an educational workshop for high school teachers, July 2010, Chicago, IL

An educational outreach workshop was held as part of the 2010 ACA meeting in Chicago. This workshop was geared towards school teachers, and carried Illinois continuing education certification. Sponsorship by the ACA, the USNCCr and a supplement from the NSF allowed the teachers to attend the workshop at no charge. Unfortunately, due to inclement weather that caused severe flooding, many of the 29 registered attendees could not make it to the workshop.

The workshop combined presentations from a number of instructors with hands-on exercises that could be taken back to the classroom. As an intriguing start to the day, Bruce Knoll (Bruker) demonstrated that with modern instrumentation, users need very little training to mount a crystal and start collection of a dataset. By lunchtime, the diffractometer had finished collecting data on aspirin, and the automated software had gone through all steps of structure solution without requiring user input. While the data were being collected, Claudia Rawn (Oak Ridge National Lab/UT Knoxville) took the teachers on a Materials Discovery Tour, introducing them to basic concepts of bonding, structure, and structure-property relationships. Her powerpoint presentation was balanced with a number of hands-on exercises, including stacking of closed-packed layers of spheres, identifying several metal, ceramic and polymer spheres based on known

After lunch, Katherine Kantardjieff (CSU Pomona) introduced remotely enabled instrumentation and how it can be used to expose a wider audience to crystallographic experiments. This was followed by a highly entertaining presentation by Jim Kaduk (IIT), who showed that powder diffraction can provide interesting information about everyday materials like peanut butter, oreos and many other frequently encountered compounds. The last two sessions by Colin Groom (CCDC) and David Goodsell (Scripps Research Institute, representing the RCSB PDB) gave the workshop participants a chance to learn about crystallographic databases, and to try them out hands-on.

A few of the teachers stayed for Venki Ramakrishnan's opening lecture to the ACA meeting, and were thrilled to have a chance to meet him and get his autograph.

The workshop feedback from the participants was very positive, encouraging us to

properties like density, and polymer chain entanglement. A highlight of her session was the construction of basic crystal structures (face-centered cubic material and rocksalt) from legos, which was so popular that all other presenters and even the AV staff participated!

These activities were followed up by Cora Lind's (Univ. of Toledo) introduction to diffraction basics, which exposed the teachers to waves and slit experiments and drew analogies to diffraction experiments. With the aid of diffraction grating slides and lasers, the concepts of repeat distance and atom identity determining spot location and spot intensity were demonstrated.

run similar workshops in the future. Teacher attitudes changed from apprehension in the morning to excitement after the first few hands-on exercises, and by the end of the day, several participants were making plans for how to use what they learned in their own classrooms! Several of them also took additional hands-on kits to share with their colleagues.

workshop organized by Cora Lind (Univ. Toledo) and Claudia Rawn (ORNL, UT Knoxville)

Diffraction Basics

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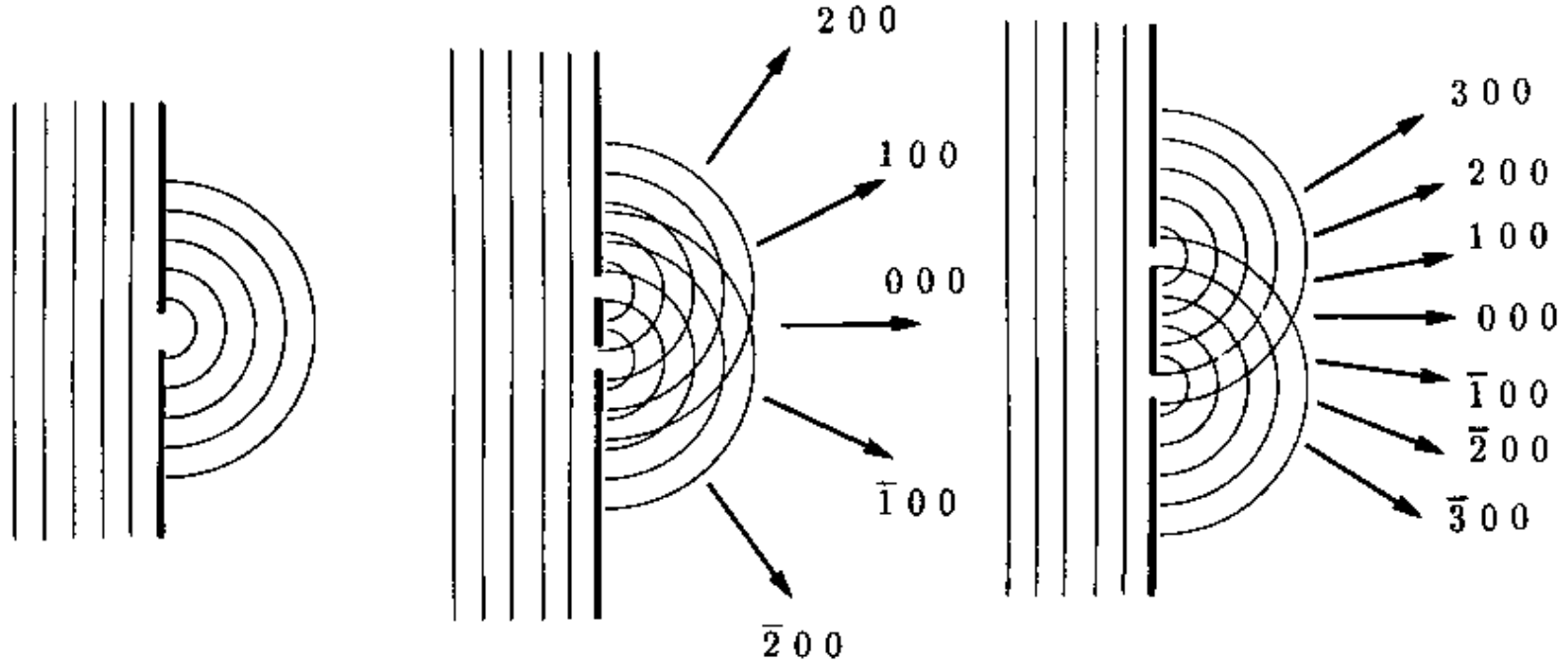
Diffraction

- Definition (from "Cambridge Advanced Learner's Dictionary"):
  - **diffraction** noun [U] SPECIALIZED
    (a pattern caused by) a change in the direction of light, water or sound waves
  - **diffract** verb [T]
    to separate light into coloured strips or into light and dark strips
- Definition (from "Merriam Webster's Dictionary"):
  - Main Entry: **dif·frac·tion**; Function: *noun*
    Etymology: New Latin *diffraction-, diffractio,* from Latin *diffringere* to break apart, from *dis-* + *frangere* to break
  - a modification which light undergoes in passing by the edges of opaque bodies or through narrow slits or in being reflected from ruled surfaces and in which the rays appear to be deflected and to produce fringes of parallel light and dark or colored bands; *also* **:** a similar modification of other waves (as sound waves)

X-ray diffraction

- "Scattering of X-rays by the atoms of a crystal that produces an interference effect so that the diffraction pattern gives information on the structure of the crystal or the identity of a crystalline substance" (Webster's)
  - Both light and X-rays are electromagnetic radiation – the only difference lies in the wavelength!
- X-ray diffraction can be envisioned as an equivalent process to what happens when you shine light through a grating
  - Formulism obeys the same laws as "slit experiments"

Diffraction of light by slits



- Incoming light is a plane wave
- Slit apertures result in an outgoing spherical wave
- Interference determines the diffraction pattern

"Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

Diffraction of light by masks

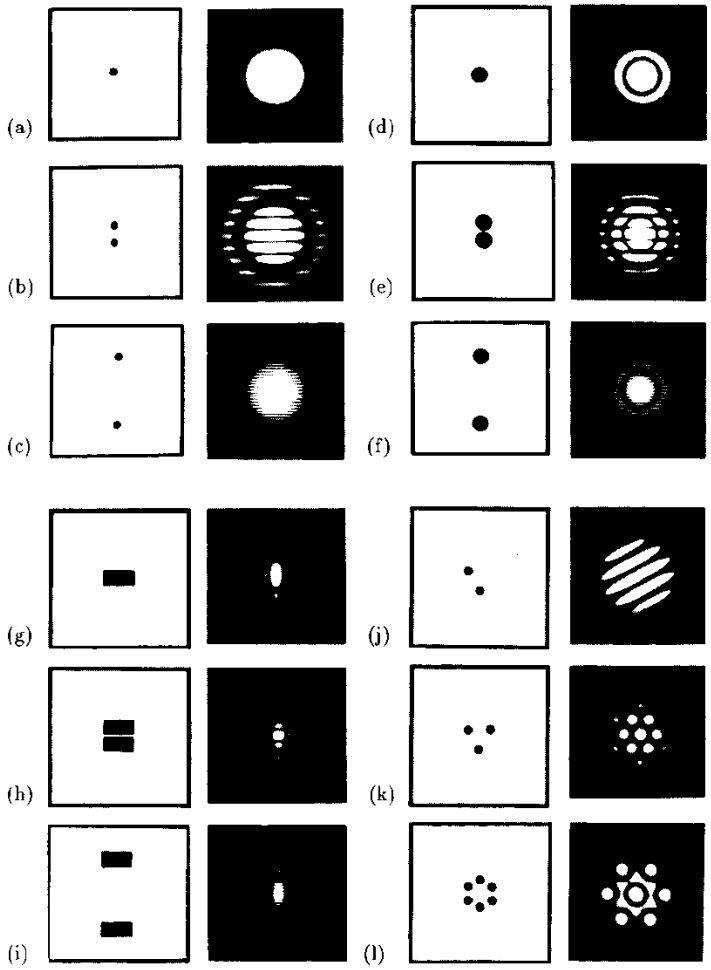
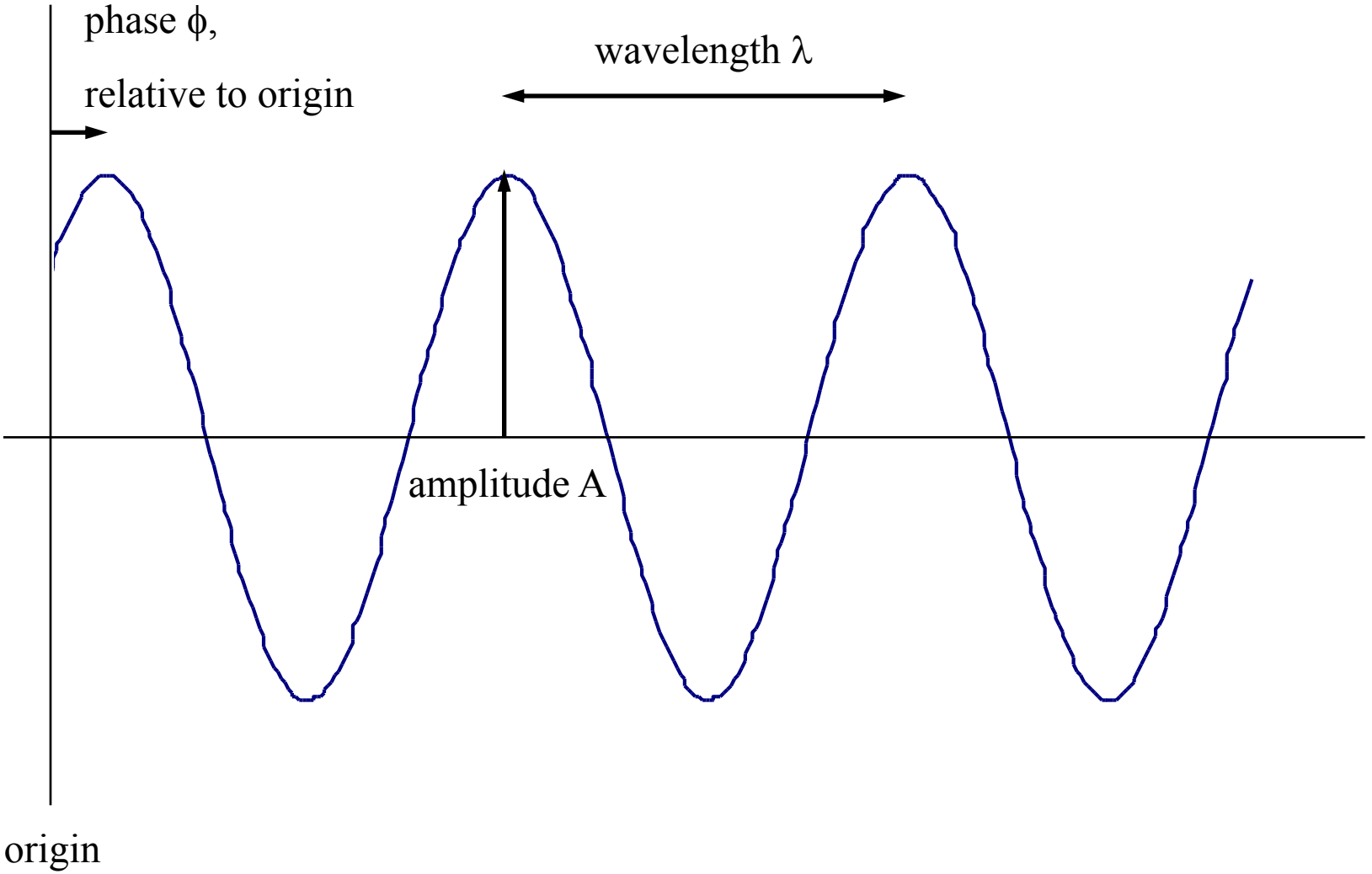


FIGURE 3.7. Examples of diffraction patterns from a variety of masks. The mask with the holes in it is to the left and its diffraction pattern is to the right. The diffraction pattern of (a) a single round hole in a mask, (b) two round holes, and (c) two round holes further apart. Note that in (b) and (c) the diffraction pattern is sampled (in lines) with a spacing that is inversely proportional to the distance between holes in the mask. The diffraction effects when the sizes of the holes in the masks are increased are shown in (d), (e), and (f). The holes are larger than in (a), (b), or (c), but the spacings between their centers are the same. Recall that the experimental diffraction pattern is now more compact, illustrating the reciprocal relationship between the size of an object and its diffraction pattern. Since the spacings of holes in the masks are the same in (a), (b), and (c), and in (d), (e), and (f), the distances between sampling regions are also the same. The effect of changing the shape of the holes in the mask is shown in (g), (h), and (i), where the holes are rectangular in shape. Again, the reciprocal relationship between dimensions in real space and in the diffraction pattern is shown. The wider part of the hole gives a narrower diffraction pattern. The spacings between holes are the same as in (a) to (f). Finally, the effect of different arrangements of holes on the diffraction pattern. In (j) there are two holes in the mask, and the resulting diffraction pattern is similar to that in (b), although one mask is rotated with respect to the other. In (k) the effect of three holes, equivalent to three superpositions of the diffraction pattern in (j), each at 120° to each other is seen. In (l) the diffraction pattern of six holes is shown. (Reprinted from G. Harburn, C. A. Taylor and T. R. Welberry: *Atlas of Optical Transforms*.)

"Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

Waves

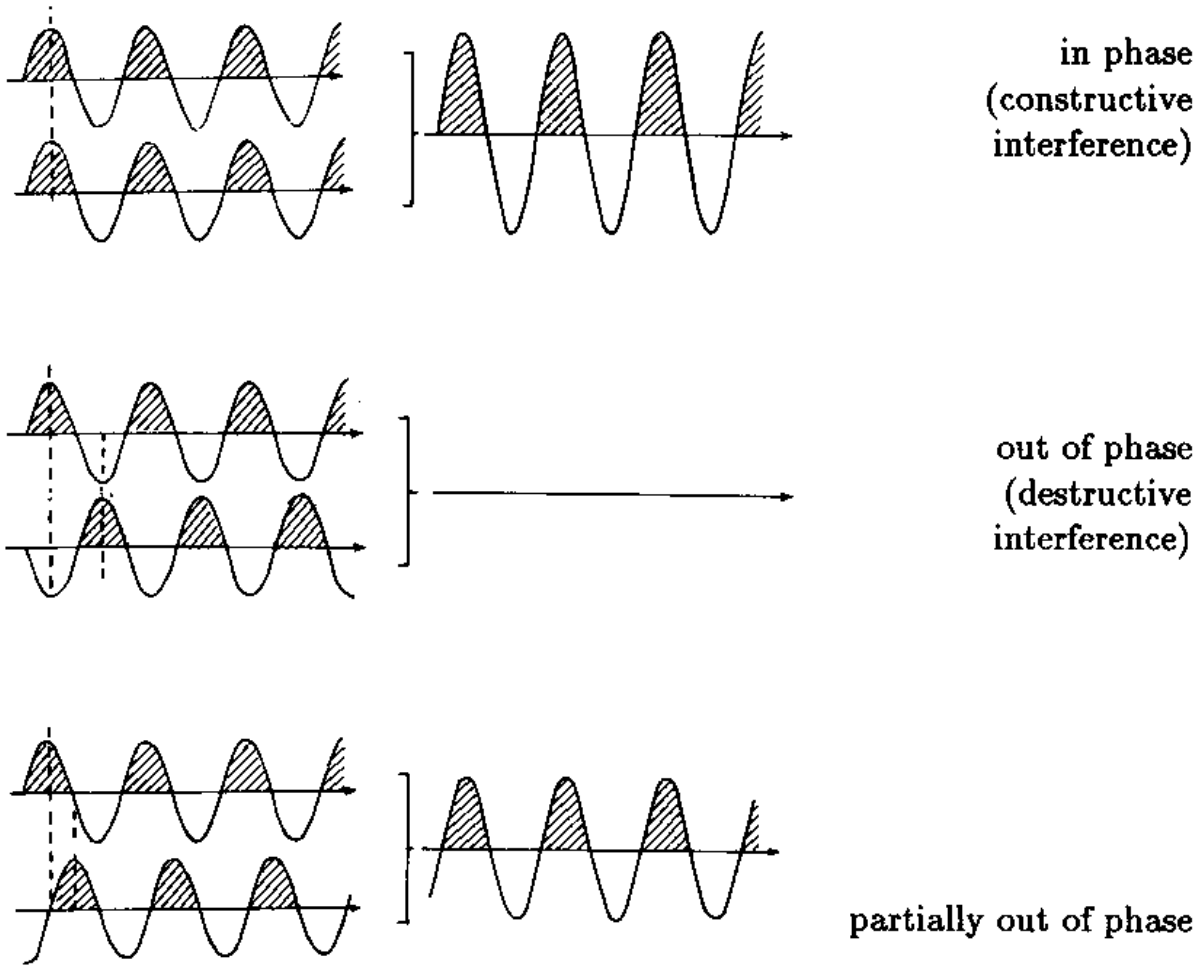


Wave equations

- A wave can be described by a cosine function
  - amplitude is position dependent (x)
  - amplitude is time dependent (t)

    $\Rightarrow A = \cos(kx-\omega t)$
- Two parallel waves will interact with each other
  - called interference
  - constructive interference if waves are in phase
  - destructive interference if waves are exactly out of phase
- The interaction between waves can be envisioned by addition of their wave equations

Adding wave functions



"Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

Interference

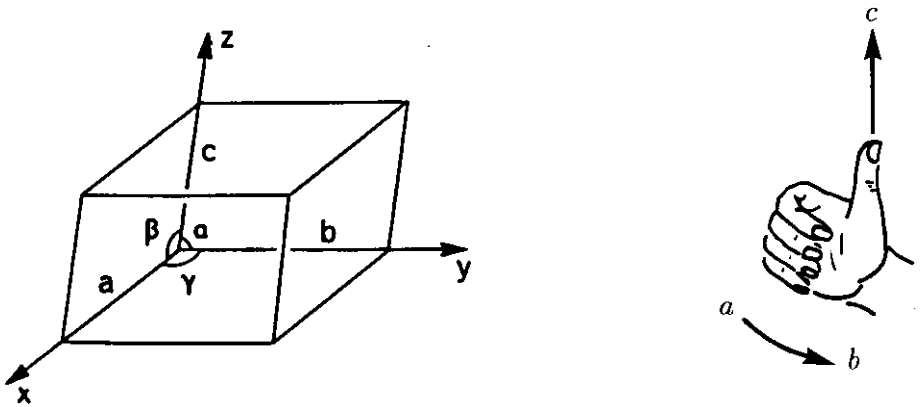
- For waves with the same frequency and amplitude, we see *constructive interference* when two waves have a phase difference of $n\lambda$, where $(n \in \mathbb{Z})$
  - called "in phase"
- *Destructive interference* is observed for a phase difference of $(n + ½)\lambda$, where $(n \in \mathbb{Z})$
  - called "out of phase"
- A phase difference can result from a path difference
  - happens in slit experiments
  - the same thing happens when X-rays are diffracted by a crystal

What is a crystal?

- Historic definition before the advent of crystallography
  - A solid with well-defined faces
- Crystallographic definition
  - A material with a regularly repeating structural motif
- The strict definition is more vague
  - Any material that gives a diffraction pattern with sharp peaks

Repeating motif: The unit cell

- The repeating structural motif in a crystal is referred to as a unit cell
  - Only the size and contents of one unit cell are necessary to describe the entire crystal
- Remember to use a right-handed axis system!

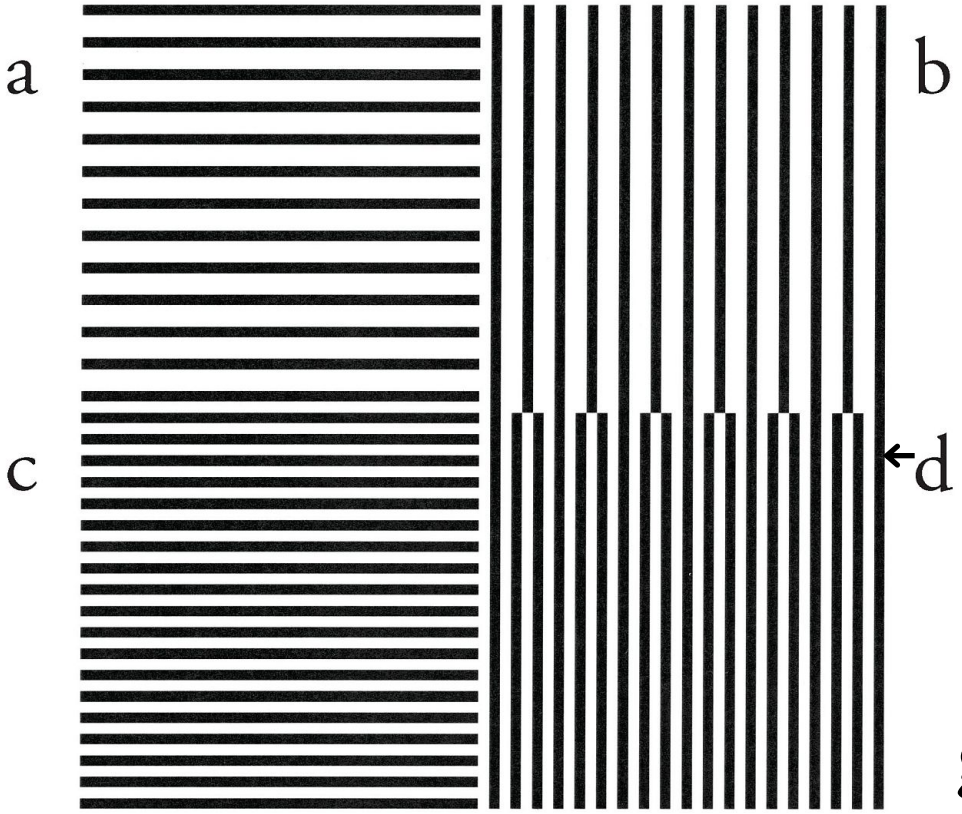


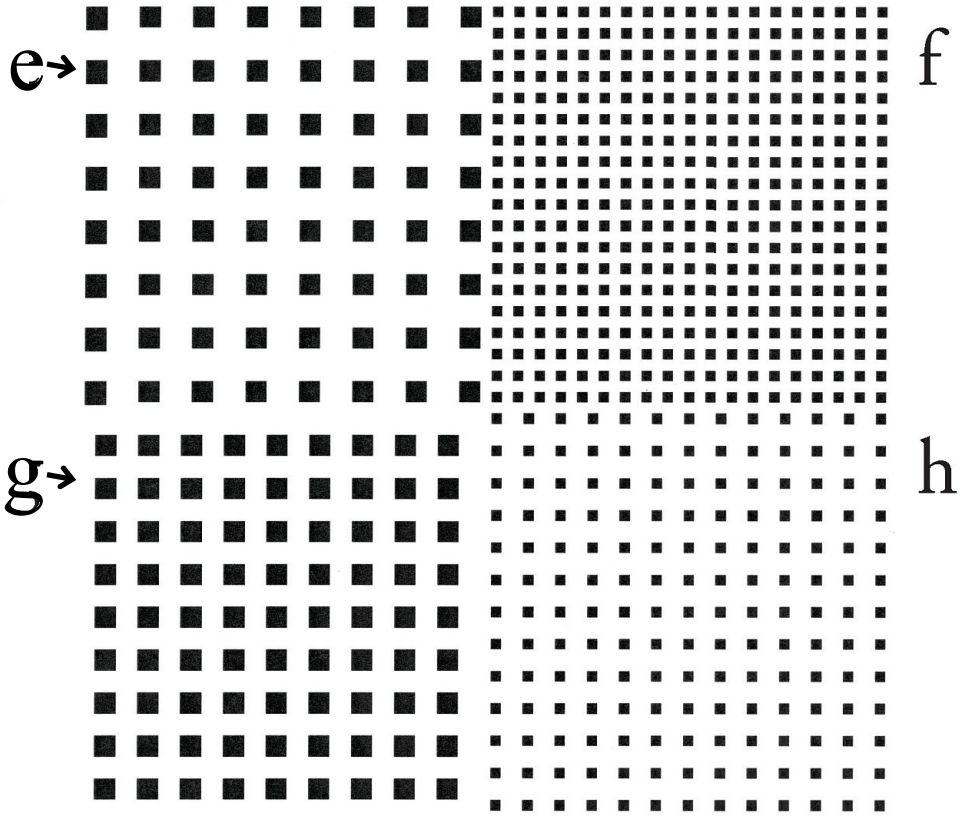
"Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

