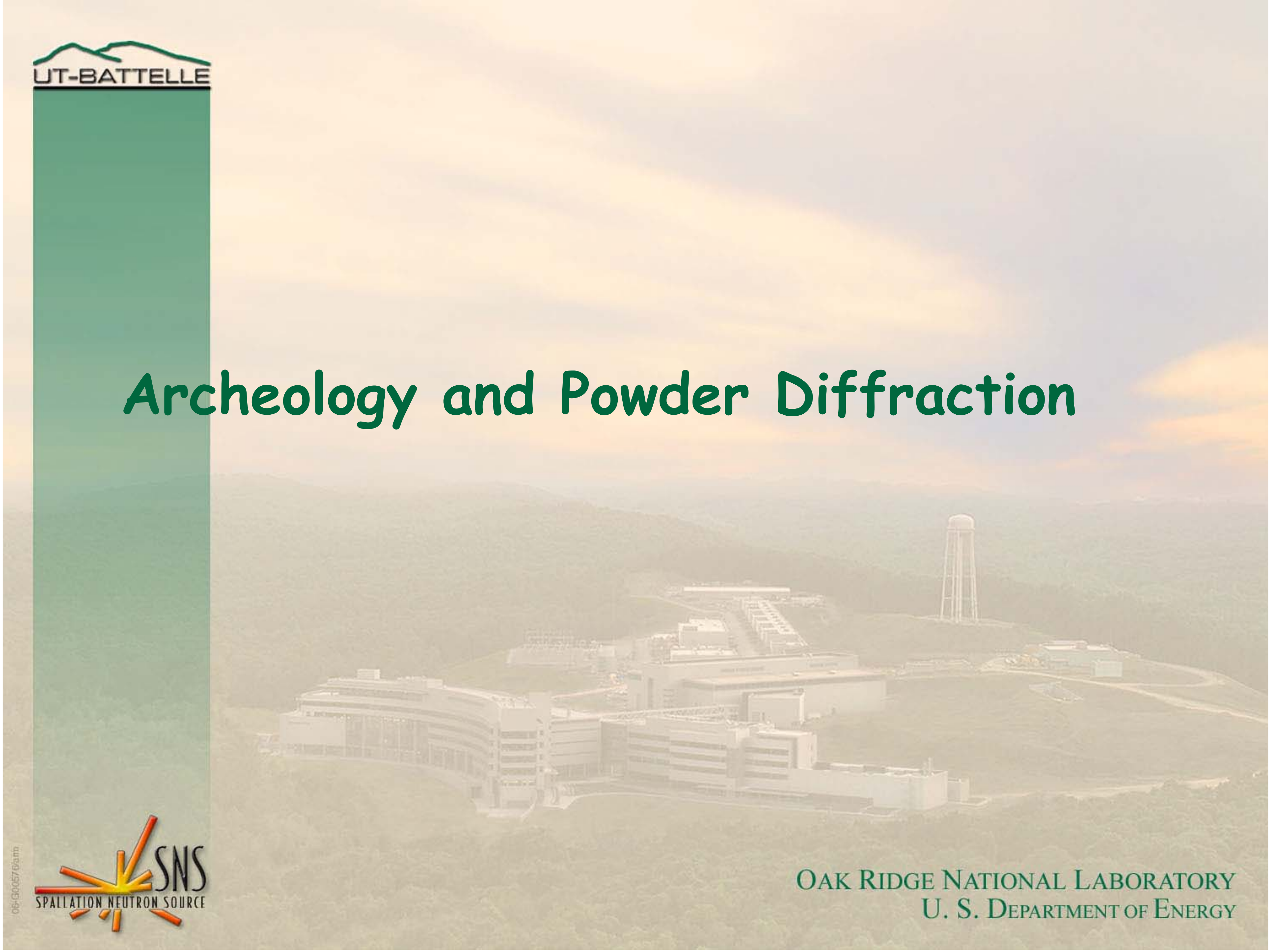
Powder Diffraction Applications

Ashfia Huq
Spallation Neutron Source
National School on Neutron and X-ray Scattering
October 2, 2008

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

- 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



UT-BATTELLE

Archeology and Powder Diffraction

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06-G00576/arm

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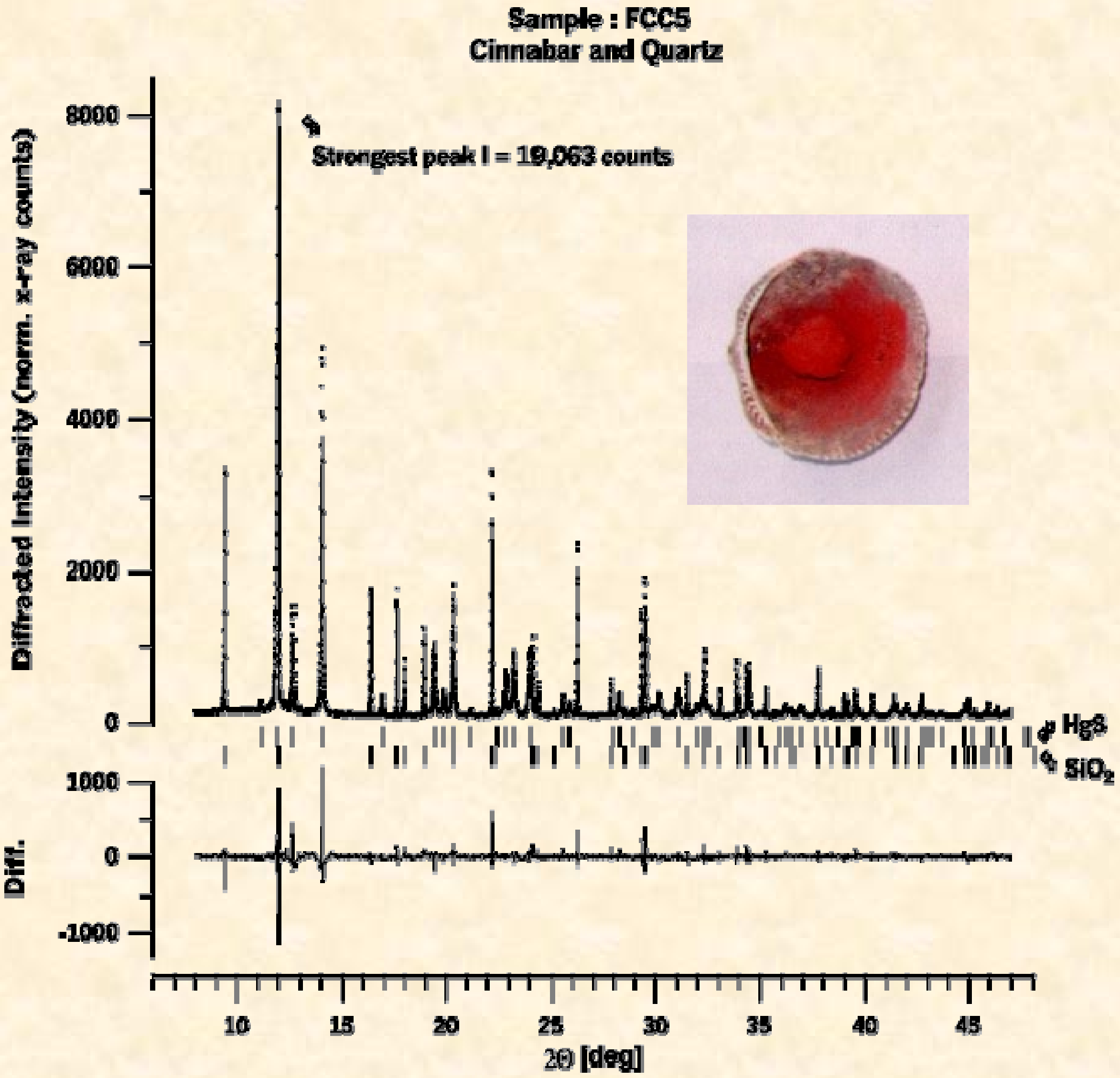
Phase ID: "Finger Printing"

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

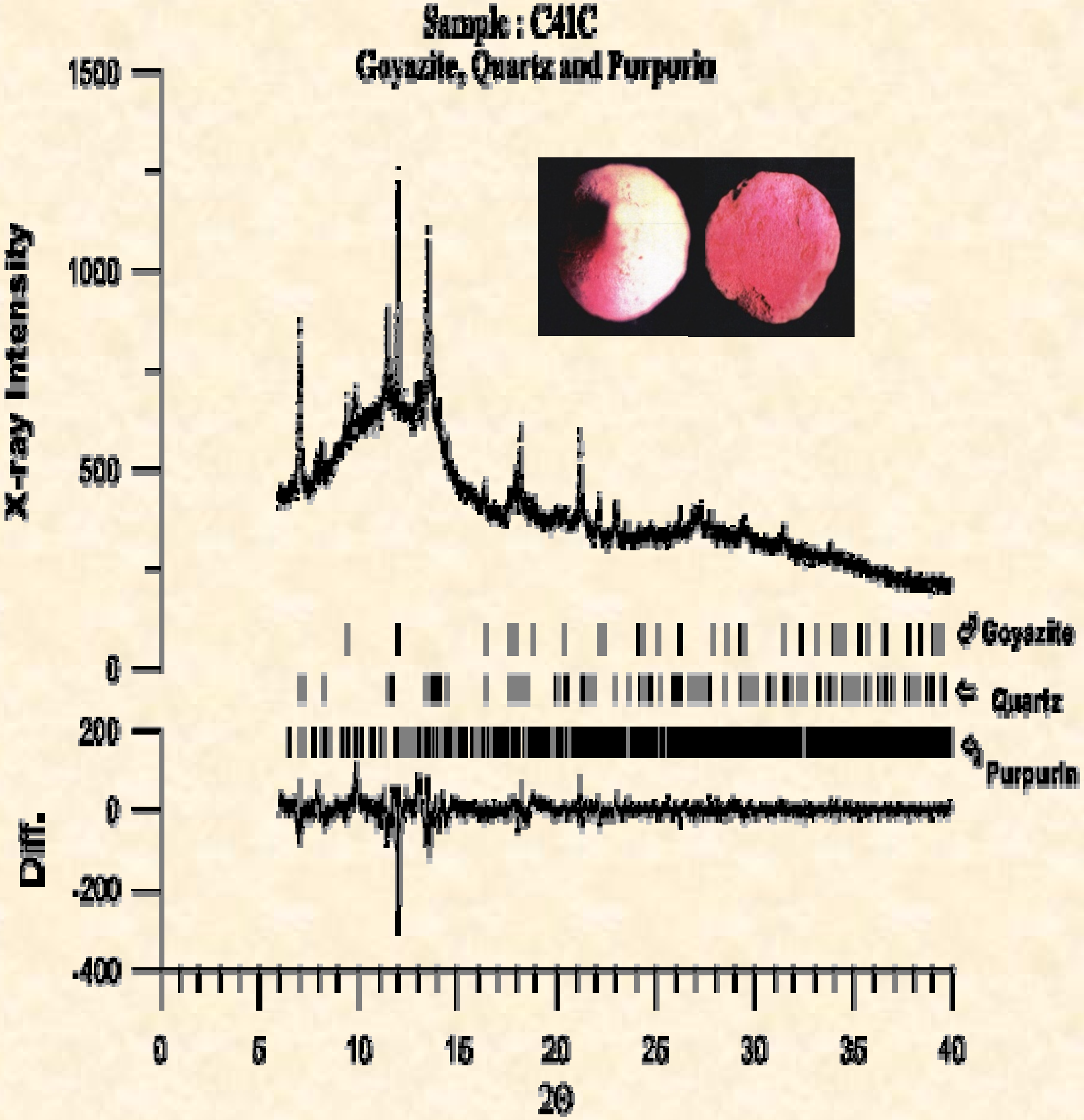
Natural antique colorants include red pigments such as cinnabar and ochre and pink pigments such as madder. These archaeological pigments have been used as ritual and cosmetic make-up and they are a material proof of handcraft activities and trade in the Mediterranean.

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

KERKOUANE ♥



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



Conclusions

Ten punic make-up samples were studied with SR-XRD using a 2D CDD 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.

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

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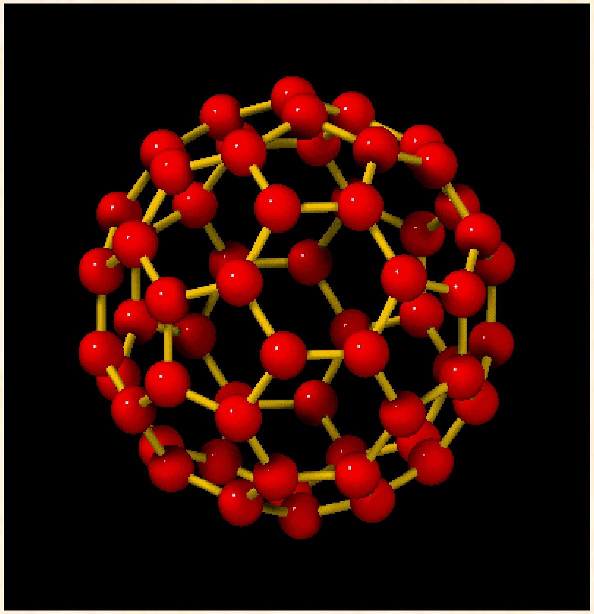
Buckminsterfullerene



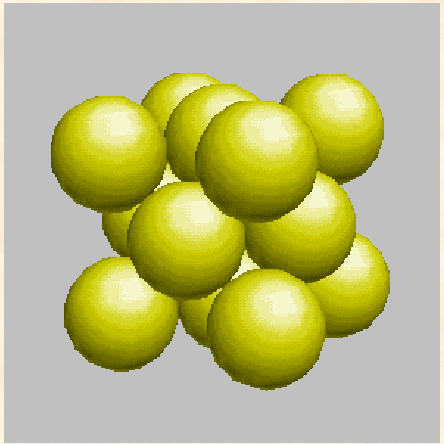
1985 : R.F Curt, H.W. Croto & R.E. Smalley discover C_{60}. They are awarded the Nobel prize in Chemistry in 1996.

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

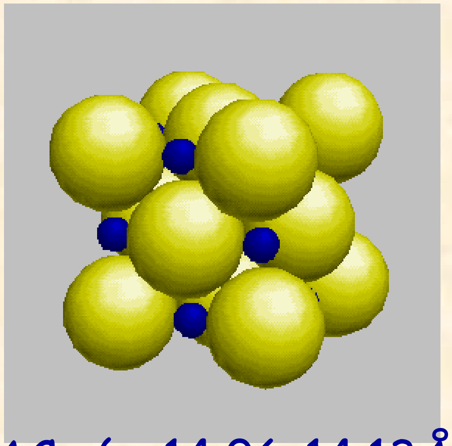
1991 : A group at AT&T Bell labs, finds superconductivity in alkali doped C_{60} with Tc=18K for K_3C_{60}. Later Tc=28K is observed for Rb_3C_{60}

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

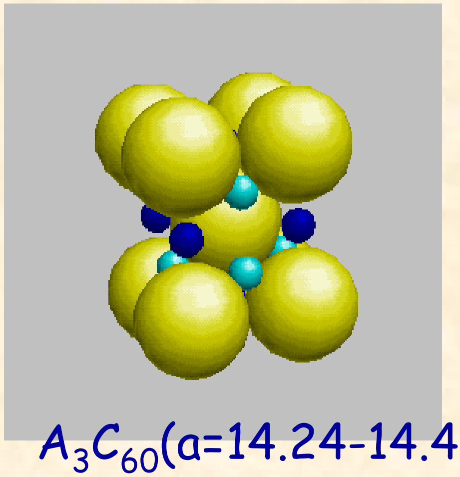
Alkali(K,Rb,Cs) doped C_{60}



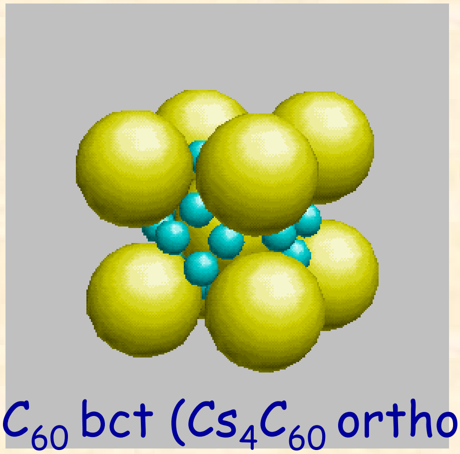
fcc C_{60}(a=14.17Å)



AC_{60}(a=14.06-14.13Å)
A in octahedral site

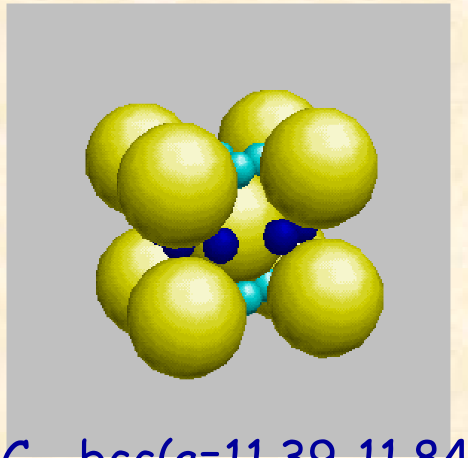


A_3C_{60}(a=14.24-14.44)
A in both tetrahedral
And octahedral site



2/3 filling
but a band
insulator !

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



A_6C_{60} bcc(a=11.39-11.84Å)

C_{60} base Superconductors:

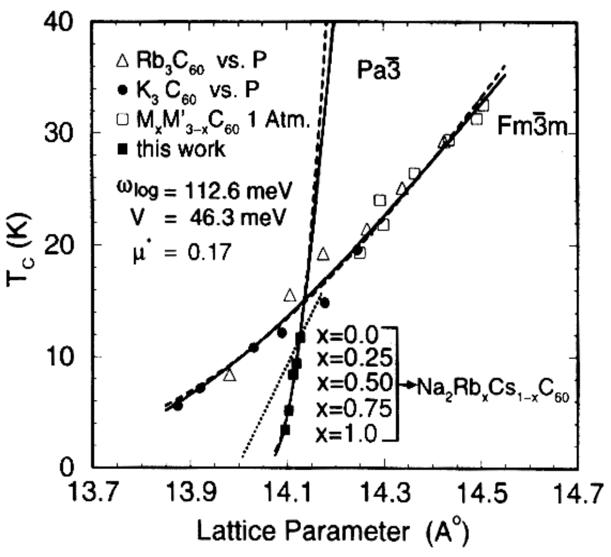
Changing the lattice parameter in Alkali doped fullerides, (either decreasing it with pressure or increasing it by substitution of a larger cation) increases the DOS $N(E_F)$.

