The zigzag stripe patterns in the triangular layers consist not only of alternating octahedra and tetrahedra but also of charge ordered Co^{2+} and Co^{3+} ions. However, charge balance suggests each Kagomé layer would contain $\text{Co}^{3+}:\text{Co}^{2+}=5:1$, however we find this number to be 4:1.

Side by side views of triangular and Kagomé layers of YBaCo_4O_7 (left) & YBaCo_4O_8 . Large red and small blue circles represent Ba and Y ions.

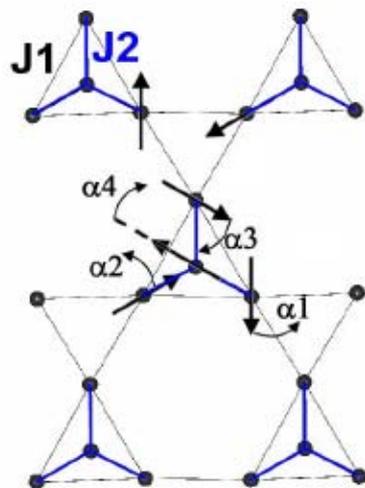
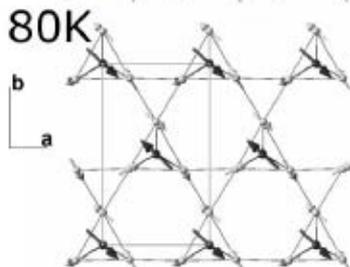
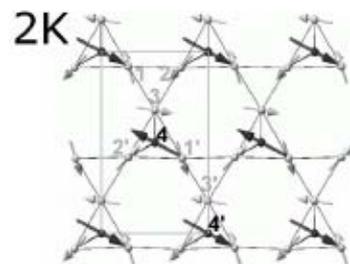
What about magnetism?



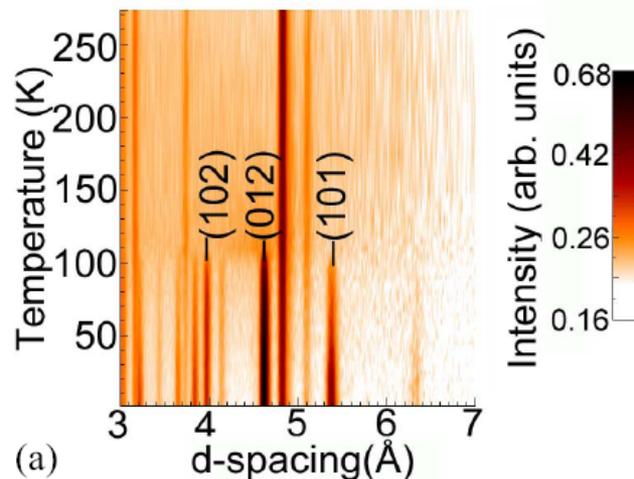
Portion of the Neutron data collected at GEM at 90K(dashed) and 1.7K (solid). Magnetic scattering due to AF order in the 1.7K pattern is marked by two observed propagation vectors.

Since $k_2=(1/2,0,0)$ is missing in $R=Y$, it is reasonable to conclude that k_2 involves a contribution of rare earth. Magnetic structure not yet solved.

Non magnetic rare earth R=Y



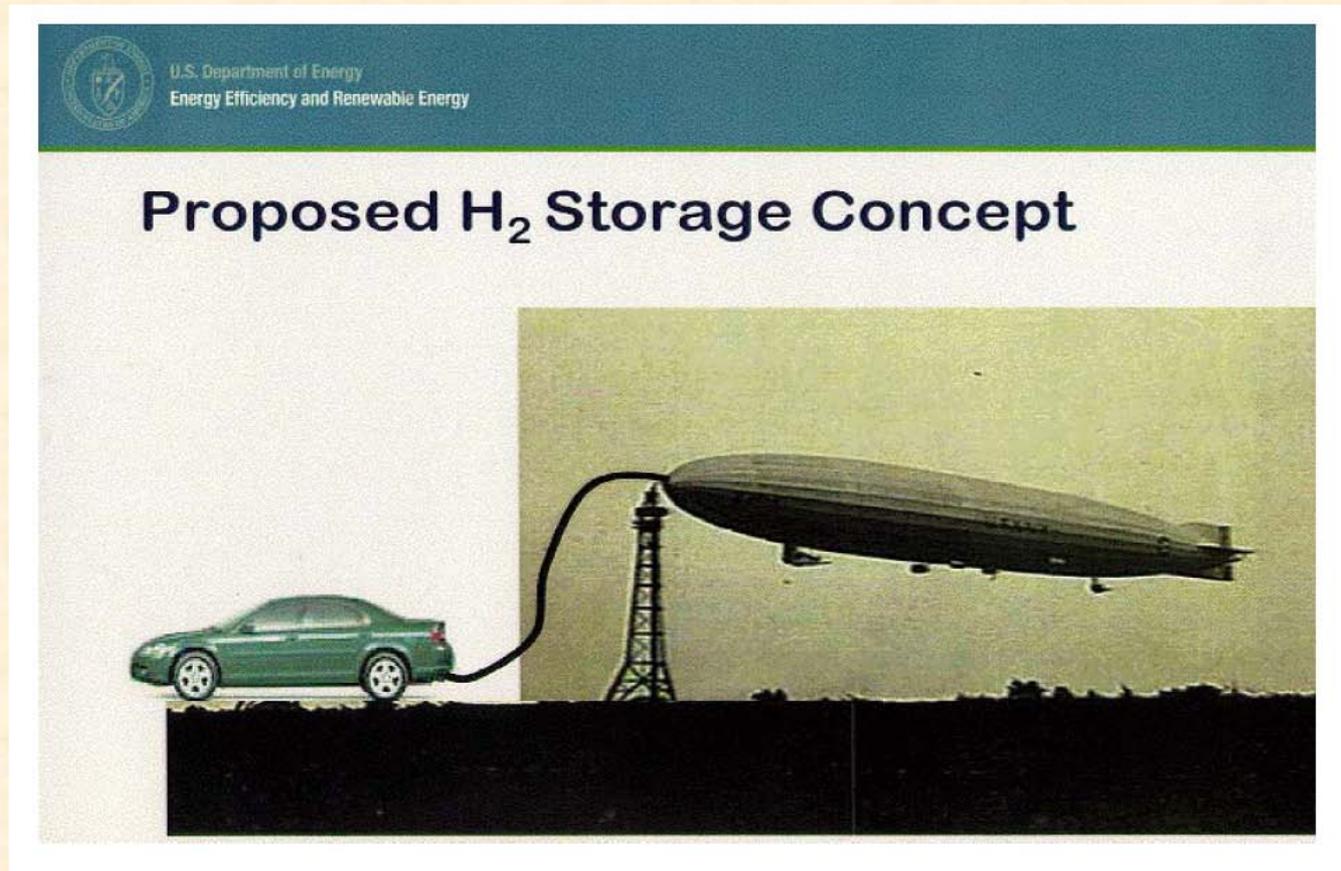
When $R = Y$ and $x=0$, (Y114), the system also transforms from trigonal to orthorhombic, with $T_s = 310$ K. Magnetic superlattice reflections appear at $T_N = 108$ K. The complex magnetic structure was solved by simulated annealing and exhibits features of the negative chirality ordering pattern in the Kagome plane.



Time resolved Powder Diffraction: Hydrogen Storage Materials for mobile application

What will your 'Fuel Tank' for this car look like ?

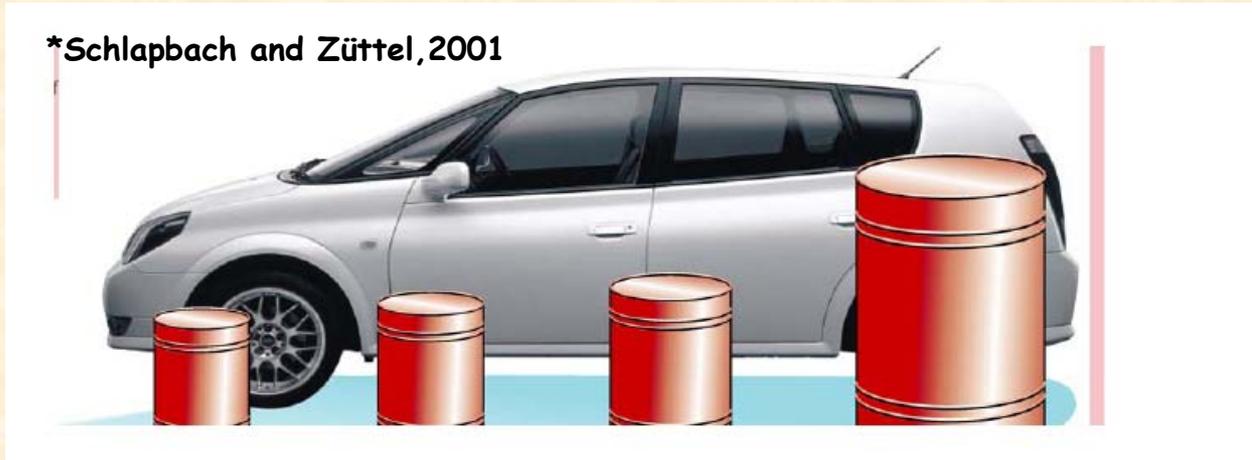
At STP 1kg of H₂ gas has a volume of 11m³.



Picture
borrowed from
J. Eckert
Presentation

SNS
SPALLATION NEUTRON SOURCE

What are the DOE requirements for the "Freedom Car"? (Challenge: Pack Hydrogen as close as possible)



4kg H₂ in

Mg₂NiH₄

LaNi₅H₆

liquid H₂

H₂ at 100atm

2005

2010

2015

Capacity (wt % H₂):

4.5

6.0

9.0

Energy density (MJ/l):

4.3

5.4

9.7

Specific Energy (MJ/kg):

5.4

7.2

10.8

Operating Temp (C):

-20/50

Delivery pressure (bar):

2.5

Refueling rate (H₂/min):

0.5

1.5

2.0

LOW COST!!!!