The seven crystal systems

Crystal system	Unit cell edges	Unit cell angles
Cubic	$a = b = c$	$\alpha = \beta = \gamma = 90°$
Hexagonal	$a = b \neq c$	$\alpha = \beta = 90°, \gamma = 120°$
Rhombohedral	$a = b = c$	$\alpha = \beta = \gamma \neq 90$ or $120°$
Tetragonal	$a = b \neq c$	$\alpha = \beta = \gamma = 90°$
Orthorhombic	$a \neq b \neq c$	$\alpha = \beta = \gamma = 90°$
Monoclinic	$a \neq b \neq c$	$\alpha = \gamma = 90°, \beta \neq 90$ or $120°$
Triclinic	$a \neq b \neq c$	$\alpha \neq \beta \neq \gamma \neq 90$ or $120°$

Diffraction – Discovery Slide





Reciprocal space

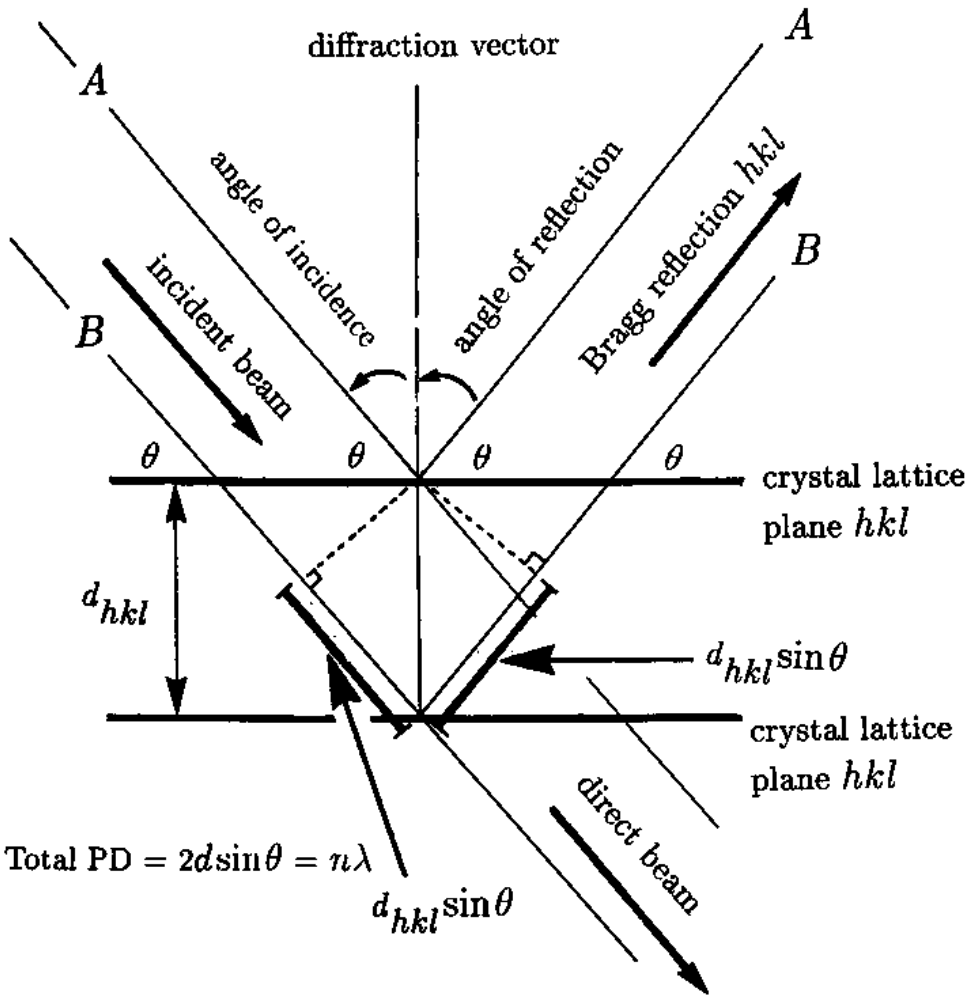
- We do not see the periodic electron density directly during a diffraction experiment
  - we only observe the intensity distribution of X-ray scattering from the crystal(s)
- The diffraction intensity is correlated to the electron density in the crystal by a Fourier transform
  - often referred to as direct space and reciprocal space
- This means that we sample reciprocal space with our diffraction experiments
  - we can define a reciprocal lattice that corresponds to the direct (crystal) lattice

Laue equations

- In 1912, Max von Laue realized that the path differences PD_1, PD_2 and PD_3 for waves diffracted by atoms separated by one unit cell translation have to be a multiple of the diffraction wavelength for constructive interference
  - $PD_1 = h\,\lambda$, $PD_2 = k\,\lambda$, $PD_3 = l\,\lambda$
  - $h, k, l \in \mathbb{Z}$
- He showed that these three conditions have to be fulfilled simultaneously

Bragg's law

- Reflection of X-rays from parallel lattice planes
  - families of planes have equal spacing
- Constructive interference when PD = nλ
- The Laue equations can be rewritten as $2d_{hkl}\sin\theta_{hkl} = n\lambda$



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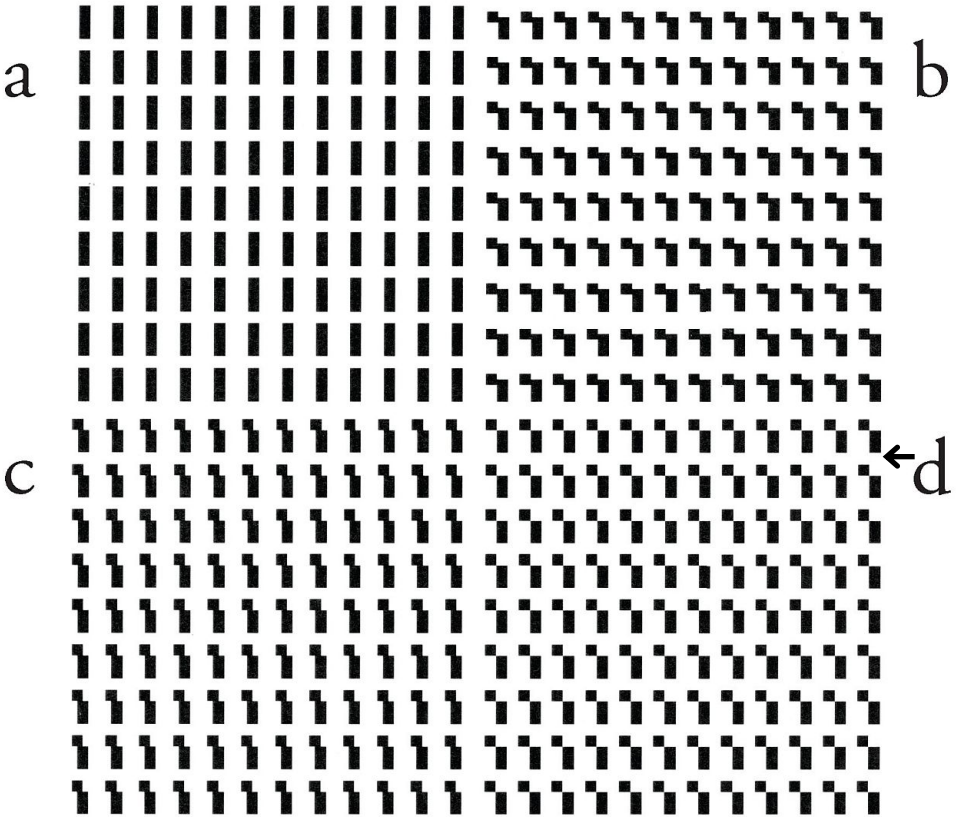
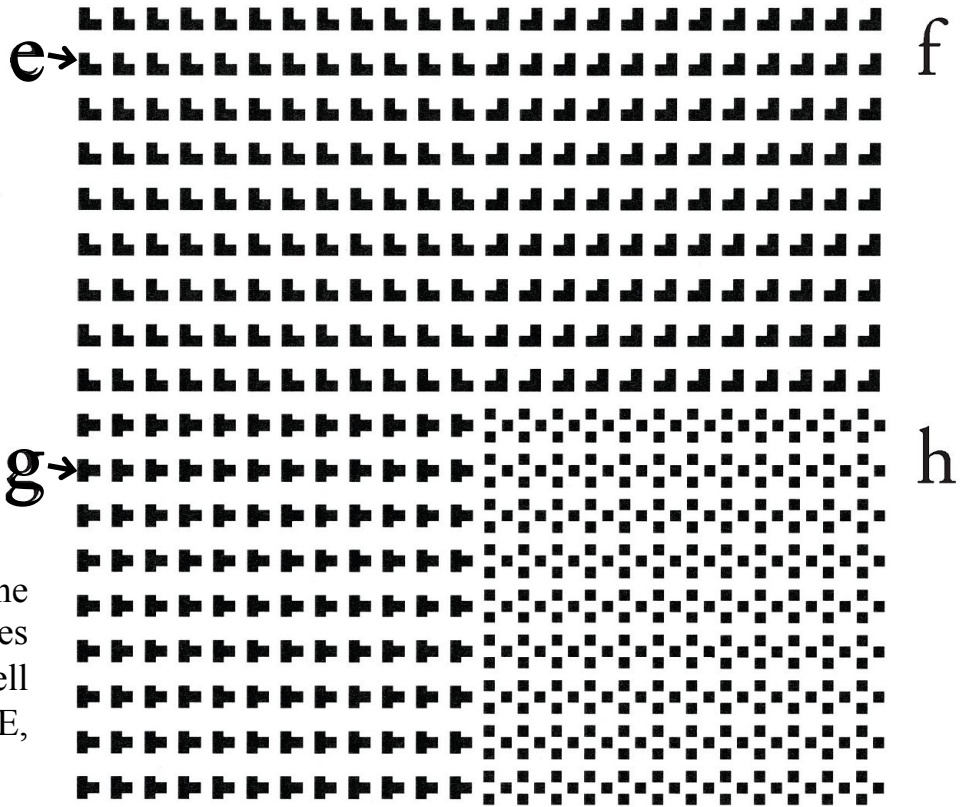
Diffraction Intensities – VSEPR Slide





What's a hole got to do with a crystal?

- The hole scatters the light rays
- An electron scatters X-rays
- Imagine a crystal as a "hole pattern" of electrons!

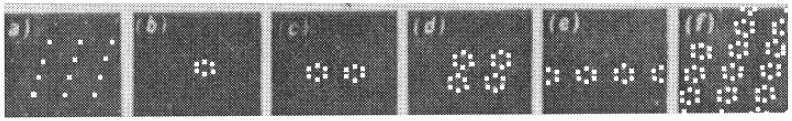
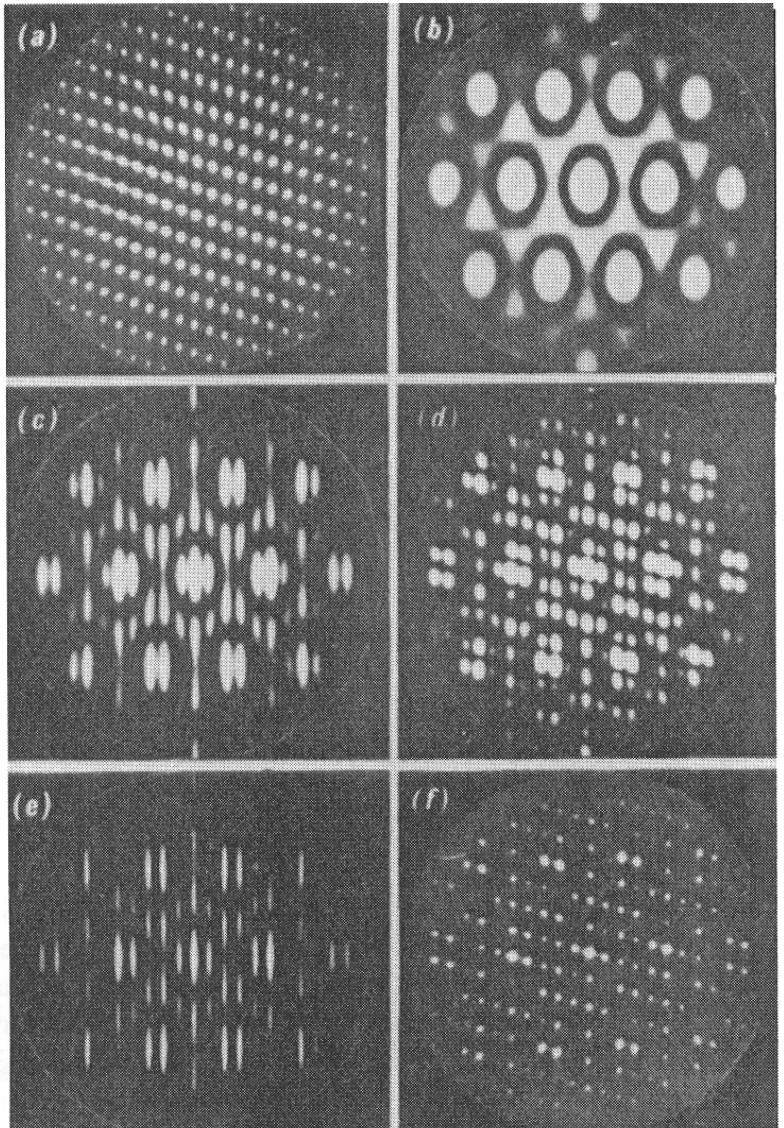


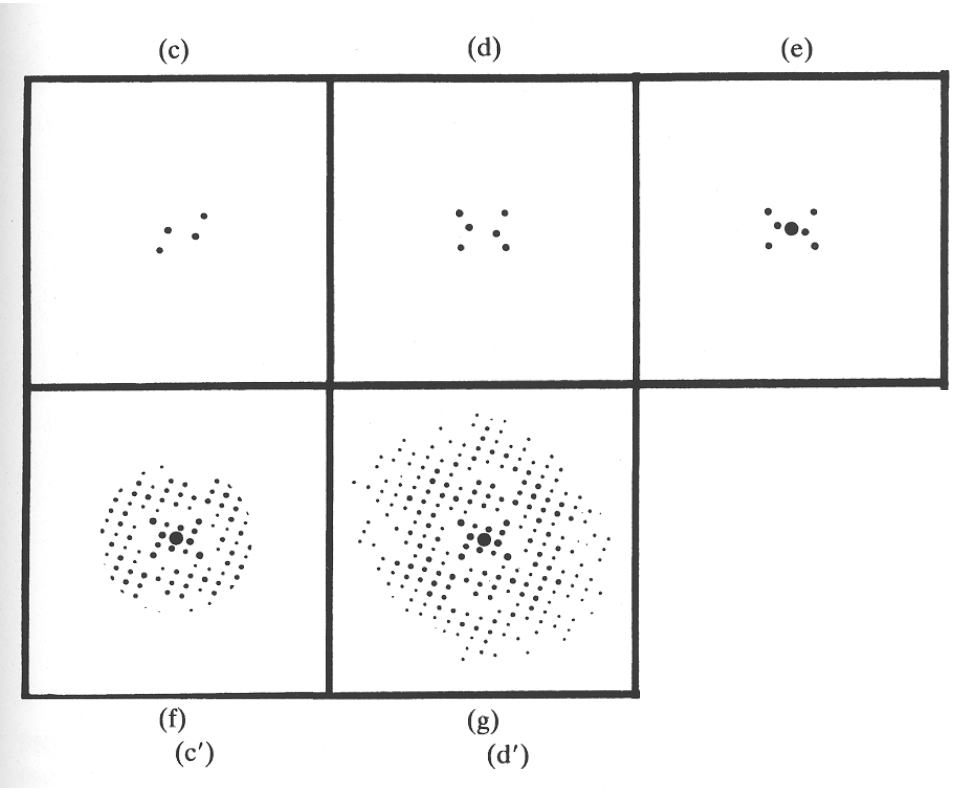
FIGURE 5.6. Optical diffraction patterns illustrating scattering by (a) a two-dir monatomic lattice (portion only), (b) a single molecule (benzene ring), (c) two n (d) four molecules, (e) a "row" of six molecules (portion only), (f) a net of (portion only). [Reproduced from *Optical Transforms*, by C. A. Taylor and H. Lip the permission of Routledge.]

"Structure Determination by X-ray Crystallography", Ladd and Palmer, Plenum, 1994.



The opposite approach…

- …transforming your diffraction pattern into an electron density map



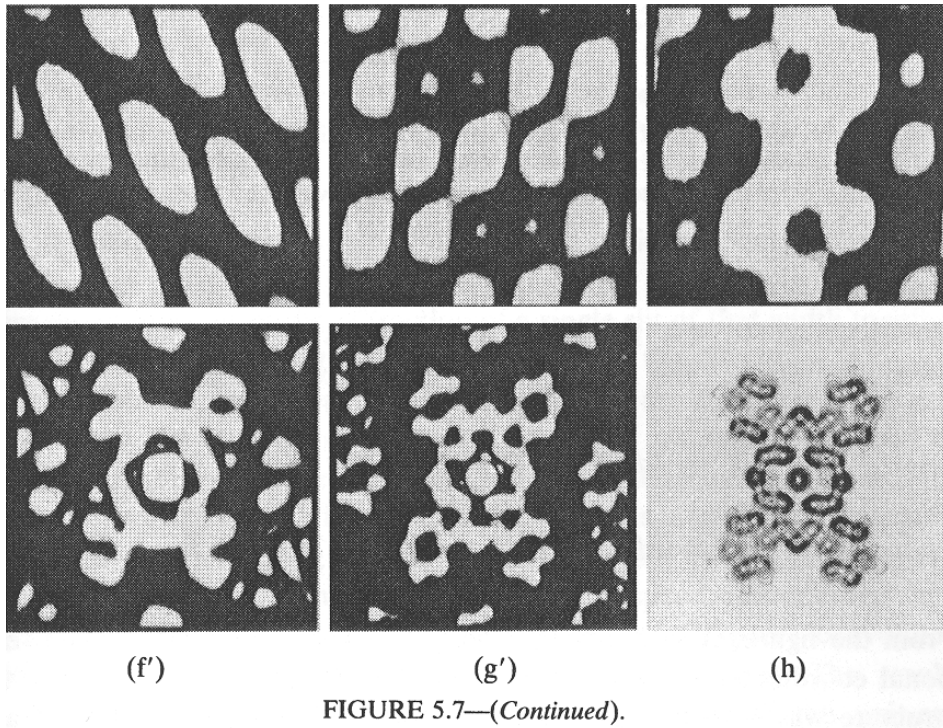


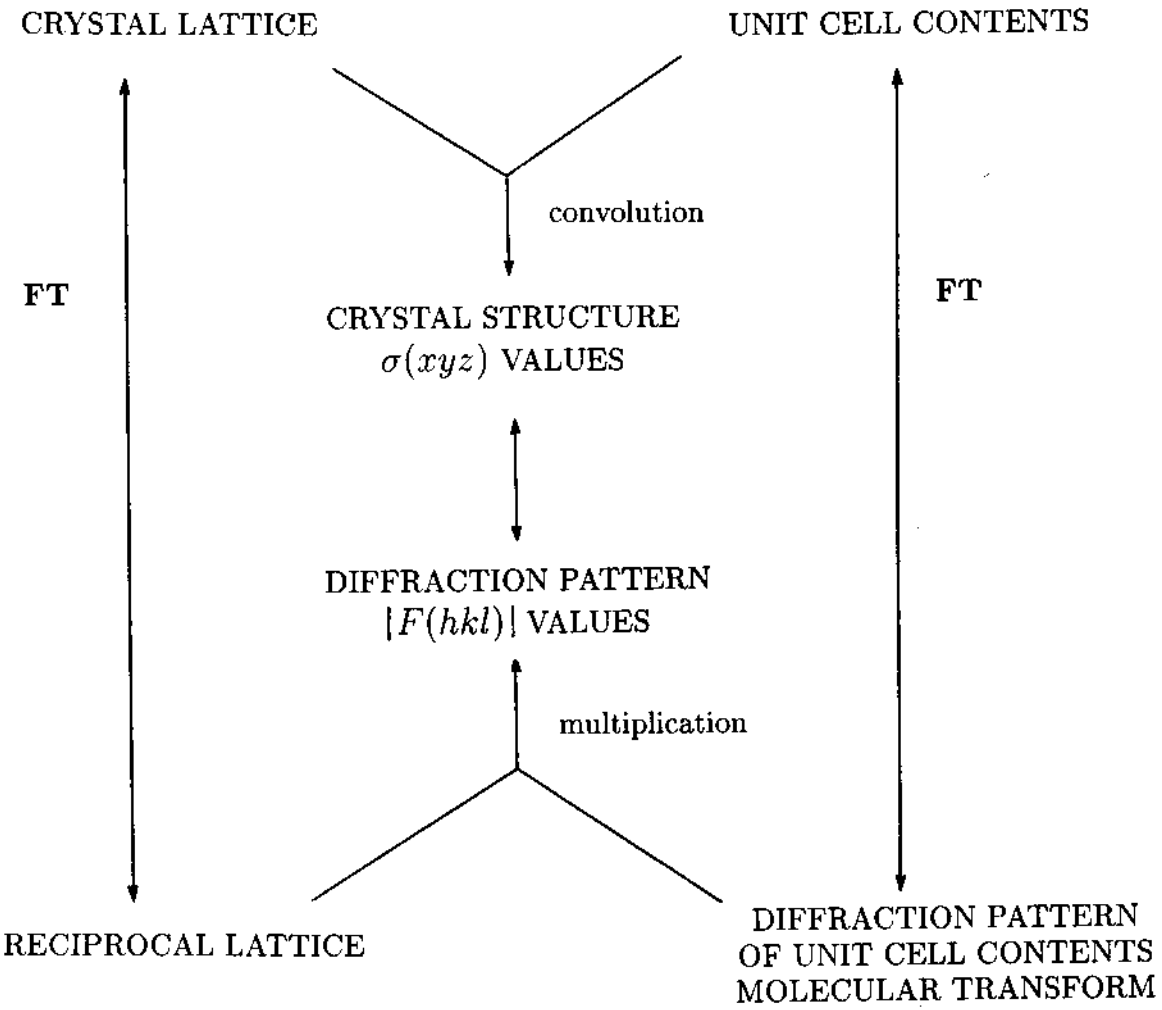
FIGURE 5.7—(*Continued*).

The resolution of your electron density map will strongly depend on how many reflections you use in creating it!

"Structure Determination by X-ray Crystallography", Ladd and Palmer, Plenum, 1994.