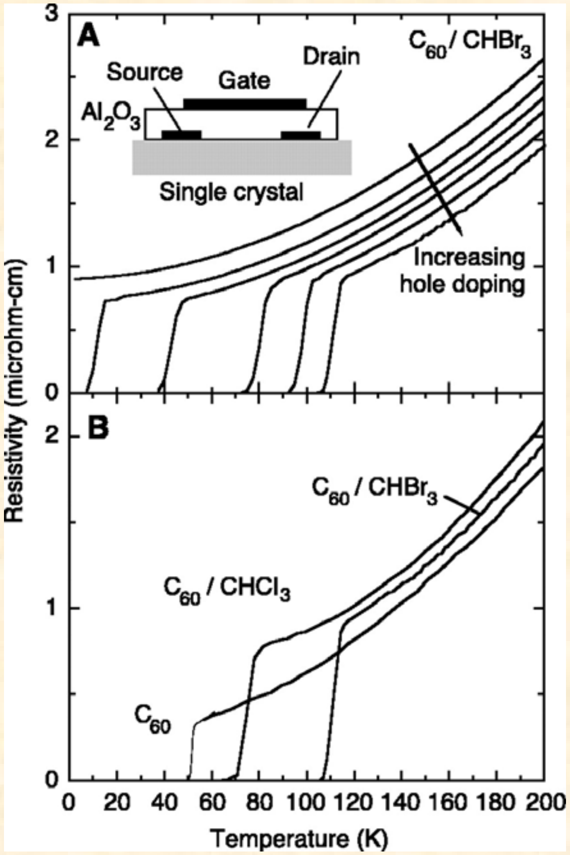
According to BCS theory

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

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

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

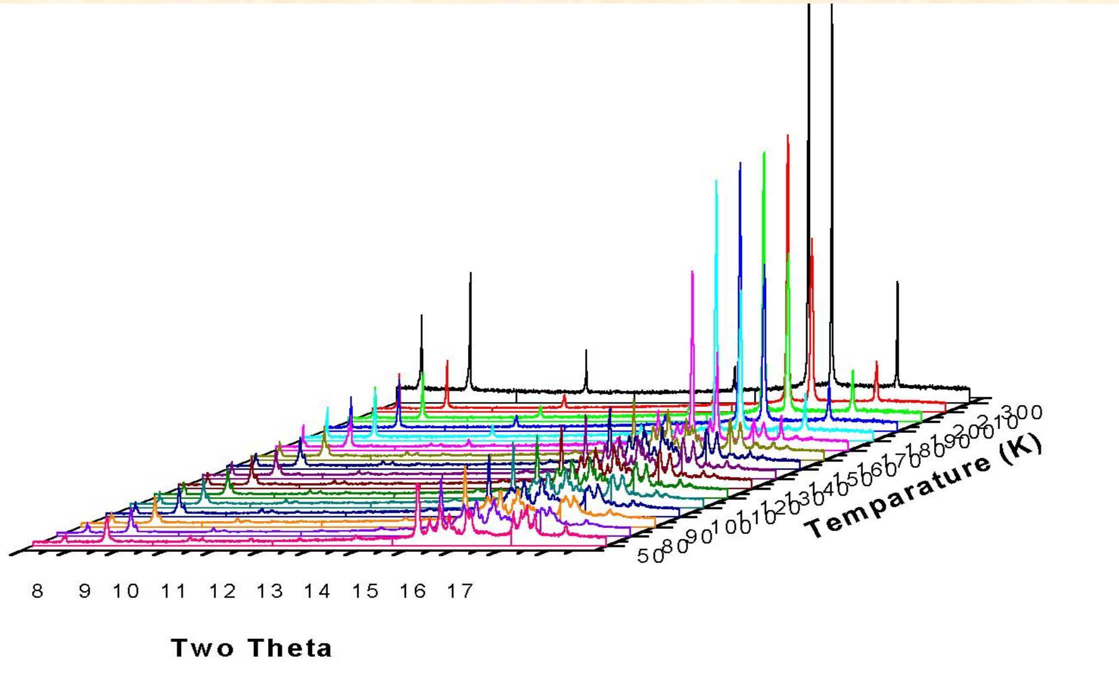




Interesting new superconductors, FET of organic materials (anthracine, pentacene, tetracene C_{60}. Record T_c=117K for C_{60} / $CHBr_3$. (Tc=80K for C60 / CHCl3)

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

What happens to the crystal structure as we decrease T ?



Hexagonal

↓

Monoclinic

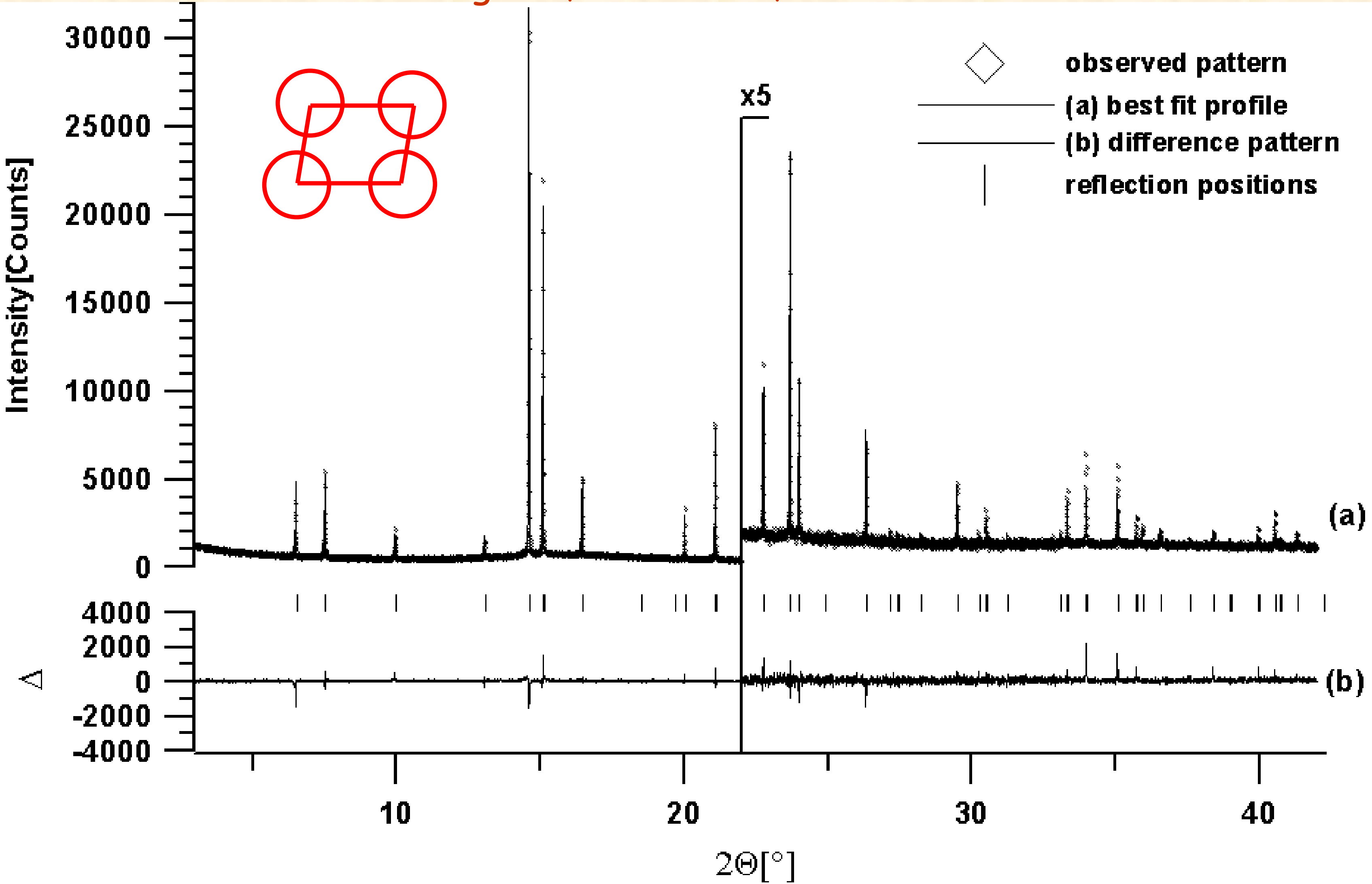
↓

Triclinic

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

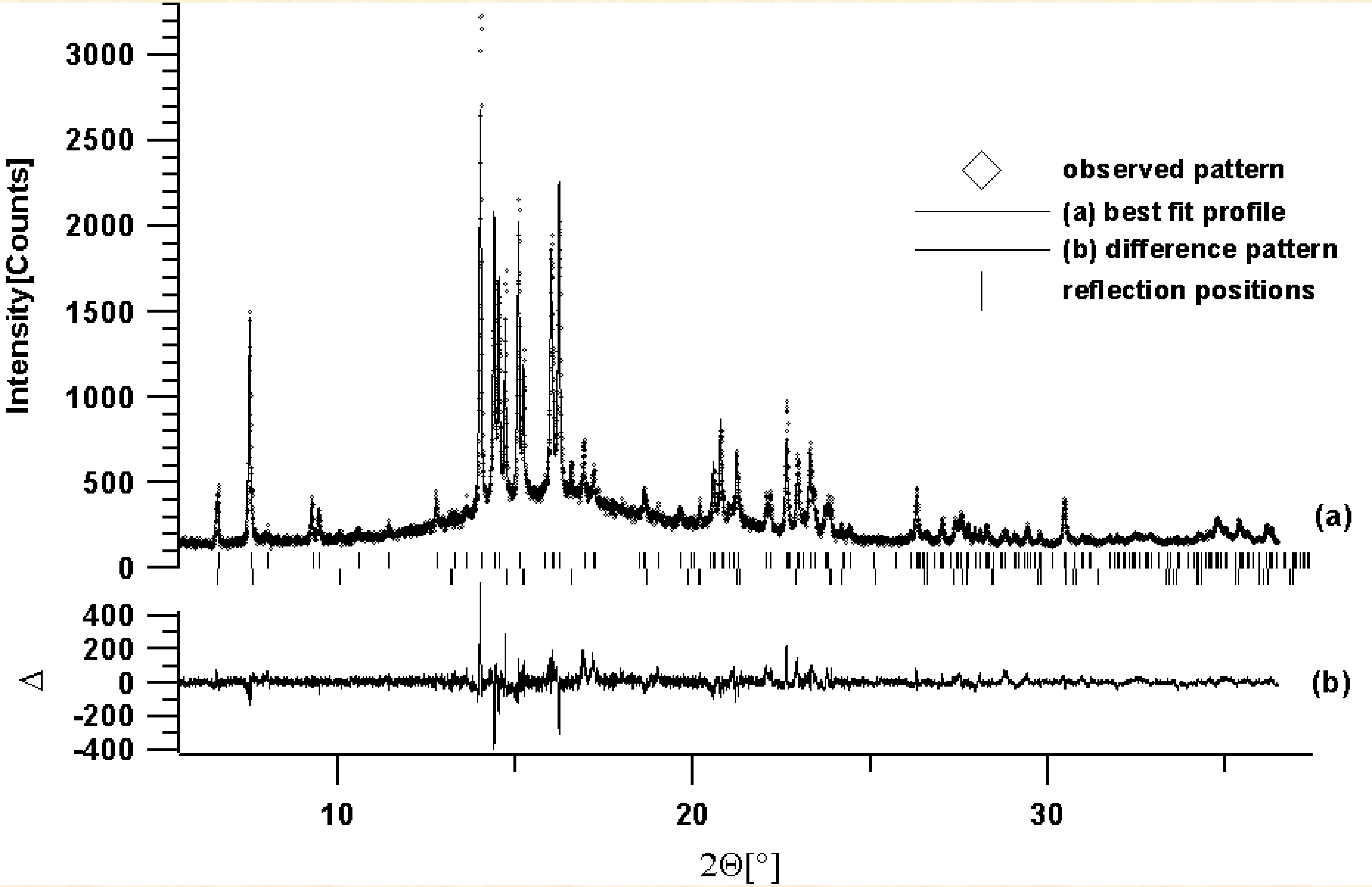
$C_{60} \cdot 2CHCl_3$ at room temp.

Hexagonal, *a* = 10.096, *c* = 10.101Å



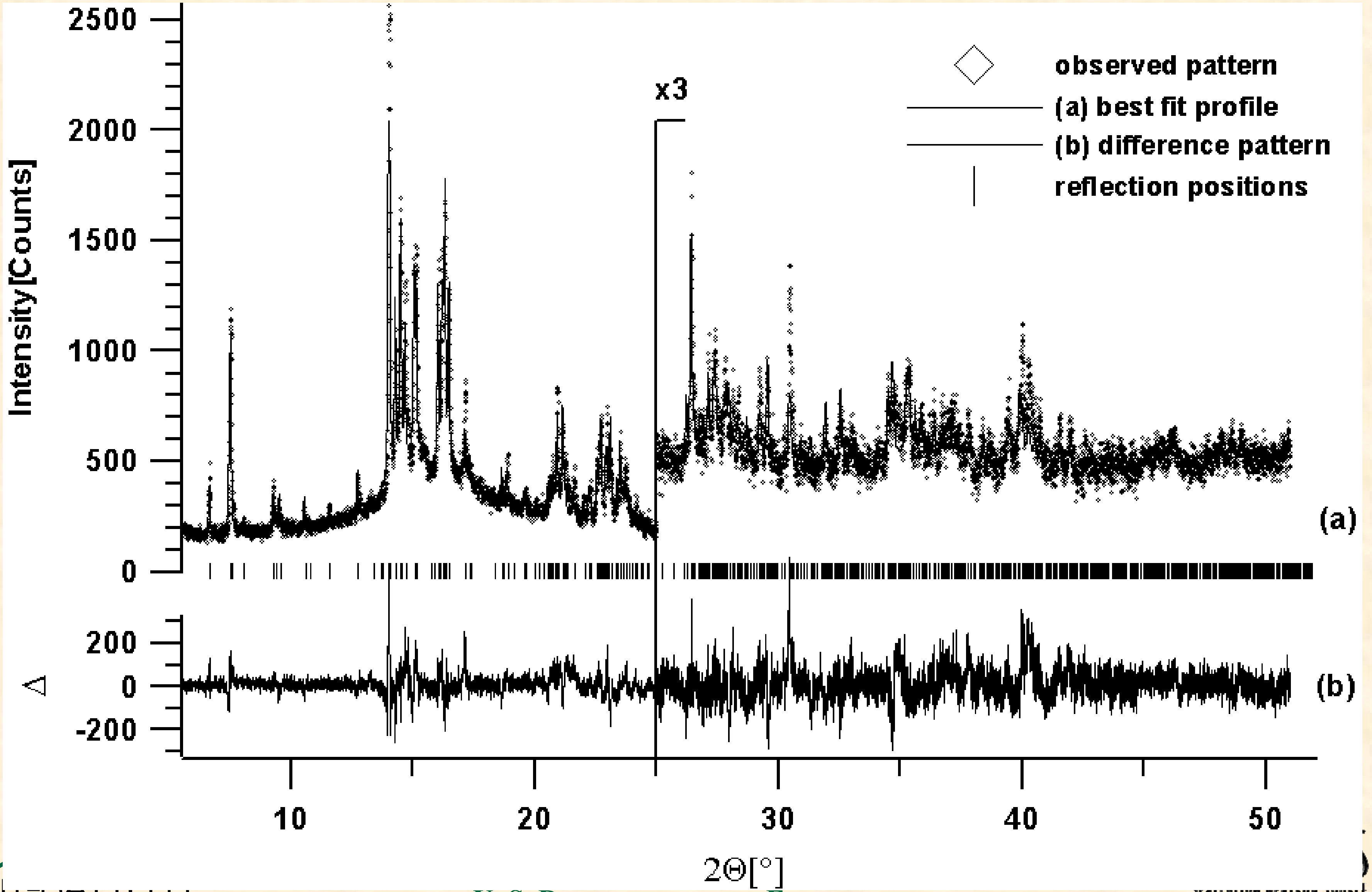
$C_{60} \cdot 2CHCl_3$ at 170K

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°)

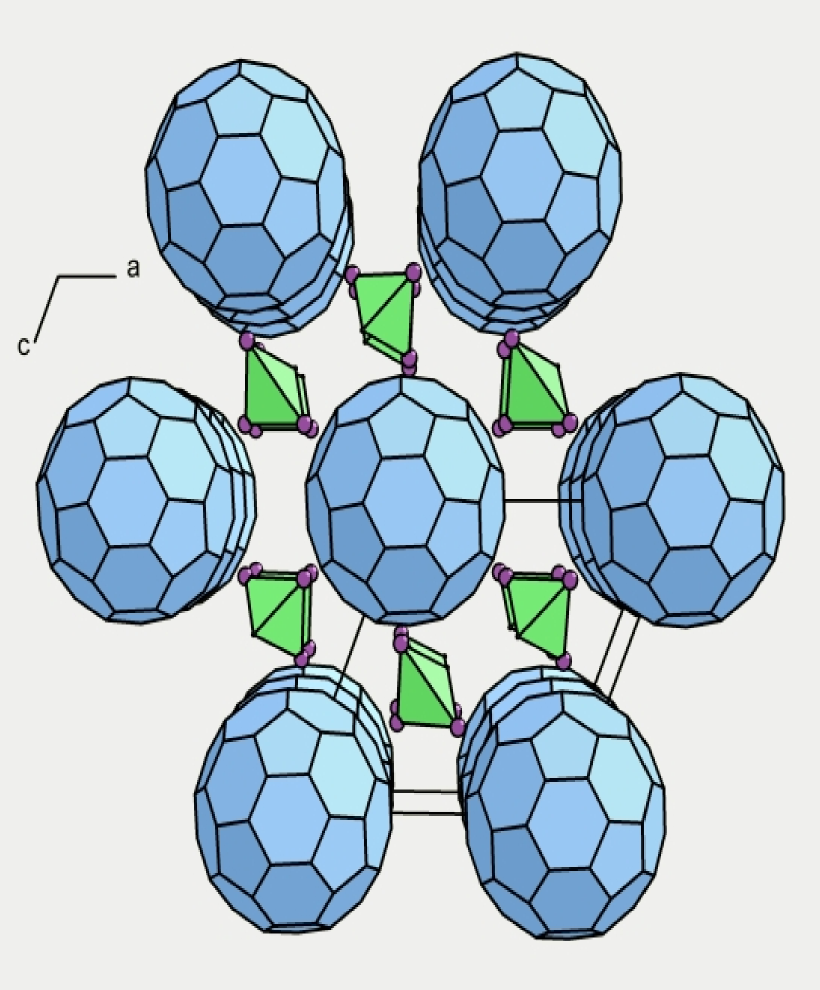


The crystal structure of C_{60} intercalated with $CHCl_3/CHBr_3$ is not **fcc** but **hcp**. More over when it is cooled it undergoes a phase transition and at ~150K they are converted into a fully order triclinic phase.