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

Li₃N : Hydrogen Storage Candidate

Chen et. al: (Nature Nov 2002)

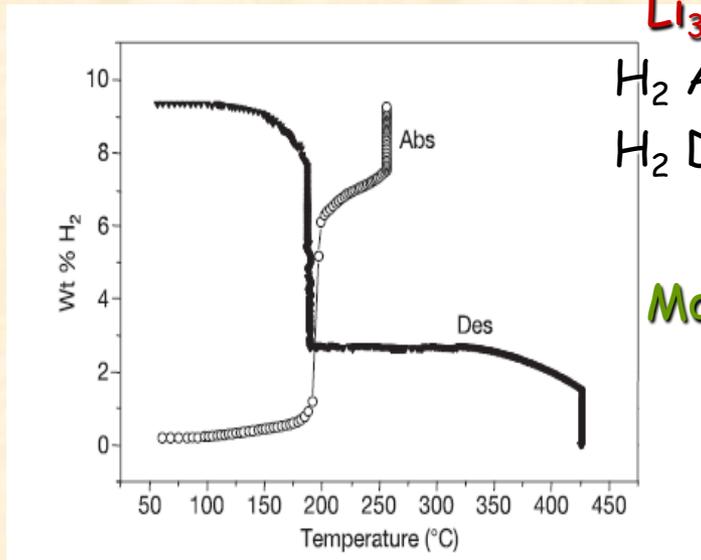
Li Imide

Li Amide



H₂ Absorption 9.3 wt% gain at 255°C

H₂ Desorption 6.3 wt% at 200°C + 3wt % above 320°C



More recently (2004-2005) Meisner et. al. & others:



(~5.2% cyclable H₂)

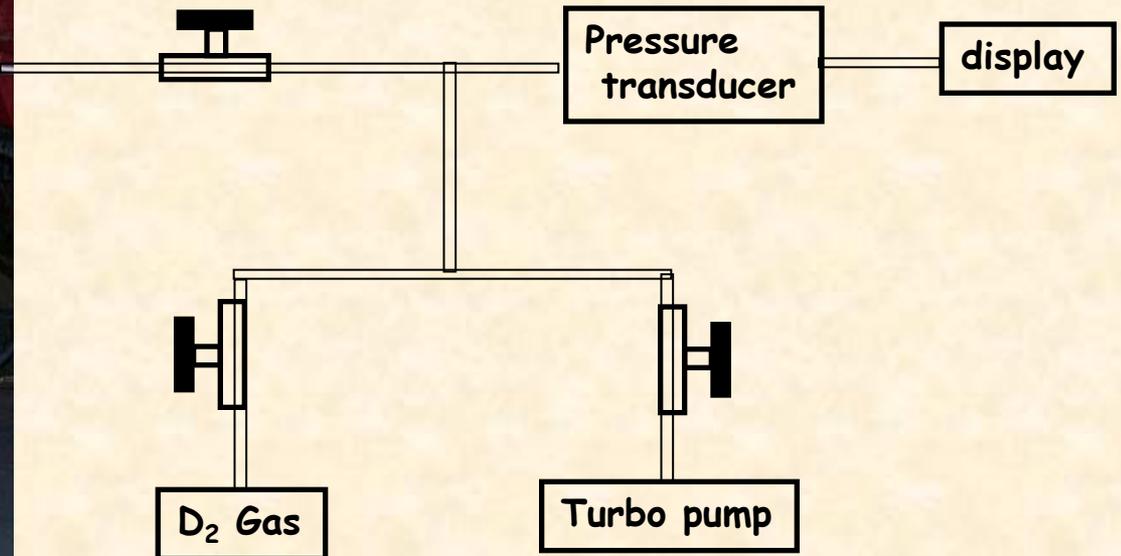
Our Goal: To study this reaction in-situ in bulk material.

Huq et. al. J. Phys. Chem. C 111, 10712 (2007)

Experimental Setup for in-situ measurements

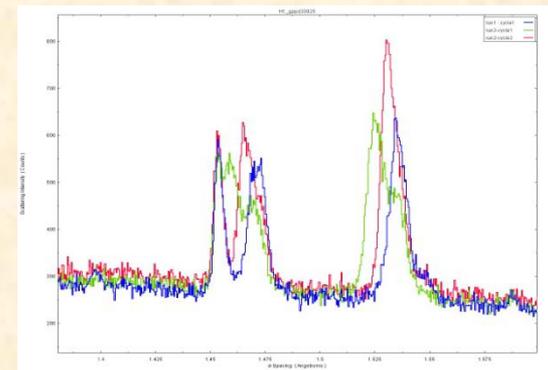
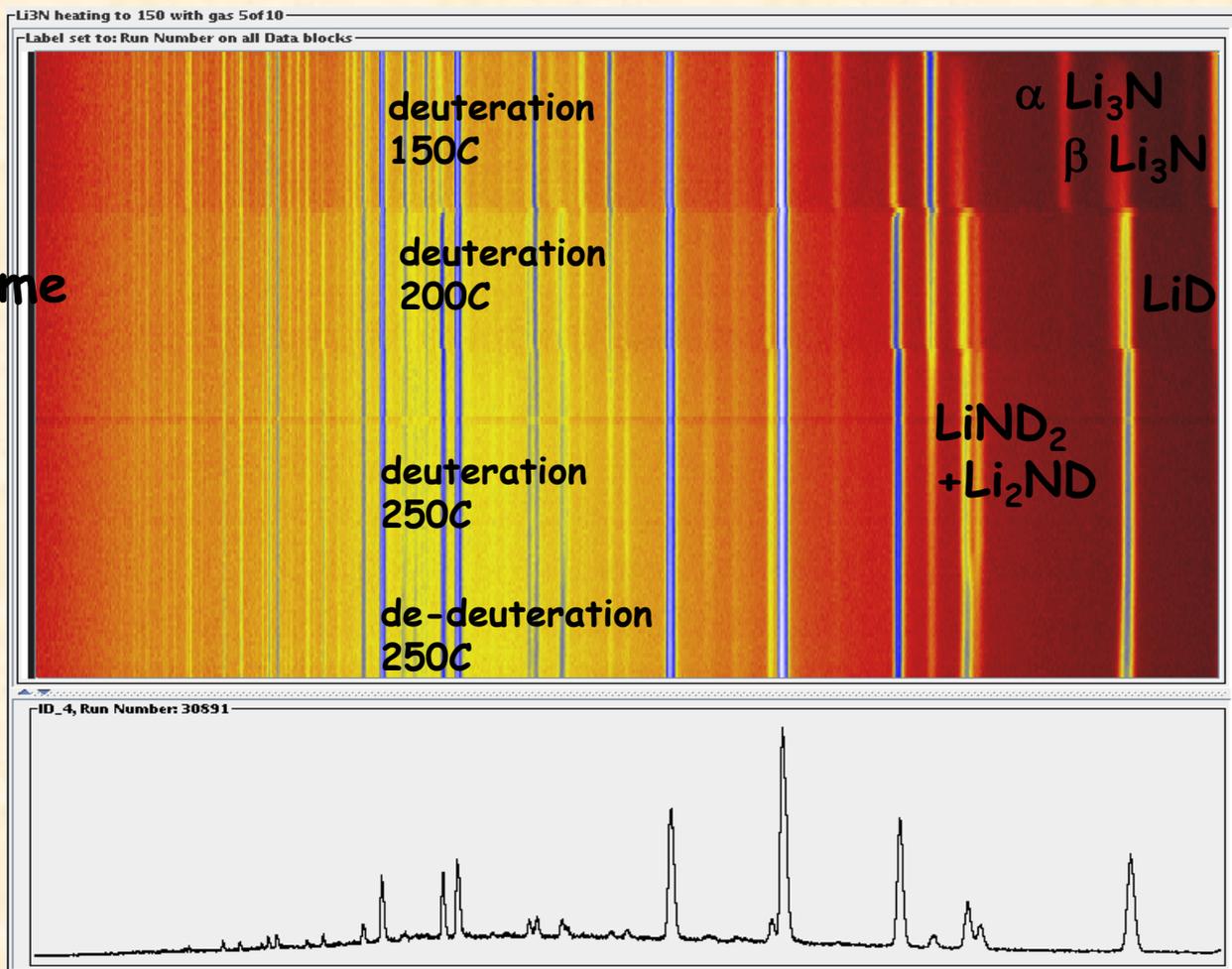


Note: We cannot use null scattering material e.g. V or TiZr for sample holder as they are great H getter and often used for catalysis.