Real and reciprocal space

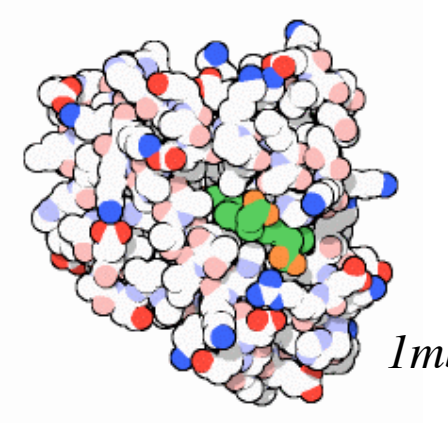
Crystal space



Diffraction space

"Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

Getting the Most out of the Protein Data Bank

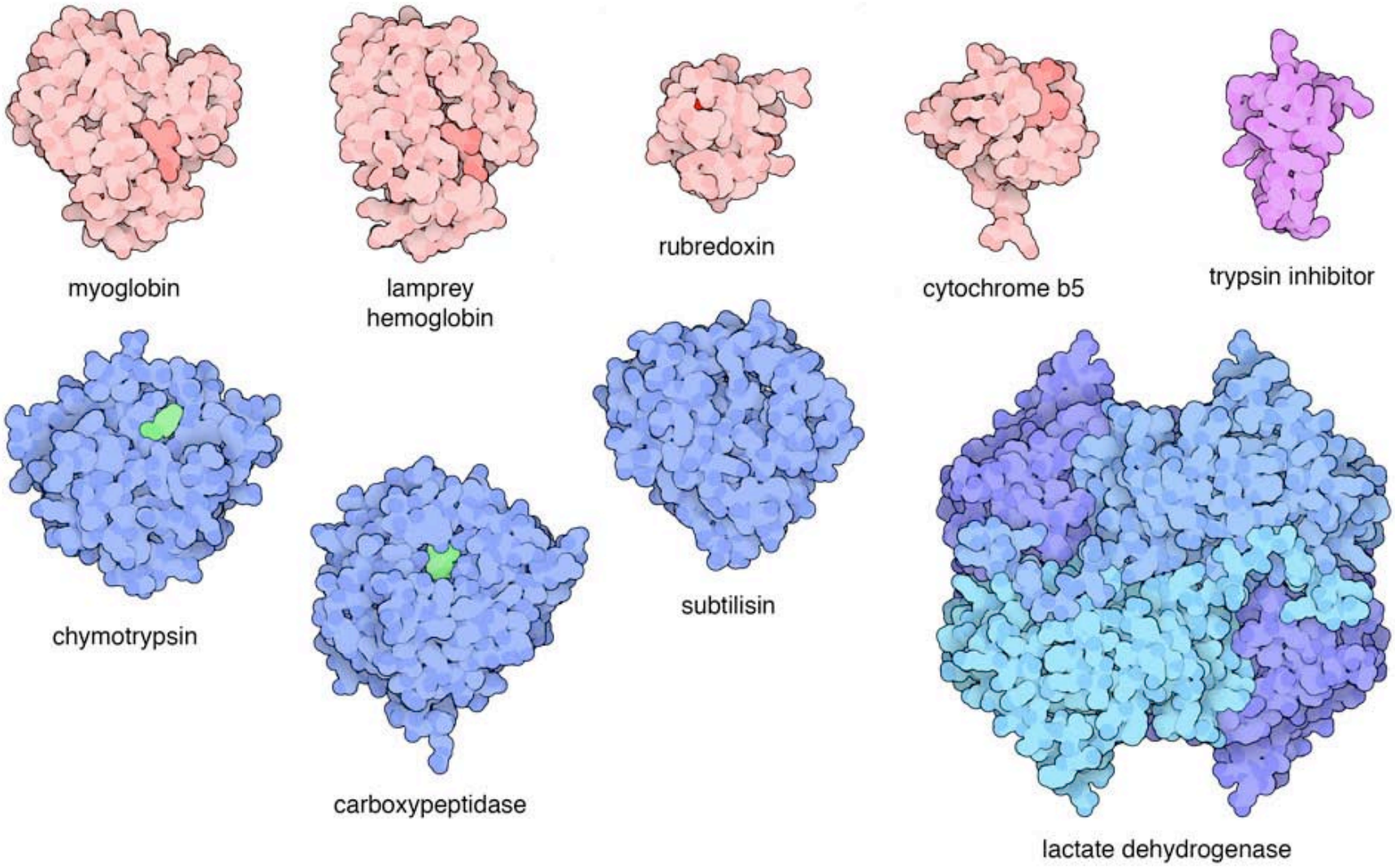


1mbn

David S. Goodsell

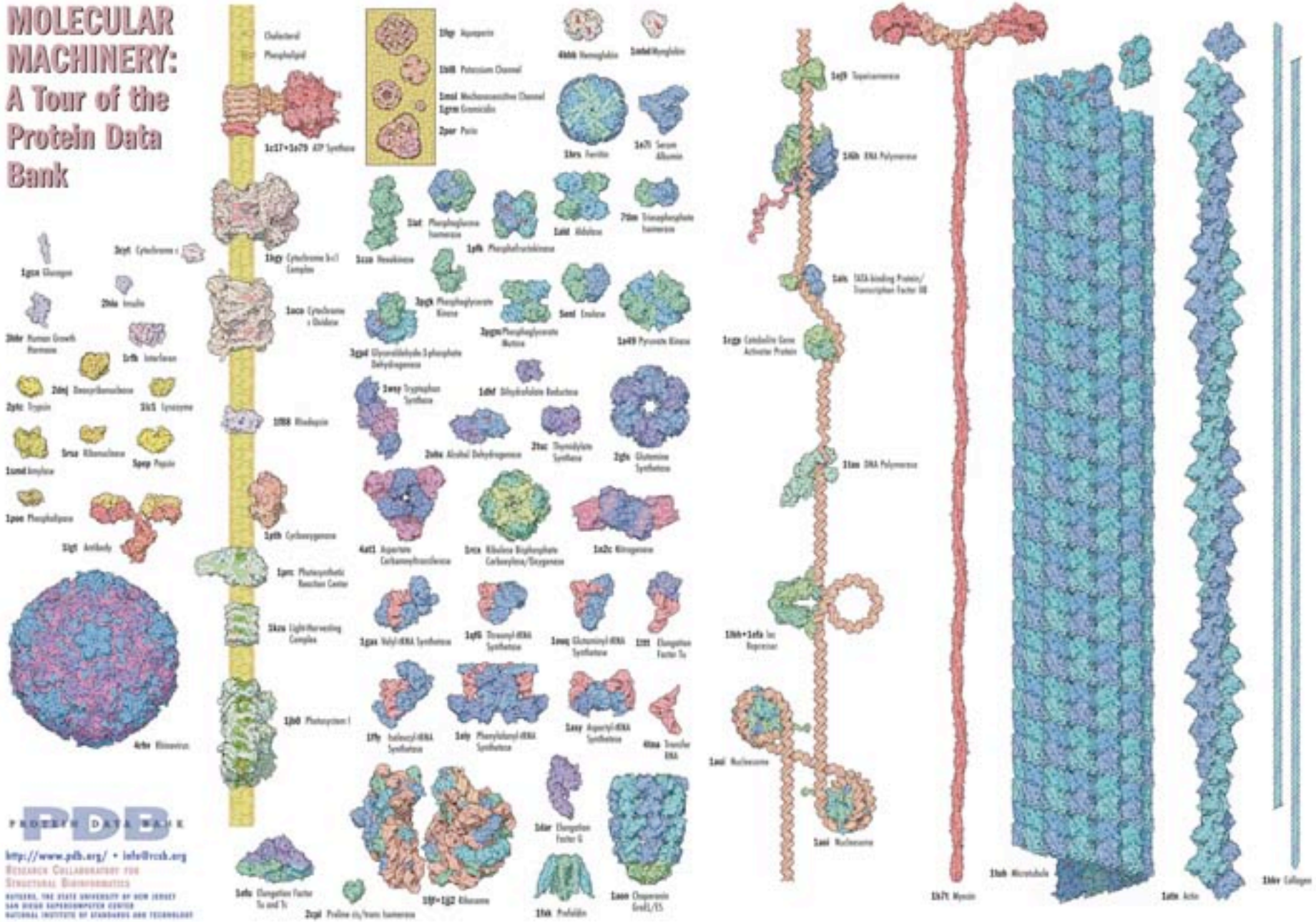
The Scripps Research Institute

RCSB Protein Data Bank



Protein Data Bank in 1973

MOLECULAR MACHINERY: A Tour of the Protein Data Bank



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The PDB archive contains information about experimentally-determined structures of proteins, nucleic acids, and complex assemblies. As a member of the **wwPDB**, the RCSB PDB curates and annotates PDB data according to agreed upon standards.

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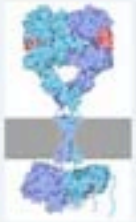

Molecule of the Month:

Epidermal Growth Factor

The cells in your body constantly communicate with each other, negotiating the transport and use of resources and deciding when to grow, when to rest, and when to die. Often, these messages are carried by small proteins, such as epidermal growth factor (EGF). EGF is a message telling cells that they have permission to grow. It is released by cells in areas of active growth, then is either picked up by the cell itself or by neighboring cells, stimulating their ability to divide. The message is received by a receptor on the cell surface, which binds to EGF and relays the message to signaling proteins inside the cell, ultimately mobilizing the processes needed for growth.

Full Article...

Protein Structure Initiative Featured Molecule:

RBBP9 (Retinoblastoma Binding Protein 9)

Researchers at **NESG** are using structural genomics to reveal the secrets of signaling within cancer cells. The structure of retinoblastoma binding protein 9 showed that it is a serine protease as well as a partner of retinoblastoma protein.

Full Article | **PSI Structural Genomics Knowledgebase**

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Baba, S., Someya, T., Kumasaka, T., Kawai, G., Nakamura, K.

Crystal structure of YmaH (Hfq) from Bacillus subtilis in complex with an RNA aptamer
Abstract not available.

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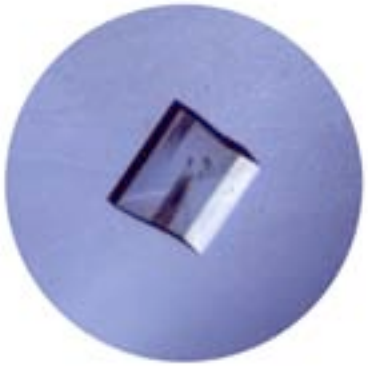
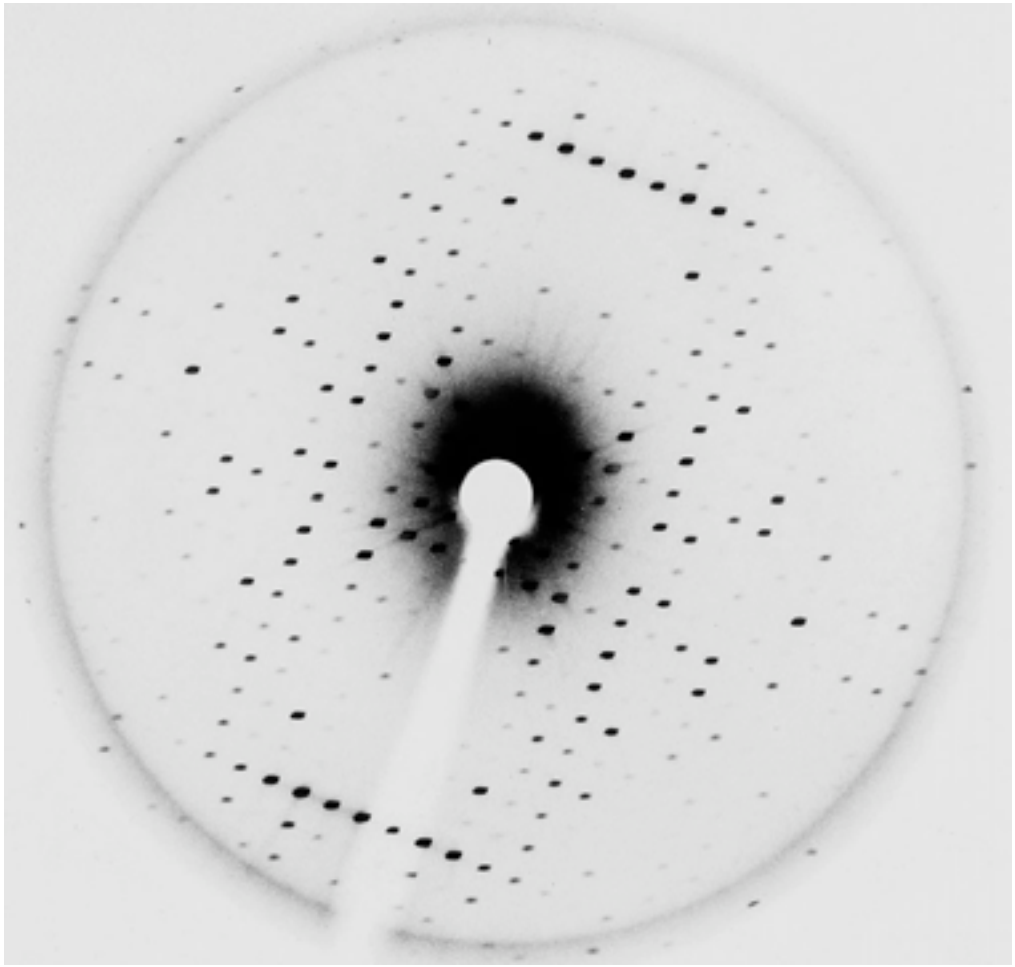
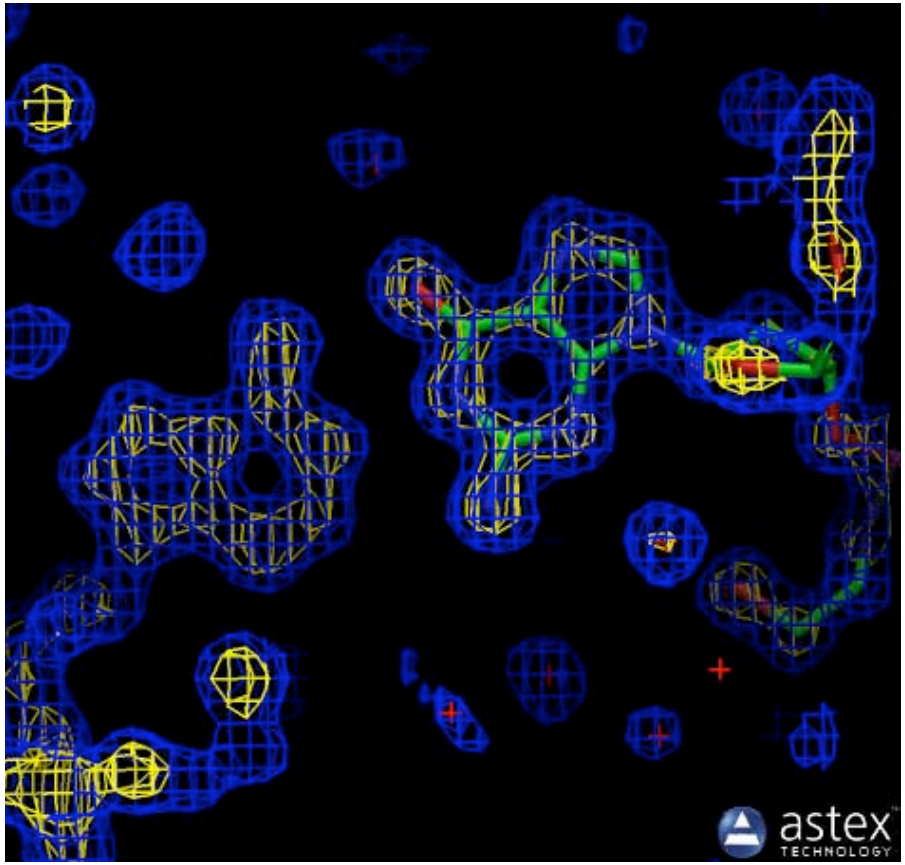


How do we see molecules?

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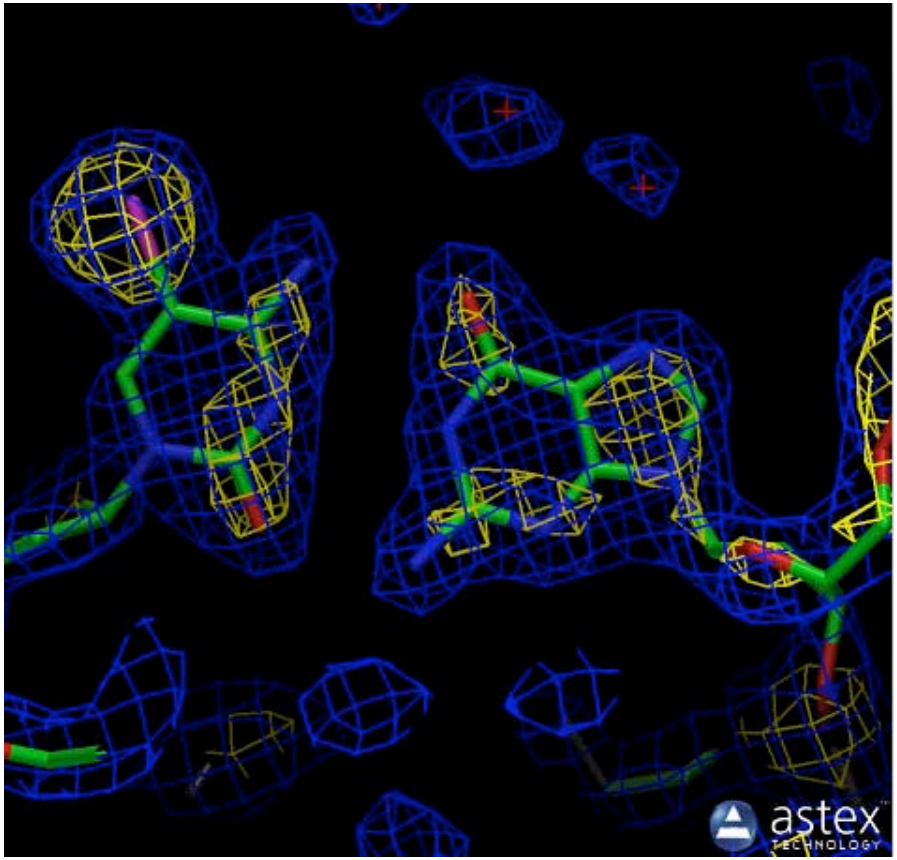
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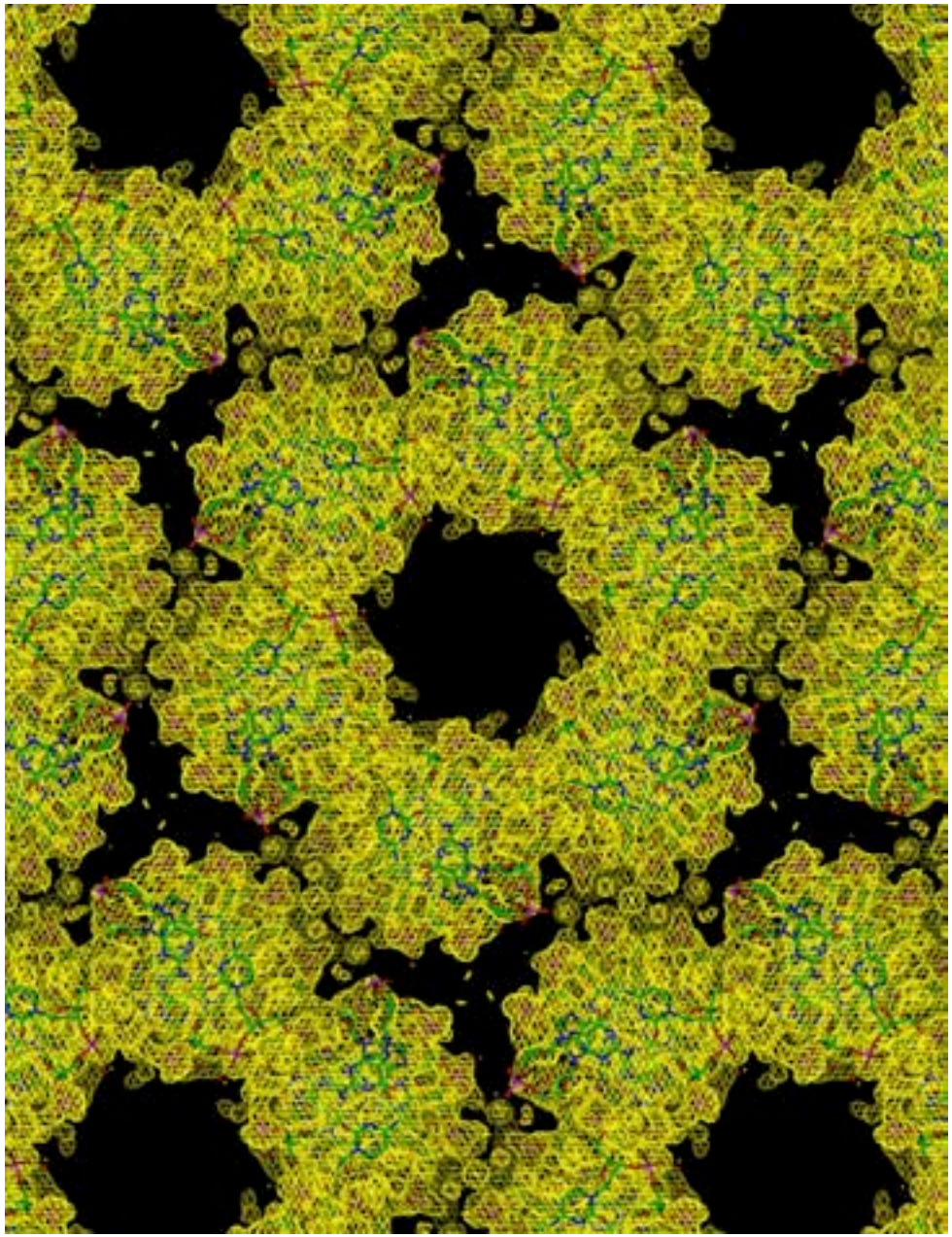

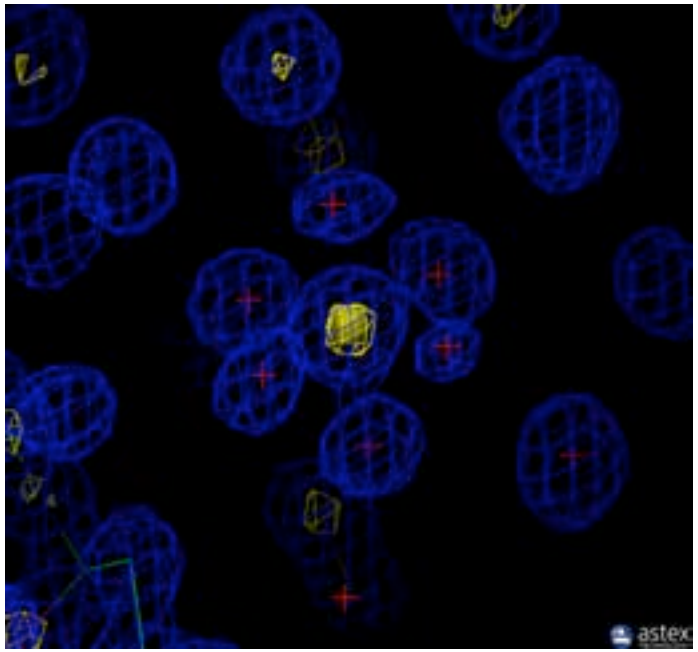
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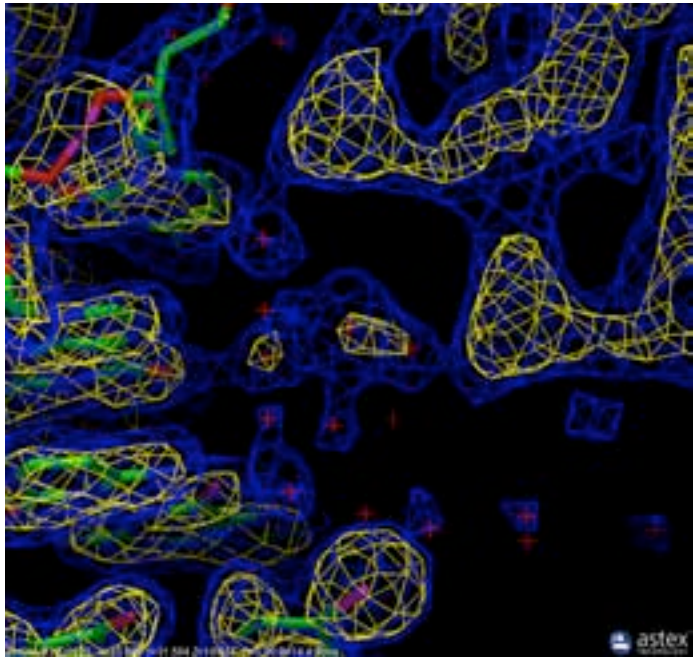
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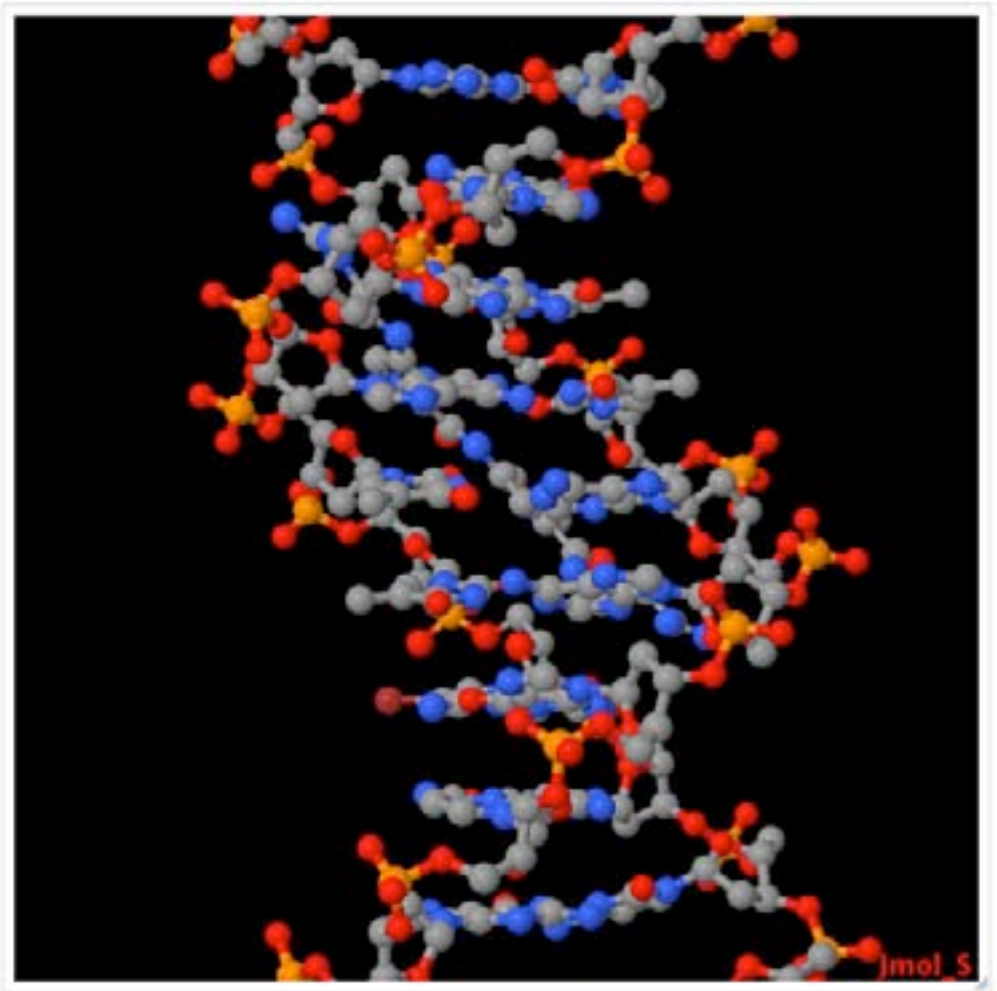
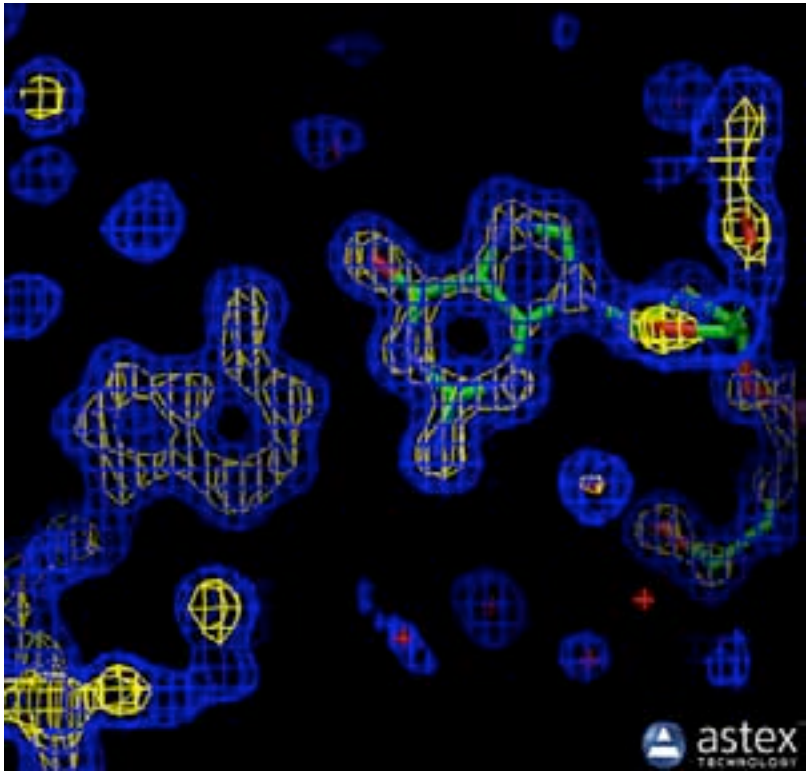
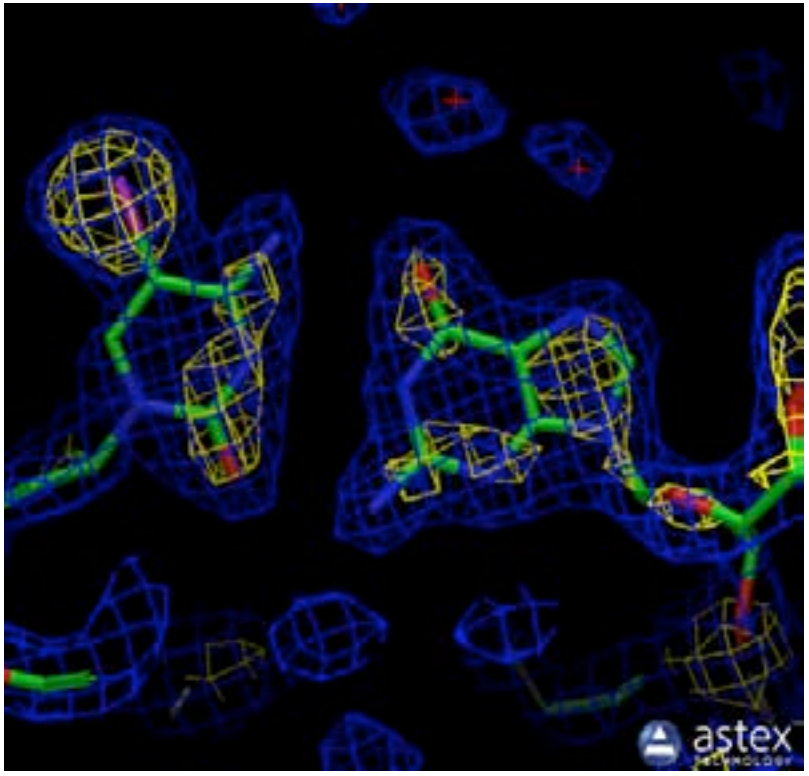