	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
$C_{60}.2CHCl_3$	P 6/mmm	10.09, 10.095	80	10.09
$C_{60}.2CHBr_3$	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

C60.2CHCl3 (P –1)



Interfullerene distances

$C_{60} \cdot 2CHCl_3$
In plane:
9.82, 9.84, 10.35
Between plane: 10.09

$C_{60} \cdot 2CHBr_3$
In plane:
9.90, 9.90, 10.50
Between plane: 10.34

cf. C_{60}: 9.93 (5K)
K_3C_{60}: 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}

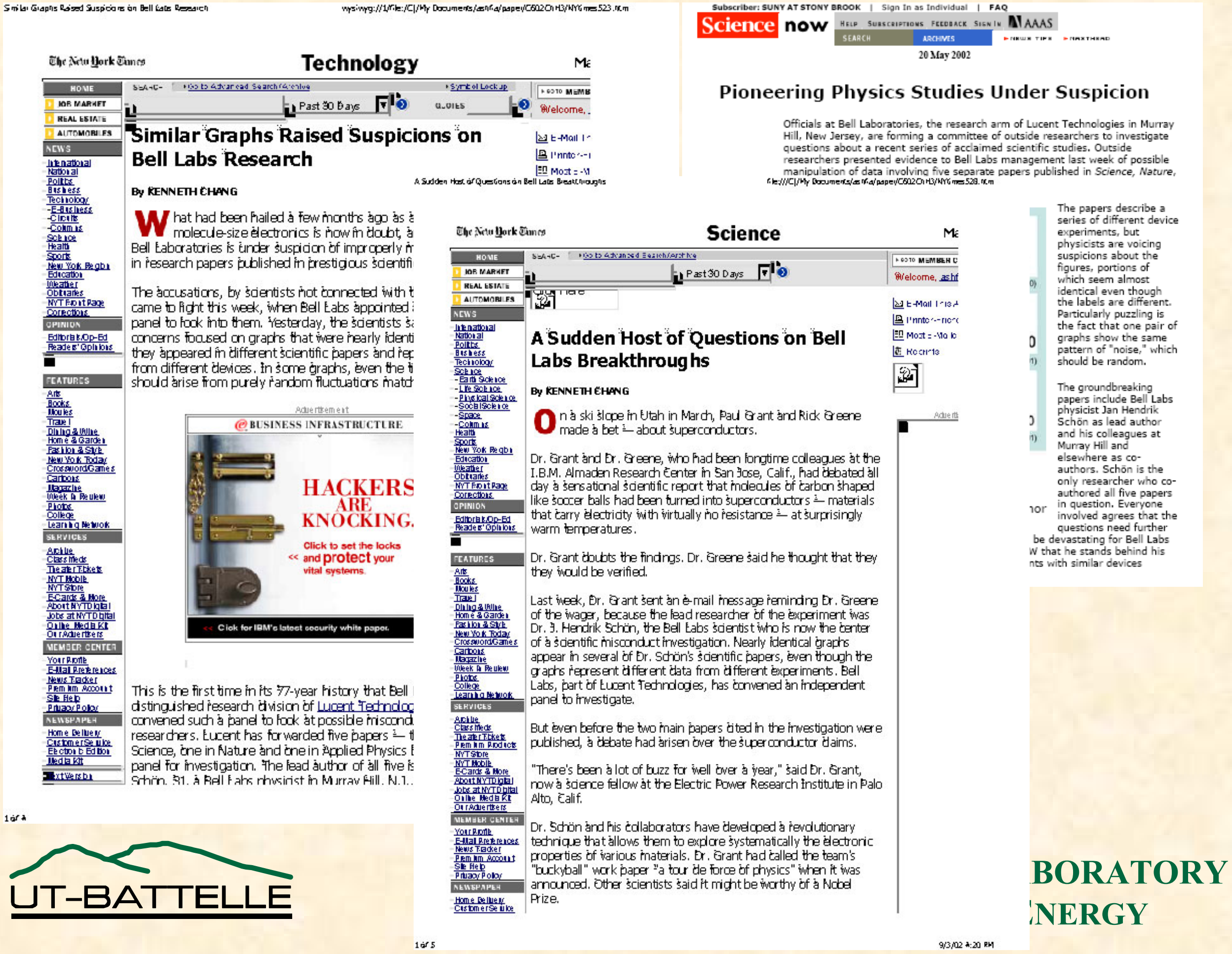
R. E. Dinnebier[1], O. Gunnarssson[1], H. Brumm[1], E. Koch[1], A. Huq[2],

P. W. Stephens[2], 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 Suspicions on Bell Labs Research

The New York Times — Technology

Similar Graphs Raised Suspicions on Bell Labs Research

By KENNETH CHANG

What had been hailed a few months ago as
molecule-size electronics is now in doubt,
Bell Laboratories is under suspicion of improperly
in research papers published in prestigious scientifi

The accusations, by scientists not connected with
came to light this week, when Bell Labs appointed
panel to look into them. Yesterday, the scientists
concerns focused on graphs that were nearly identi
they appeared in different scientific papers and rep
from different devices. In some graphs, even the
should arise from purely random fluctuations matc

This is the first time in its 77-year history that Bell
distinguished research division of Lucent Technolog
convened such a panel to look at possible miscond
researchers. Lucent has forwarded five papers — t
Science, one in Nature and one in Applied Physics
panel for investigation. The lead author of all five is
Schön, 31, a Bell Labs physicist in Murray Hill, N.J.

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Science now

20 May 2002

Pioneering Physics Studies Under Suspicion

Officials at Bell Laboratories, the research arm of Lucent Technologies in Murray Hill, New Jersey, are forming a committee of outside researchers to investigate questions about a recent series of acclaimed scientific studies. Outside researchers presented evidence to Bell Labs management last week of possible manipulation of data involving five separate papers published in *Science, Nature,*

The papers describe a series of different device experiments, but physicists are voicing suspicions about the figures, portions of which seem almost identical even though the labels are different. Particularly puzzling is the fact that one pair of graphs show the same pattern of "noise," which should be random.

The groundbreaking papers include Bell Labs physicist Jan Hendrik Schön as lead author and his colleagues at Murray Hill and elsewhere as co-authors. Schön is the only researcher who co-authored all five papers in question. Everyone involved agrees that the questions need further
be devastating for Bell Labs
W that he stands behind his
nts with similar devices

A Sudden Host of Questions on Bell Labs Breakthroughs

The New York Times — 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 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 "buckyball" work paper "a tour de force of physics" when it was announced. Other scientists said it might be worthy of a Nobel Prize.

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.

Neutron Powder Diffraction

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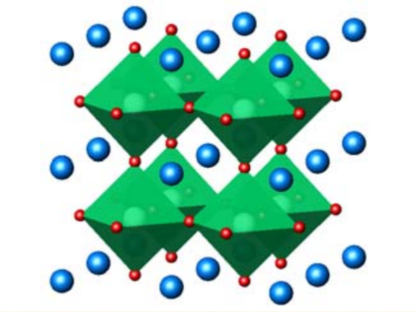
Why Neutrons ?

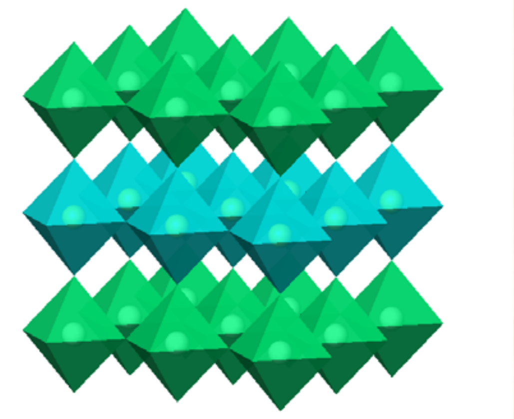
- Electrically neutral; penetrates centimeters of bulk material (allows non-destructive bulk analysis)
- Detects light atoms even in the presence of heavy atoms (organic crystallography) – H is special!
- Distinguishes atoms adjacent in Periodic table and even isotopes of the same element (changing scattering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

Ba_2CuWO_6: An Ordered Tetragonal Perovskite

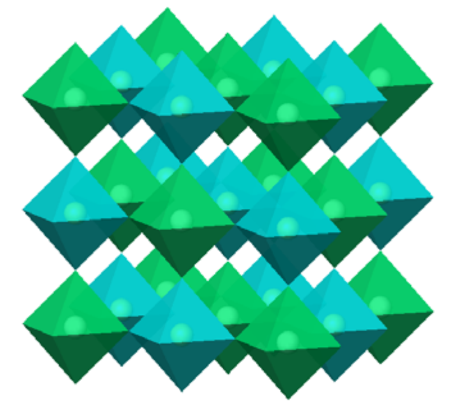


Simple cubic AMX_3 perovskite: a = 3.8045.

Double Perovskites $A_2MM'O_6$: Out of 3 possible ordering only 2 observed



Model #1: Ordered alternation of MO_6 and $M'O_6$octahedra in one direction, leading to formation of layered perovskite.



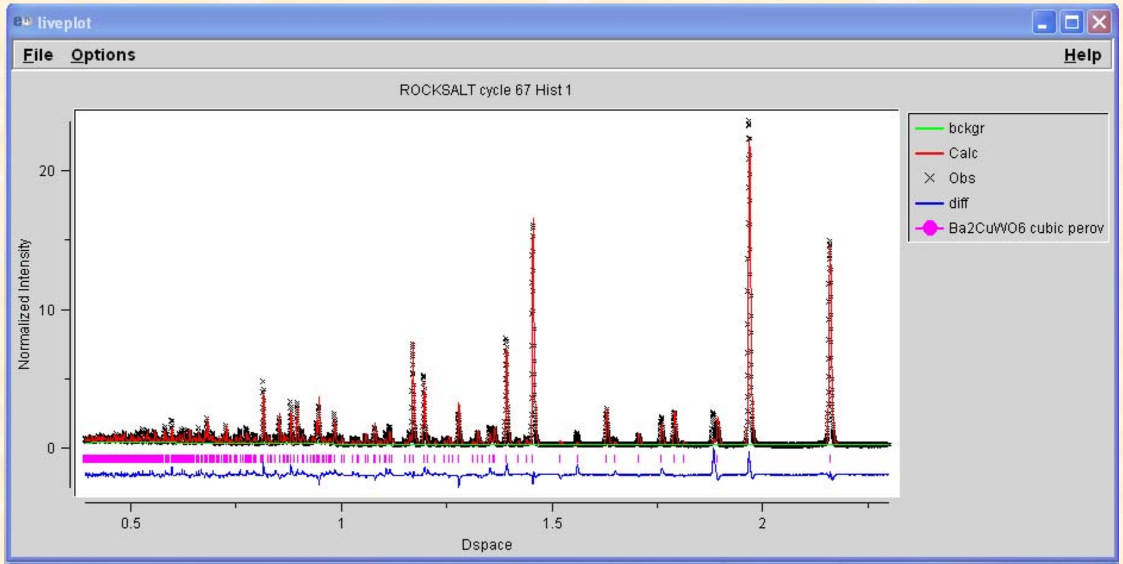
Model #2: Ordered alternation in the three directions of space, resulting in rock-salt ordered superstructure.

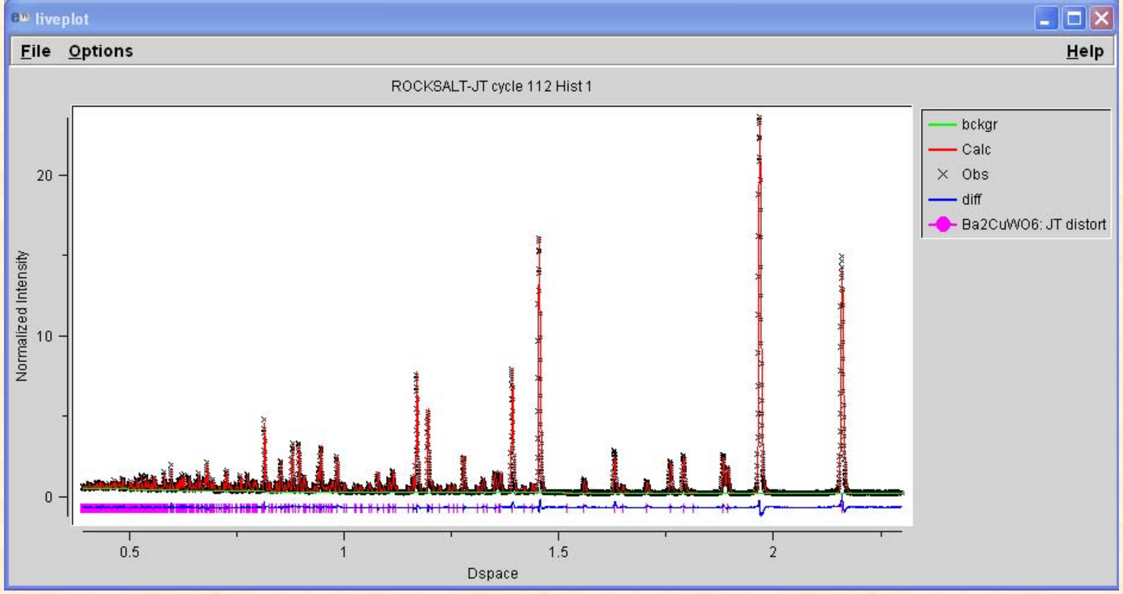
Model #1 – Layered Ordering:

Space Group	*P4/mmm*			
Lattice	*a* = 3.94 Å; *c* = 8.64 Å			
Atom	***x***	***y***	***z***	**Occupancy**
Ba	**¼**	**¼**	**½**	**1**
Cu	**0**	**0**	**0**	**1**
W	**0**	**0**	**0**	**1**
O(1)	**0**	**0**	**¼**	**1**
O(2)	**½**	**0**	**0**	**1**
O(3)	**½**	**0**	**½**	**1**

Model #2 – Rock Salt Type Ordering:

Space Group	*I4/m*			
Lattice	*a* = 5.57 Å; *c* = 8.64 Å			
Atom	***x***	***y***	***z***	**Occupancy**
Ba	**0**	**½**	**¼**	**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**





Jahn Teller Distortion?

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

Recall Cu^{2+} electronic configuration $(t_{2g})^6(e_g)^3$: So in fact CuO_6 octahedra are elongated along the c axis. The e_g orbital is split into

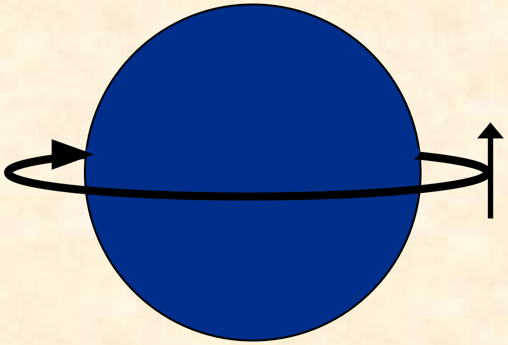
$(d_{x^2-y^2}$ and $d^2_{z^2})$

Magnetism & powder diffraction

Introduction to Magnetism

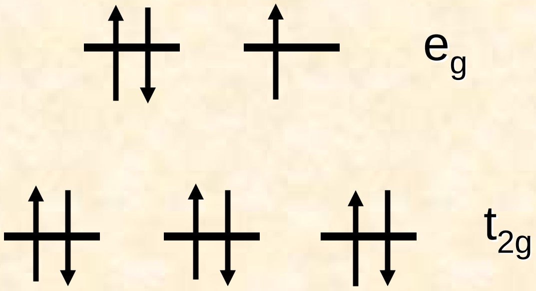
- Origin of magnetism – electrons.
  - Electrons have a magnetic moment (dipole; μ_S). Magnetic moments arise from two properties of an electron:

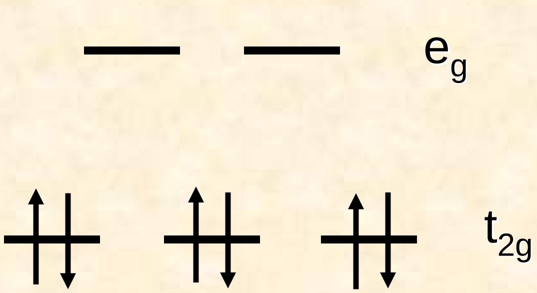
1. Motion around the nucleus (gyromagnetic ratio)
2. Total spin quantum number ($S = \Sigma s$; $s = \pm\frac{1}{2}$)



  - Dipole unit – Bohr magnetons (μ_B). 1 $\mu_B = 9.2742\times10^{-24}$ 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)