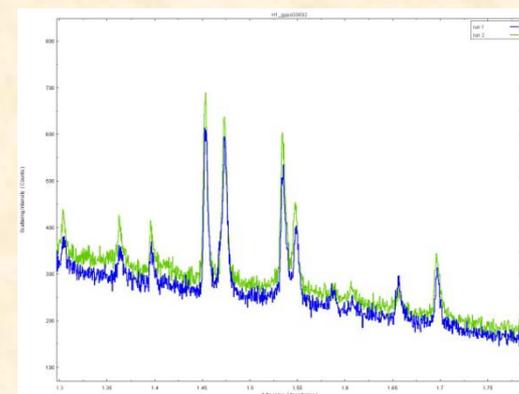


Inconel cell: 100 bar and 500C

In situ Deuteration & De-deuteration

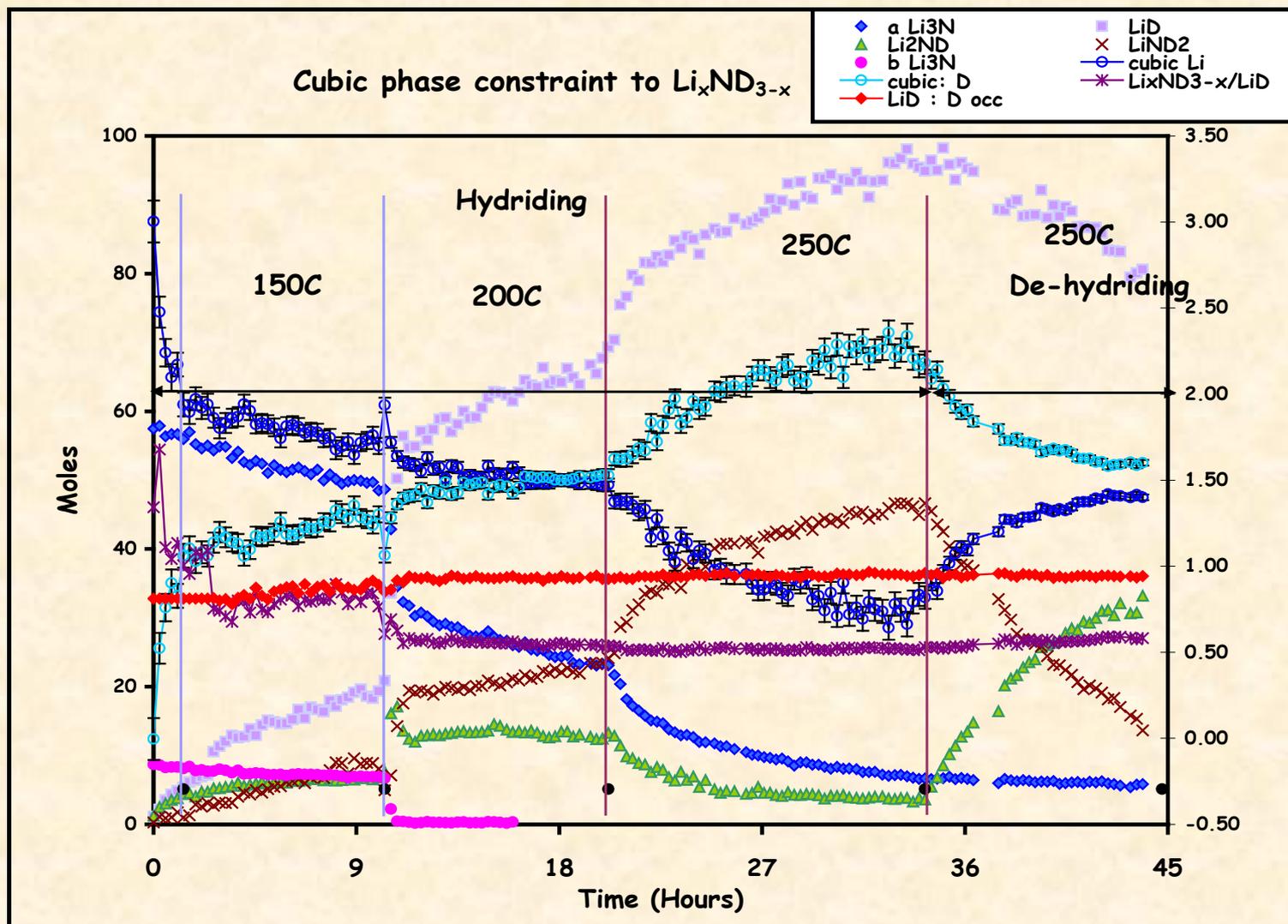


34h: deuteration
10h: pumping



→ d spacing

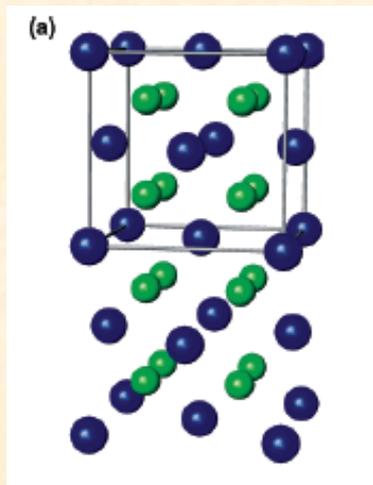
Moles of Phases Present



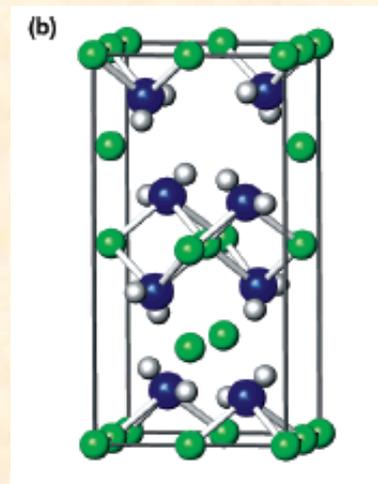
Imide - Amide : Structural relationship

David et. al., JACS 129,1594

Anti fluoride
cubic imide



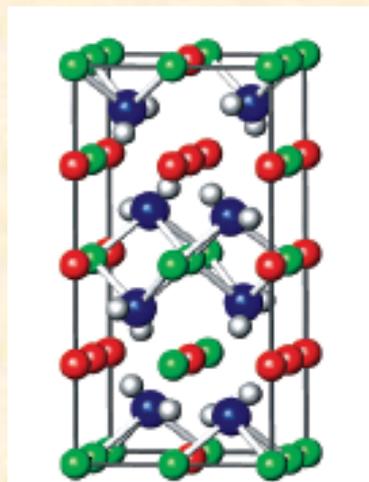
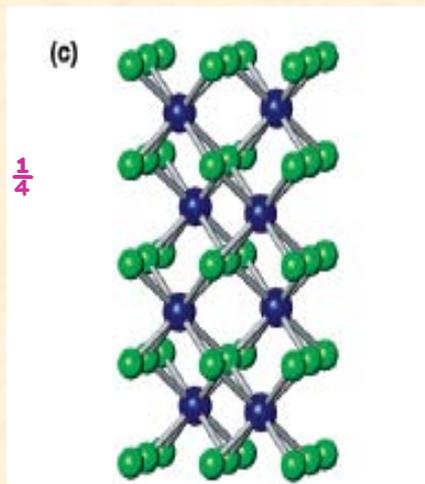
blue: N
green: Li
grey: H



Tetragonal
amide

shift origin $\frac{1}{4} \frac{1}{4} \frac{1}{4}$

$a \times a \times 2a,$

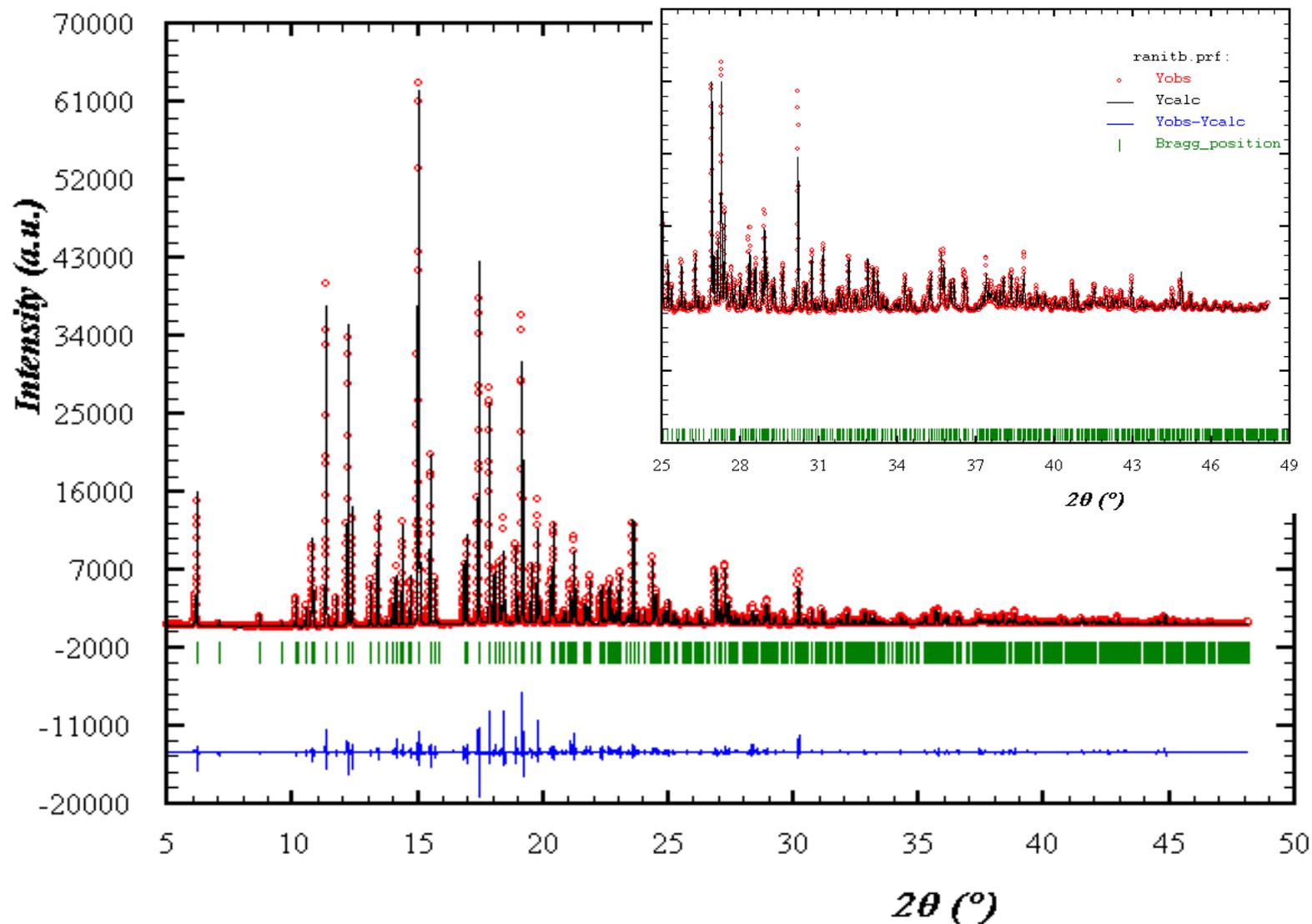


show unoccupied
Li sites by red

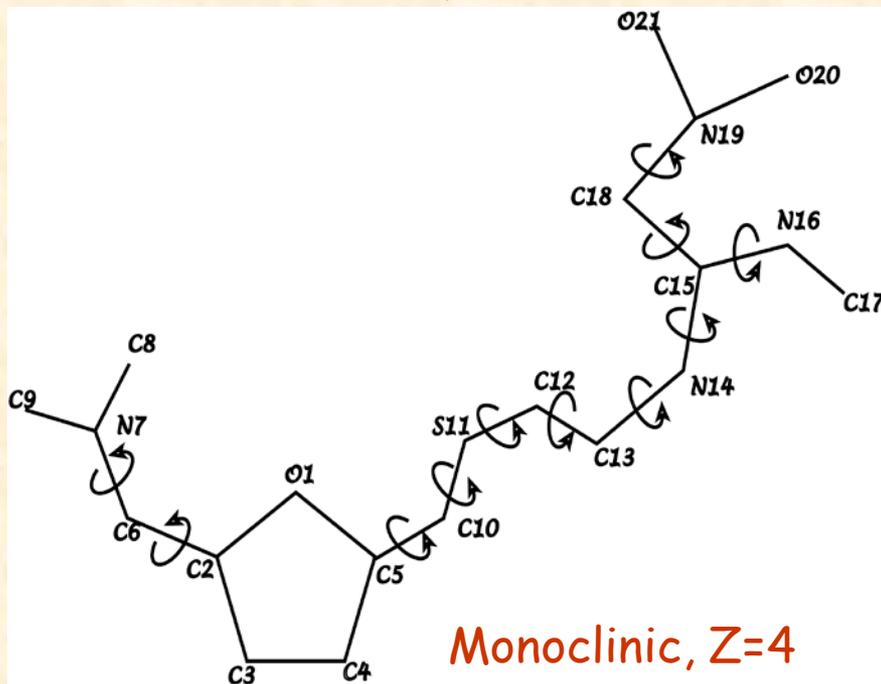
Ab-initio Structure Solution from Powder Diffraction

Undertake a project like this with very good data

Ranitidine HCL form II



Ranitidine HCl (Zantac®) is a very widely used drug for ulcers, excess production of stomach acid. There is an interesting subtlety in its crystal structure. Huq et. al. J. Pharm. Sci. 92, 244 (2003)