Coordinates deposited in the PDB are the result of:

1) Crystallographic electron density
2) Model constraints and biological information
3) Interpretation

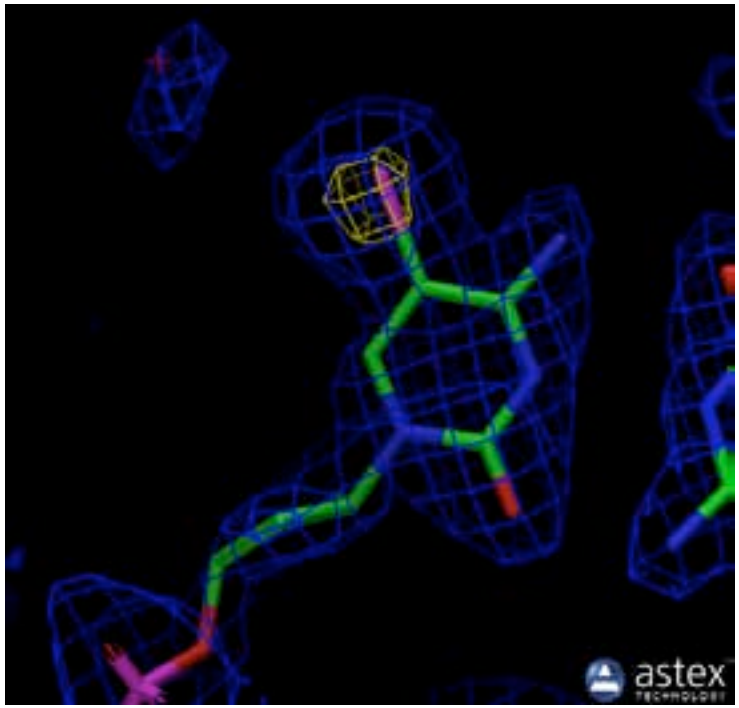


PDB entry 3dnb; 1.3 Å resolution



PDB entry 6bna; 2.21 Å resolution

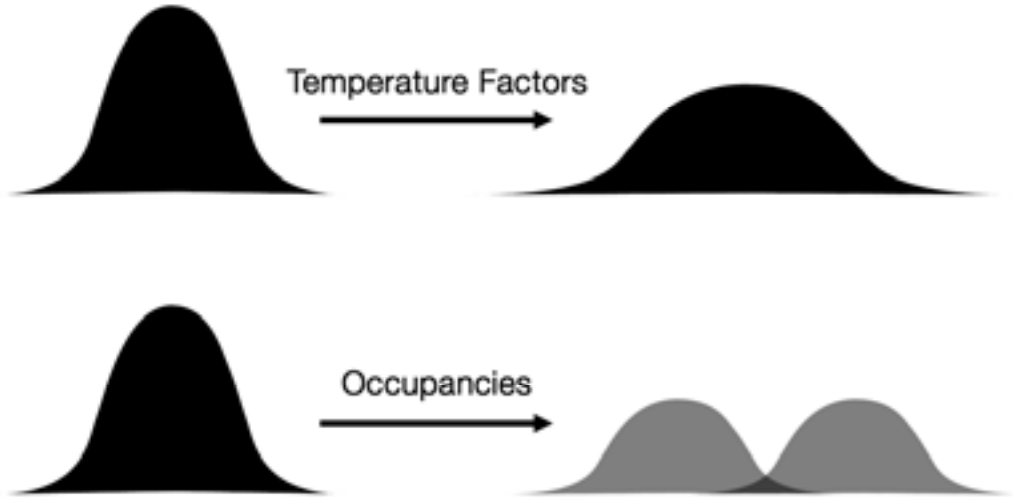
The final model is a compromise between the electron density and the model constraints.



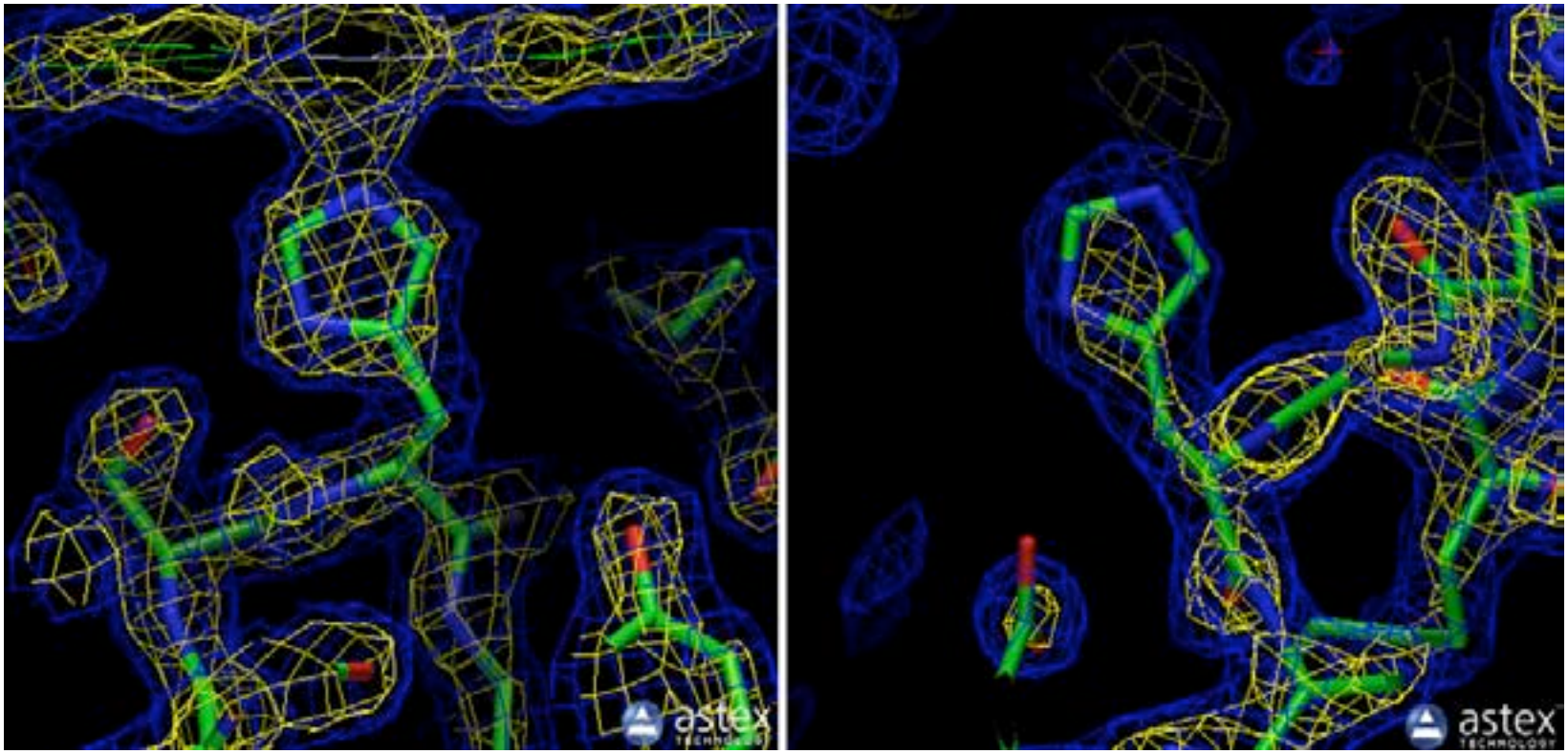
PDB entry 1bna

The atomic model is typically composed of:

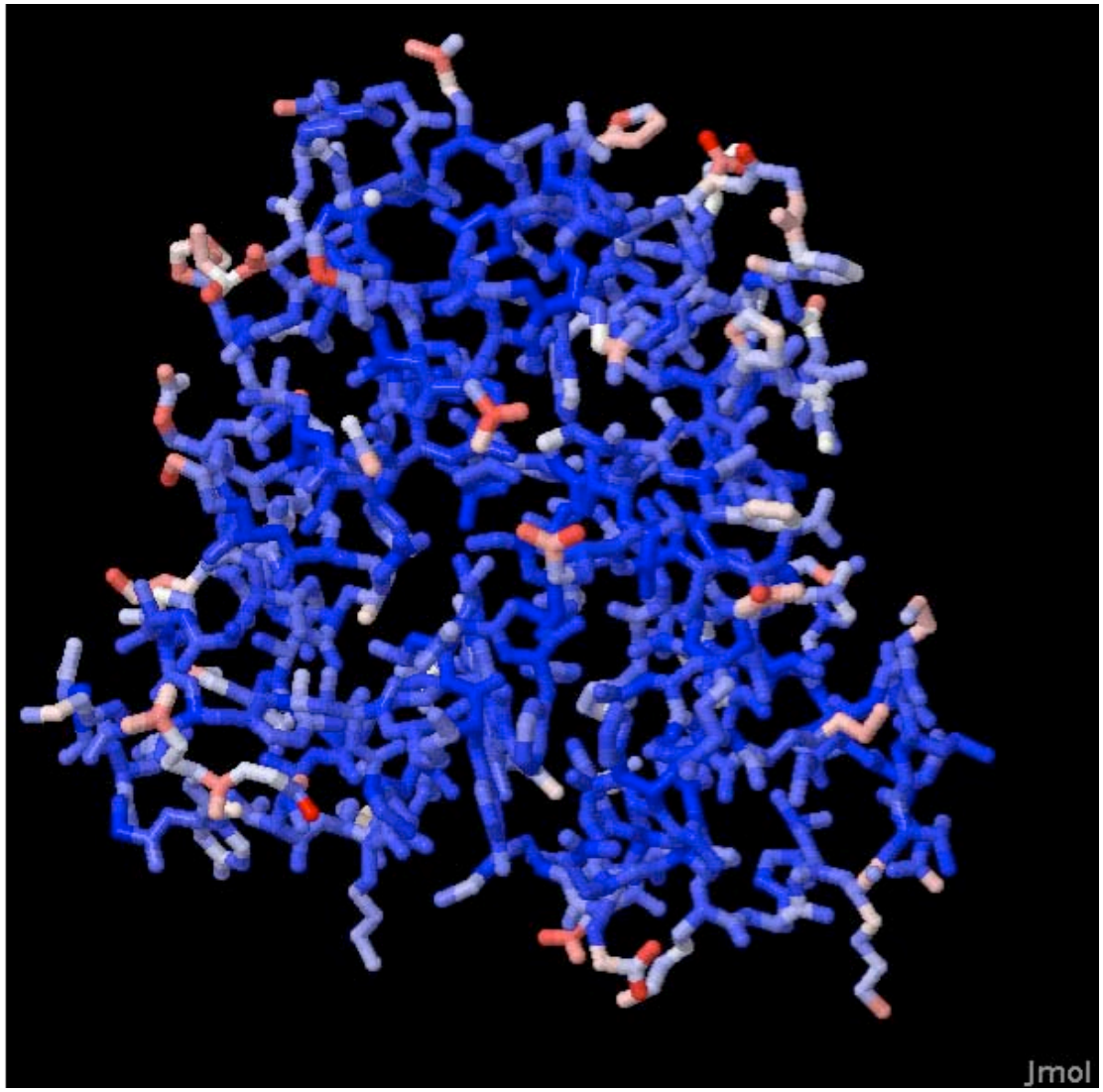
1) Coordinates of the atom centers
2) Atom types (number of electrons)
3) Temperature factors
4) Occupancies



Temperature factors model (primarily) vibrational disorder

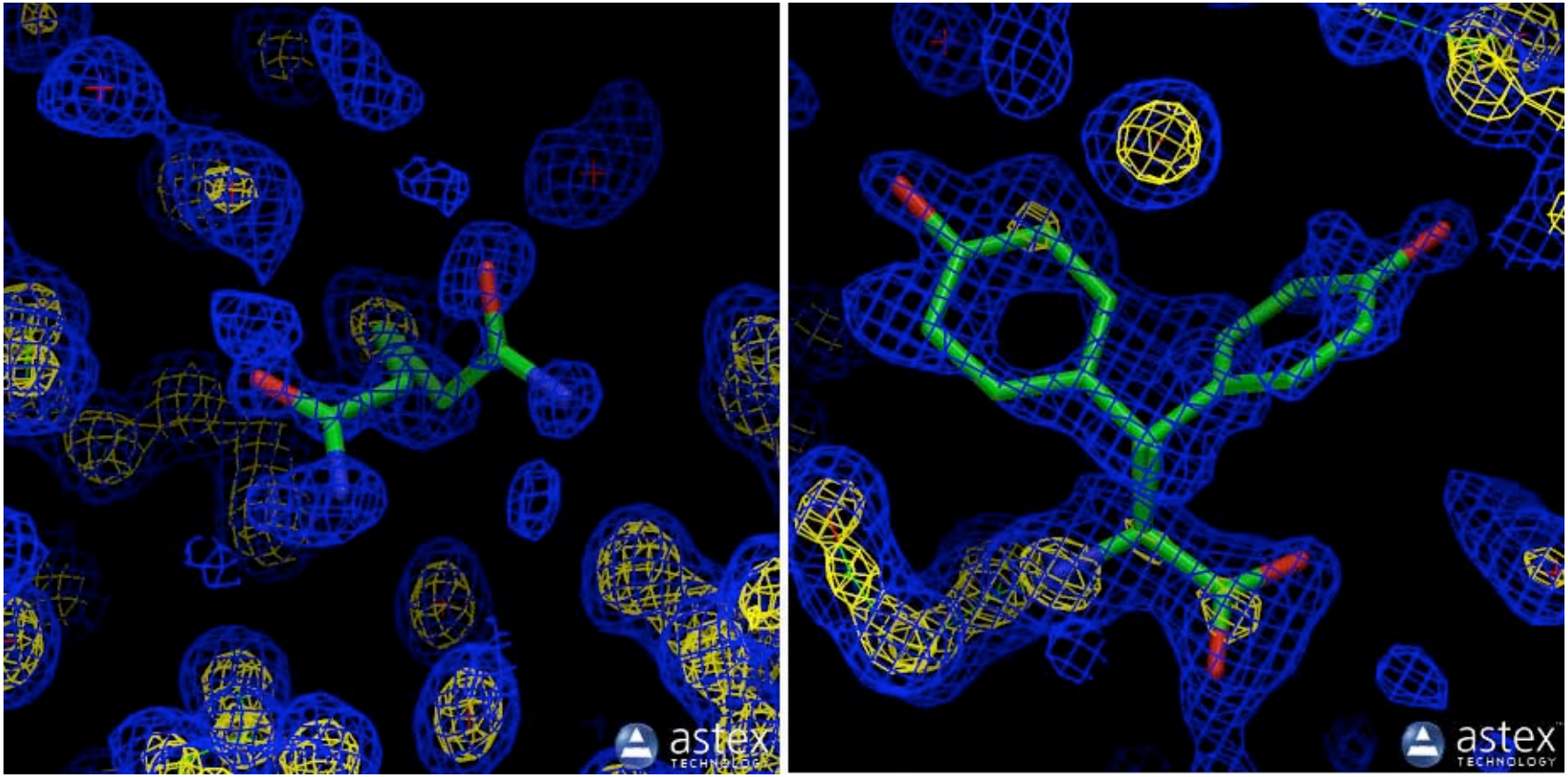


Two histidines from myoglobin (1mbi)



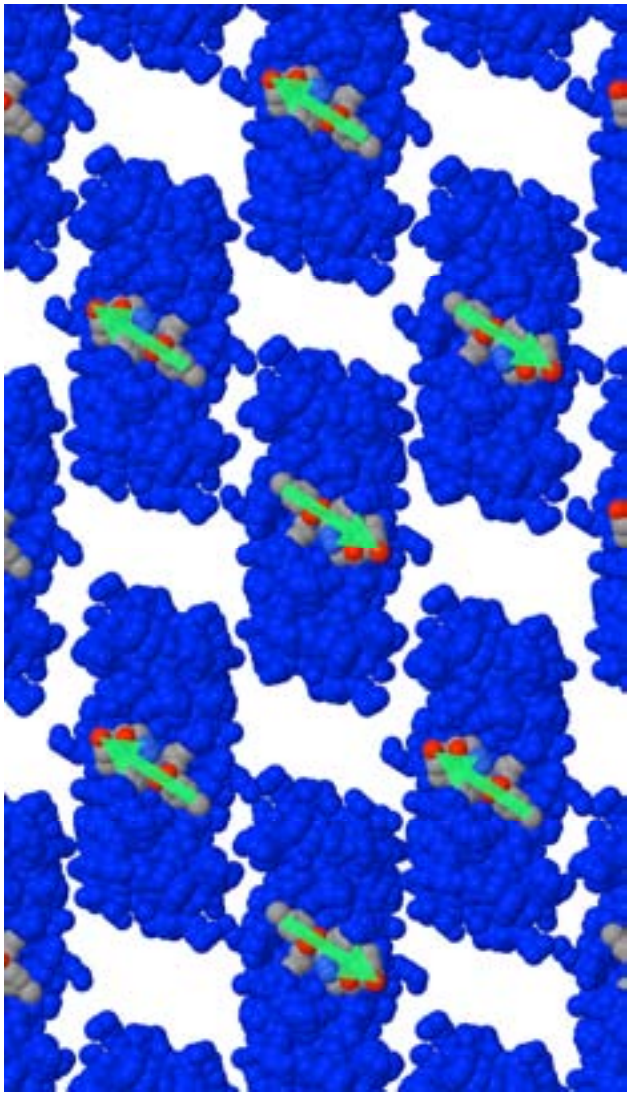
PDB entry 1mbi

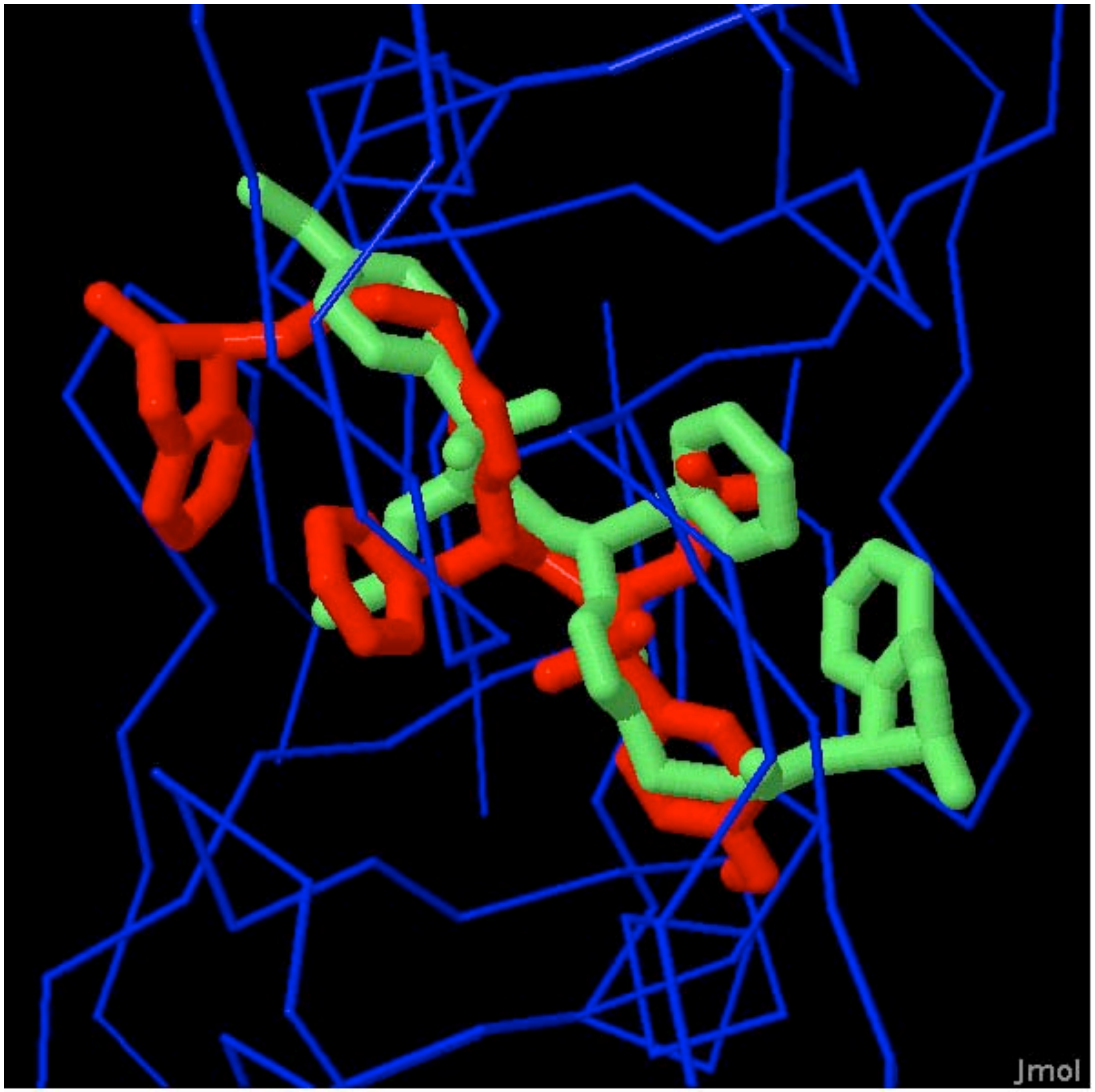
Occupancy values are used for cases of static disorder



PDB entry 1a6m

Static Disorder in HIV Protease

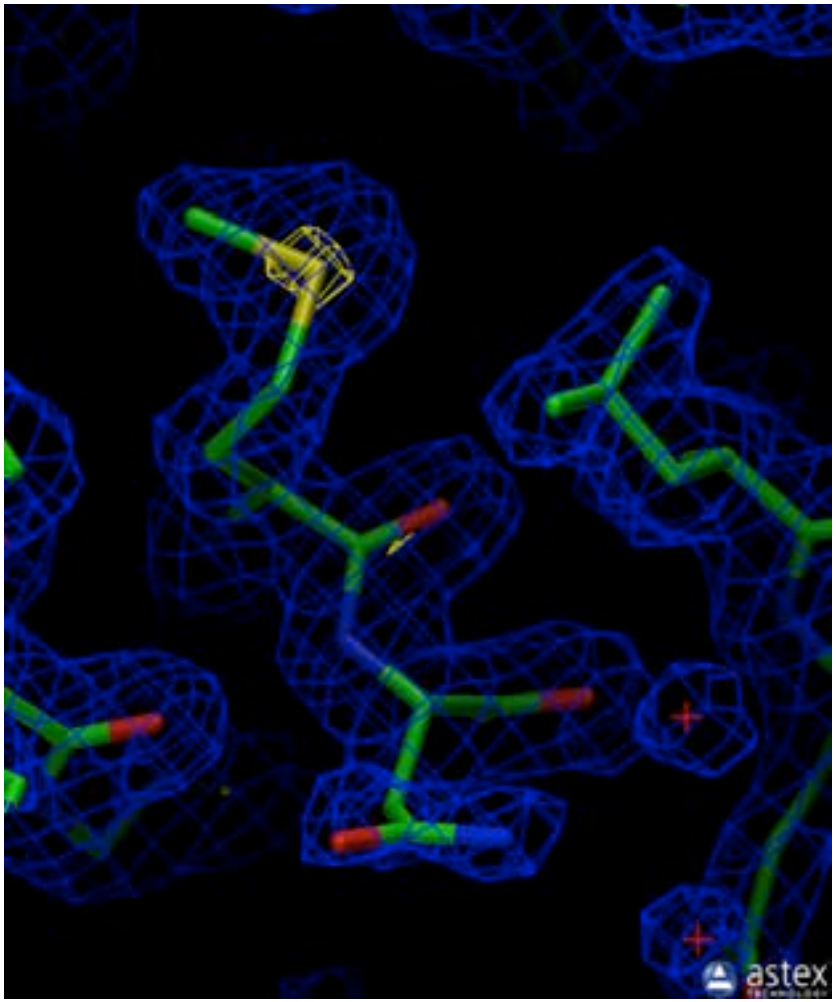




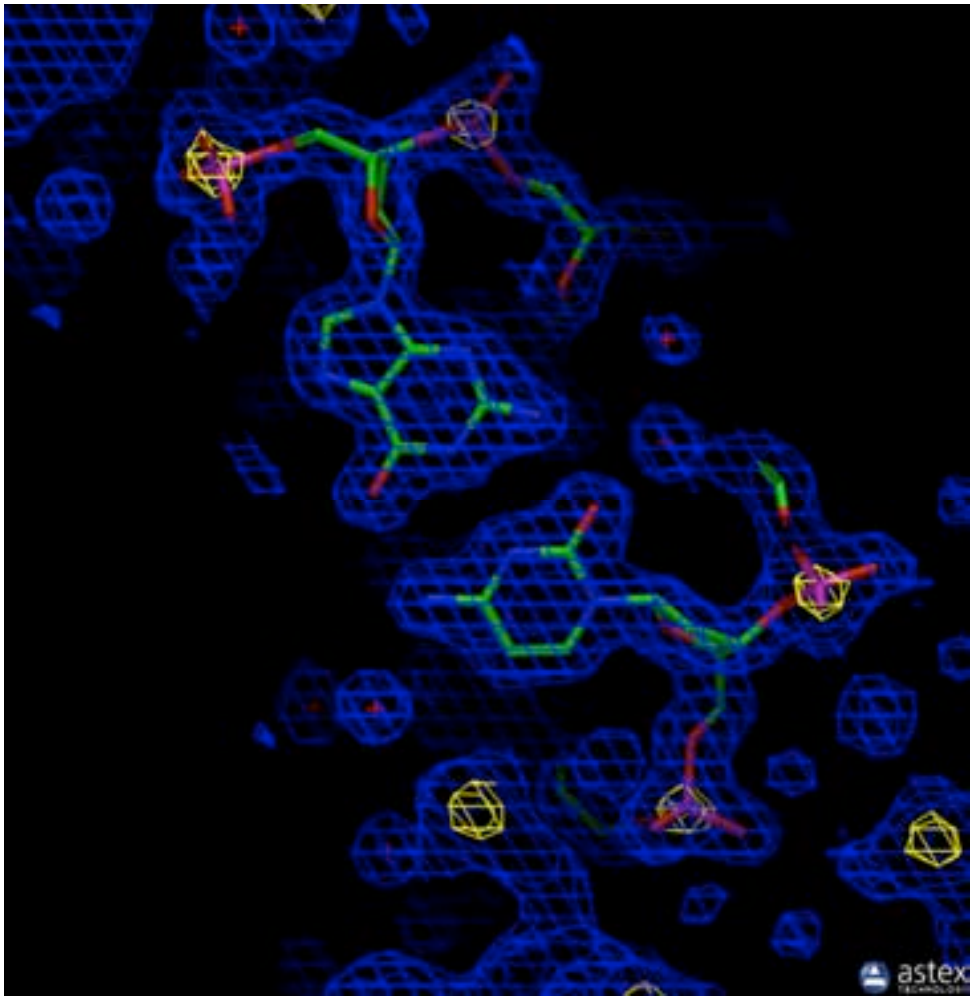
PDB entry 2hc0

Determining the Atom Type

Interpretation is often based on shape and chemistry.