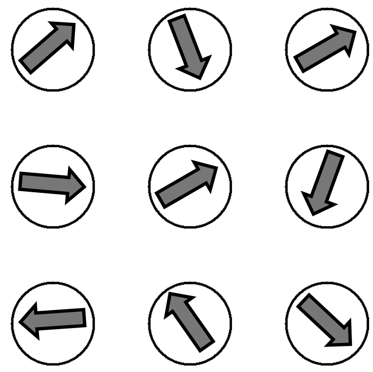
d^6 ion

$S = 0$

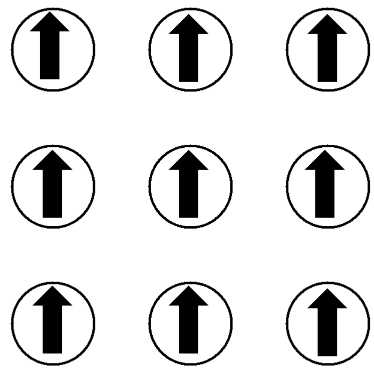
No magnetic moment

Diamagnetic ion

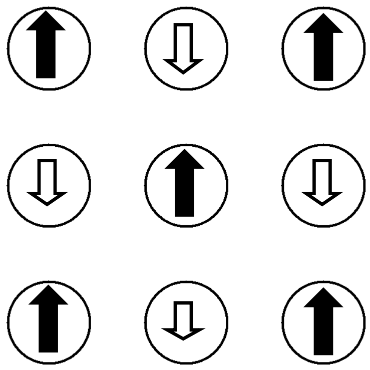
Magnetic Ordering Types



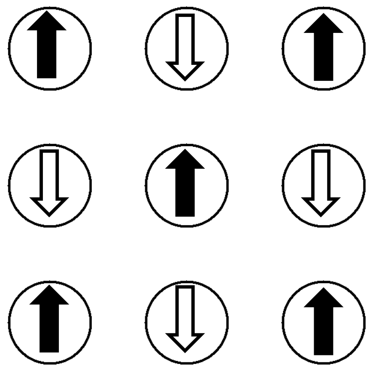
Paramagnetic (PM)



Ferromagnetic (FM)



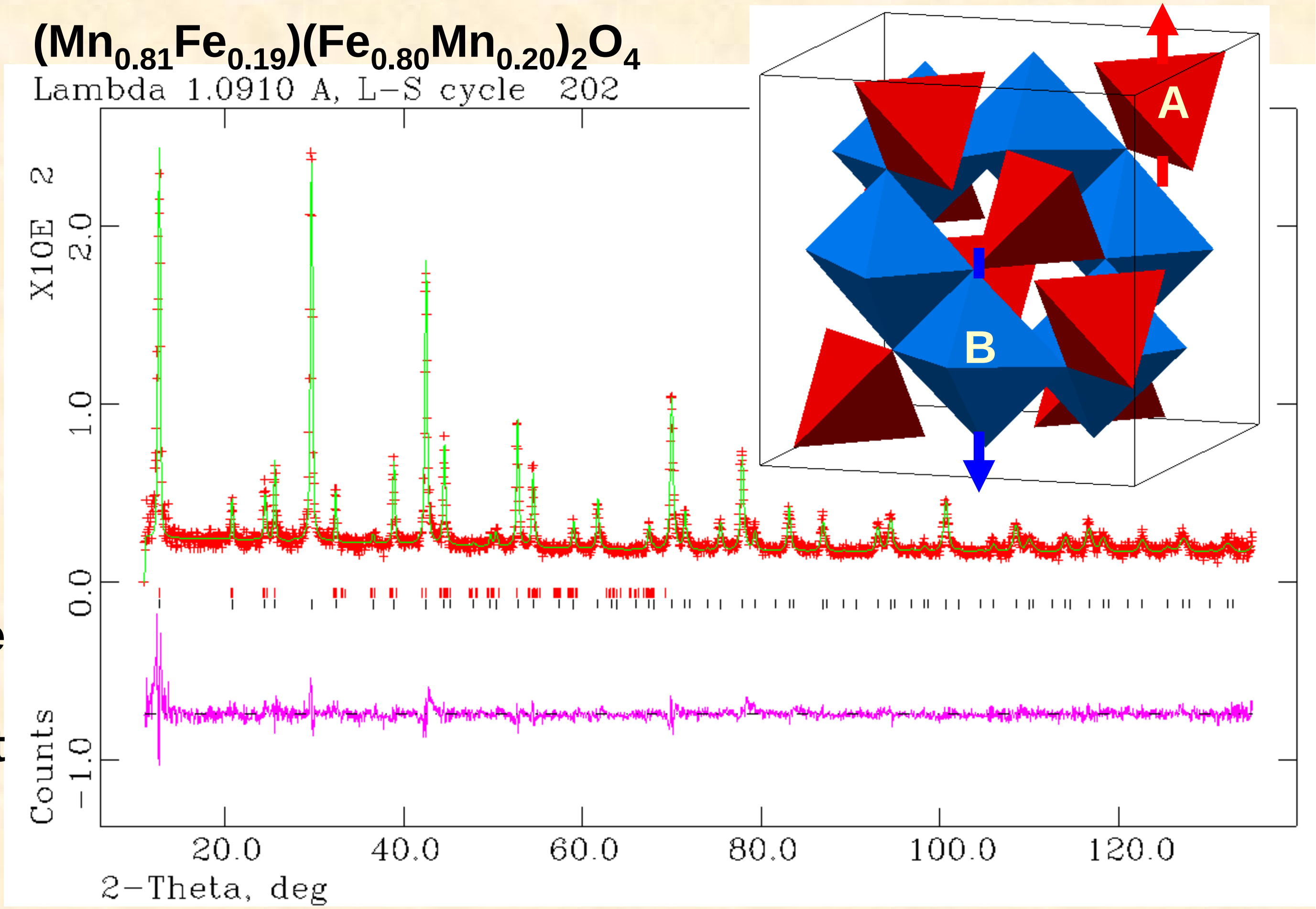
Ferrimagnetic



Antiferromagnetic (AFM)

FERRIMAGNETIC AB_2O_4 SPINEL STRUCTURE

$(Mn_{0.81}Fe_{0.19})(Fe_{0.80}Mn_{0.20})_2O_4$

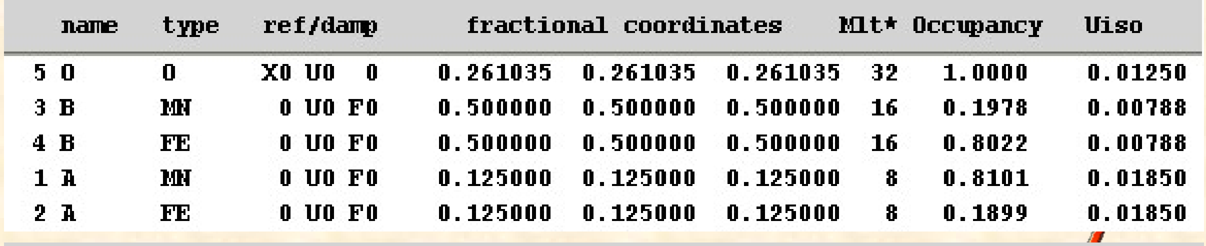


2-phase refinement nuclear + magnetic structure

Results give

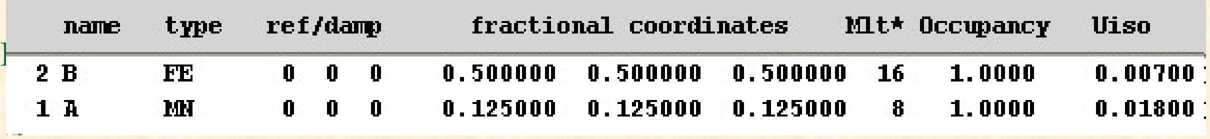
- **lattice parameter**
- **oxygen position**
- **distribution of Mn/Fe on T and O sites**
- **atomic displacement parameters**
- **magnetic moments on the T and O sites (e.g., -2.9 and 2.0 μ_B)**

nuclear phase



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

magnetic phase



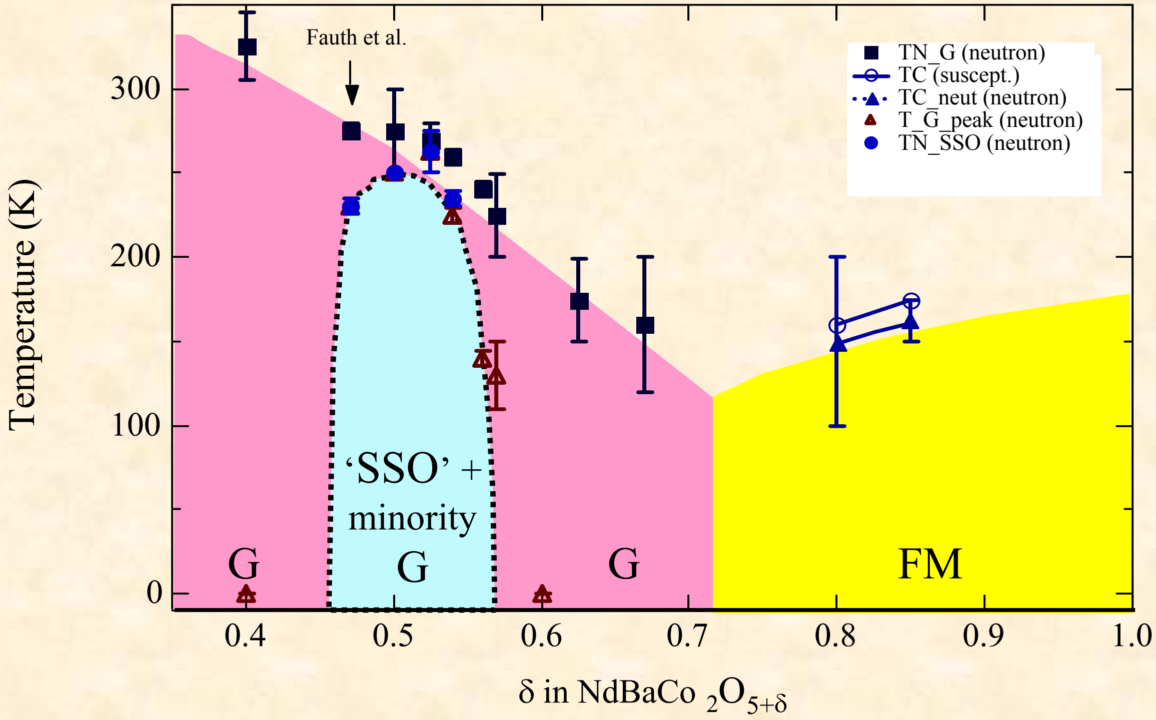
name	type	ref/damp			fractional coordinates			Mlt*	Occupancy	Uiso
2 B	FE	0	0	0	0.500000	0.500000	0.500000	16	1.0000	0.00700
1 A	MN	0	0	0	0.125000	0.125000	0.125000	8	1.0000	0.01800

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

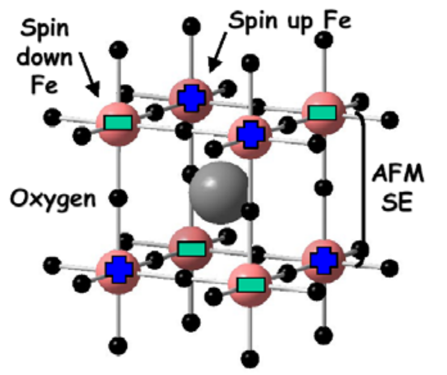
UT-BATTELLE

Magnetic Ordering: Oxygen-deficient A-site Layered Perovskite $NdBaCo_2O_{5+\delta}$

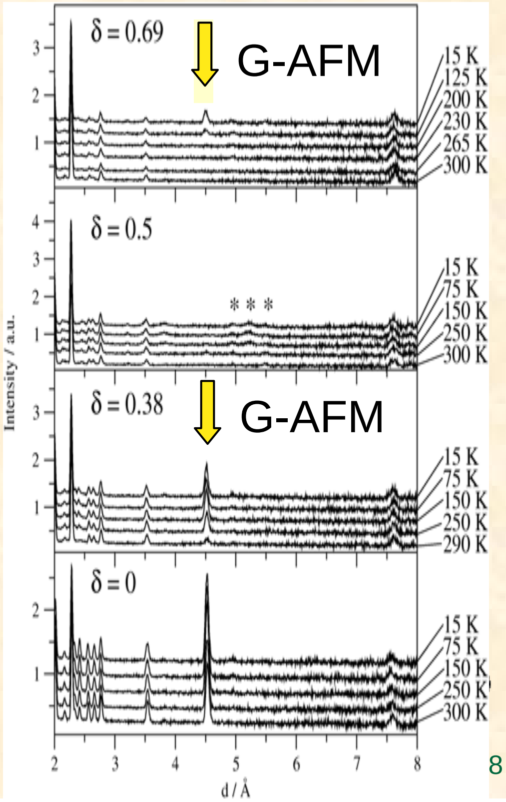
Burley et. al. J. Solid State. Chem. 170, 339 (2003)



Magnetic phase diagram



G type AF ordering in a simple perovskite



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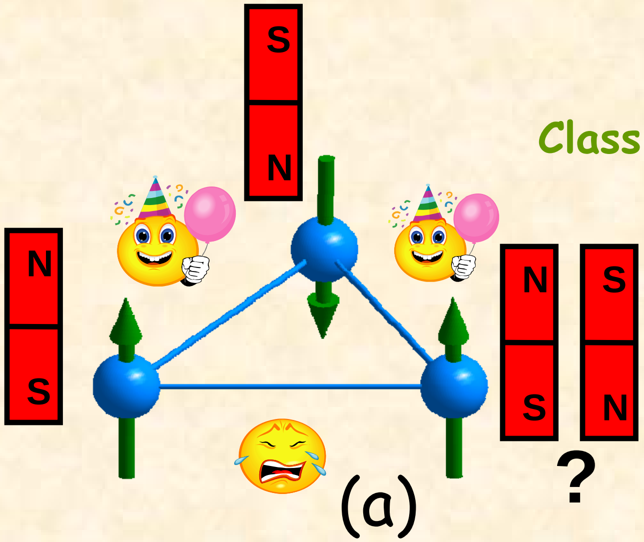
Very often life is not so simple and one has to use both X-rays and Neutrons to get to the right picture

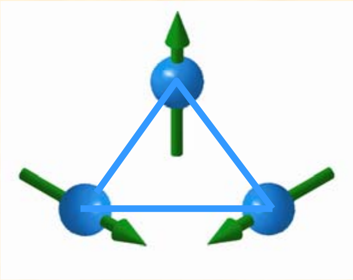
Frustrated Lattice:



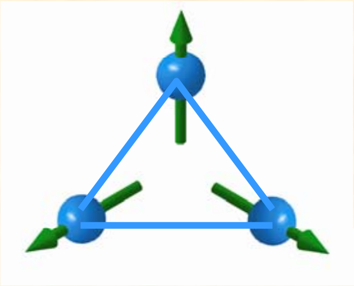
(a)

Classic example of geometric frustration (Triangular lattice)

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



(b)

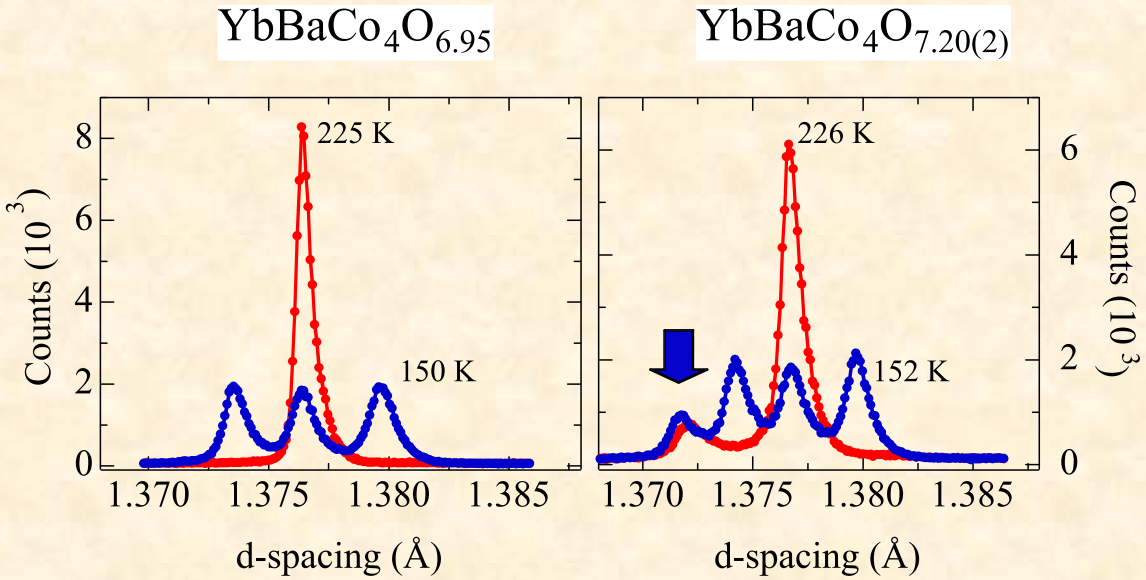


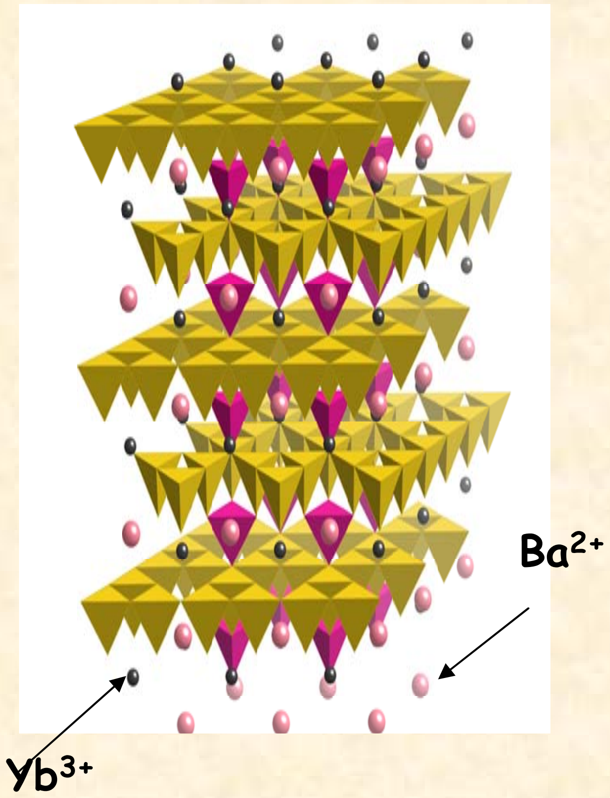
(c)

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

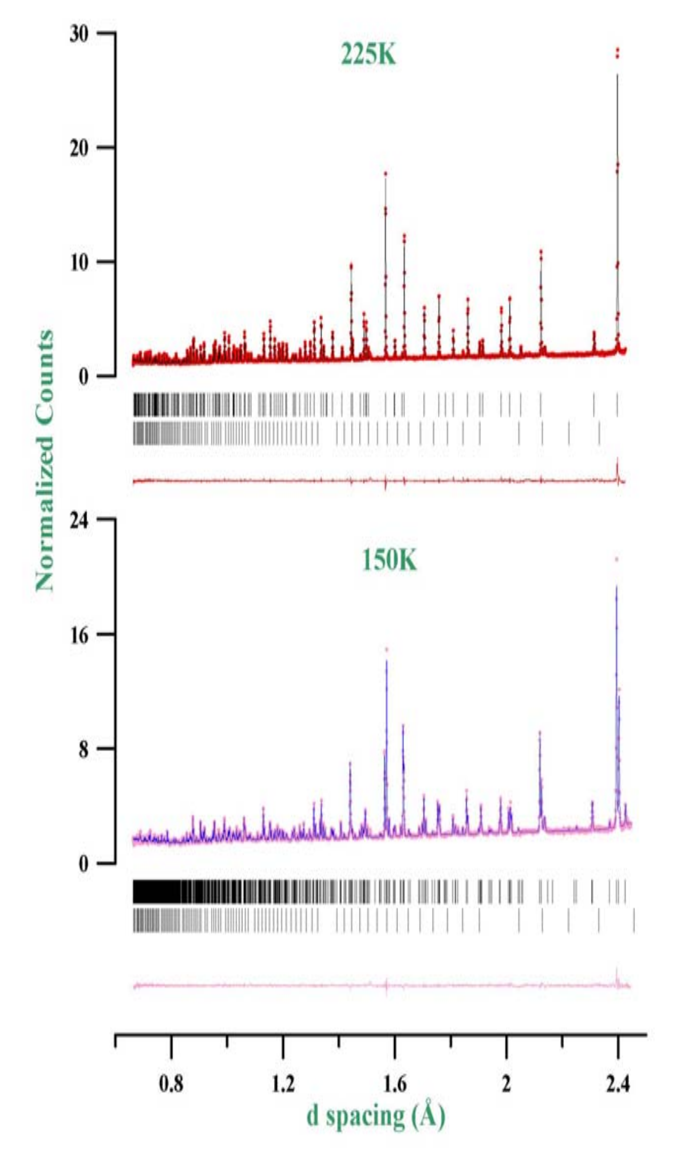
Mechanisms lifting the degeneracy often determine the phase diagrams and other physical properties of these systems.