Monoclinic, Z=4

$a=18.808 \text{ \AA}$,

$b=12.981 \text{ \AA}$,

$c=7.211 \text{ \AA}$

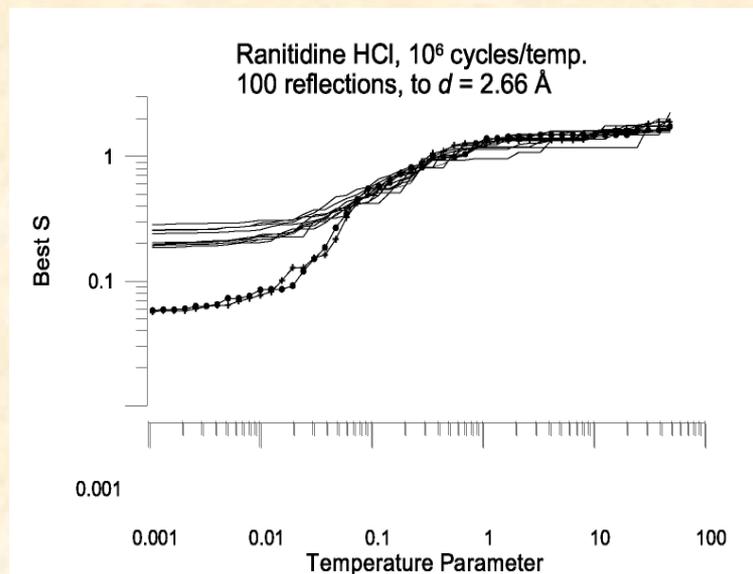
$\beta=95.057^\circ$,

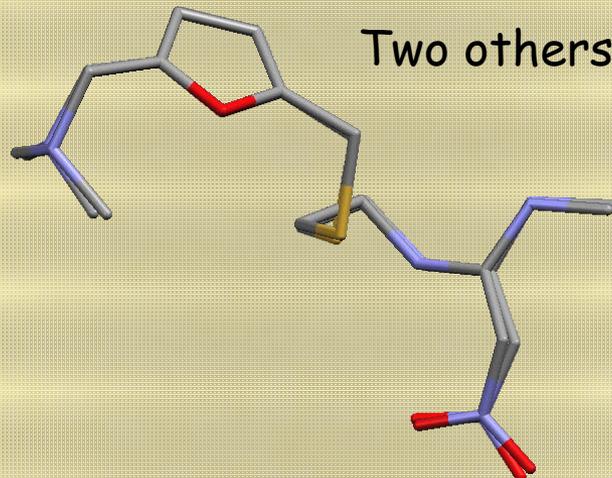
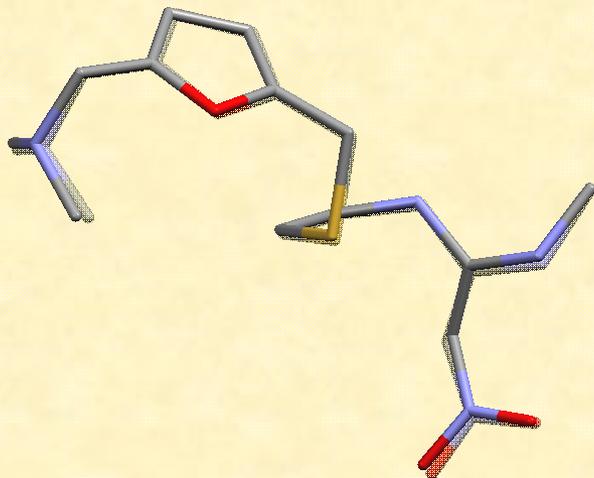
Space Group : $P 2_1/n$

3 spatial coordinates : position

3 Eulerian angles : orientation

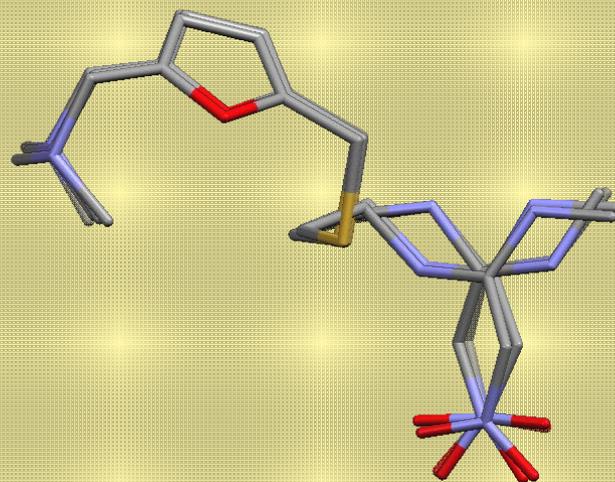
11 torsions.





Two others

Two candidate solutions from PSSP

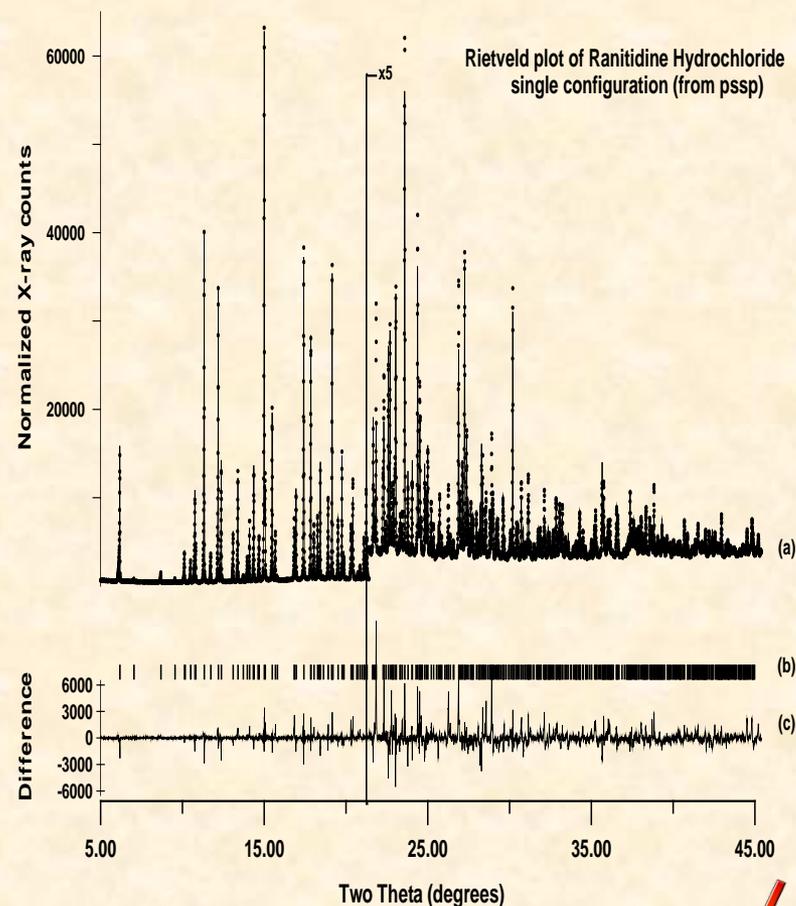
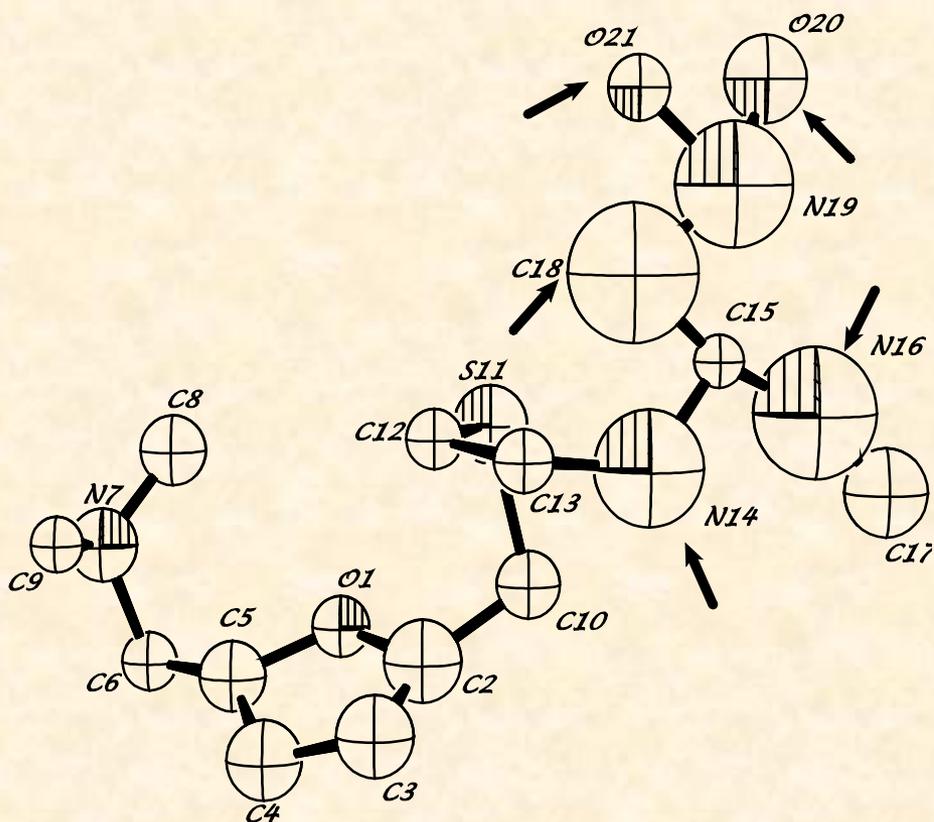


All four,
superimposed.

Disorder,
or inability of powder
data to distinguish
a few of the atoms?