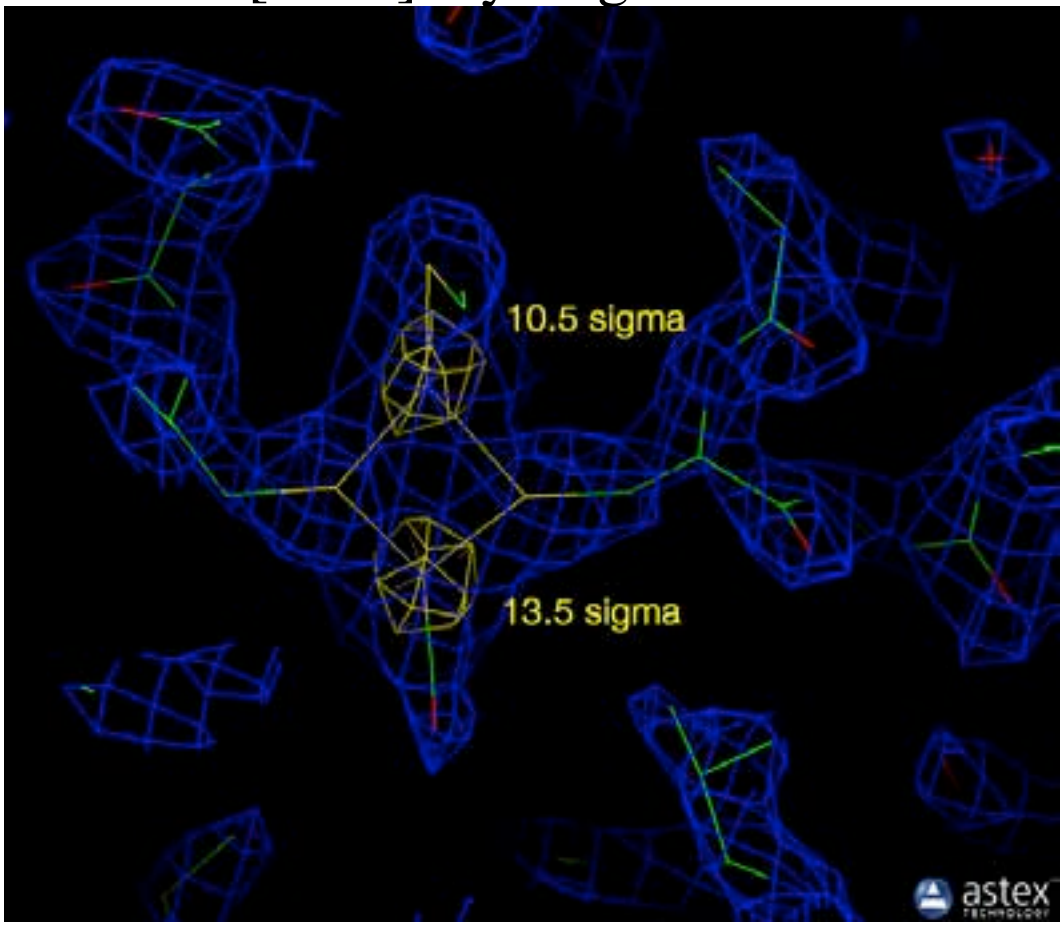
PDB entry 1mbi



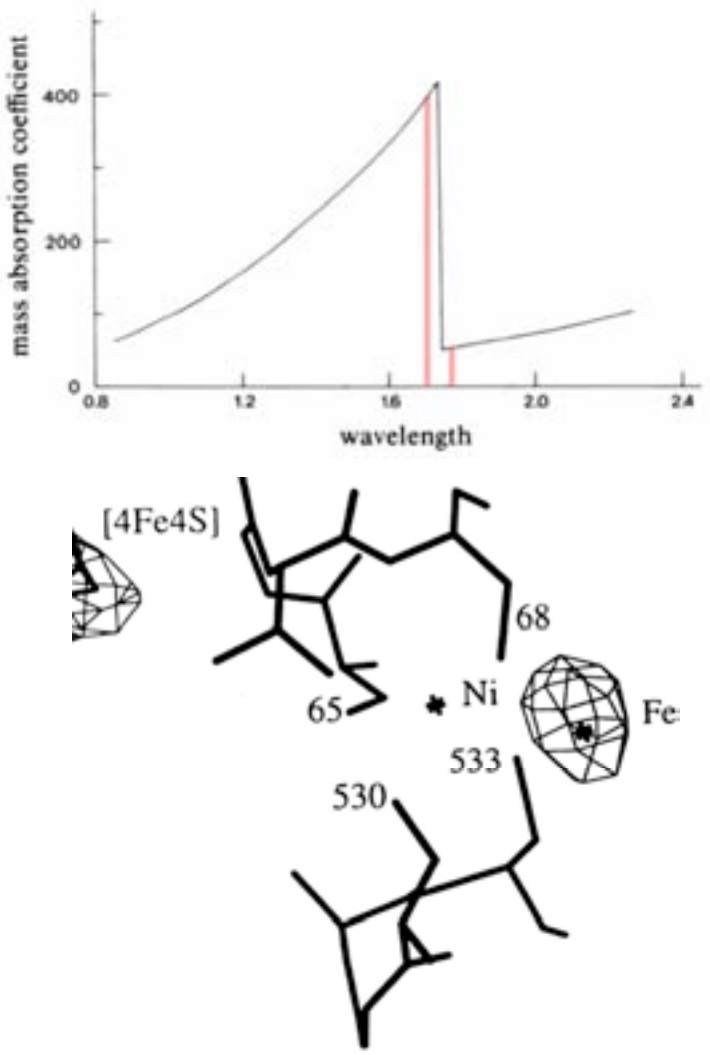
PDB entry 158d

Determining the Atom Type

[NiFe] Hydrogenase

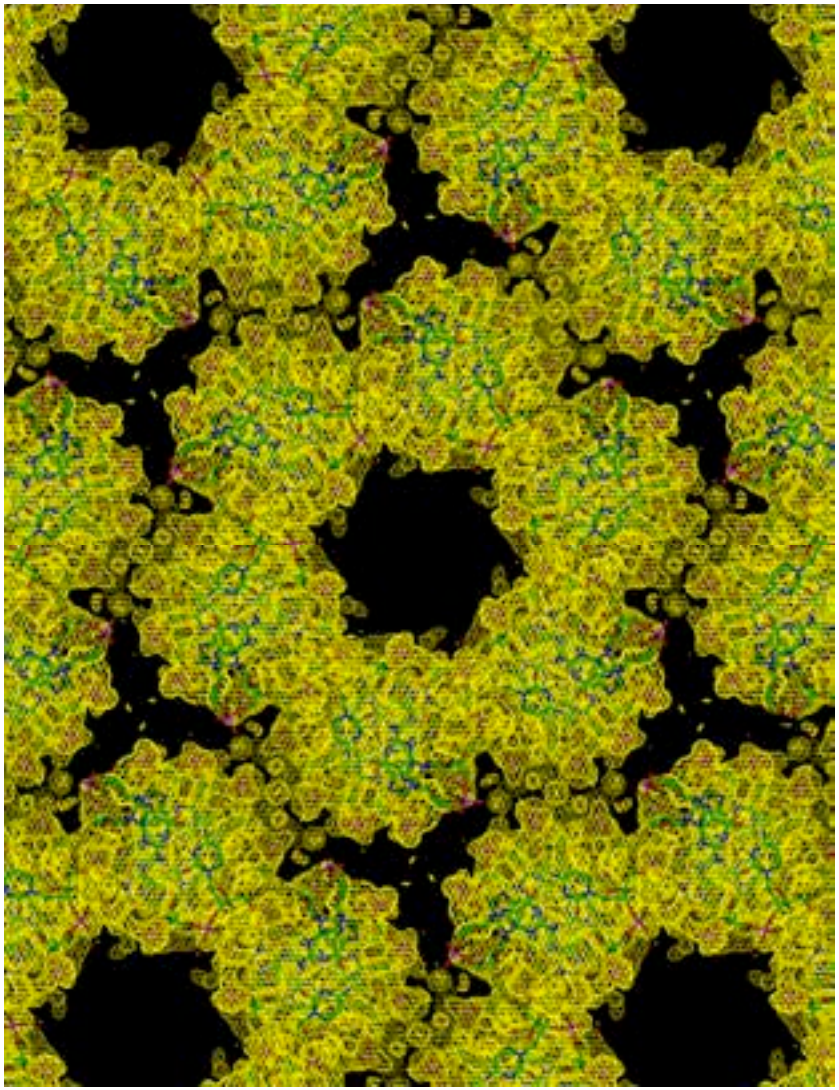


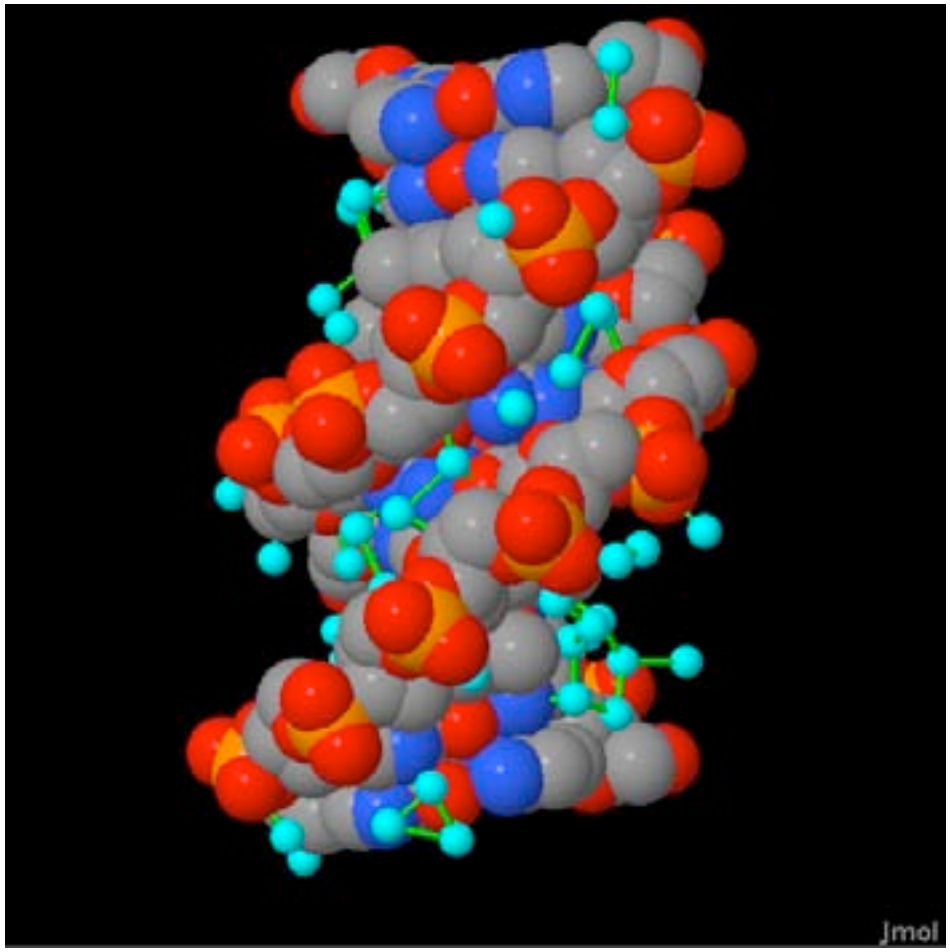
PDB entry 1yq9



JACS 118, 12989 (1996)

Ordered water molecules are typically observed in first and second coordination shells





PDB entry 158d

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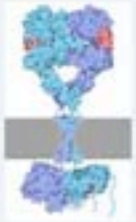

Molecule of the Month:

Epidermal Growth Factor

The cells in your body constantly communicate with each other, negotiating the transport and use of resources and deciding when to grow, when to rest, and when to die. Often, these messages are carried by small proteins, such as epidermal growth factor (EGF). EGF is a message telling cells that they have permission to grow. It is released by cells in areas of active growth, then is either picked up by the cell itself or by neighboring cells, stimulating their ability to divide. The message is received by a receptor on the cell surface, which binds to EGF and relays the message to signaling proteins inside the cell, ultimately mobilizing the processes needed for growth.

Full Article...

Protein Structure Initiative Featured Molecule:

RBBP9 (Retinoblastoma Binding Protein 9)

Researchers at **NESG** are using structural genomics to reveal the secrets of signaling within cancer cells. The structure of retinoblastoma binding protein 9 showed that it is a serine protease as well as a partner of retinoblastoma protein.

Full Article | **PSI Structural Genomics Knowledgebase**

Latest Structures Hide

3hsb - Crystal structure of YmaH (Hfq) from Bacillus subtilis in complex with an RNA aptamer
Baba, S., Someya, T., Kumasaka, T., Kawai, G., Nakamura, K.

Crystal structure of YmaH (Hfq) from Bacillus subtilis in complex with an RNA aptamer
Abstract not available.

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2010-06-22

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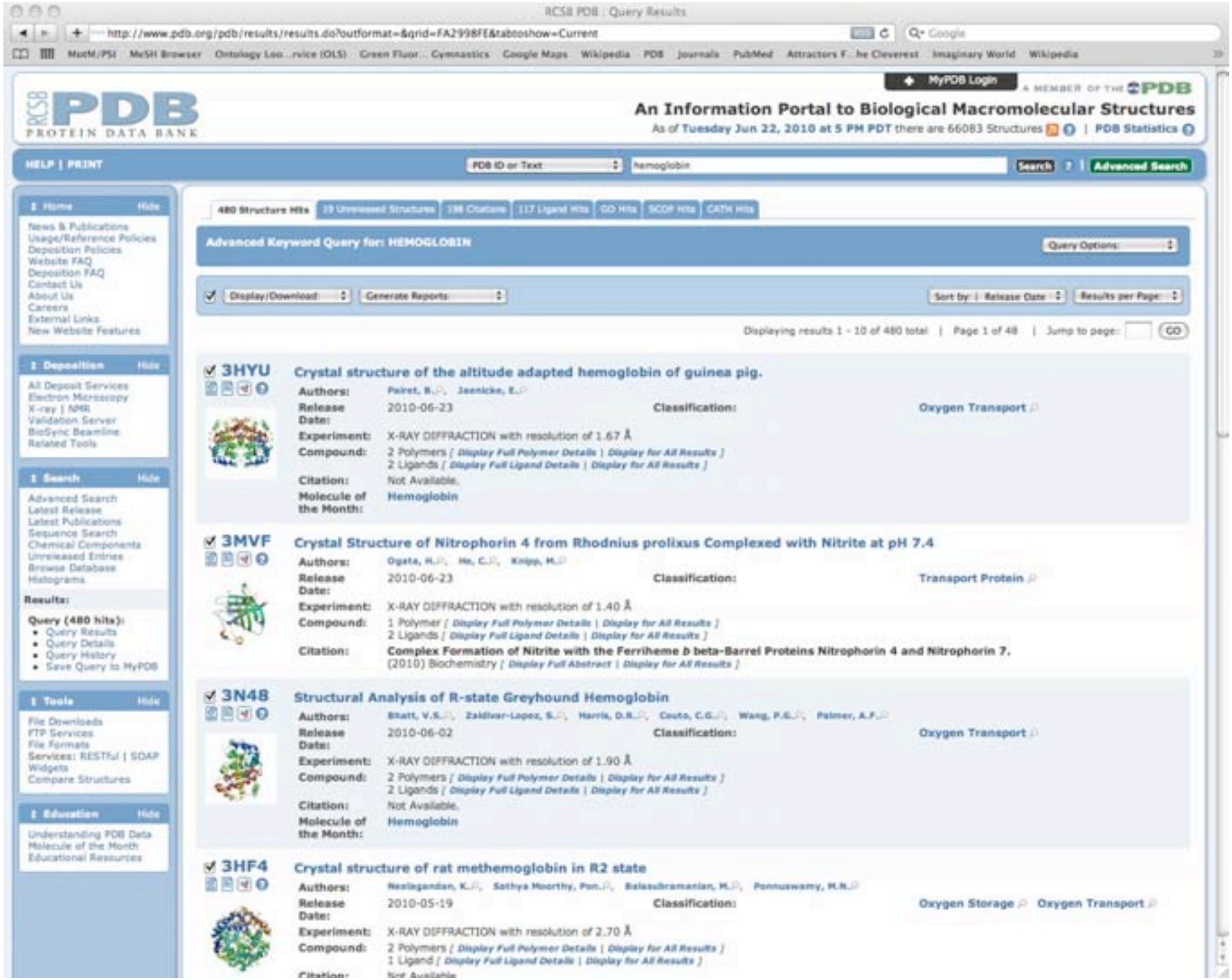
ftp://snapshots.wwpdb.org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Generate Reports:
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Results per Page:
Displaying results 1 - 10 of 480 total | Page 1 of 48 | Jump to page:
GO
3HYU
Crystal structure of the altitude adapted hemoglobin of guinea pig.
Authors: Peiret, B., Jaenicke, E.
Release Date: 2010-06-23
Classification: Oxygen Transport
Experiment: X-RAY DIFFRACTION with resolution of 1.67 Å
Compound: 2 Polymers [Display Full Polymer Details | Display for All Results]
2 Ligands [Display Full Ligand Details | Display for All Results]
Citation: Not Available.
Molecule of the Month: Hemoglobin
3MVF
Crystal Structure of Nitrophorin 4 from Rhodnius prolixus Complexed with Nitrite at pH 7.4
Authors: Ogata, H., He, C., Knipp, M.
Release Date: 2010-06-23
Classification: Transport Protein
Experiment: X-RAY DIFFRACTION with resolution of 1.40 Å
Compound: 1 Polymer [Display Full Polymer Details | Display for All Results]
2 Ligands [Display Full Ligand Details | Display for All Results]
Citation: Complex Formation of Nitrite with the Ferriheme b beta-Barrel Proteins Nitrophorin 4 and Nitrophorin 7. (2010) Biochemistry [Display Full Abstract | Display for All Results]
3N48
Structural Analysis of R-state Greyhound Hemoglobin
Authors: Bhatt, V.S., Zaldivar-Lopez, S., Harris, D.R., Couto, C.G., Wang, P.G., Palmer, A.F.
Release Date: 2010-06-02
Classification: Oxygen Transport
Experiment: X-RAY DIFFRACTION with resolution of 1.90 Å
Compound: 2 Polymers [Display Full Polymer Details | Display for All Results]
2 Ligands [Display Full Ligand Details | Display for All Results]
Citation: Not Available.
Molecule of the Month: Hemoglobin
3HF4
Crystal structure of rat methemoglobin in R2 state
Authors: Neelagandan, K., Sathya Moorthy, Pon., Balasubramanian, M., Ponnuswamy, M.N.
Release Date: 2010-05-19
Classification: Oxygen Storage, Oxygen Transport
Experiment: X-RAY DIFFRACTION with resolution of 2.70 Å
Compound: 2 Polymers [Display Full Polymer Details | Display for All Results]
1 Ligand [Display Full Ligand Details | Display for All Results]

RCSB PDB : Structure Summary for 3HYU – Crystal structure of the altitude adapted hemoglobin of guinea pig.

http://www.pdb.org/pdb/explore/explore.do?structureId=3HYU

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Crystal structure of the altitude adapted hemoglobin of guinea pig.

DOI:10.2210/pdb3hyu/pdb

3HYU

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Primary Citation

Crystal structure of the altitude adapted hemoglobin of guinea pig.

B., Pairet, E., Jaenicke

Journal: To be Published

Not in PubMed

Molecular Description

Classification: Oxygen Transport

Structure Weight: 32812.88

Molecule: Hemoglobin subunit alpha
Polymer: 1 Type: polypeptide(L) Length: 141
Chains: A

Molecule: Hemoglobin subunit beta
Polymer: 2 Type: polypeptide(L) Length: 146
Chains: B

Source

Polymer: 1
Scientific Name: Cavia porcellus Common Name: Guinea pig

Polymer: 2
Scientific Name: Cavia porcellus Common Name: Guinea pig

Ligand Chemical Component

Identifier	Name	Formula	Interaction View
HEM	PROTOPORPHYRIN IX CONTAINING FE	C34 H32 Fe N4 O4	Ligand Explorer
PO4	PHOSPHATE ION	O4 P	Ligand Explorer

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Biological Assembly

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Biological assembly assigned by authors

Deposition Summary

Authors: Pairet, B., Jaenicke, E.

Deposition: 2009-06-23
Release: 2010-06-23

Experimental Details

Method: X-RAY DIFFRACTION
Experimental Data:
Resolution(Å): 1.67
R-Value: 0.179 (obs.)
R-Free: 0.203
Space Group: C 2 2 2_1
Unit Cell:

Length (Å)	Angles (°)
a = 83.54	α = 90.00
b = 89.95	β = 90.00
c = 82.72	γ = 90.00

Structure Links

Molecule of the Month Features:

- Hemoglobin

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The RCSB PDB is managed by two members of the RCSB: Rutgers and UCSD, and is funded by NSF, NIGMS, DOE, NLM, NCI, NINDS, and NIDDK.

June 2010 **Molecule of the Month** by David Goodsell **Previous Features**
doi: **10.2210/rcsb_pdb/mom_2010_6**

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Epidermal Growth Factor

Keywords: cell signaling, cancer, ErbB, HER

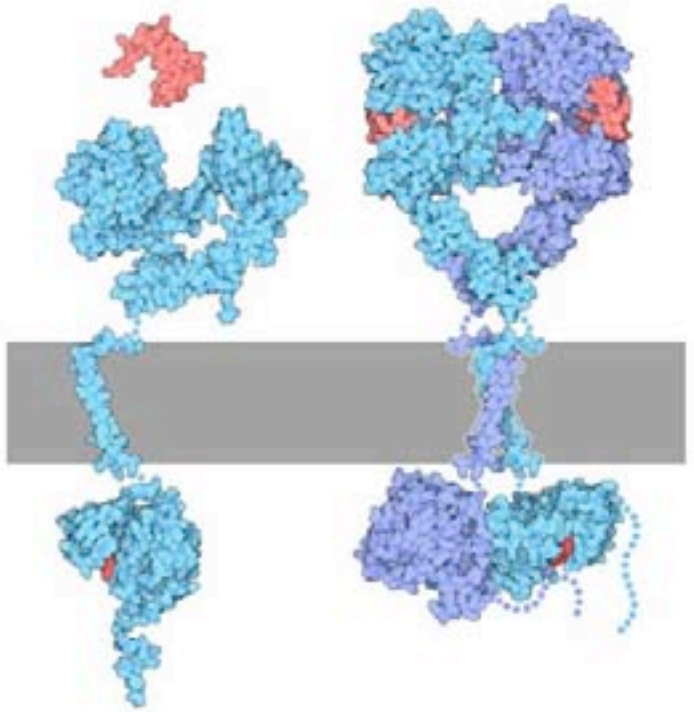
Contents

1. Introduction

The cells in your body constantly communicate with each other, negotiating the transport and use of resources and deciding when to grow, when to rest, and when to die. Often, these messages are carried by small proteins, such as epidermal growth factor (EGF), shown here in red from PDB entry 1egf. EGF is a message telling cells that they have permission to grow. It is released by cells in areas of active growth, then is either picked up by the cell itself or by neighboring cells, stimulating their ability to divide. The message is received by a receptor on the cell surface, which binds to EGF and relays the message to signaling proteins inside the cell, ultimately mobilizing the processes needed for growth.