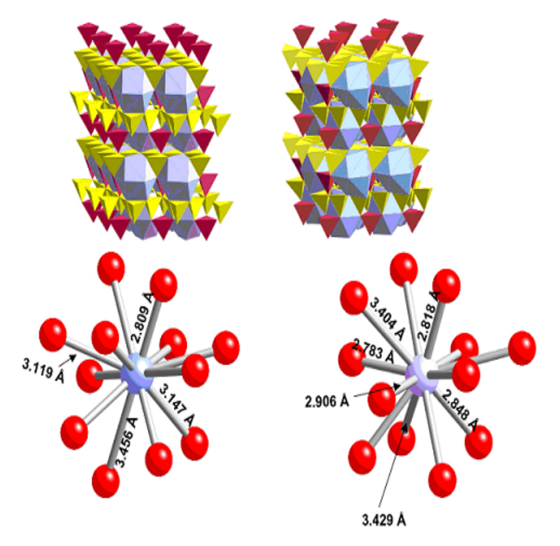
$RBaCo_4O_{7\pm\delta}$: Phase transition





Comparison of synchrotron data collected above and below structural phase transition in a single phase $YbBaCo_4O_{6.95}$ and biphasic $YbBaCo_4O_{7.2}$ 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.

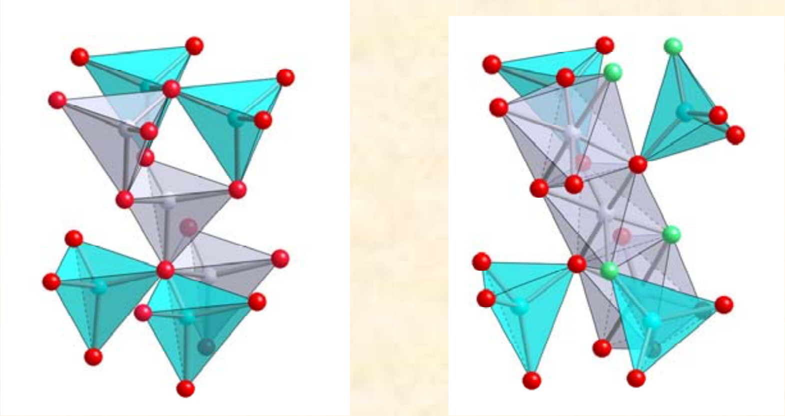




Above the transition temperature Ba^{2+} is severely under bonded, giving rise to a structural instability.
Bond Valence Calculation show convincingly that the O environment does not satisfy the bonding requirements of the Ba^{2+} ion.
To compensate for this under bonding Yb 114 undergoes a structural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

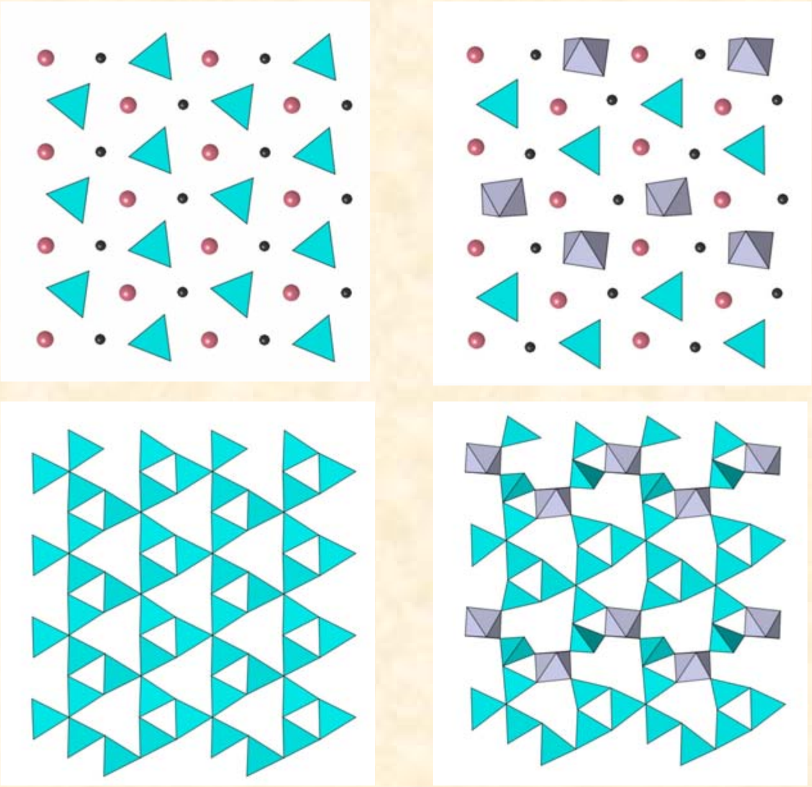
When R = Y and x=1 ($YBaCo_4O_8$)

We find that a large orthorhombic *Pbc2₁* superstructure is formed with lattice parameters $2a_o x b_o x c_o$. 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 $YBaCo_4O_7$(left) & $YBaCo_4O_8$ (right) showing drastic displacement of some oxygen atoms and the location of two new ones.

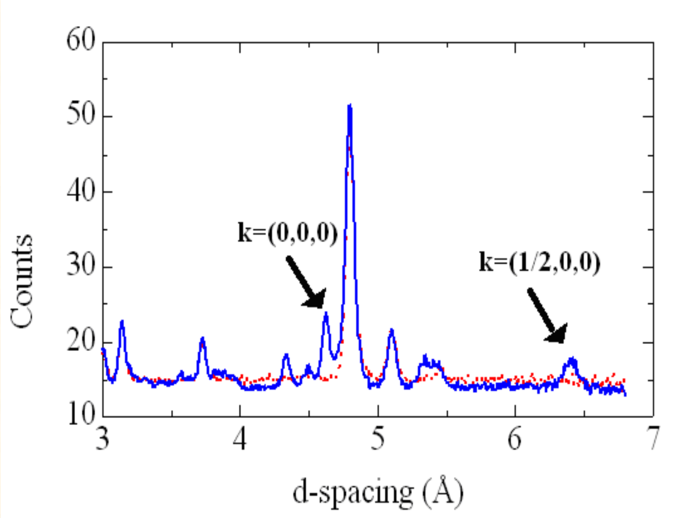
Charge ordering base on BVS



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.

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 $Co^{3+}:Co^{2+}=5:1$, however we find this number to be 4:1.

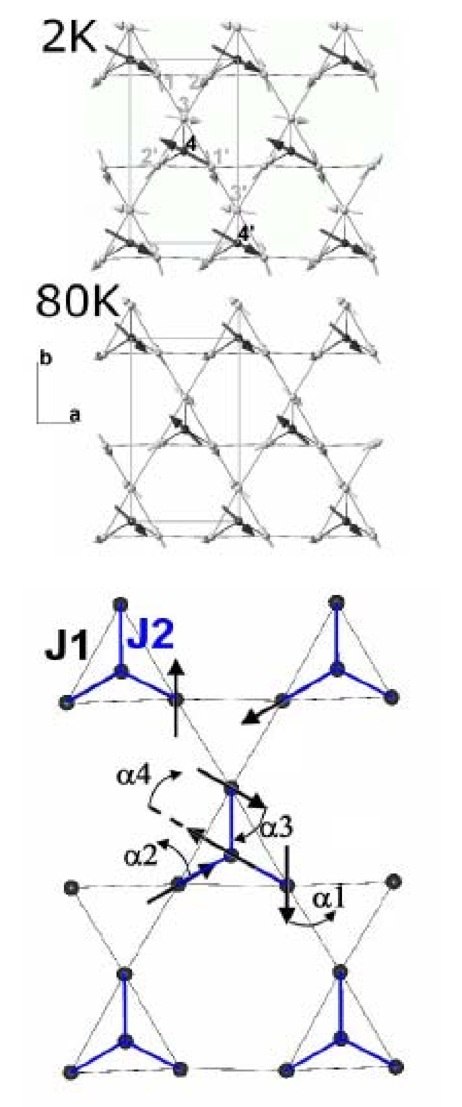
What about magnetism?



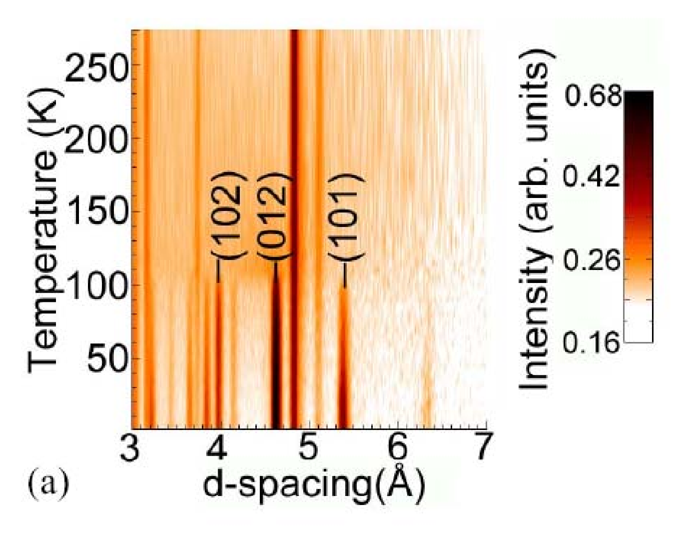
Portion of the Neutron data collected at GEM at 90K(dashed) and 1.7K (solid). Magnetic scattering due to AF order in the 1.7K pattern is marked by two observed propagation vectors.

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.

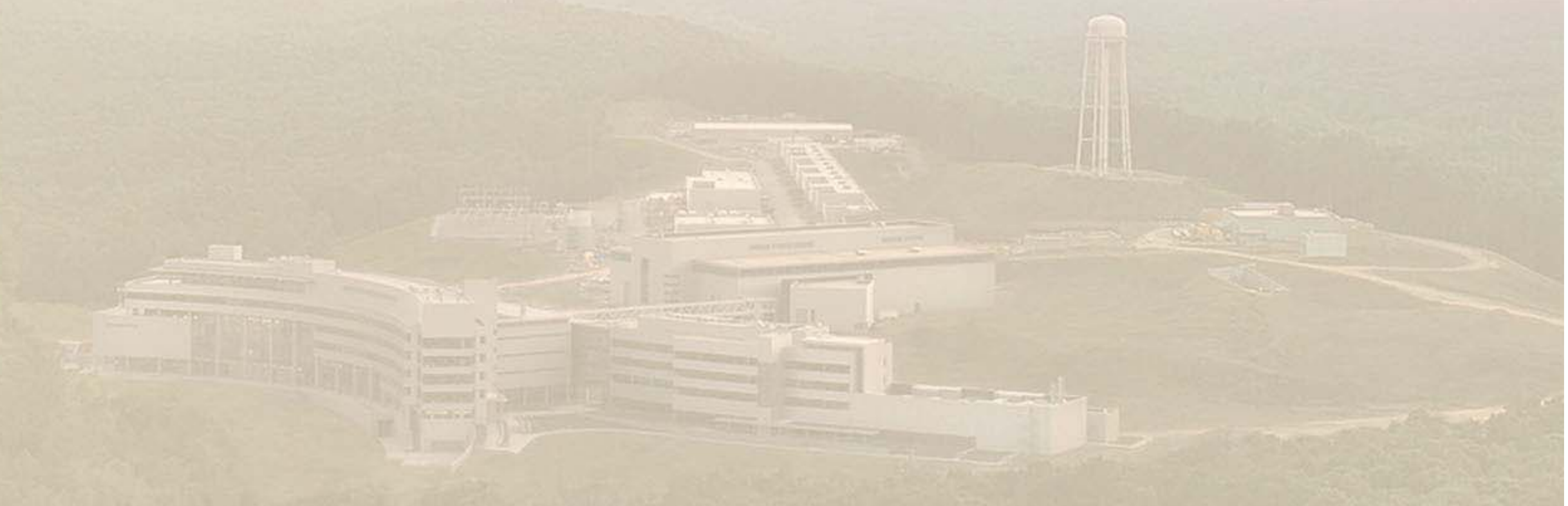
Non magnetic rare earth R=Y



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



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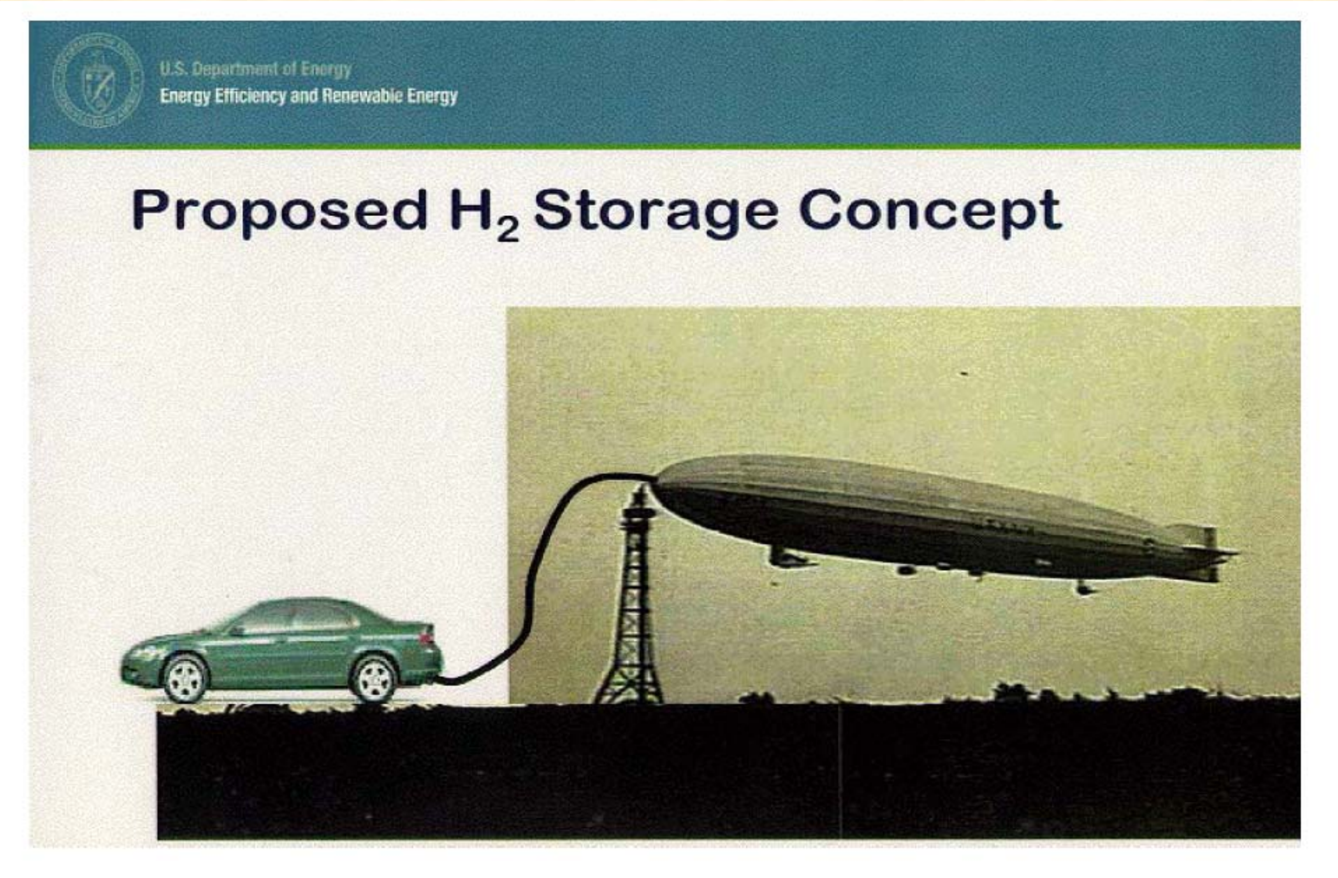
What will your 'Fuel Tank' for this car look like ?