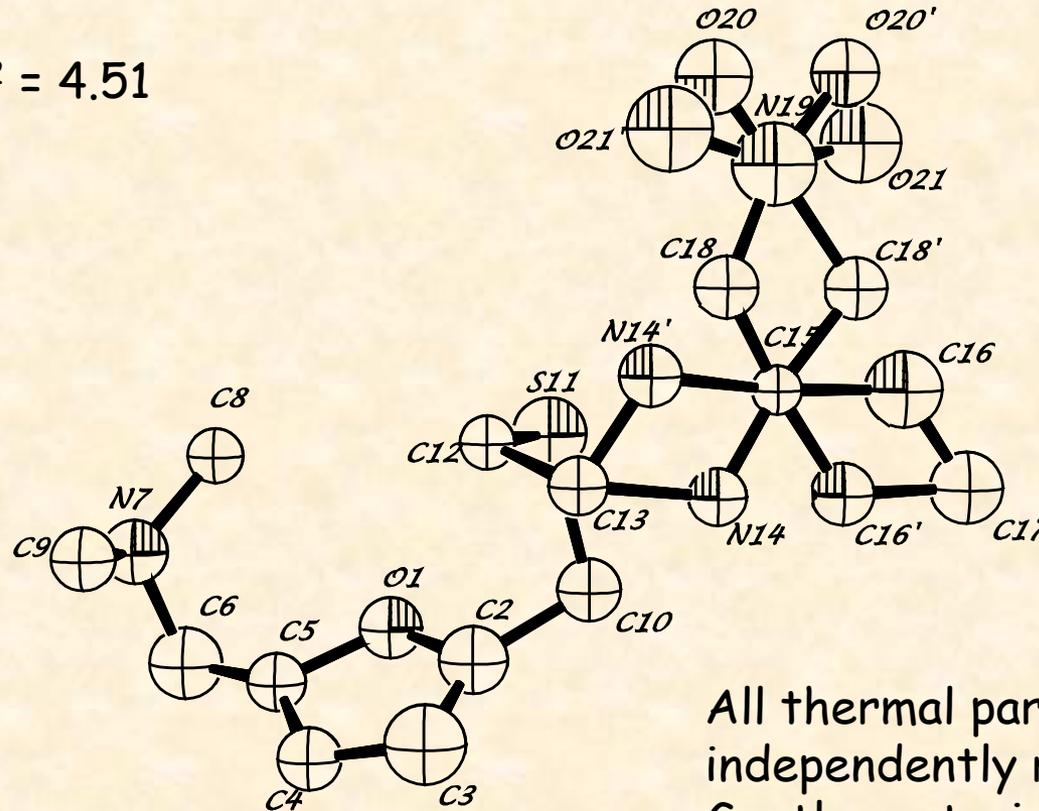
Atomic structure of our best Rietveld refinement of a single molecule. Essentially independent of which solution we start from.

$$R_{wp} = 11.12\%, \quad \chi^2 = 10.56$$



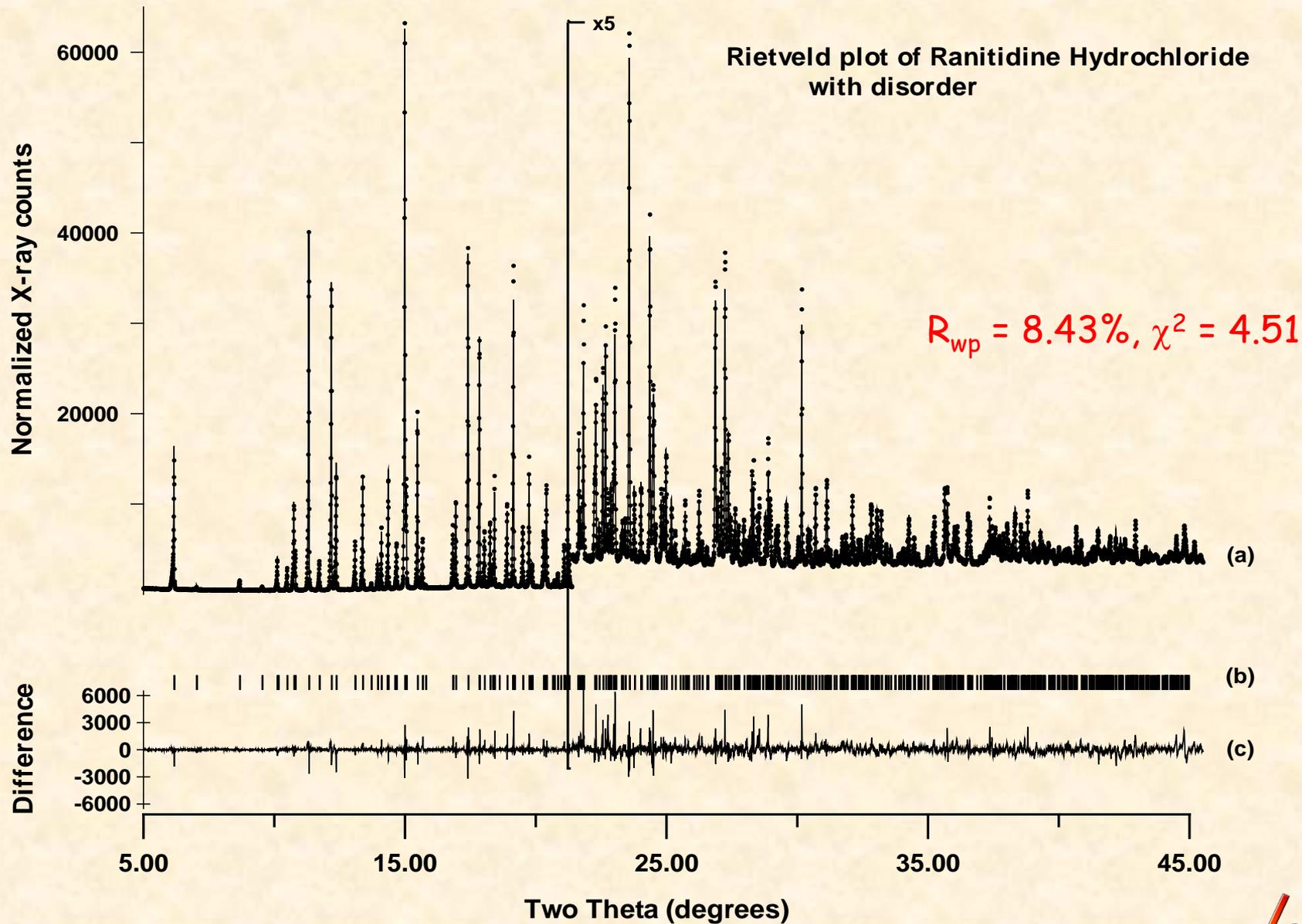
Refinement incorporating disorder. 50% occupancy of each of two sites for N14, C16, C18, O20, and O21.

$$R_{wp} = 8.39\%, \chi^2 = 4.51$$



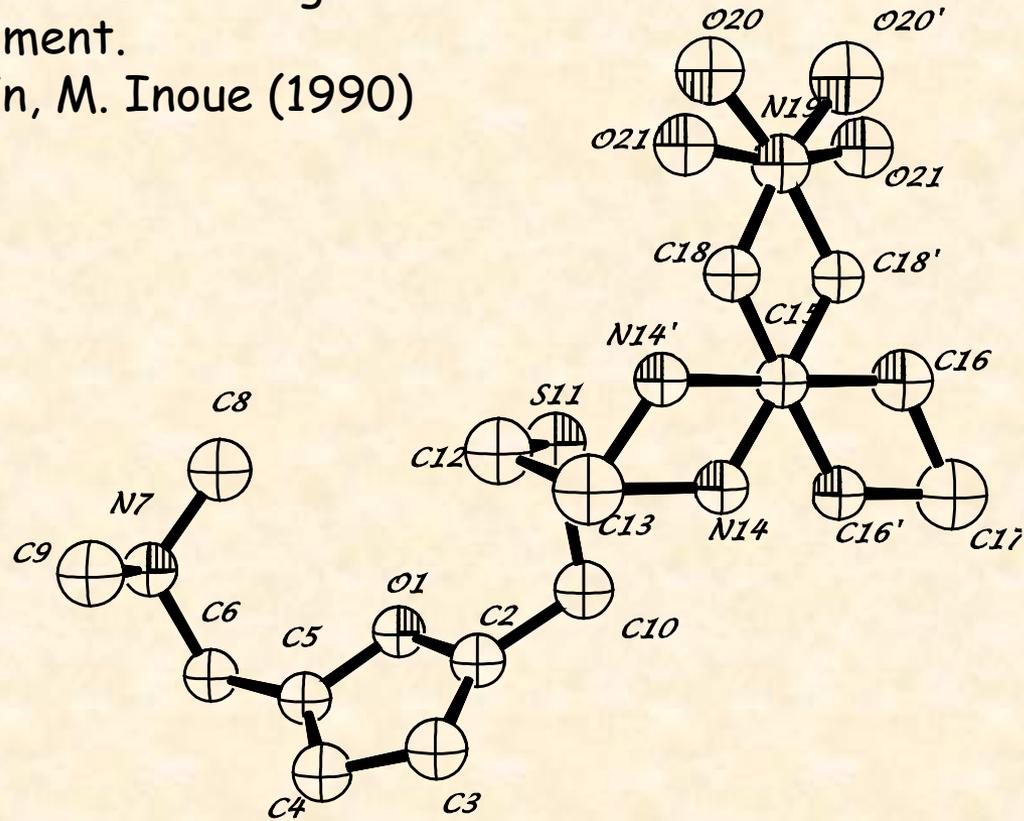
This is clearly the correct solution, which includes molecular disorder.

All thermal parameters independently refined!
Gentle restraints on bond lengths.



The answer, including disorder,
was already known from single
crystal experiment.