2. Domains and Dimers

The EGF receptor, shown here in blue, is a flexible protein with many moving parts, including a large extracellular portion, a section that crosses the cell membrane, a kinase domain and a long flexible tail. The portion facing outwards from the cell, shown at the top here, is composed of four articulated domains that recognize EGF. When EGF is not around, it folds back on itself, as shown in the structure on the left. Then, when EGF binds, the receptor opens up and binds to another copy of the receptor, forming the dimeric complex shown on the right. This brings together two copies of the kinase domain, shown at the bottom here. Since the kinase domains are close to one another, they can add phosphate groups to tyrosine amino acids on the long, flexible tails of the receptor (the tails are not seen in this structure, so they are shown here with dots). The phosphorylated tails then stimulate the signaling proteins inside the cell.



3. Structural Surprises

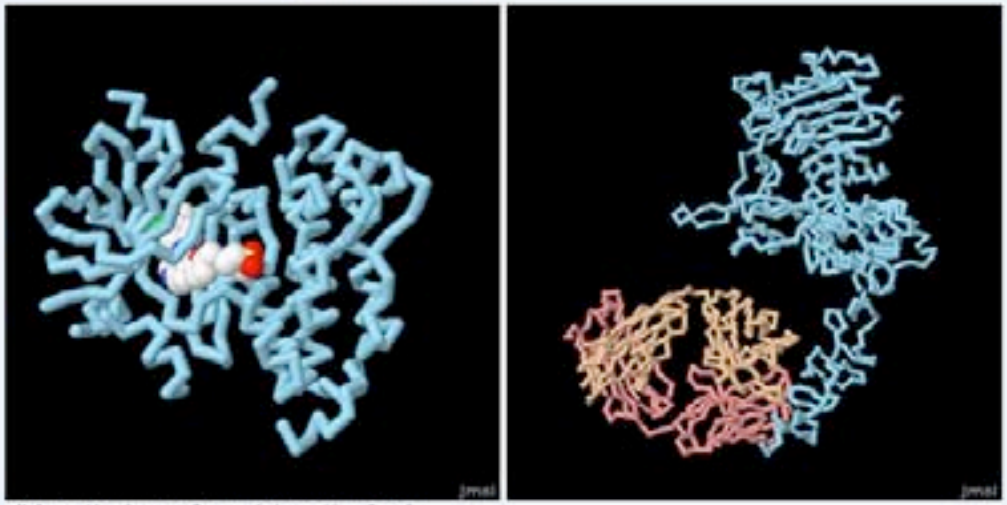
Since the EGF receptor is so flexible, it has been studied by breaking it into several pieces and studying each one separately. Consequently, several PDB files were needed to create this illustration, including 1nql, 1ivo, 2jwa, 1m17 and 2gs6. The structures of the different parts of the EGF receptor revealed several surprises. First of all, researchers found that EGF binds on either side of the receptor complex, not in the middle like other similar receptors. EGF appears to mold the receptor into the proper shape for dimerization, instead of acting like glue between the two chains. Also, careful analysis of the kinase domain showed that it is activated by association in an asymmetric head-to-tail fashion, in spite of the symmetric association of the extracellular portion.

4. Large Family

EGF and the EGF receptor are part of an extended family of proteins that together control aspects of cell growth and development. These include at least seven similar protein messages, such as transforming growth factor alpha and amphiregulin, and four receptors, collectively termed ErbB or HER receptors. These messages and receptors can mix and match, with different messages bringing together two identical receptors or two different receptors. In this way, a wide variety of messages may be carried by the system, tailored for the needs of each type of cell.

6. Exploring the Structure

Exploring the Structure | Jmol 1 | Jmol 2

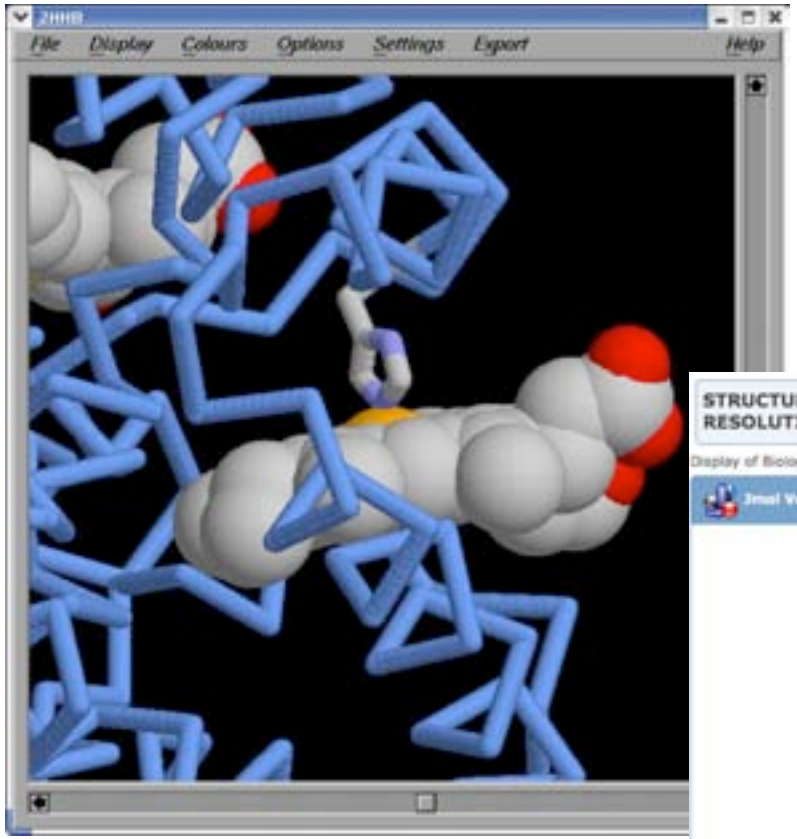


click on the image for an interactive Jmol

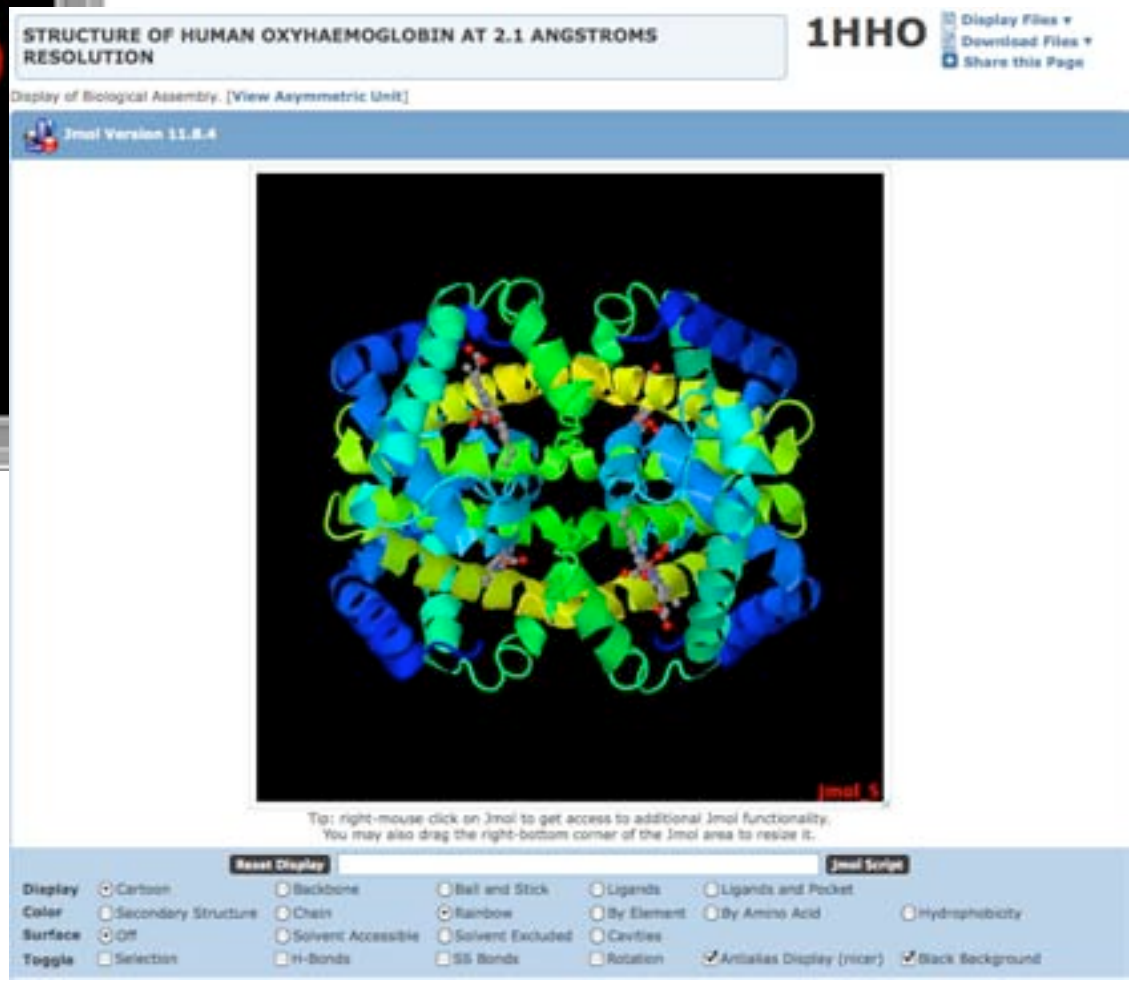
The signal carried by EGF can be dangerous if used improperly. Many forms of cancer circumvent the normal EGF signaling process, giving themselves permission to grow without control. Because of this, drugs that block EGF signaling are effective for the treatment of cancer. Two examples are shown here. At the left, the drug lapatinib is bound in the kinase domain of the receptor, blocking the signal inside the cell (PDB entry 1xkk). It is very similar to the ATP used by the receptor, and binds tightly in the active site. Therapeutic antibodies are also used for cancer treatment. Herceptin is shown on the right bound to the extracellular domain of HER2/ErbB2 (PDB entry 1n8z), and the antibody cetuximab bound to the EGF receptor may be found in PDB entry 1yy9. To explore these structures in more detail, click on the images for an interactive Jmol image.

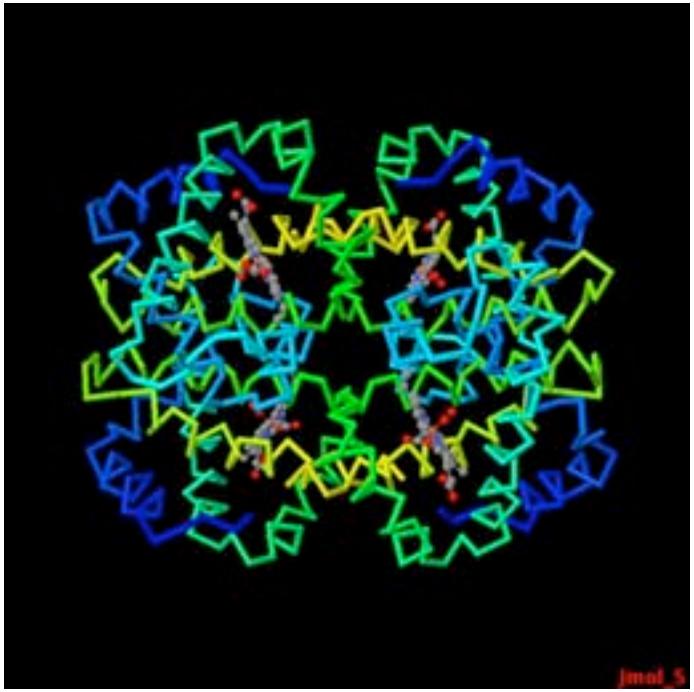
7. Topics for further exploration

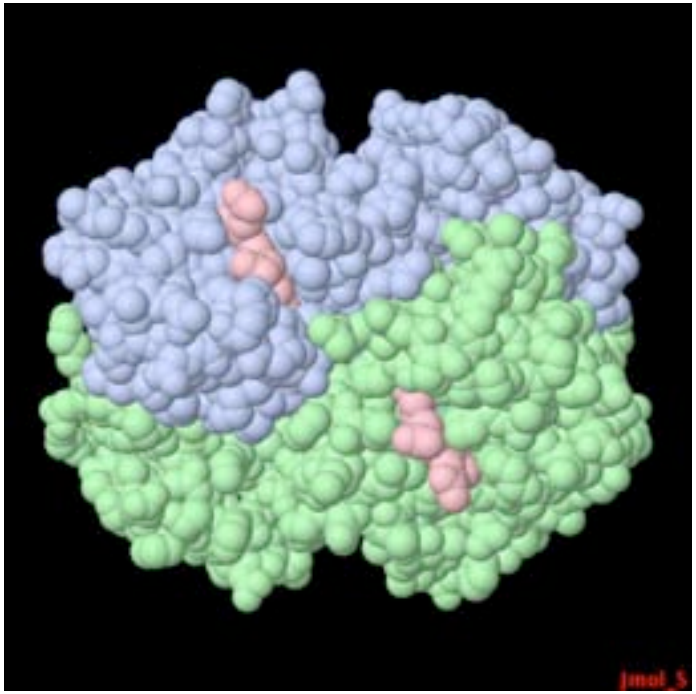
1. Compare the mode of dimerization in the EGF receptor and the **human growth hormone receptor**.
2. EGF receptor and similar receptors are currently the targets of development of drugs for cancer therapy. Can you find other examples of structures in the PDB with drugs?

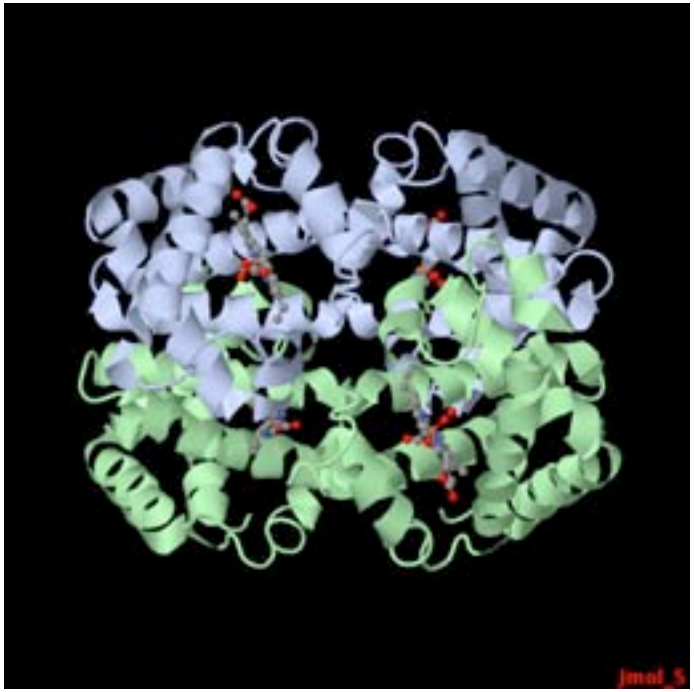


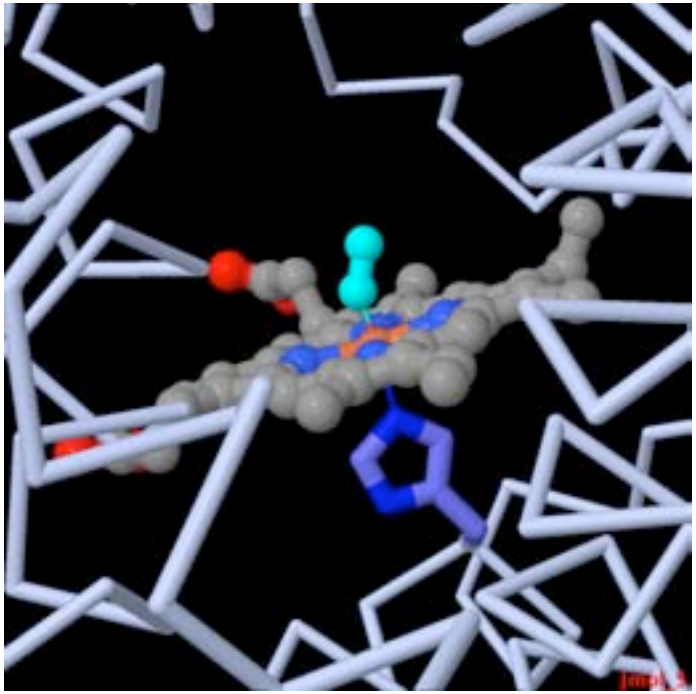
Take the time to become *really* comfortable with an interactive molecular viewer