At STP 1kg of H_2 gas has a volume of $11m^3$.

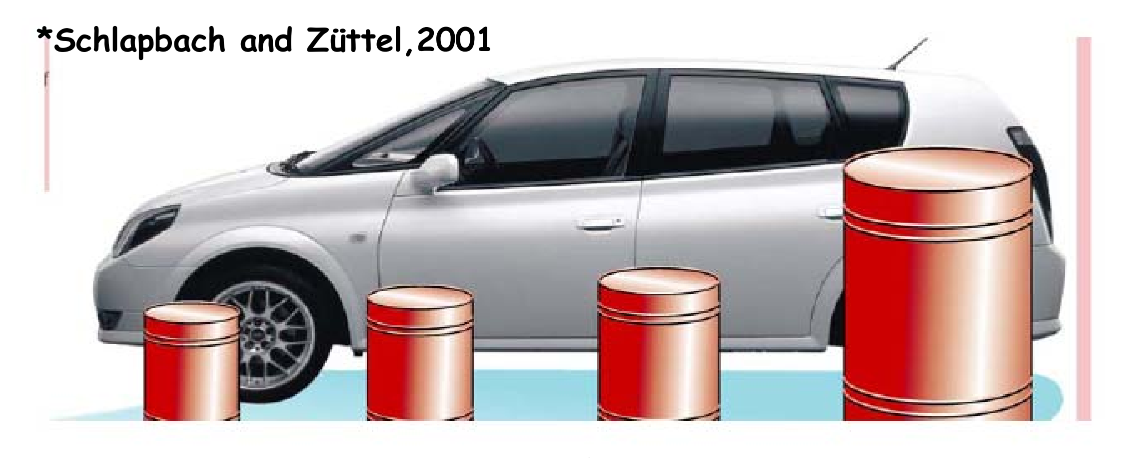


U.S. Department of Energy
Energy Efficiency and Renewable Energy

Proposed H_2 Storage Concept

Picture
borrowed from
J. Eckert
Presentation

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



*Schlapbach and Züttel,2001

4kg H_2 in Mg_2NiH_4 $LaNi_5H_6$ liquid H_2 H_2 at 100atm

	2005	2010	2015
Capacity (wt % H_2):	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 (H_2/min):	0.5	1.5	2.0

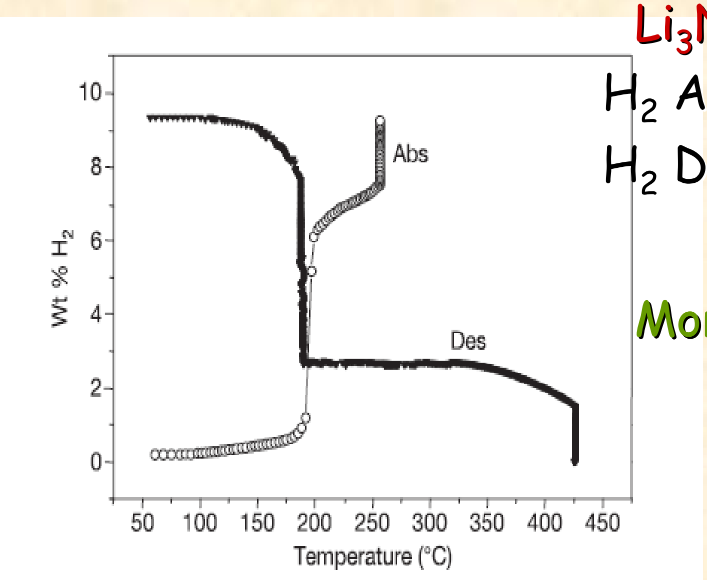
LOW COST!!!!

Li_3N : Hydrogen Storage Candidate

Chen et. al: (Nature Nov 2002)

Li Imide **Li Amide**

$Li_3N + 2H_2 \longleftrightarrow Li_2NH + LiH + H_2 \longleftrightarrow LiNH_2 + 2LiH$



H_2 Absorption 9.3 wt% gain at 255°C

H_2 Desorption 6.3 wt% at 200°C + 3wt % above 320°C

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

1. $Li_3N + 2H_2 \longrightarrow Li_2NH + LiH + H_2$
2. $Li_2NH + LiH + H_2 \longleftrightarrow LiNH_2 + 2LiH$

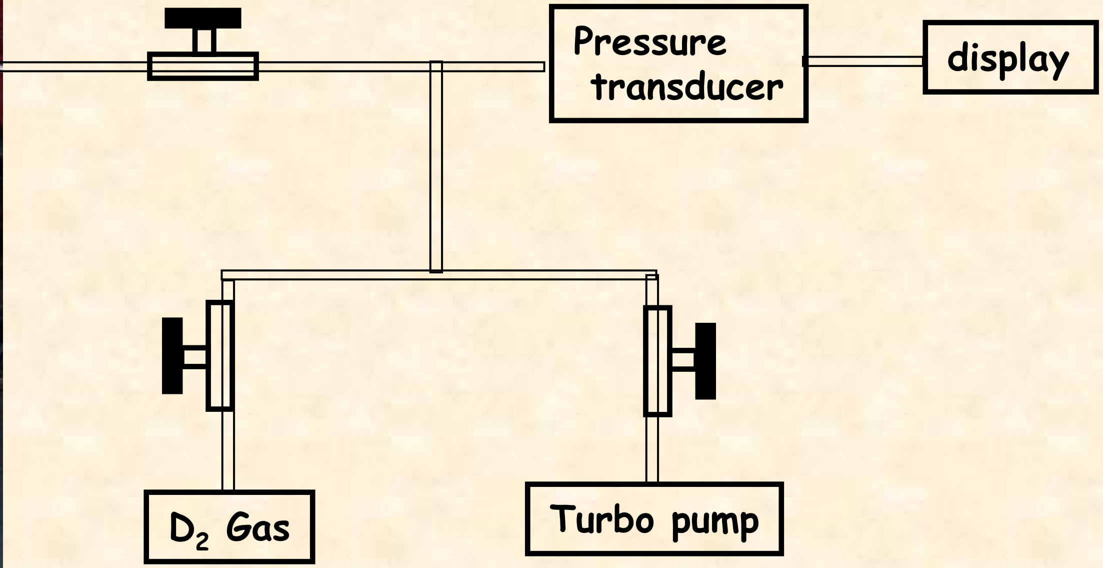
(~5.2% cyclable H_2)

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

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

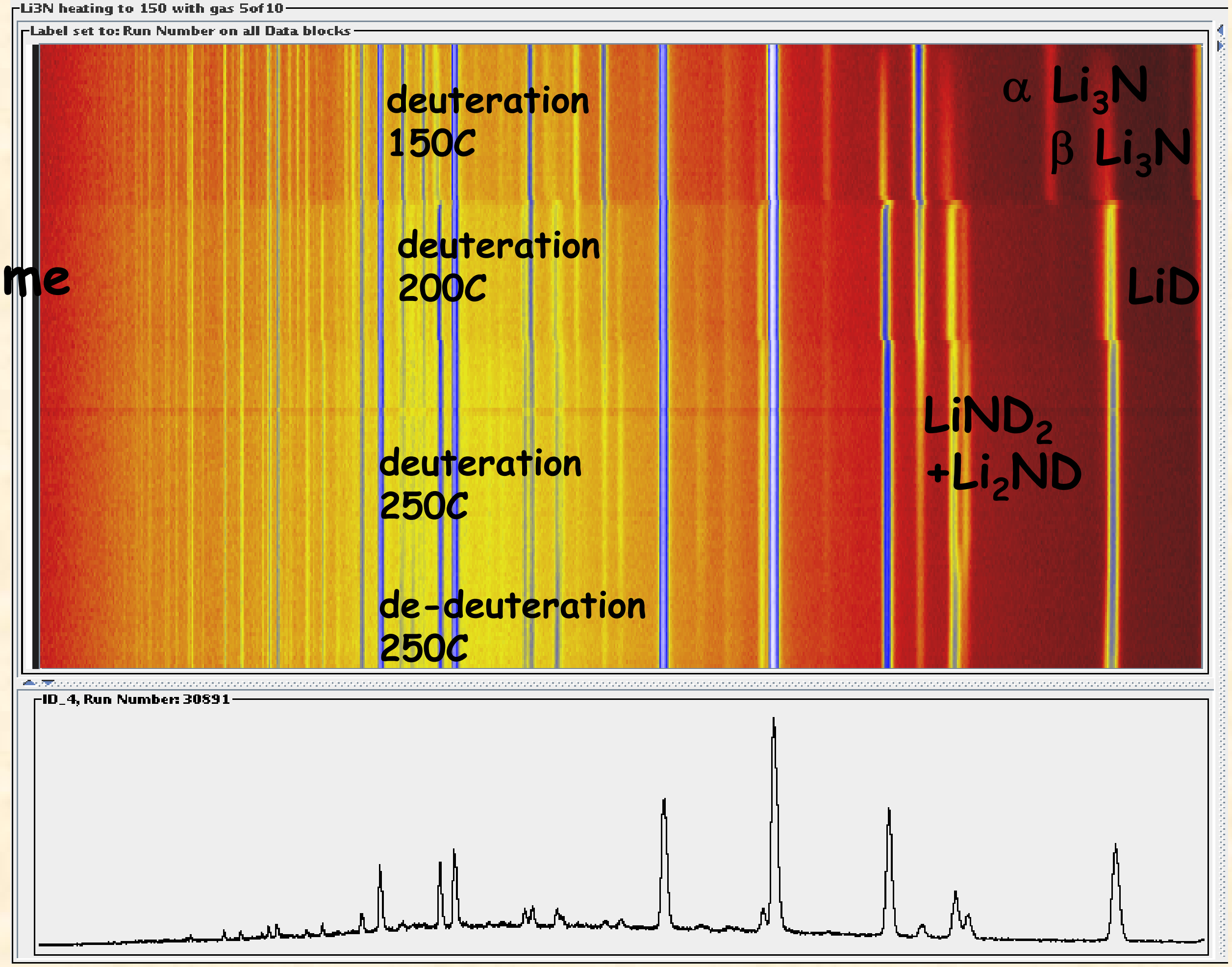
Experimental Setup for in-situ measurements

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.