T. Ishida, Y. In, M. Inoue (1990)



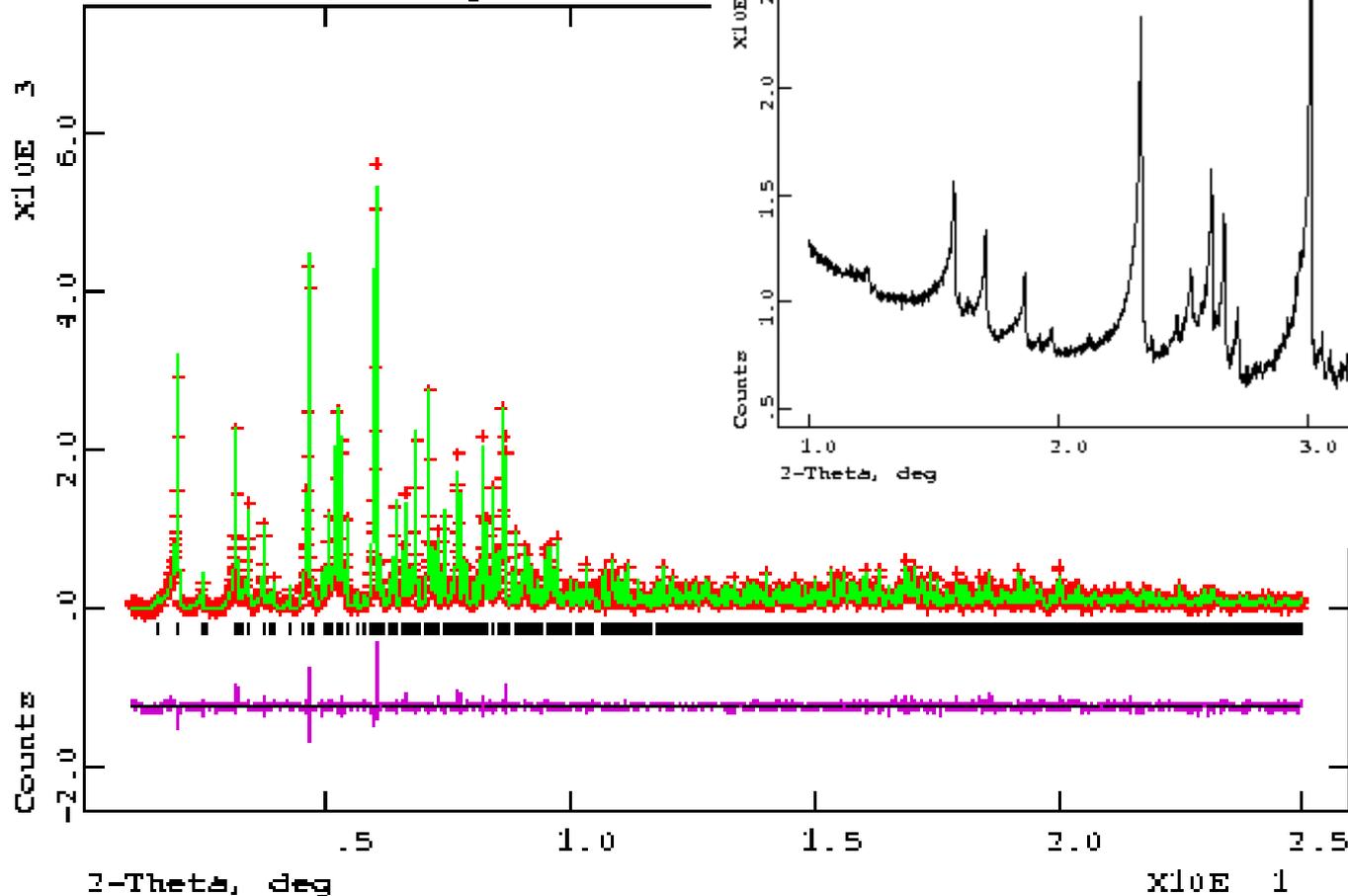
Proteins and Powder Diffraction

Extreme limit: Proteins

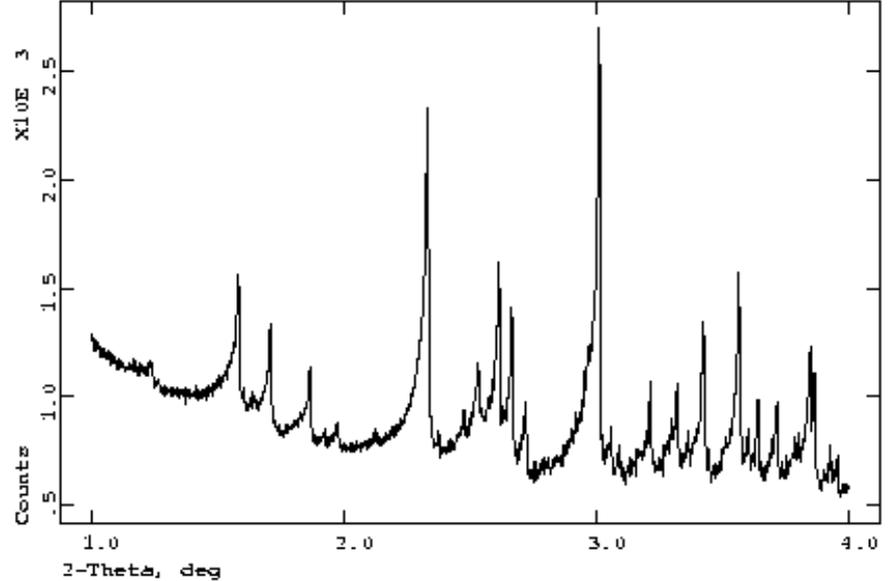
Work done by R. Von Dreele (Los Alamos) & P.W. Stephens

It is possible to get usable data, and to refine it with sufficient chemical restraints.

T3r3 HF Zn insulin RT 1.5mm cap 1.
Lambda 1.4011 Å, L-S cycle 335



T3r3 Zn insulin hard grind fresh RT 1.5mm cap 0.700233
Scan no. = 1 Lambda1, lambda2 = .700 Observed Profile



Structure solved from powder data & Rietveld refinement

Human Insulin Zn complex

Native

Ground

$a=80.96\text{\AA}$

81.28\AA

$c=37.59\text{\AA}$

73.04\AA

$N_{\text{refined}} = 1754$

2925

$N_{\text{restraints}}=3871$

7934

$N_{\text{reflections}}=9871$

12734

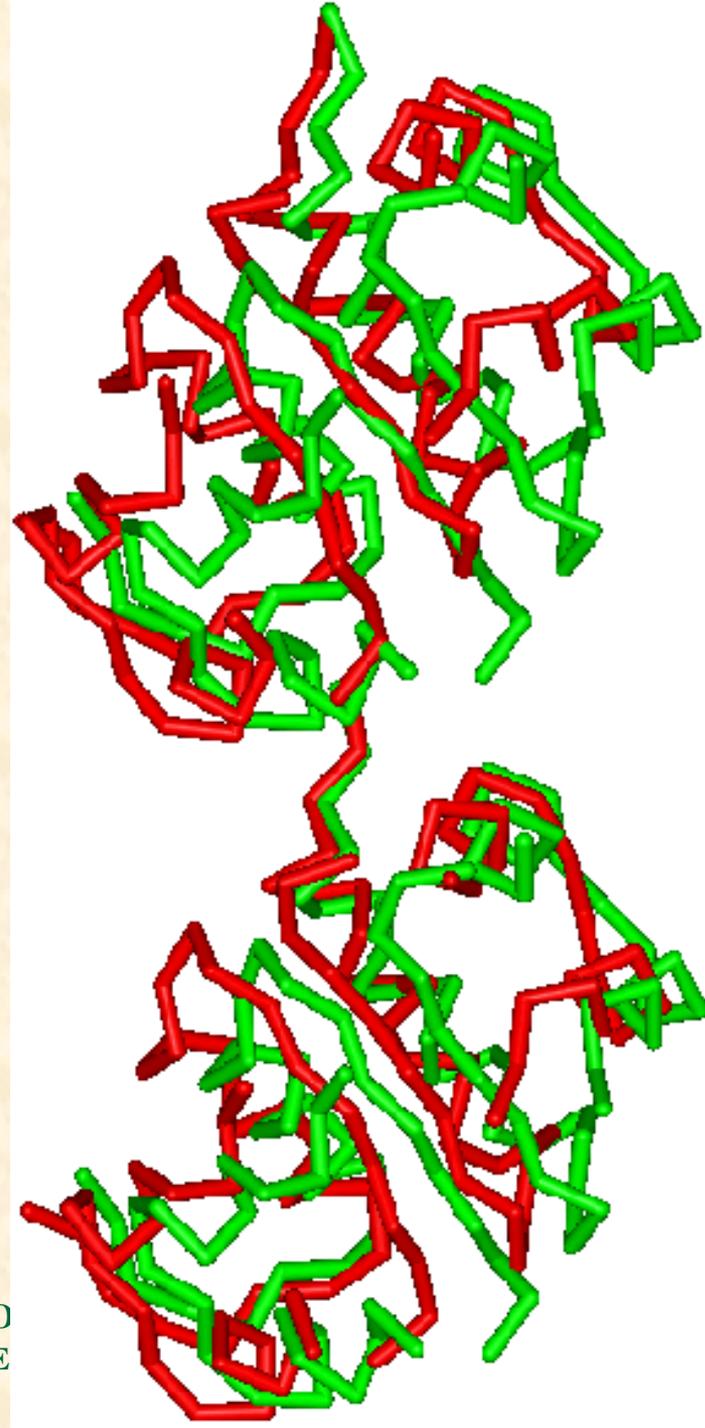
Resolution 3.06\AA

3.22\AA

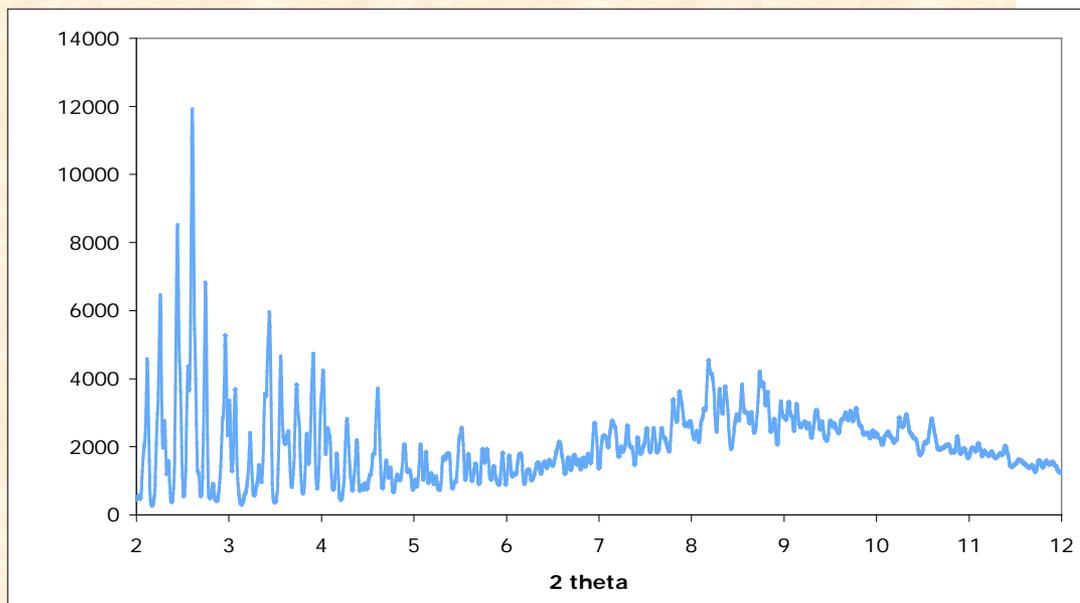
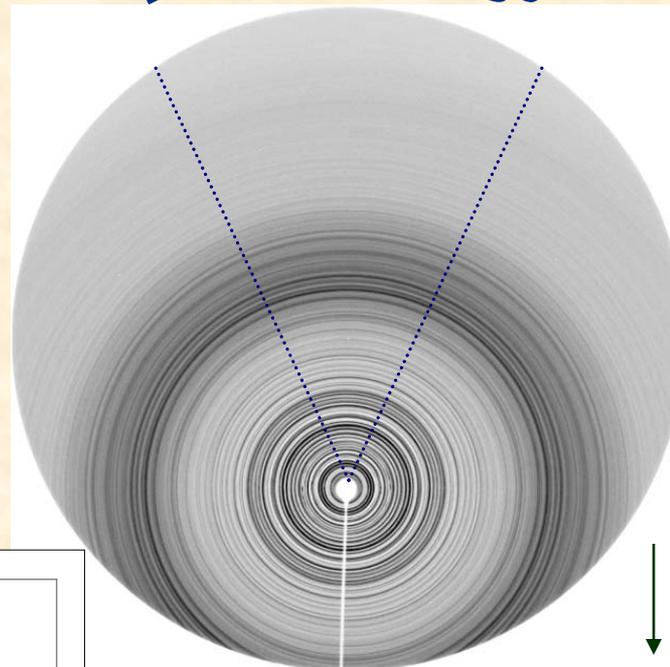
$R_{\text{wp}}=3.34\%$

3.77%

R.B. Von Dreele, P.W. Stephens, G.D. Smith, and R.H. Blessing,
"The First Protein Crystal Structure Determined from X-ray
Powder Diffraction Data: a Variant of T_3R_3 Human Insulin Zinc
Complex Produced by Grinding," *Acta Crystallographica D* 56, 1549-
53 (2000).