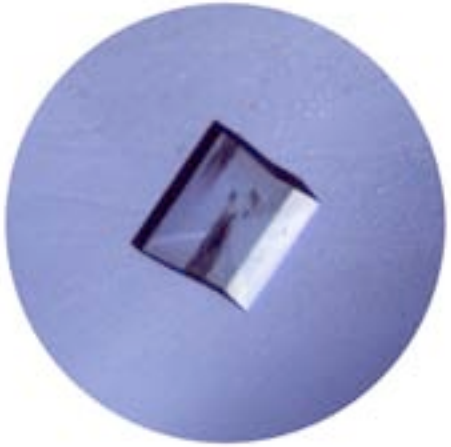








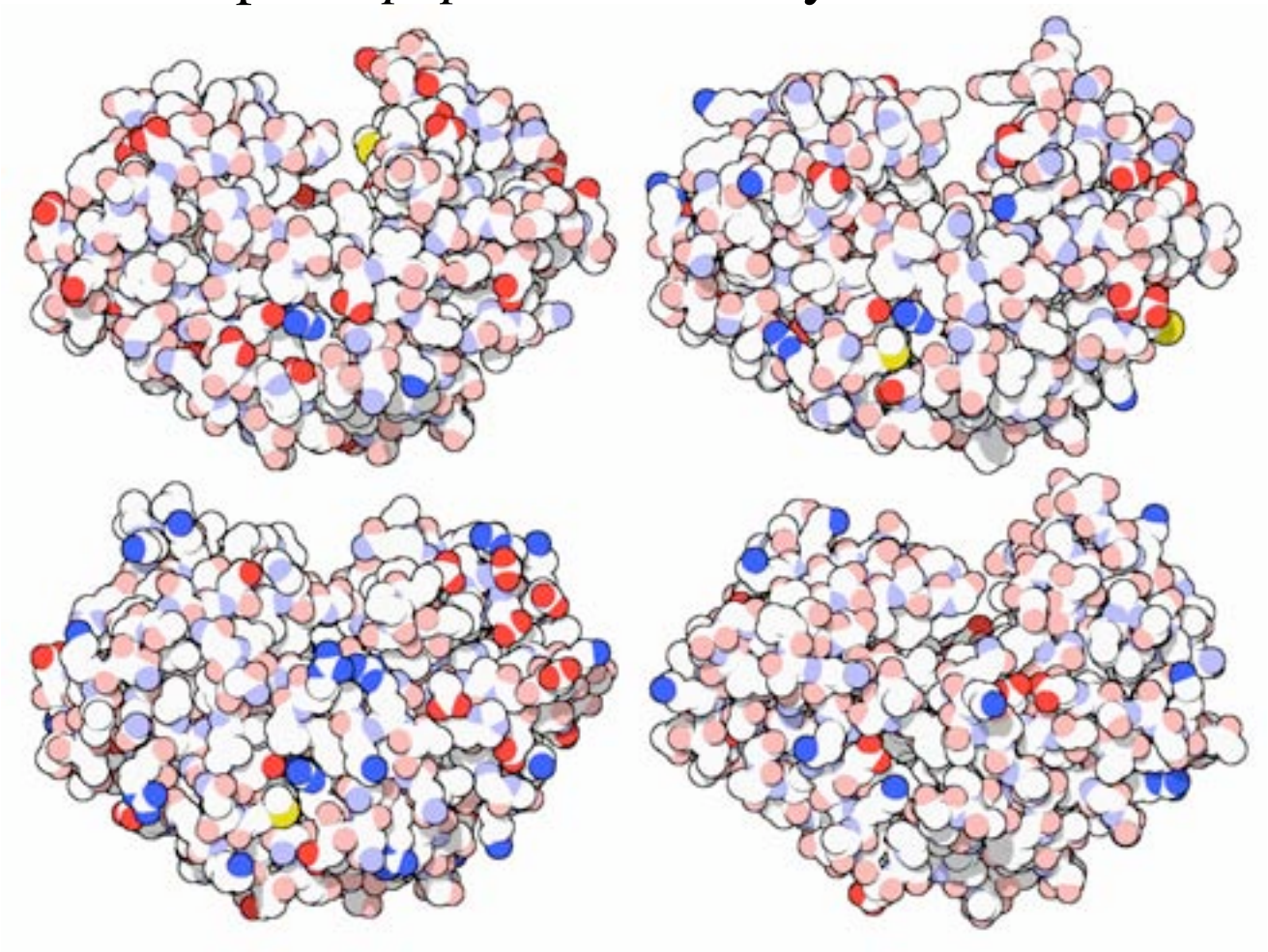




Crystallographers will do *anything* that they need to do to get crystals

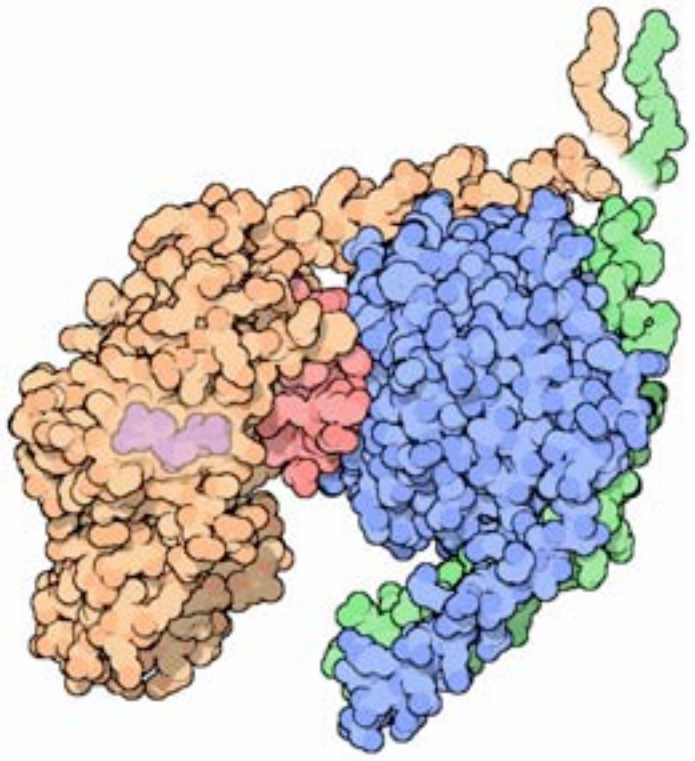
Pepsin *5pep*

Chymosin *4cms*



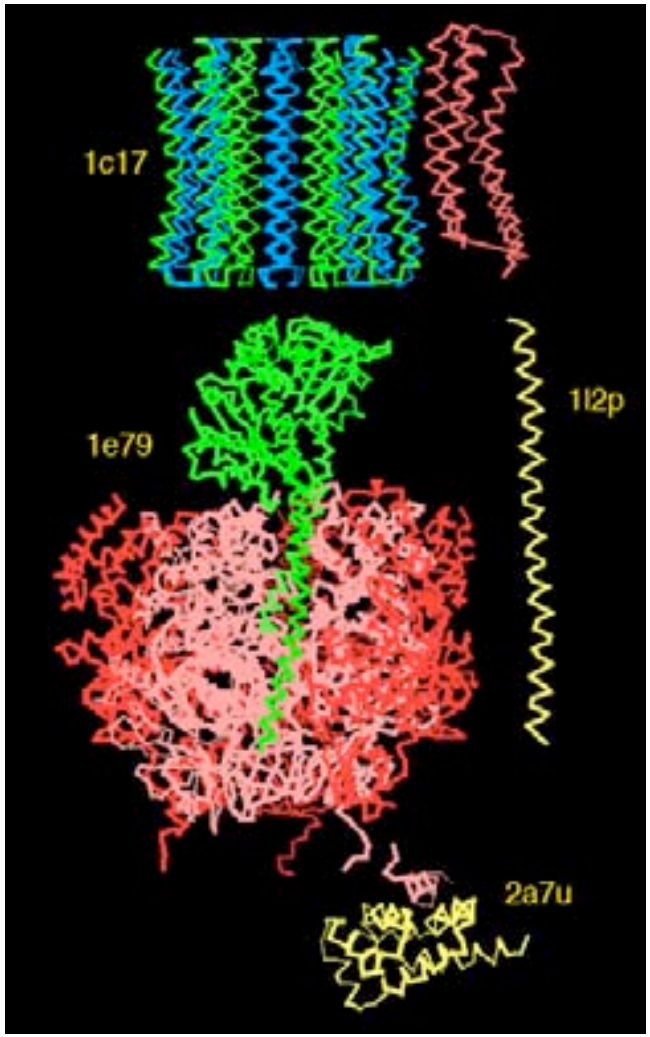
Cathepsin D *1lyb*

Endothiapepsin *4ape*



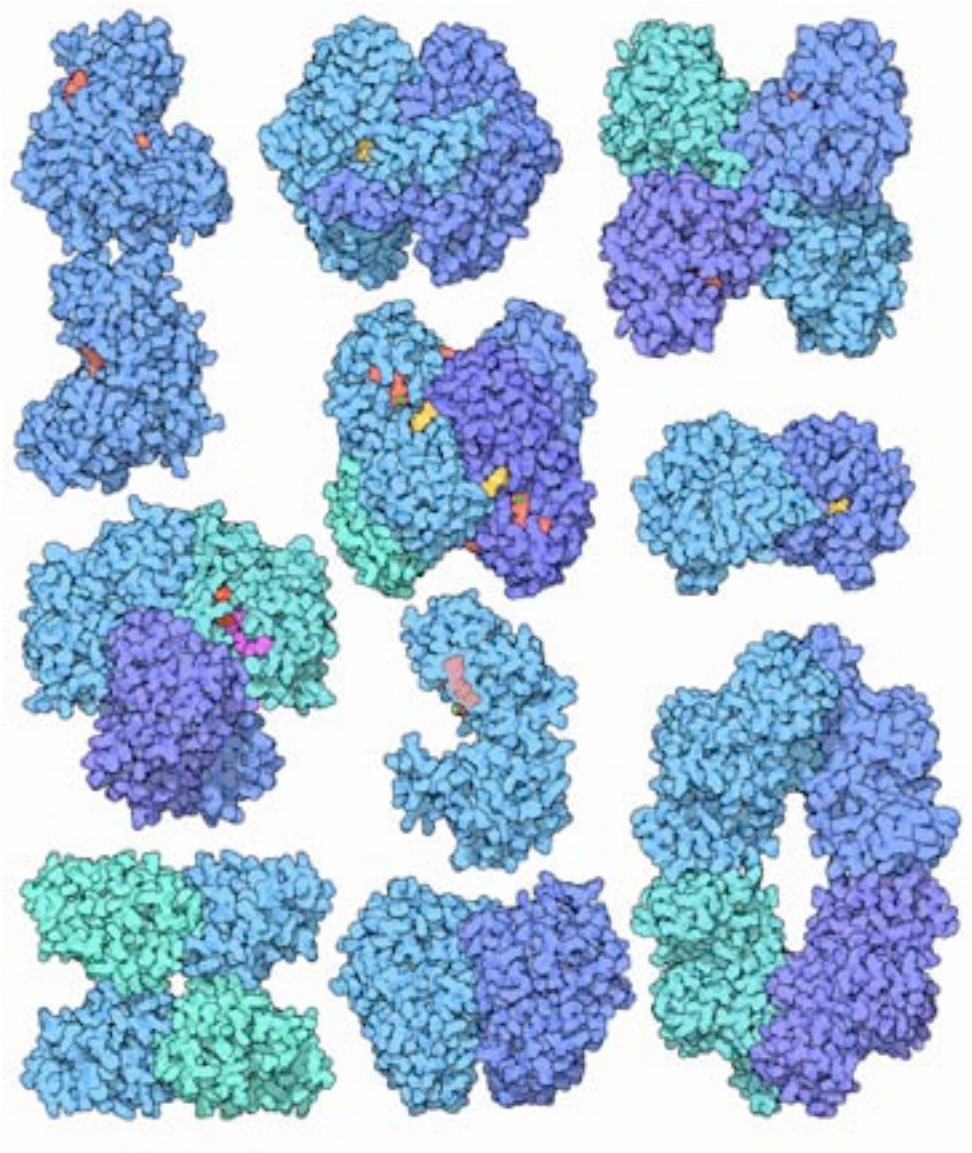
G Protein *1gg2*

Kinesin *3kin*



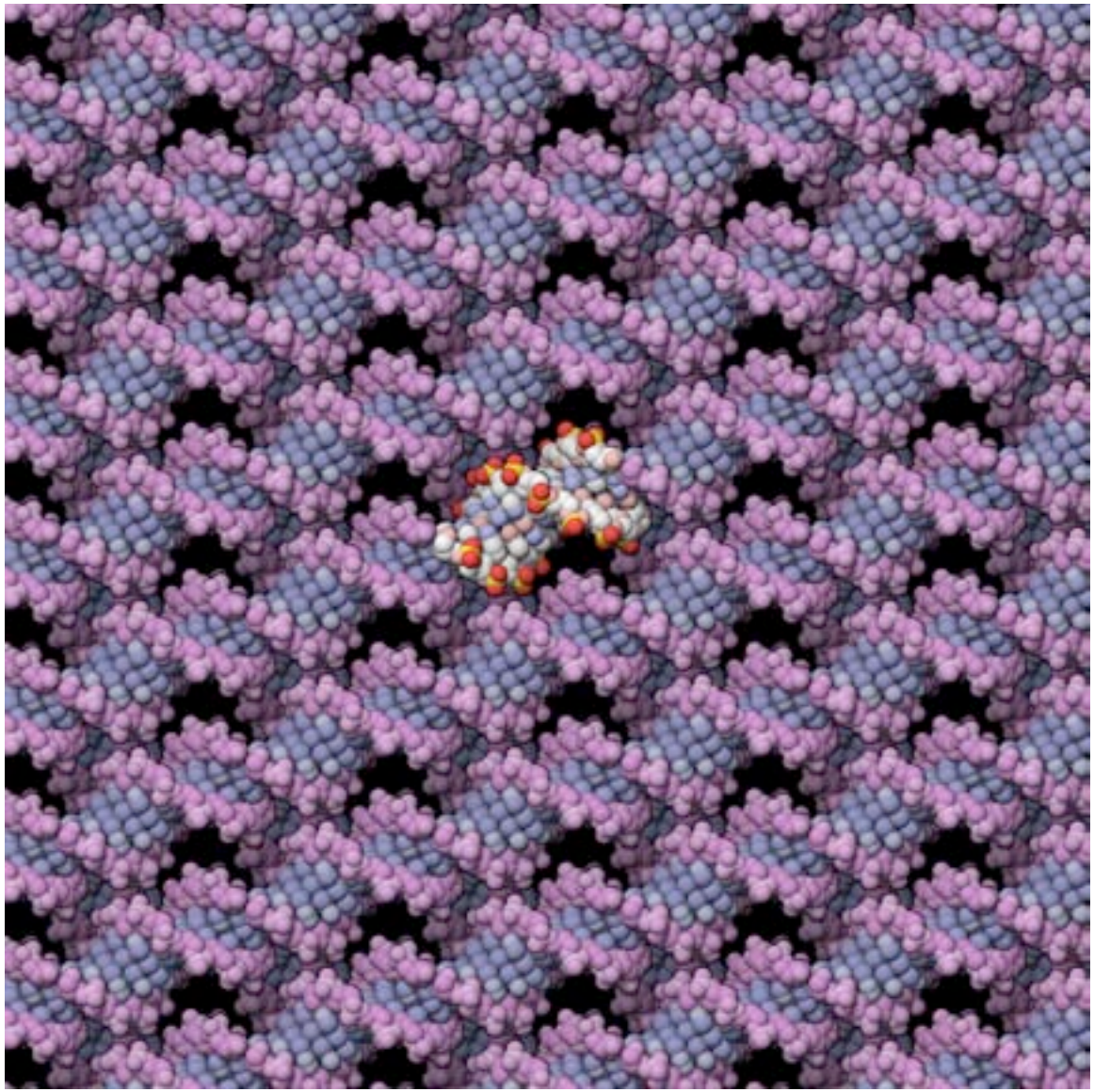
ATP synthase

The structure may include only a fragment of entire protein

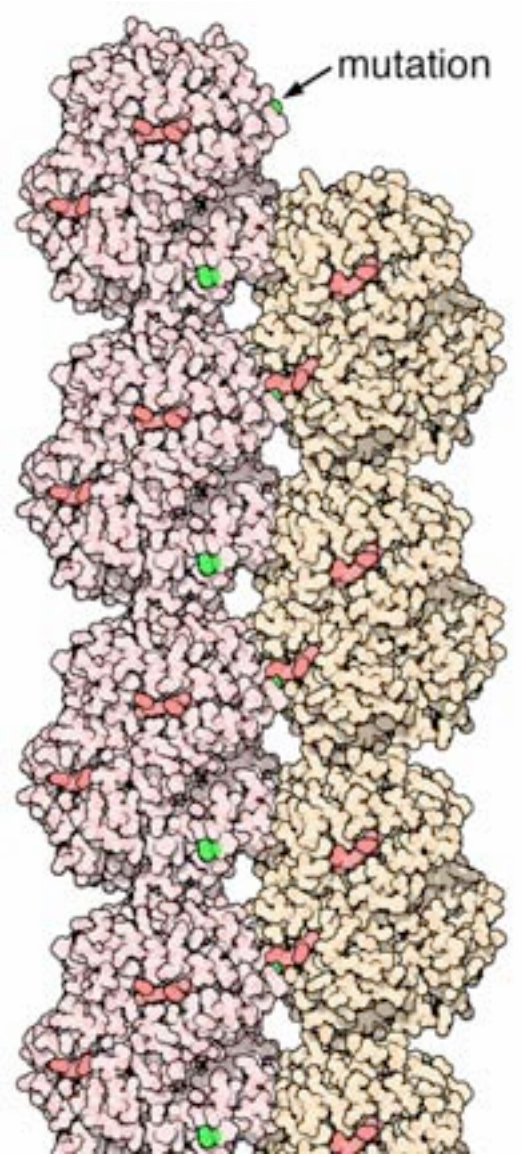


Glycolysis

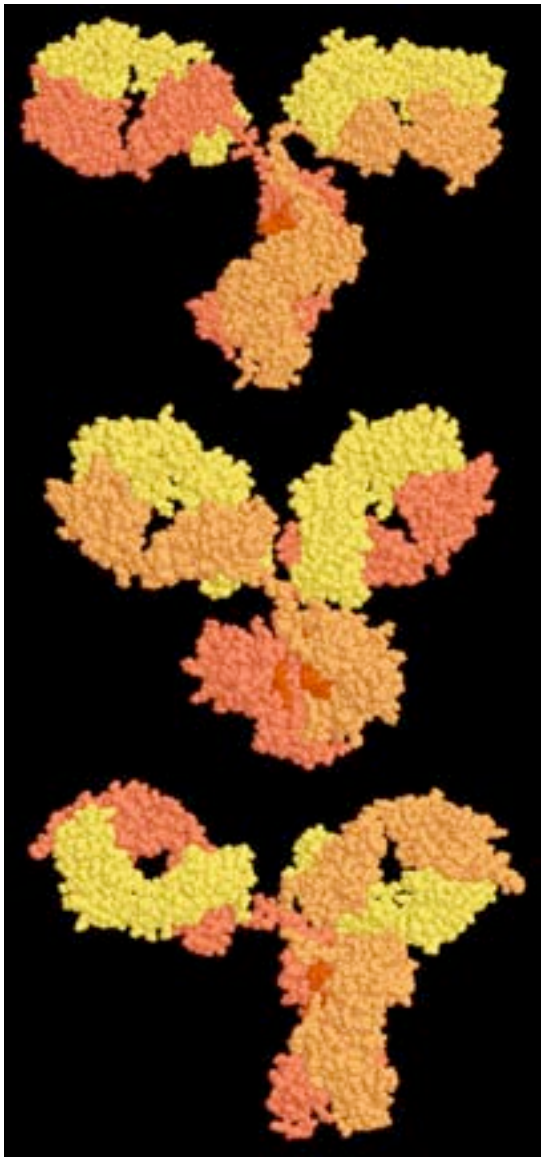
1dgk human
1hox rabbit
4pfk bacterial
4ald human
2ypi yeast
3gpd human
3pgk yeast
3pgm yeast
2one yeast
1e0u bacterial



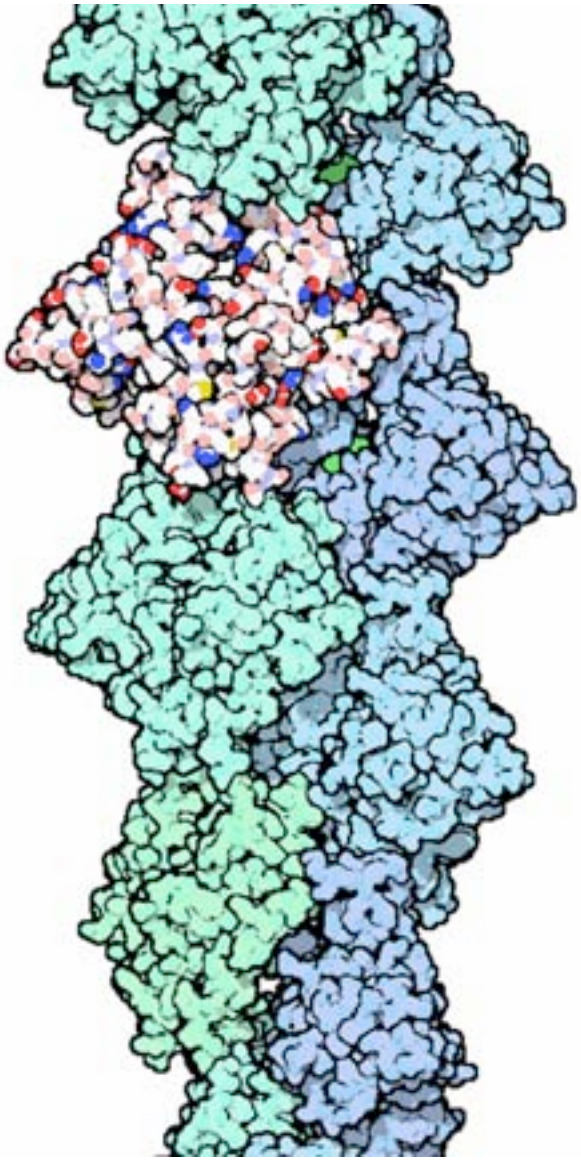
Crystals are a strange environment for biological molecules



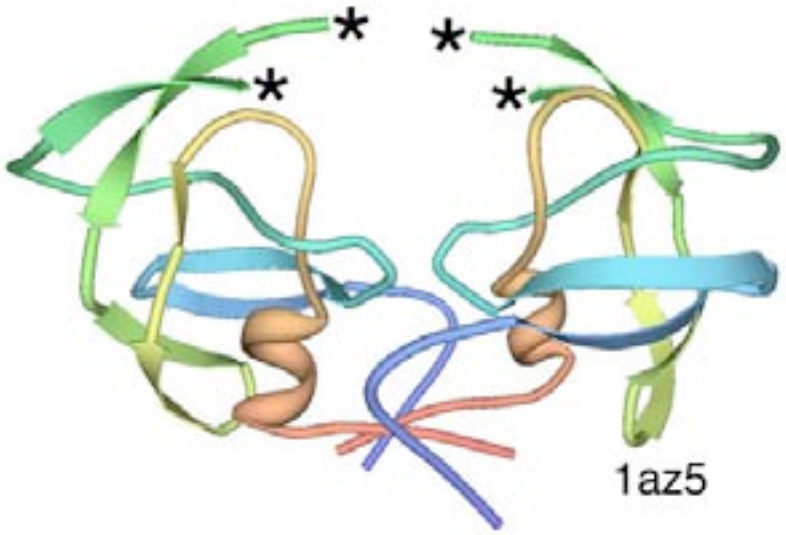
Biologically-relevant assembly
Sickle Cell Hemoglobin *2hbs*

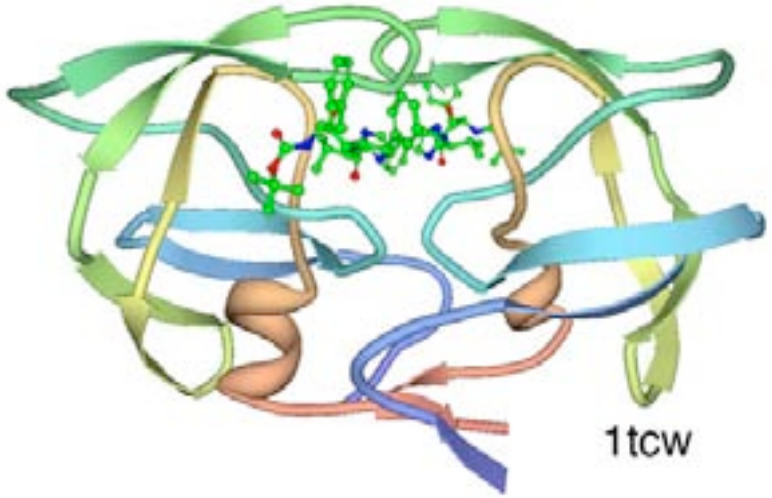


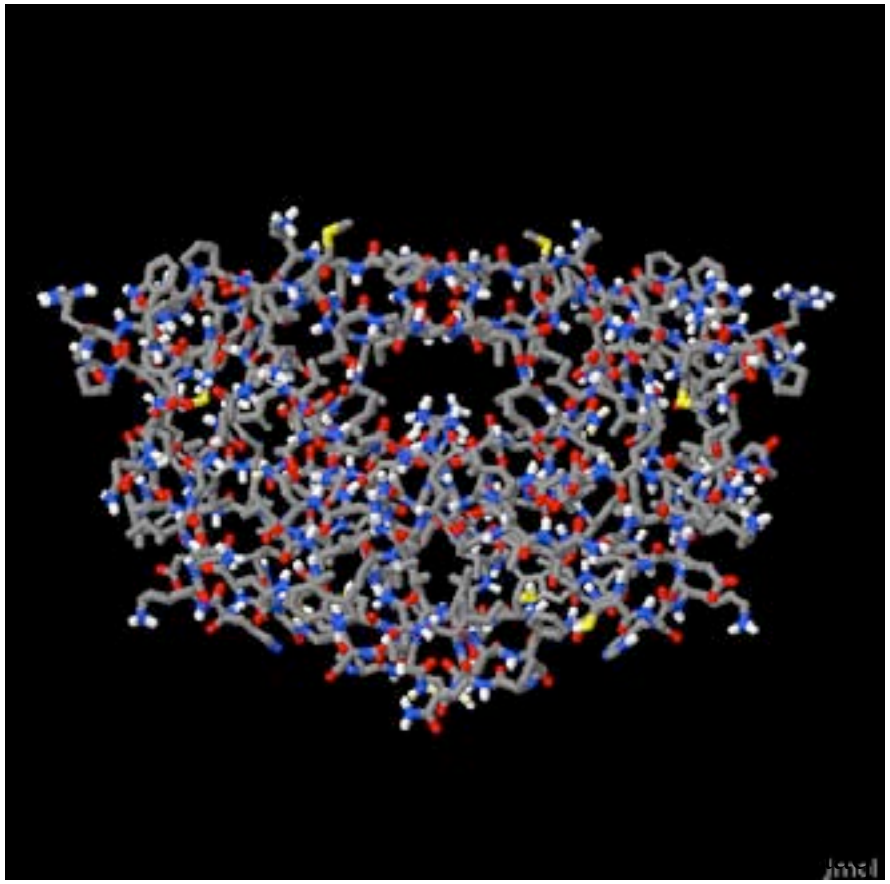
Frozen conformations
Antibodies *1igt, 1igy, 1hzh*



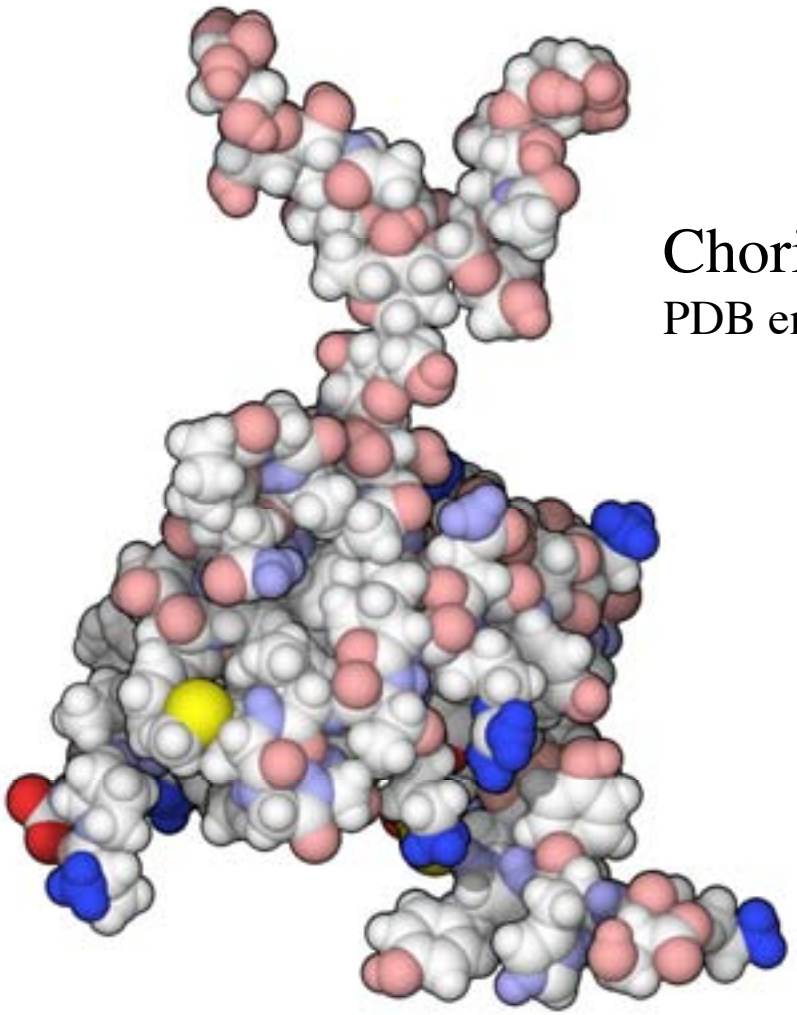
Incompatible symmetry
Actin *1atn*







Missing loops and missing hydrogen atom

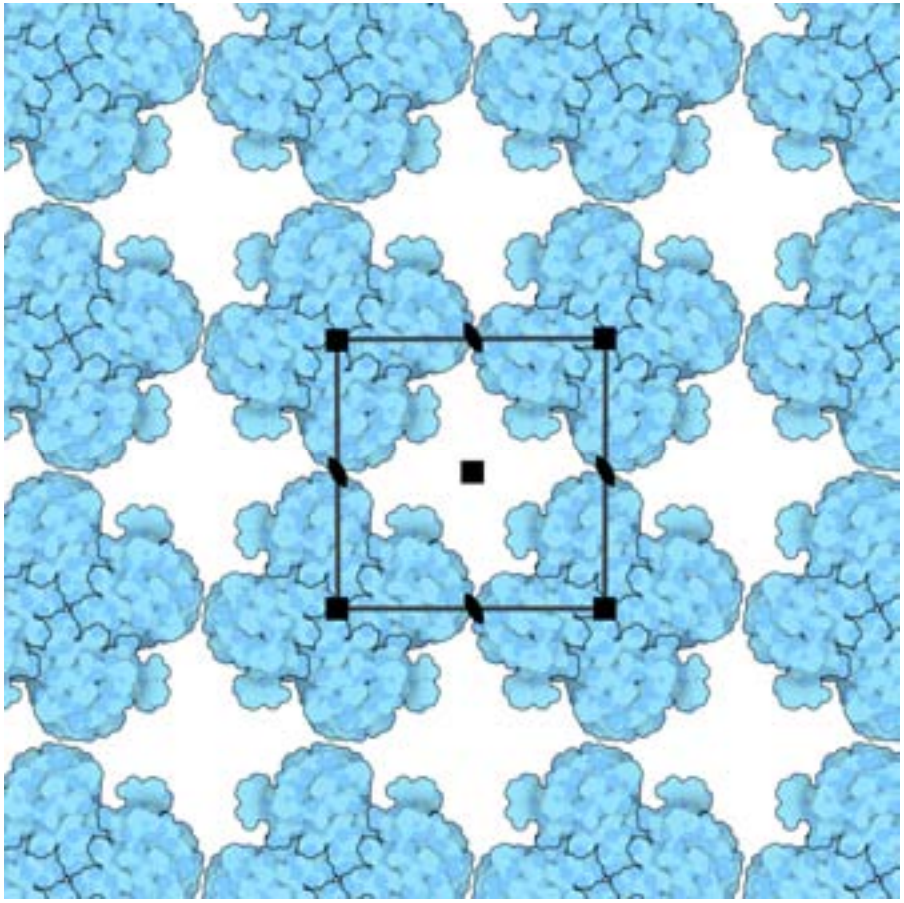


Chorionic Gonadotropin

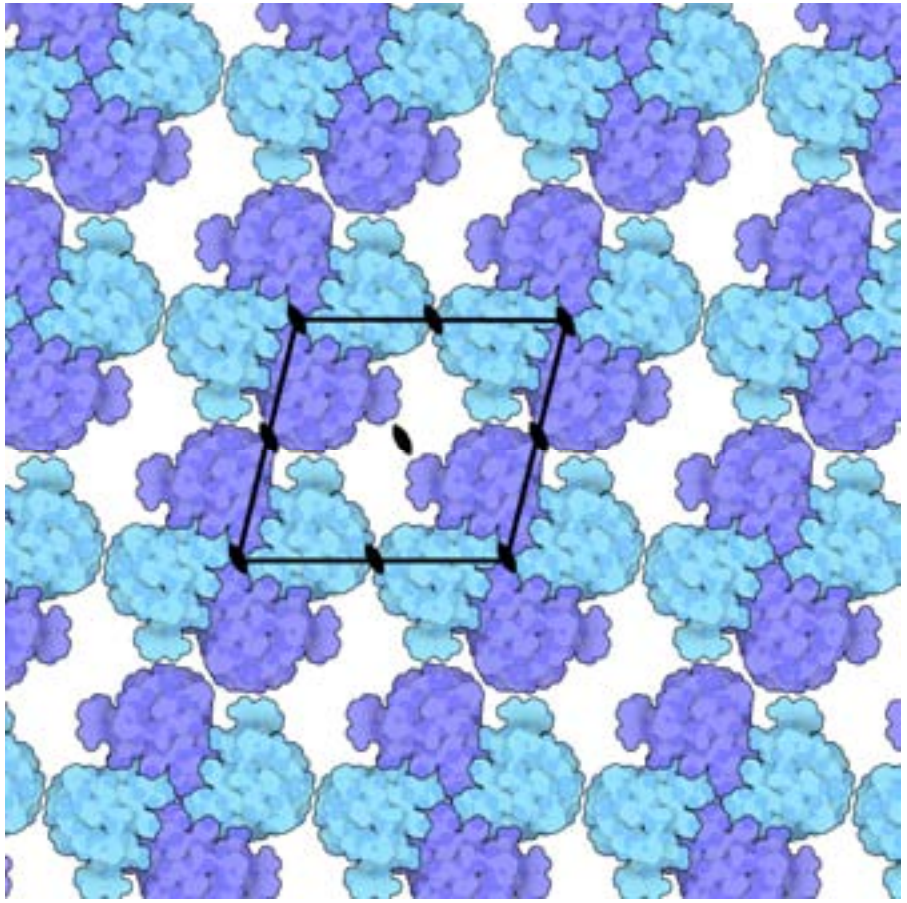
PDB entry 1hd4 (NMR)