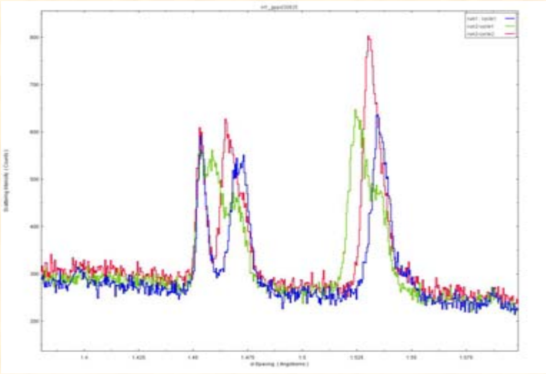
Inconel cell: 100 bar and 500C

In situ Deuteration & De-deuteration

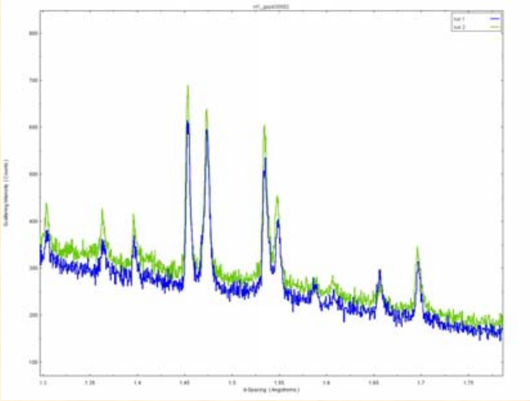


Time

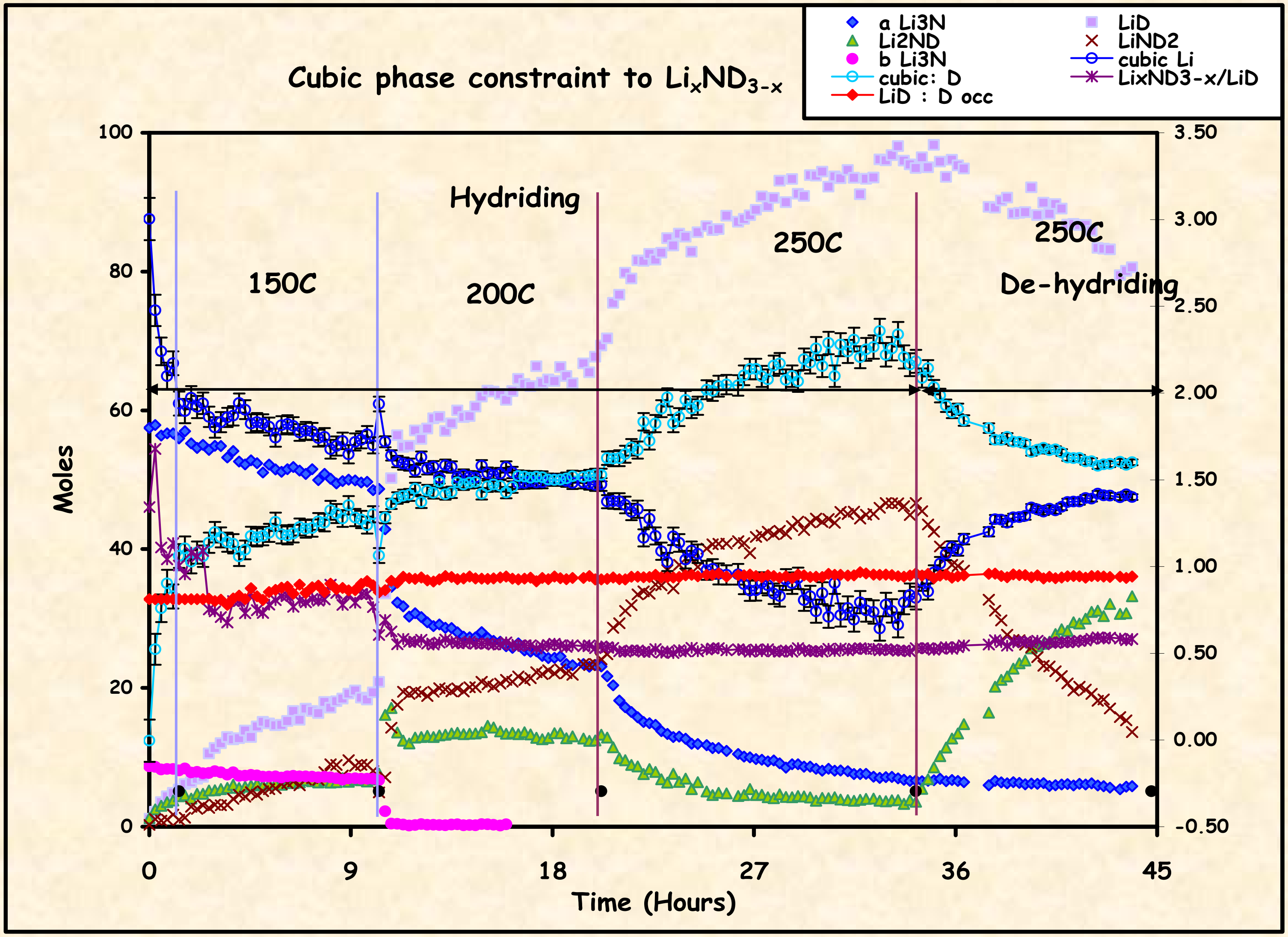
d spacing



34h: deuteration
10h: pumping



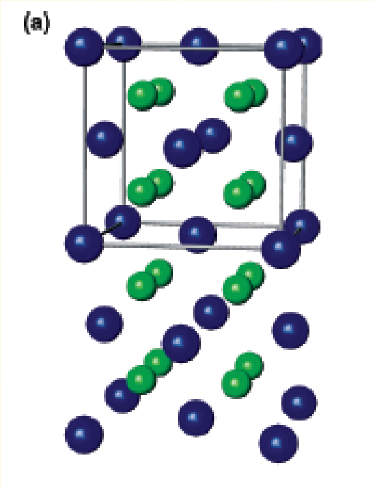
Moles of Phases Present

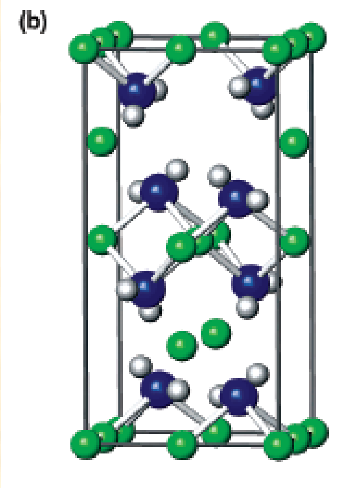


Imide – Amide : Structural relationship

David et. al., JACS 129,1594

Anti fluoride
cubic imide



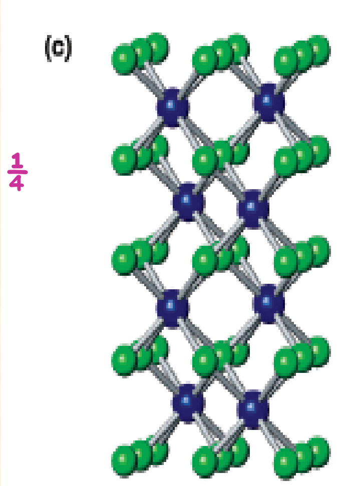


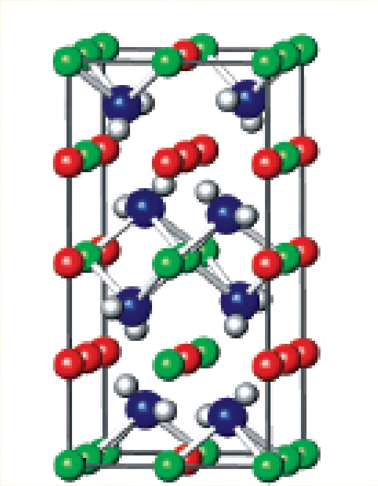
Tetragonal
amide

blue: N
green: Li
grey: H

shift origin ¼ ¼ ¼

a × a × 2a,





show unoccupied
Li sites by red

UT-BATTELLE

Ab-initio Structure Solution from Powder Diffraction

SNS
SPALLATION NEUTRON SOURCE

06-G00576/arn

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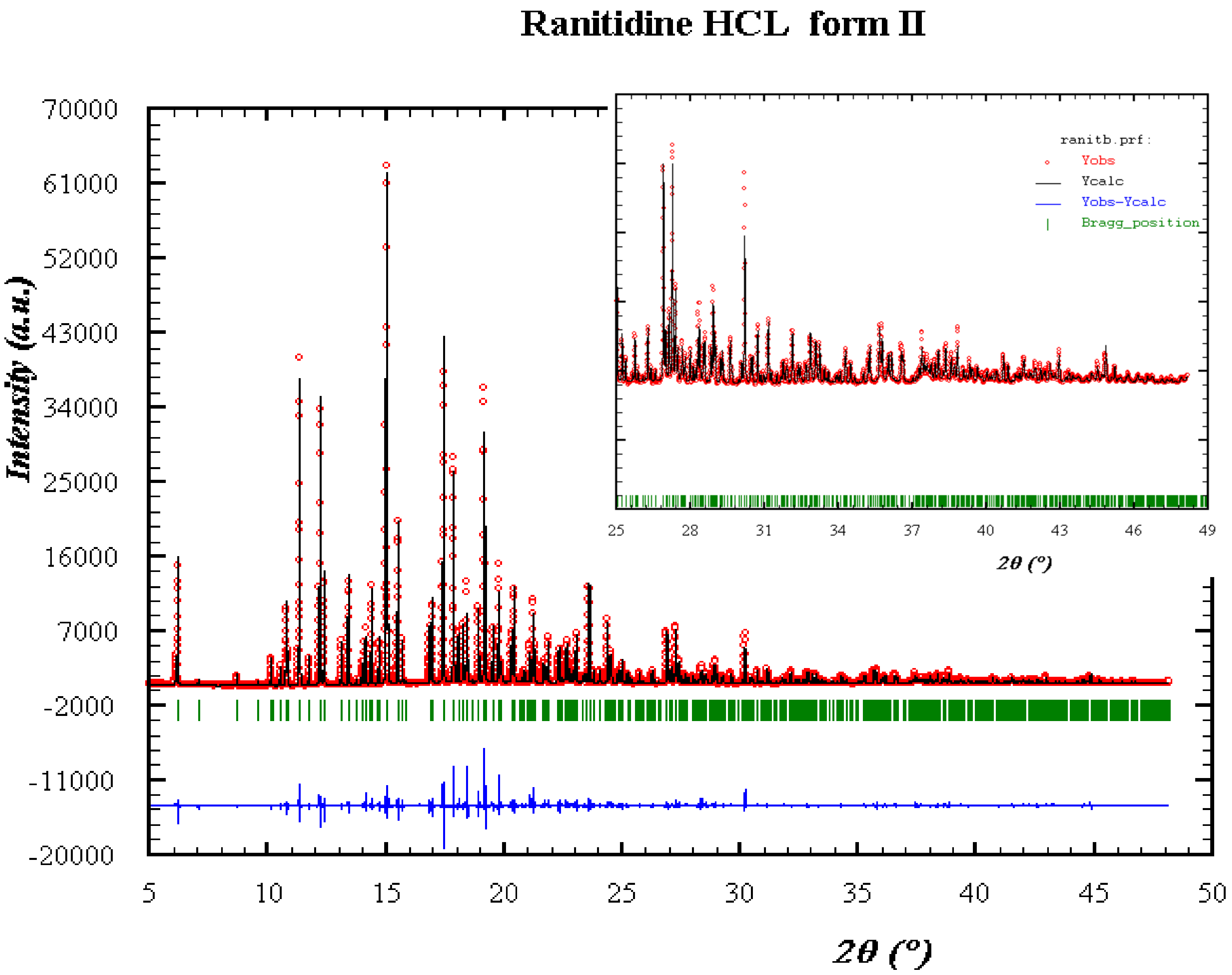
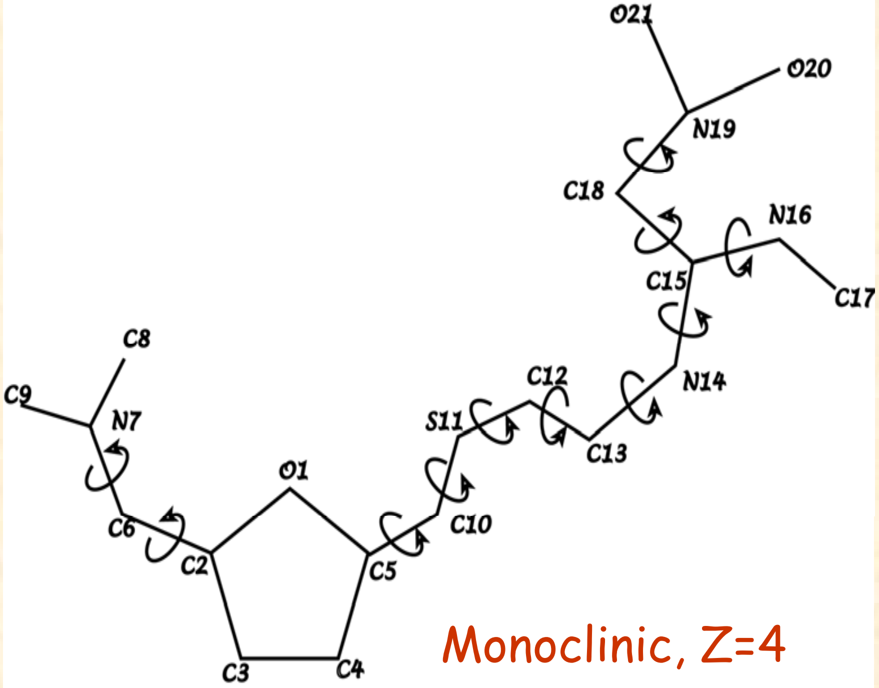
Undertake a project like this with very good data



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=18.808Å,

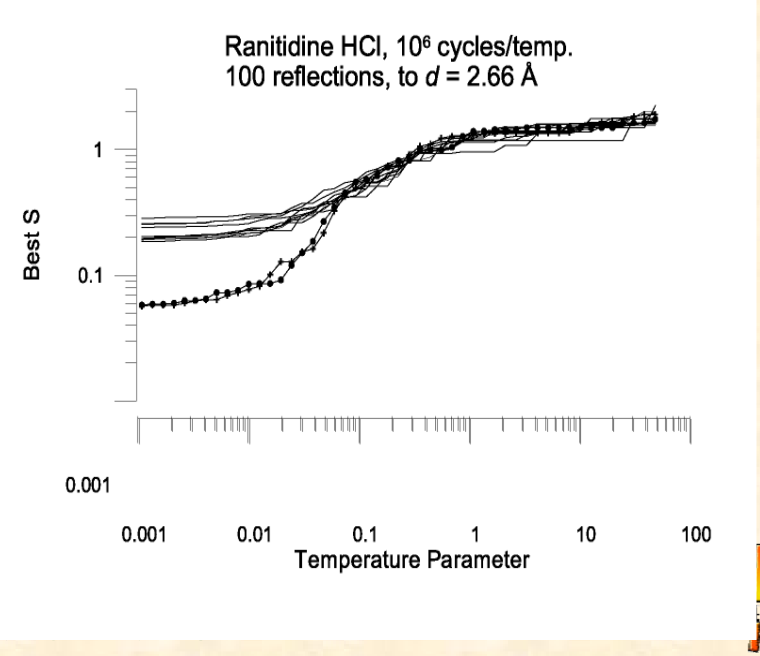
b=12.981Å,

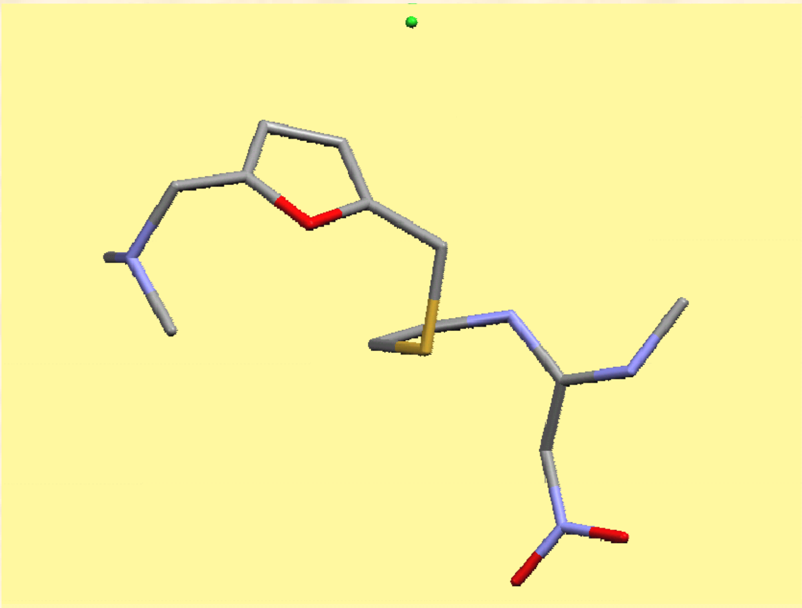
c=7.211Å

β=95.057°,

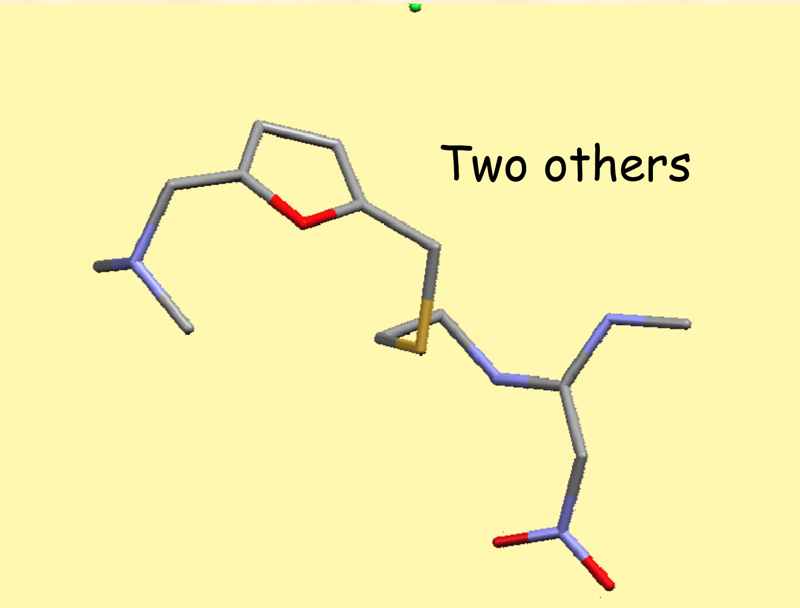
Space Group : P $2_1/n$

3 spatial coordinates : position
3 Eulerian angles : orientation
11 torsions.

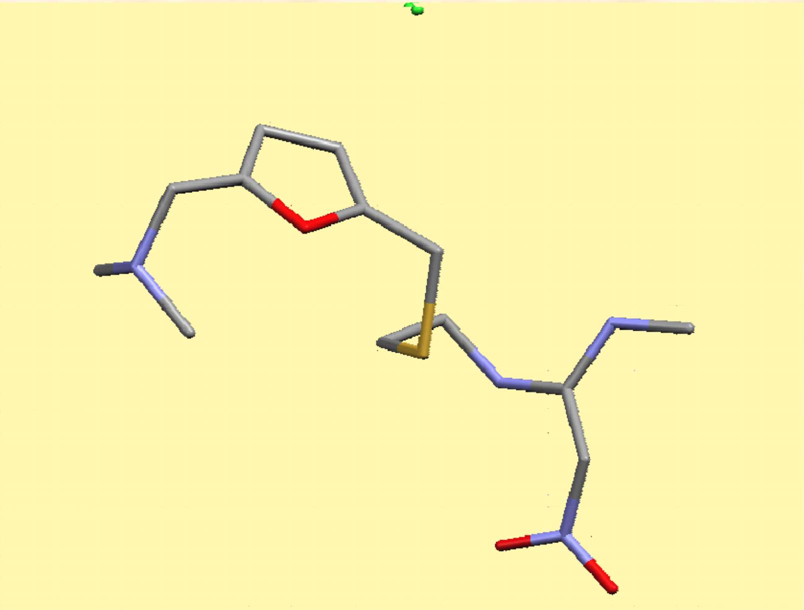

Ranitidine HCl, 10^6 cycles/temp.
100 reflections, to d = 2.66 Å



Two candidate solutions from PSSP



Two others



All four,
superimposed.

Disorder,

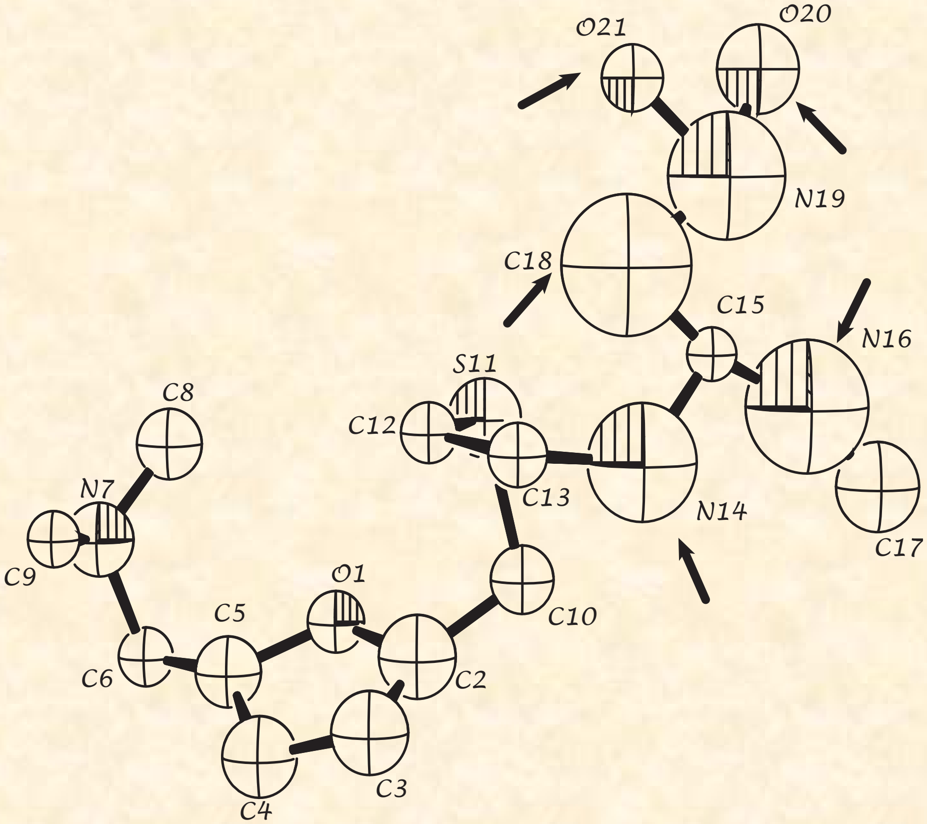
or inability of powder

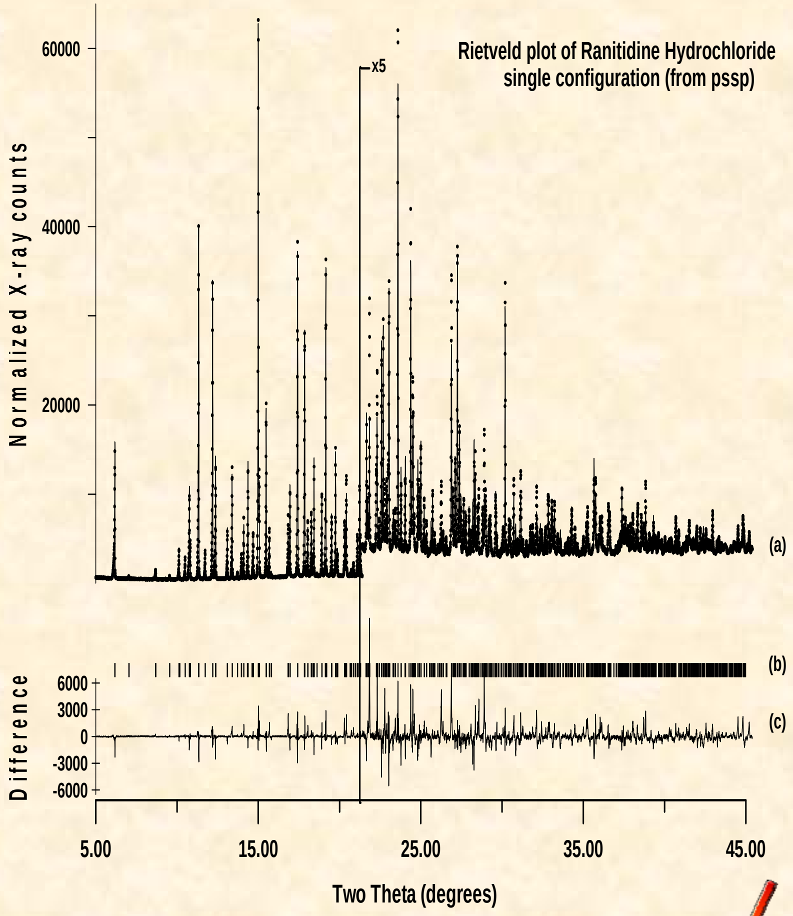
data to distinguish

a few of the atoms?