Current work at APS: Structure solution via molecular replacement



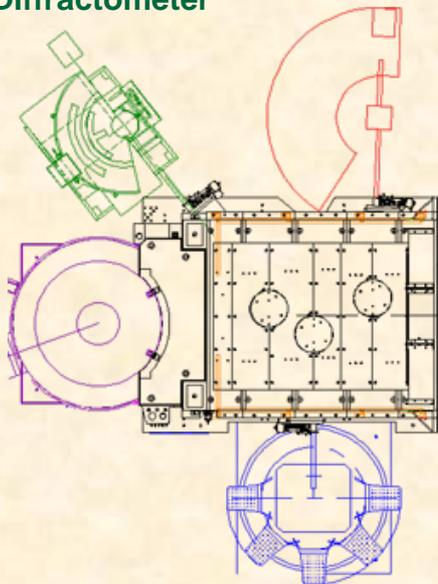
Take home message

Powder diffraction is an extremely powerful technique to study structural properties of a very wide variety of materials. To understand physical and chemical properties of materials it is crucial that we know how the "atoms are put together" and if you cannot grow those big single crystals....you can still learn quite a lot about your system using powder diffraction.

Powder Diffraction at the HFIR

WAND
Diffractometer

High-Resolution
Diffractometer

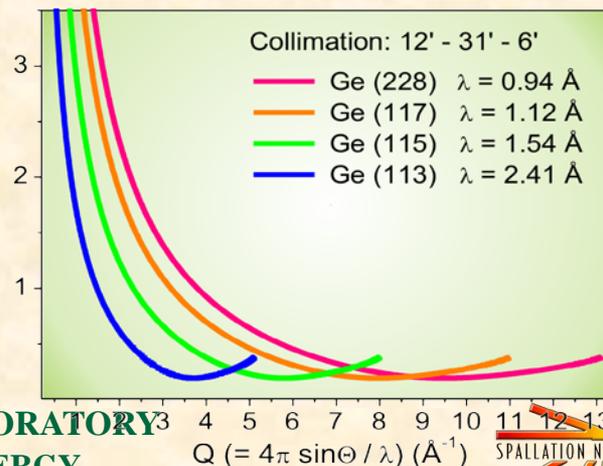


Engineering
Diffractometer

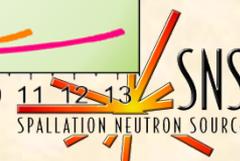


Instrument Characteristics:

(hkl)	λ (Å)	d_{\max} (Å)	$4\pi\sin\theta/\lambda$	n° flux
(115)	1.54	17.6	0.35-7.9	10^7 (n/cm ² s)
(113)	2.41	27.6	0.2-5.1	5×10^6
(117)	1.12	12.8	0.5-10.9	4×10^6



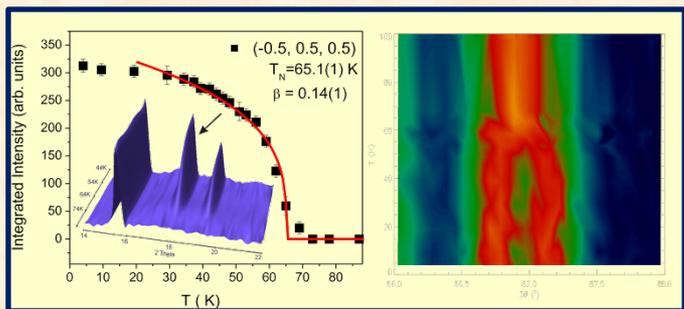
OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY



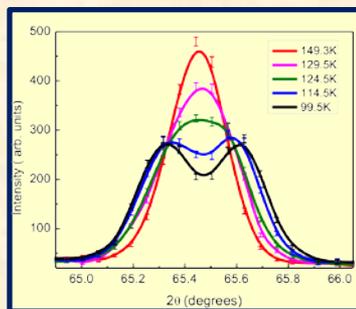
Science at HB2A Powder Diffractometer

✓ Temperature dependence of crystal and magnetic structures (0.3 K < T < 700 K)

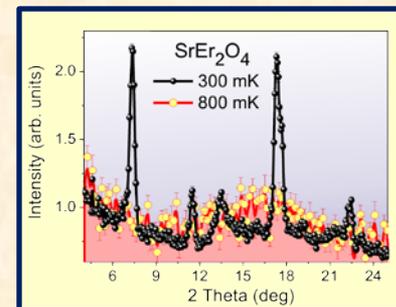
Magnetoelastic properties of MnCuO_2
O. Garlea (NSSD-ORNL) et al.



Correlated lattice effects on $\text{RFeAs}_{1-x}\text{P}_x\text{O}$ (R=La,Ce)
C. de la Cruz (NSSD-ORNL) et al.

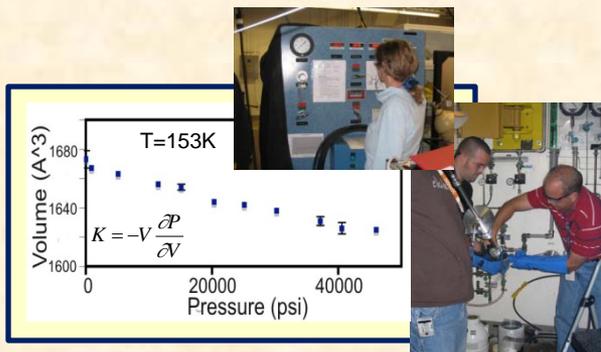


Low-temperature magnetic ordering of frustrated SrEr_2O_4
Haifeng Li (Ames Lab.) et al.



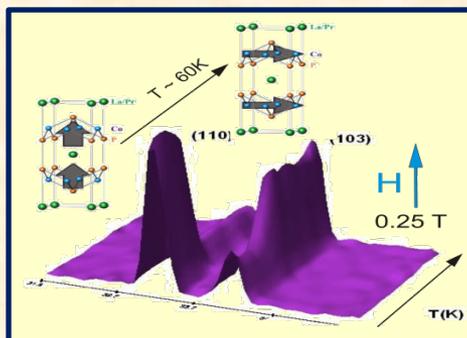
✓ Pressure studies with He gas cells up to 3 kbar

Bulk modulus of CO_2 hydrate
Claudia Rawn (MSTD-ORNL) et al.

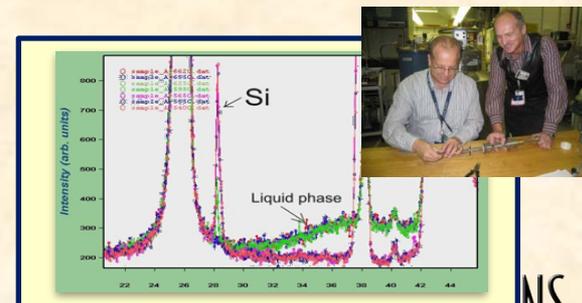


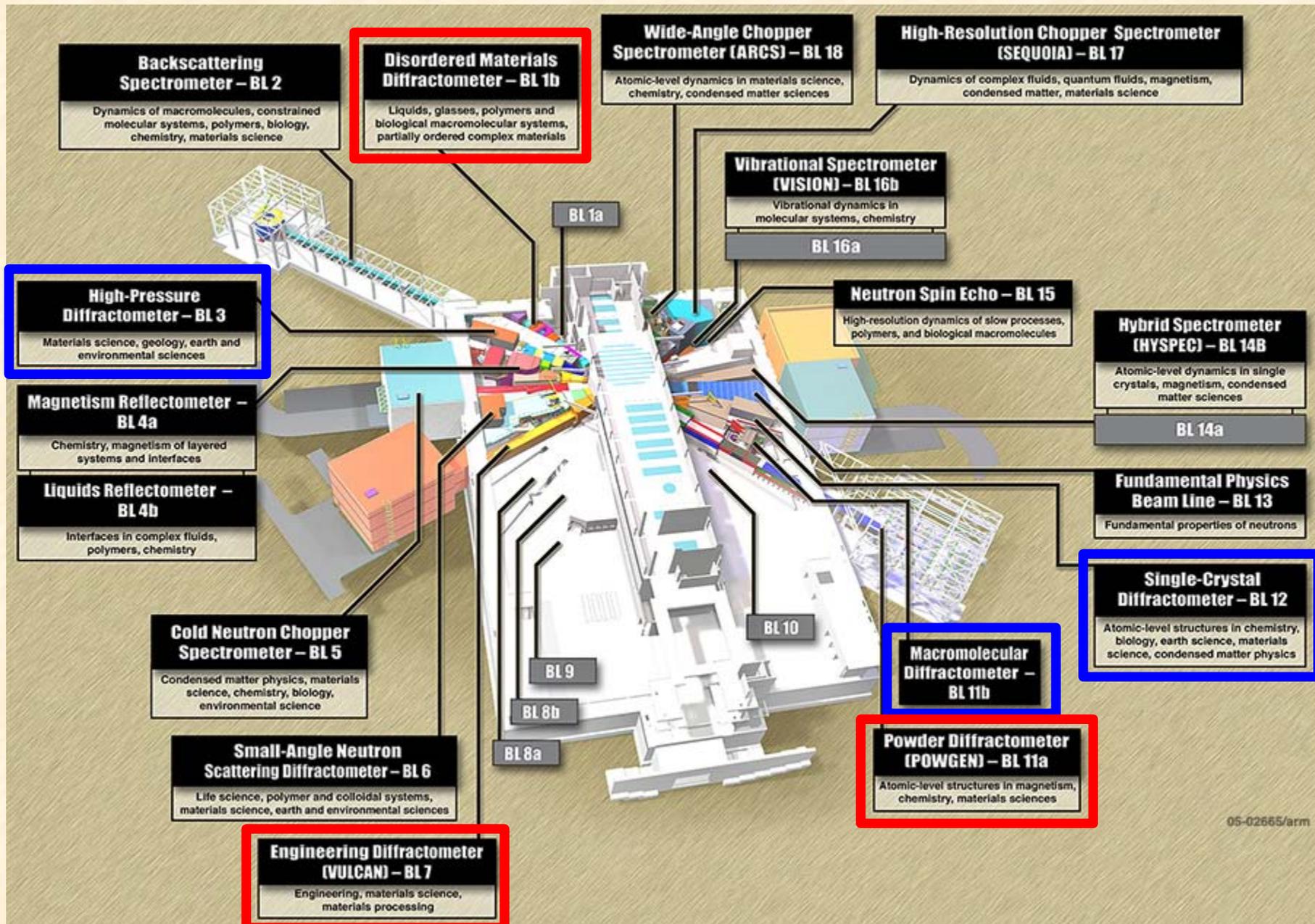
✓ Magnetic field induced phenomena, up to 5 Tesla

Magnetic pole reversal in $\text{La}_{1-x}\text{Pr}_x\text{Co}_2\text{P}_2$
M. Shatruk (FSU) et al.



✓ High-temperature studies, up to 1200 C Solidification mechanisms of industrial Al-Si Alloy *D. Sediako (NRC) et al.*



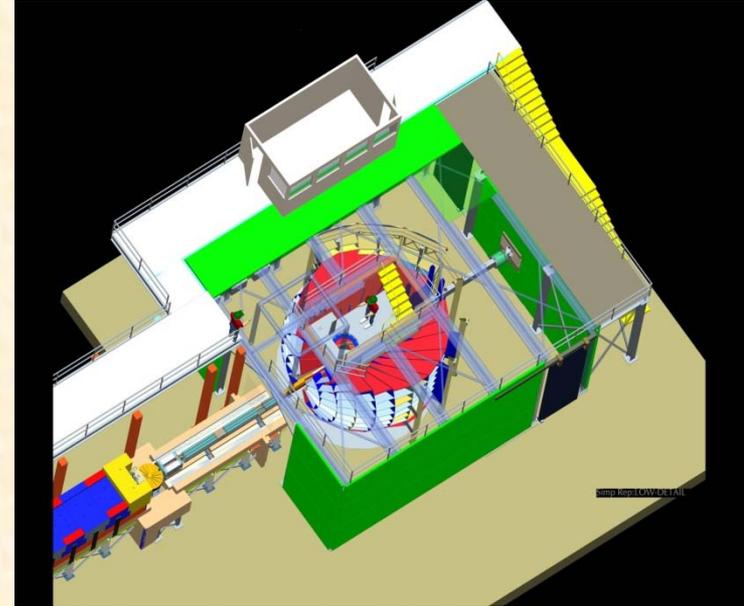


05-02665/arm

Time of flight instrument (POWGEN)



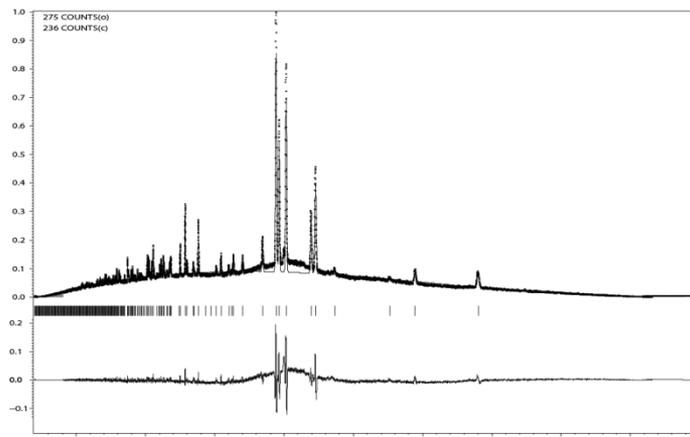
Picture of Instrument Team inside the enclosure.



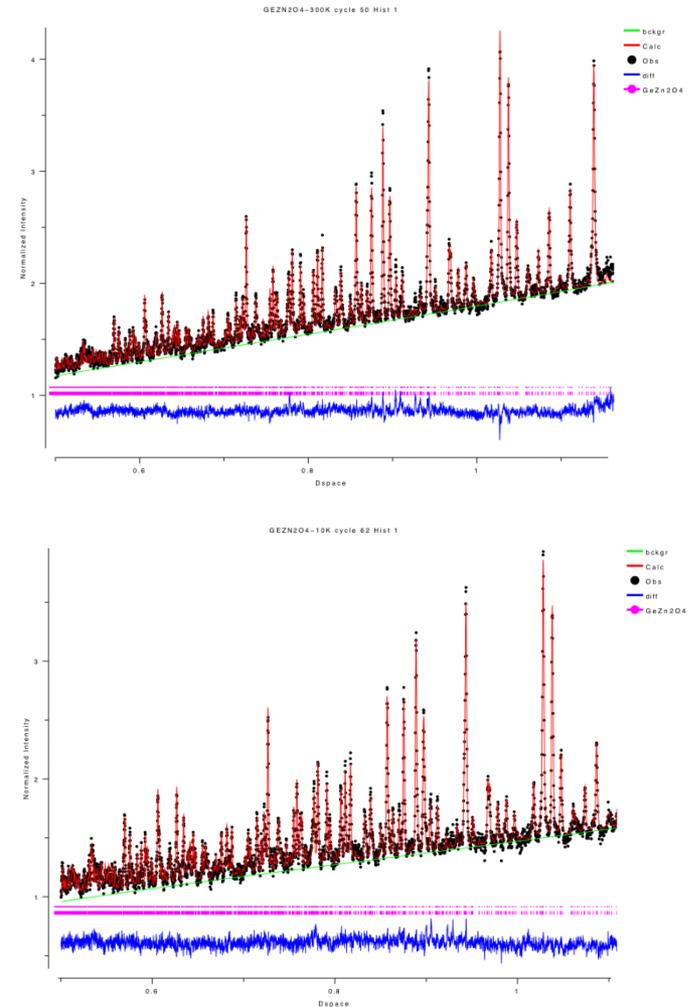
POWGEN Science

**FERNS: Fast Exchange Refrigerator
for Neutron Sciences (DOE SBIR)**

**24 samples loaded: Temp range
10K-300K**



The $Mn_{5-x}Fe_xSi_3$ series is of interest for its potential magneto caloric effect properties. FERNS has been used to map the magnetic structures as a function of temperature and Fe concentration. Above plot shows preliminary refinement data at 300K.

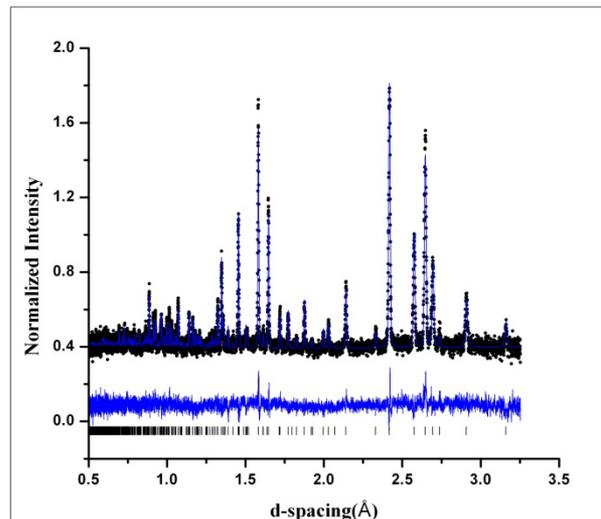
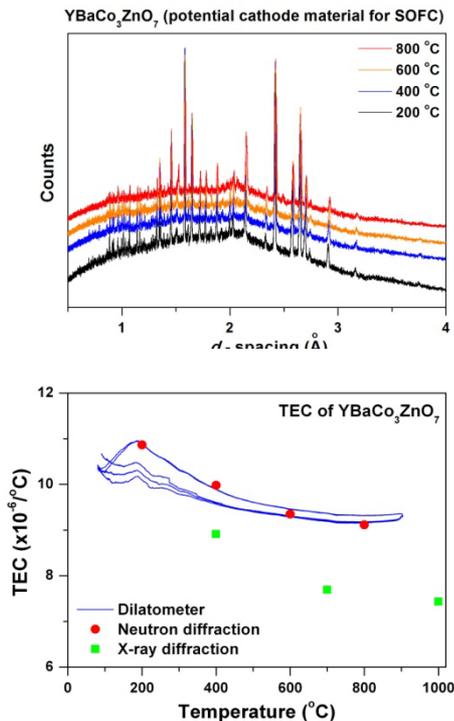


**Neutron Powder Diffraction Determination
of Two Crystal Structures of $GeZn_2O_4$**

POWGEN Science

Lucyfe Furnace : **RT -1000 C**

Data shows powder pattern of $\text{YBaCo}_3\text{ZnO}_7$, a potential candidate for cathode material for Solid Oxide Fuel Cell. The parent compound YBaCo_4O_7 is unstable at these operational temperatures. However, these doped compounds are stable up to 800°C .



Call for Proposals

Neutron Scattering Science - Oak Ridge National Laboratory Due August 25, 2010

Proposals for beam time at Oak Ridge National Laboratory's High Flux Isotope Reactor (HFIR) and Spallation Neutron Source (SNS) will be accepted via the web-based proposal system until 11:59 p.m. eastern time, Wednesday, August 25, 2010. This call is for experiments anticipated to run from January through May 2011.

Information and instructions

To learn more about submitting a proposal for beam time, go to <http://neutrons.ornl.gov/users/proposals.shtml> or directly to the proposal system at www.ornl.gov/sci/iuims/ipts/. Previously submitted proposals may be used as the basis for new proposals. All proposals will be reviewed for feasibility, safety, and the potential for high-impact science. Before beginning approved experiments, users must complete access and training requirements and ensure that the appropriate user agreements are in place.

Available instruments for general users

The ORNL Neutron Sciences web site, neutrons.ornl.gov, provides specific information about each of these instruments.

HFIR

- HB-1 Polarized Triple-Axis Spectrometer
- HB-1A Fixed-Incident-Energy Triple-Axis Spectrometer
- HB-2A Neutron Powder Diffractometer
- HB-2B Neutron Residual Stress Mapping Facility*
- HB-3 Triple-Axis Spectrometer
- HB-3A Four-Circle Diffractometer
- CG-1D Neutron Imaging Prototype Station*
- CG-2 General-Purpose SANS
- CG-3 Bio-SANS
- CG-4C Cold Neutron Triple-Axis Spectrometer*

SNS

- BL-2 Backscattering Spectrometer (BASIS)
- BL-3 Spallation Neutrons and Pressure Diffractometer (SNAP)
- BL-4A Magnetism Reflectometer (MR)
- BL-4B Liquids Reflectometer (LR)
- BL-5 Cold Neutron Chopper Spectrometer (CNCS)
- BL-6 Extended Q-Range SANS (EQ-SANS)
- BL-7 Engineering Materials Diffractometer (VULCAN)
- BL-11A Powder Diffractometer (POWGEN)
- BL-12 Single Crystal Diffractometer (TOPAZ)
- BL-15 Neutron Spin Echo Spectrometer (NSE)*
- BL-17 Fine-Resolution Fermi Chopper Spectrometer (SEQUOIA)
- BL-18 Wide Angular-Range Chopper Spectrometer (ARCS)

**Instruments with limited availability for general users*

For more information:

Neutron Scattering Science User Office, neutronusers@ornl.gov or (865) 574-4600.

These facilities are funded by the U.S. Department of Energy.

neutrons.ornl.gov