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

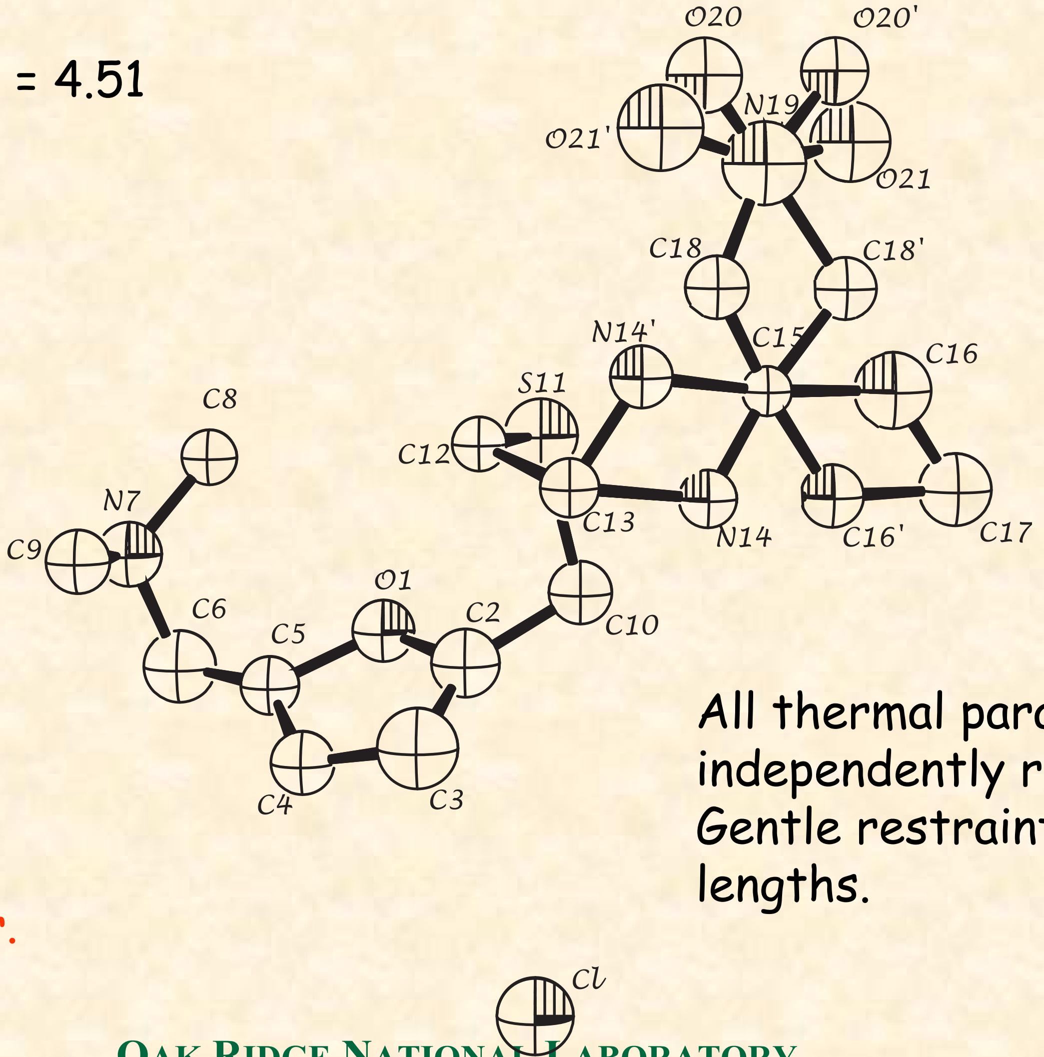
R_{wp} = 11.12%, χ^2 = 10.56





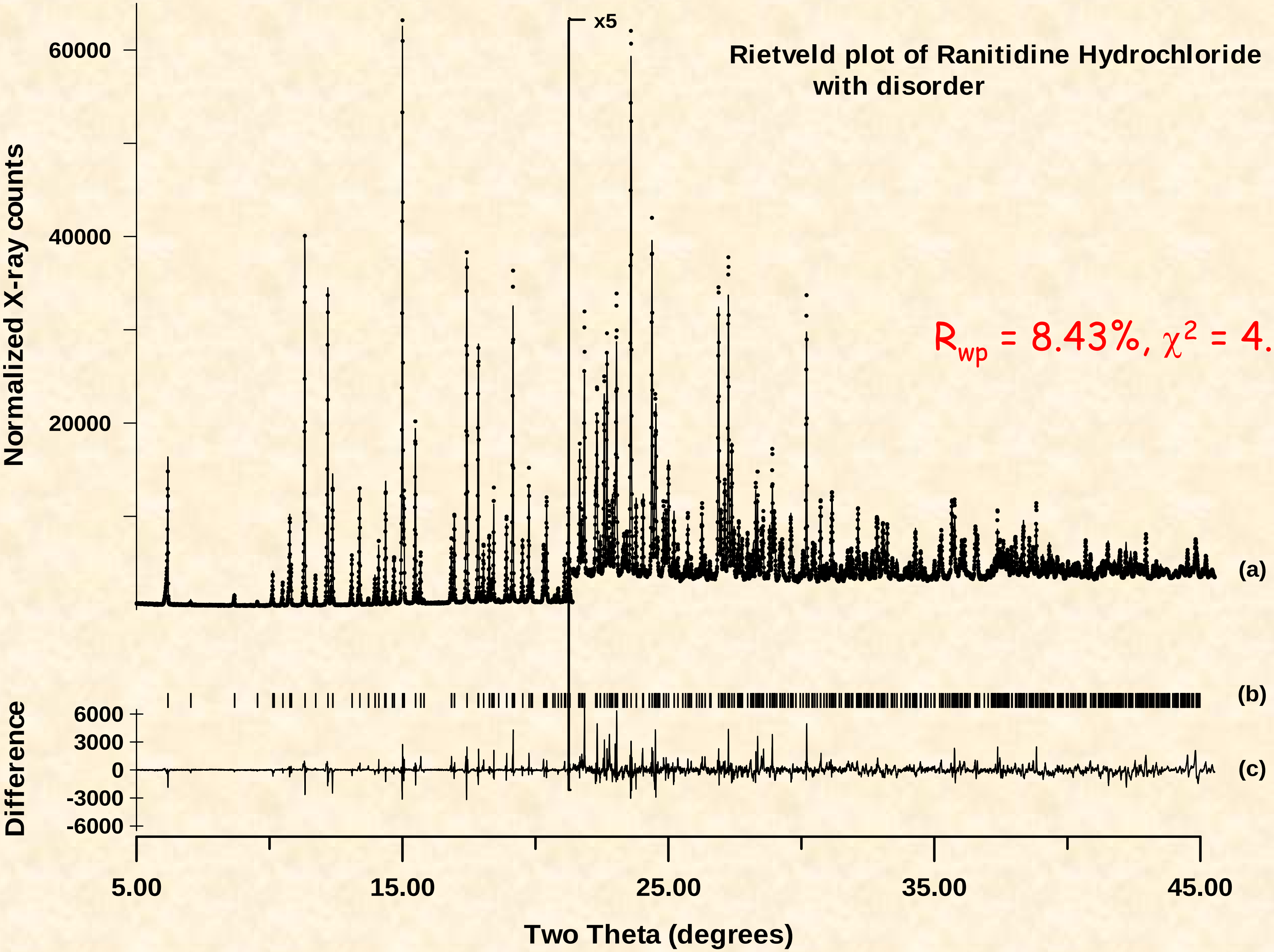
Refinement incorporating disorder. 50% occupancy of each of two sites for N14, C16, C18, O20, and O21.

R_{wp} =8.39%, χ^2 = 4.51



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

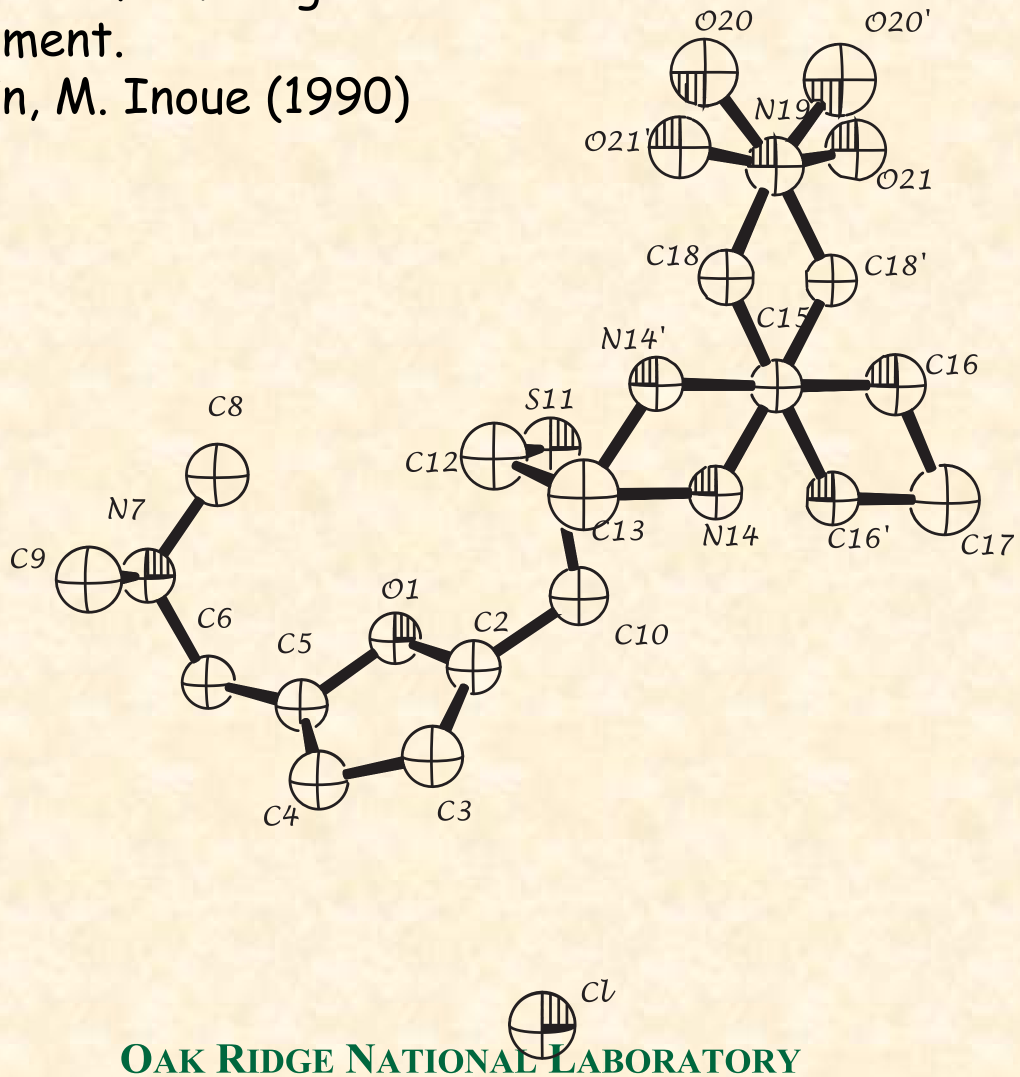
All thermal parameters independently refined! Gentle restraints on bond lengths.



R_{wp} = 8.43%, χ^2 = 4.51

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

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

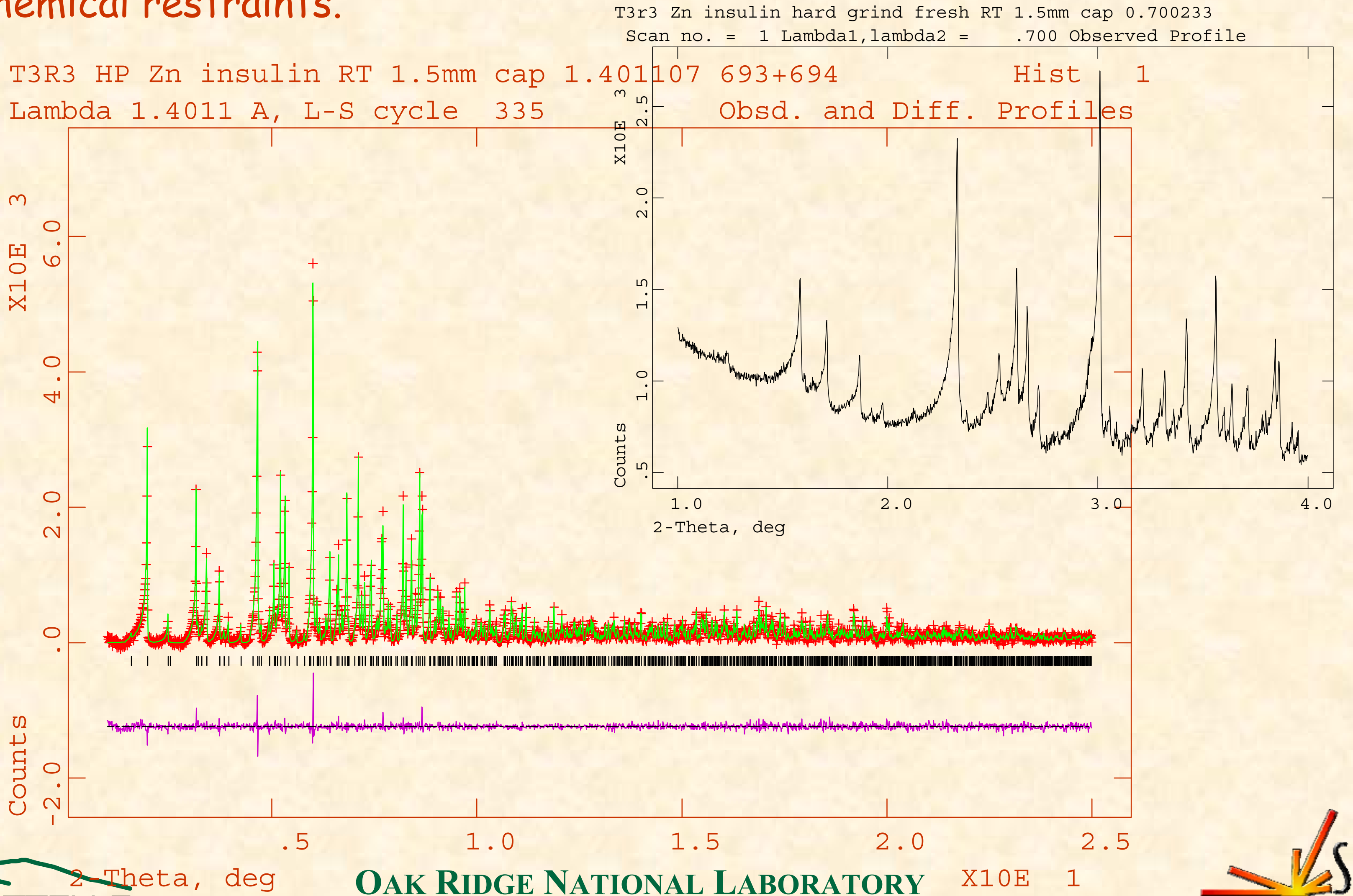


Proteins and Powder Diffraction

Extreme limit: Proteins

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

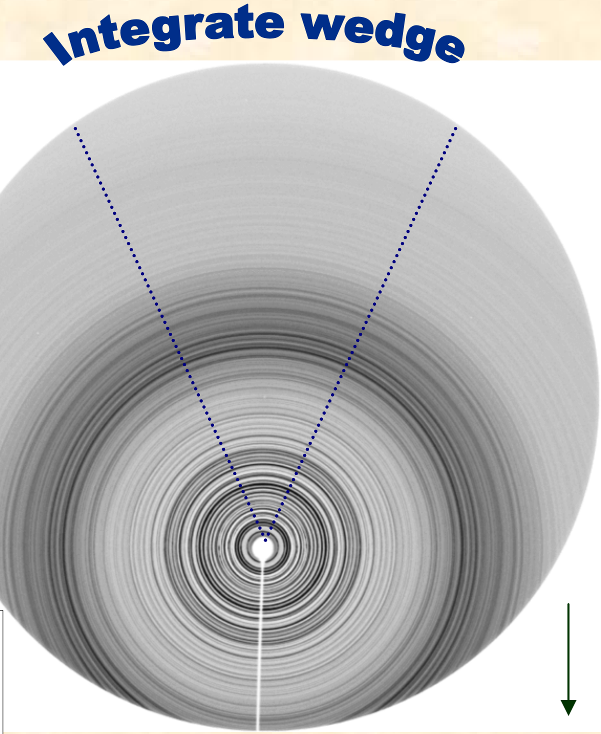
It is possible to get usable data, and to refine it with sufficient chemical restraints.

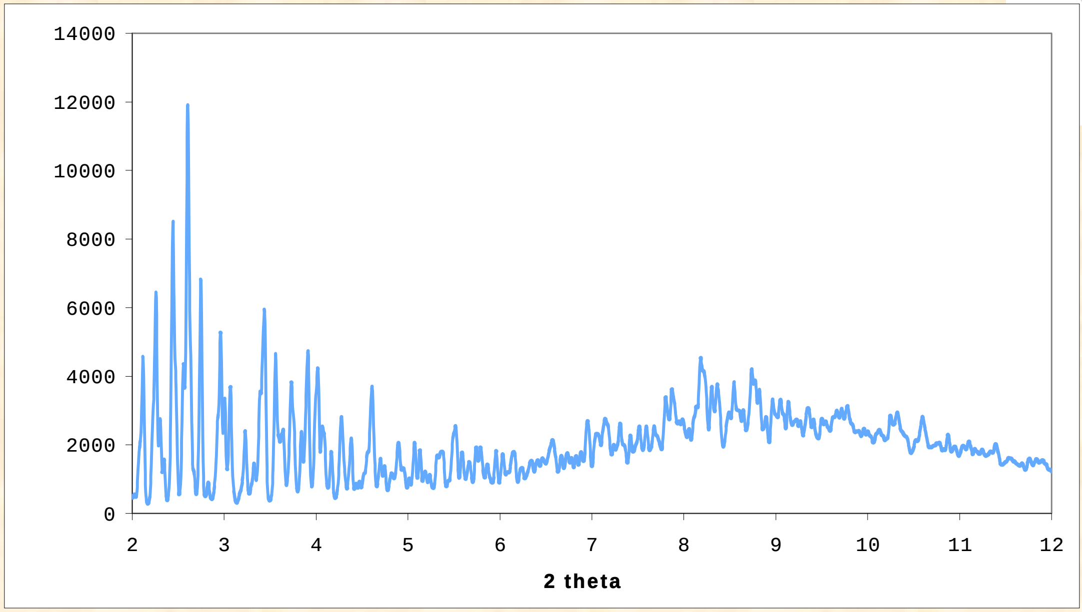


Native	Ground
a=80.96Å	81.28Å
c=37.59Å	73.04Å
Nrefined = 1754	2925
Nrestraints=3871	7934
Nreflections=9871	12734
Resolution 3.06Å	3.22Å
Rwp=3.34%	3.77%

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.