Missing glycosylation

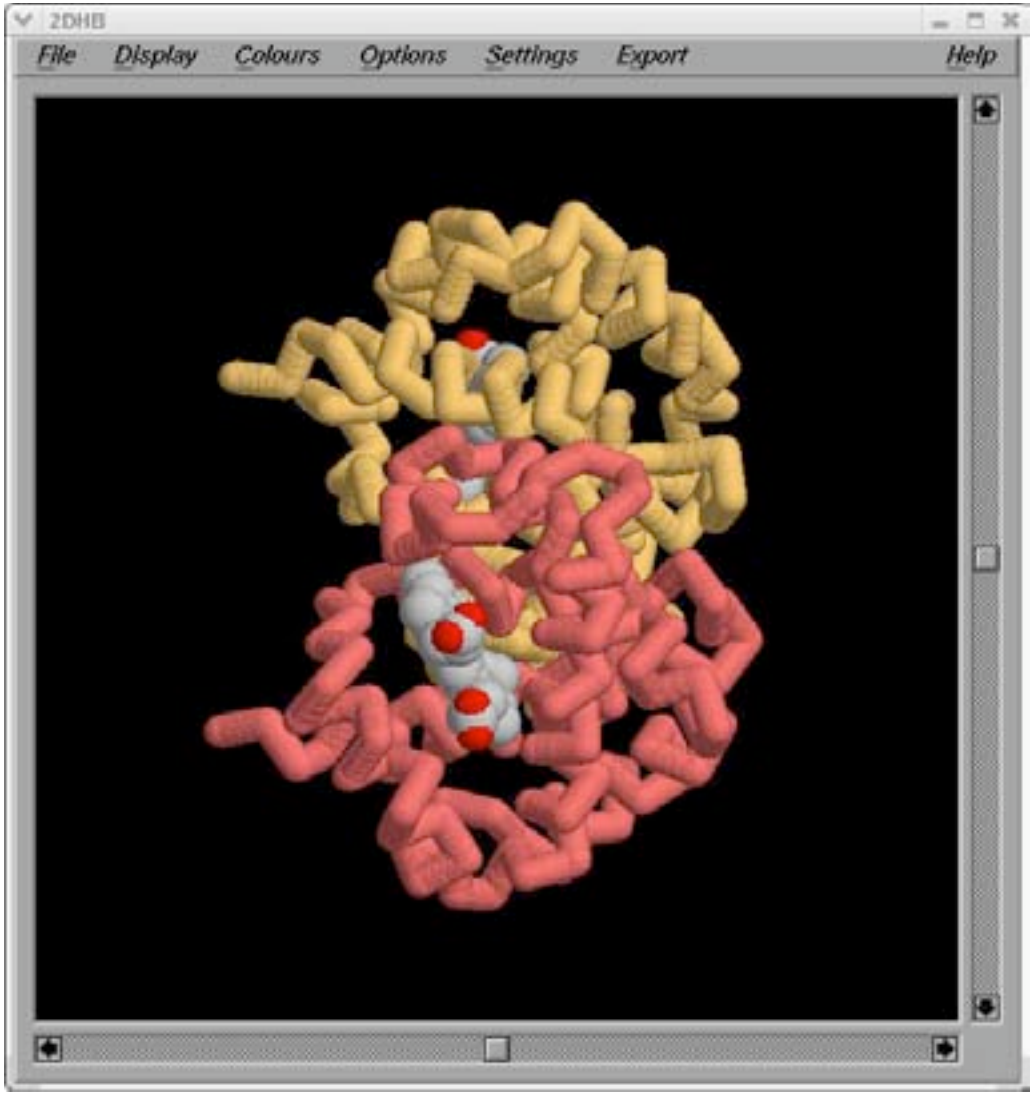
Symmetry and Biological Assemblies



Asymmetric unit = 1 chain



Asymmetric unit = 2 chains



2DHB

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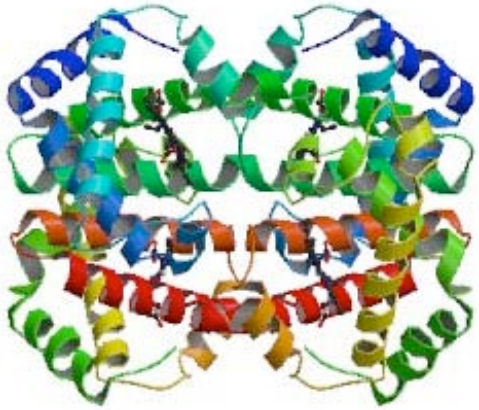
Biological Assembly ?

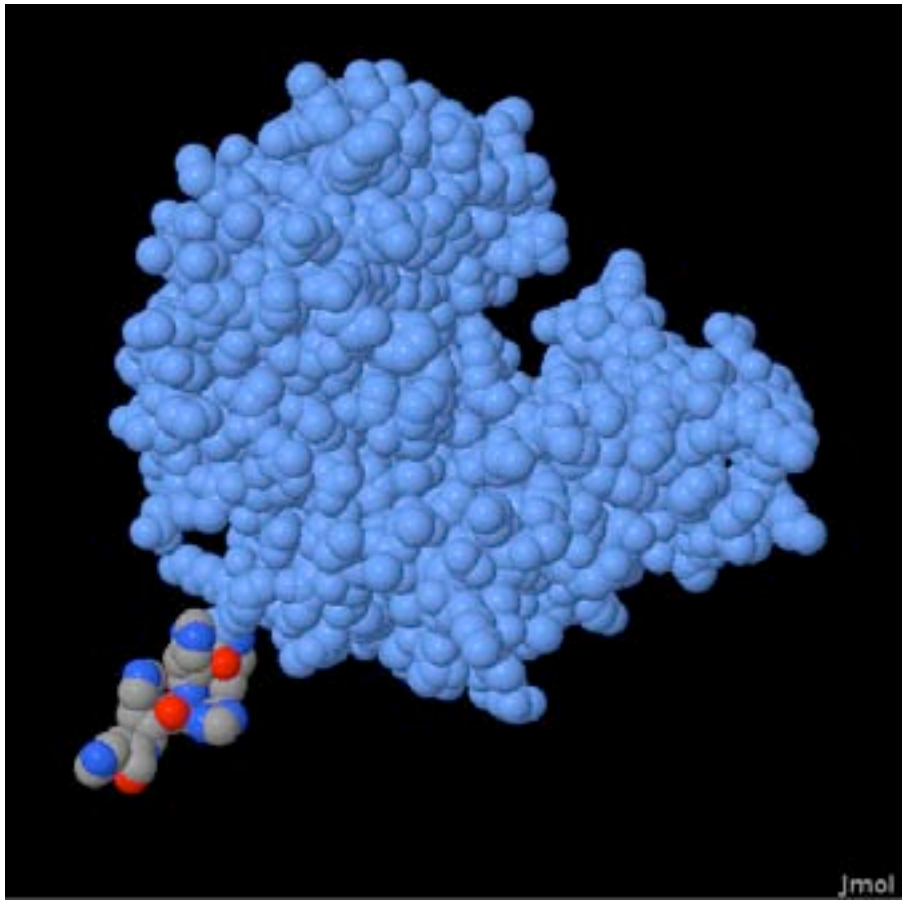


More Images...

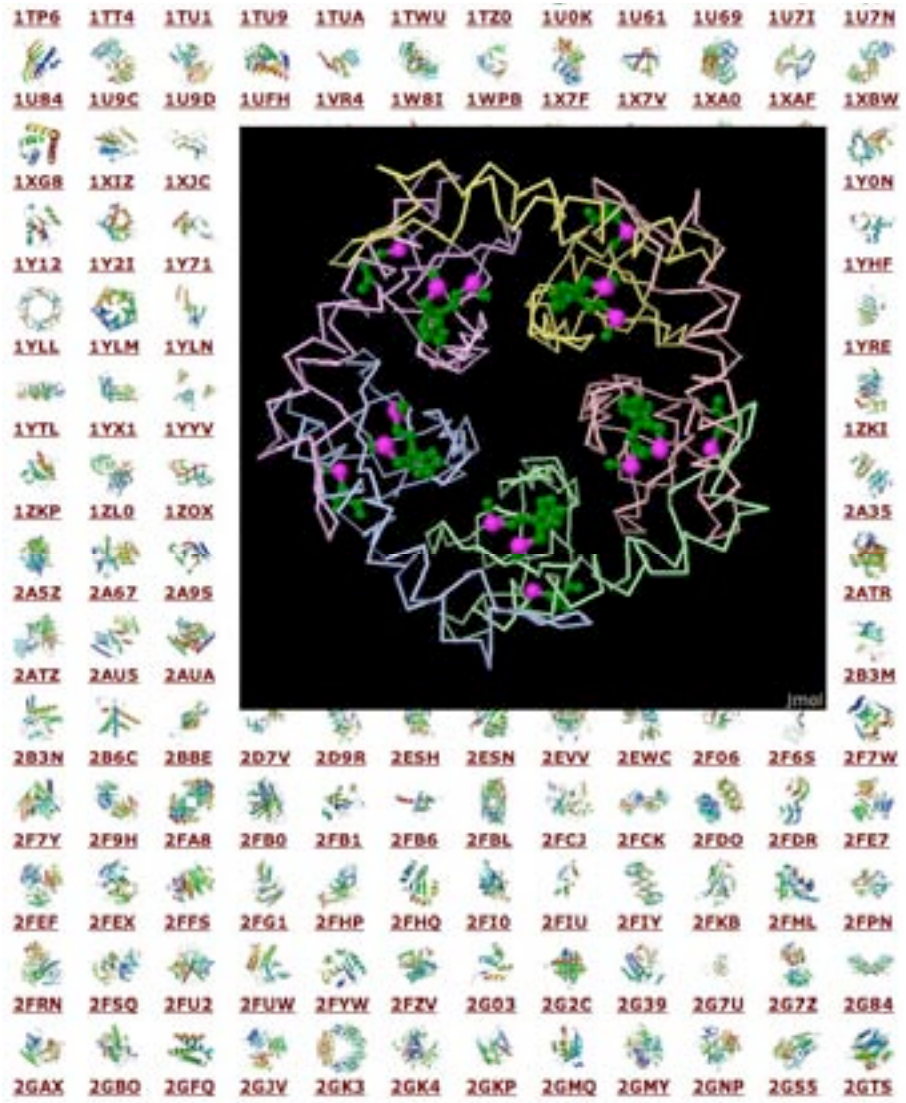
View in Jmol
Other Viewers ▾
SimpleViewer
Protein Workshop

Biological assembly assigned by authors

Octopine Dehydrogenase

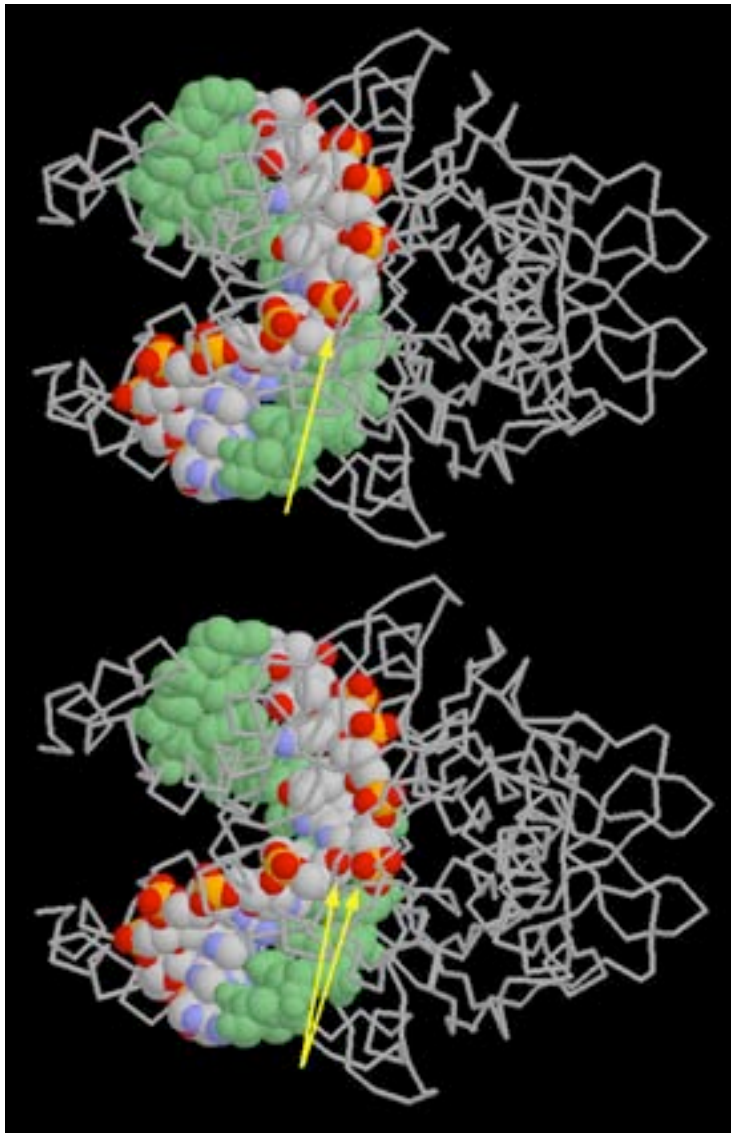


PDB entry 3c7d



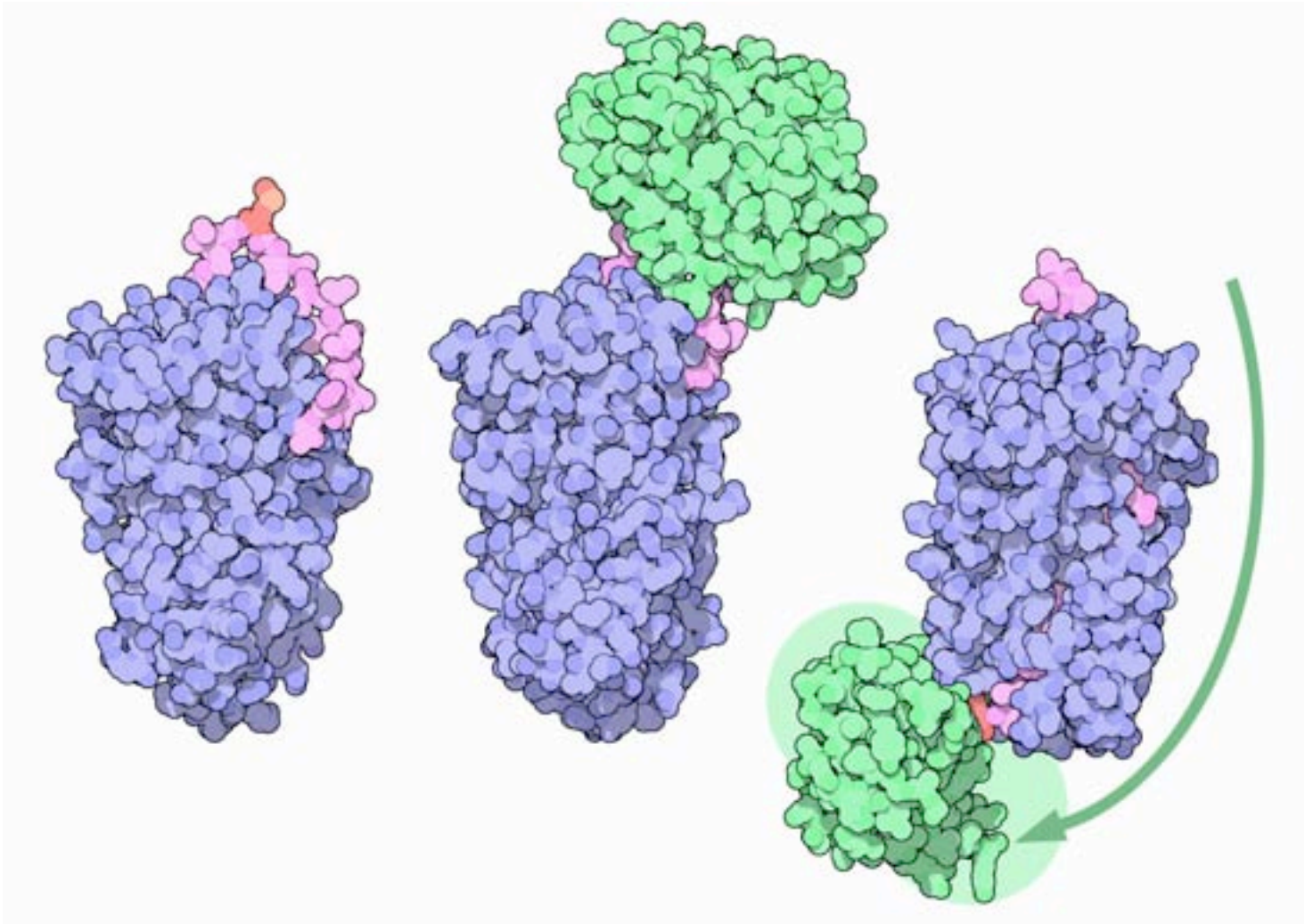
1ylm from PSI Structural Genomics Knowledgebase

Structures may include His tags or selenomethionine

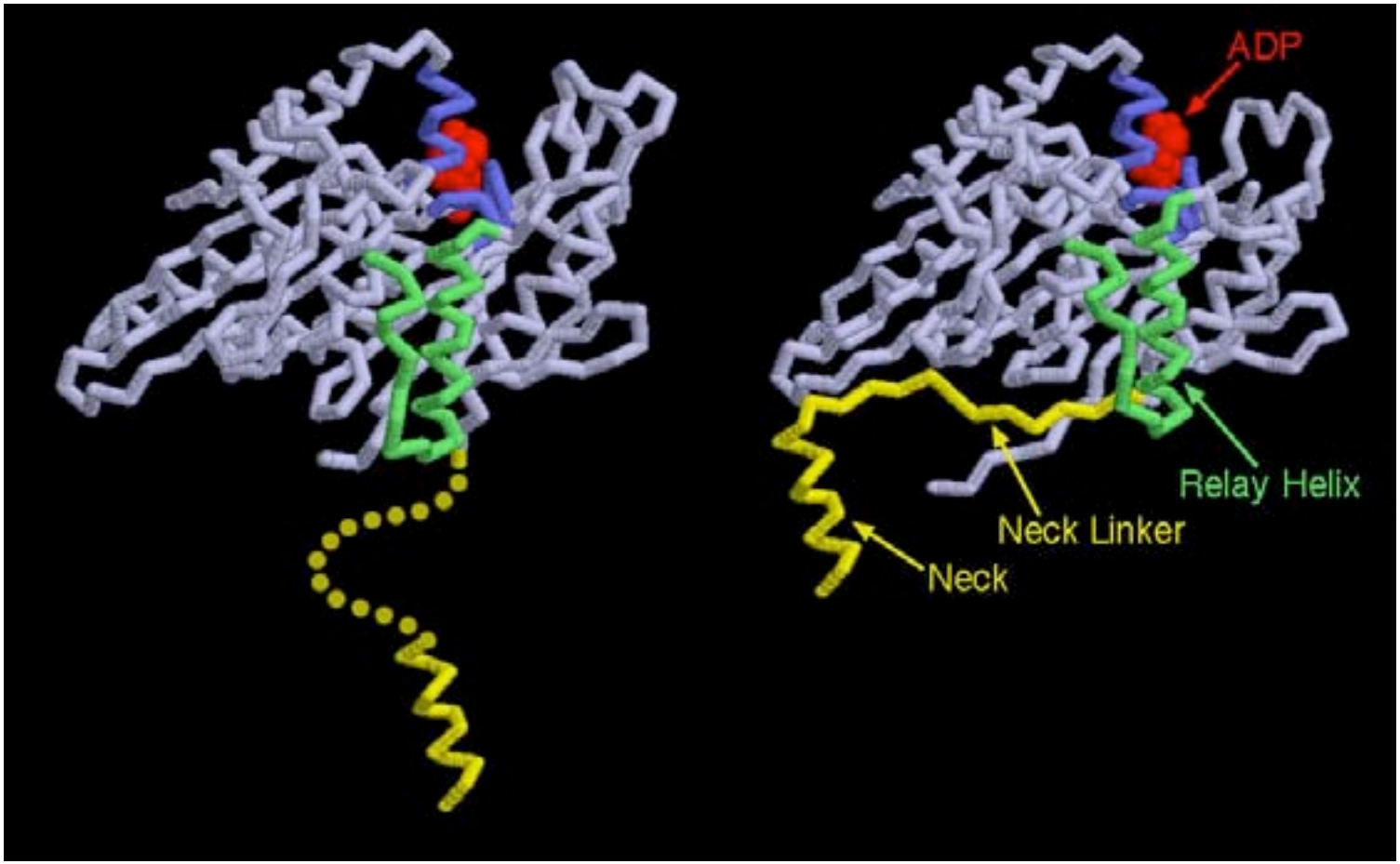


EcoRV Restriction Endonuclease

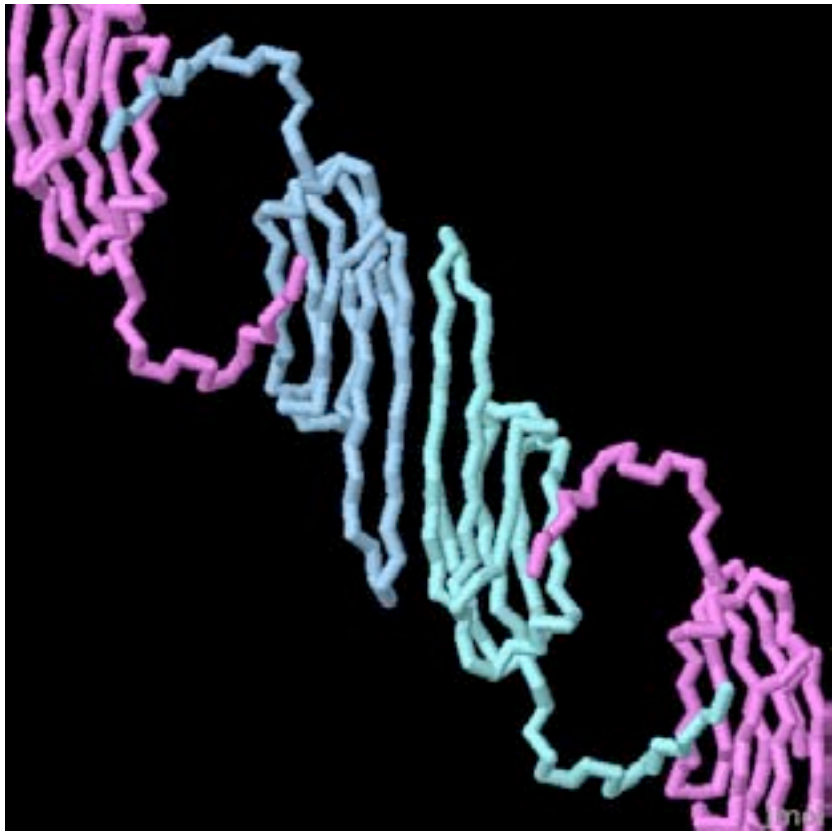
Before cutting *1rva*
After cutting *1rvc*

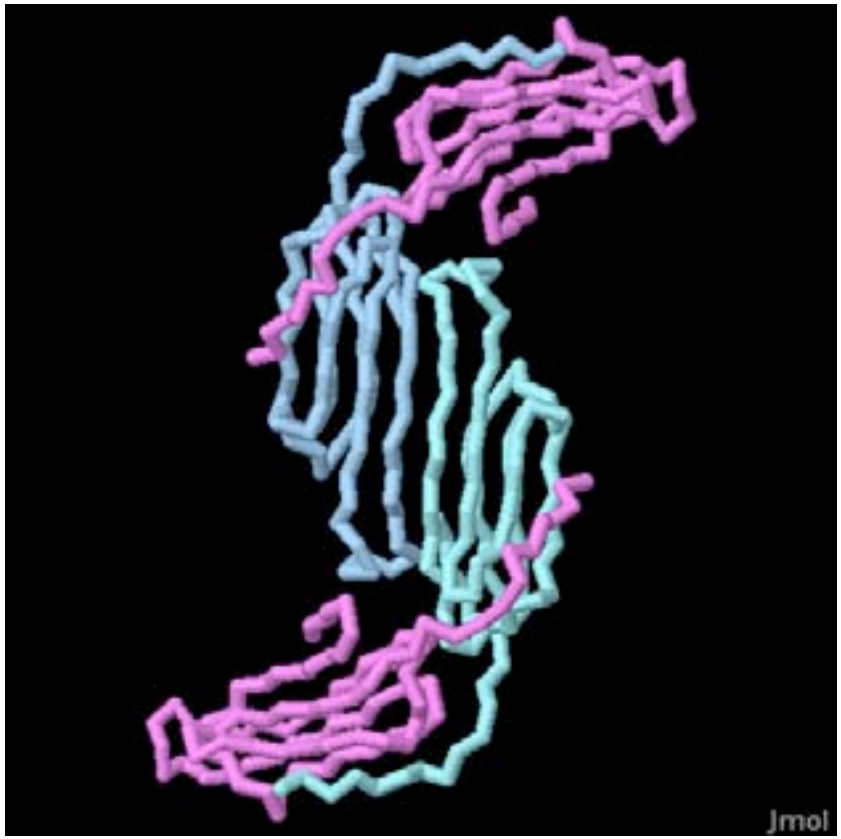


α1-antitrypsin *1psi*, *1k9o*, *1ezx*

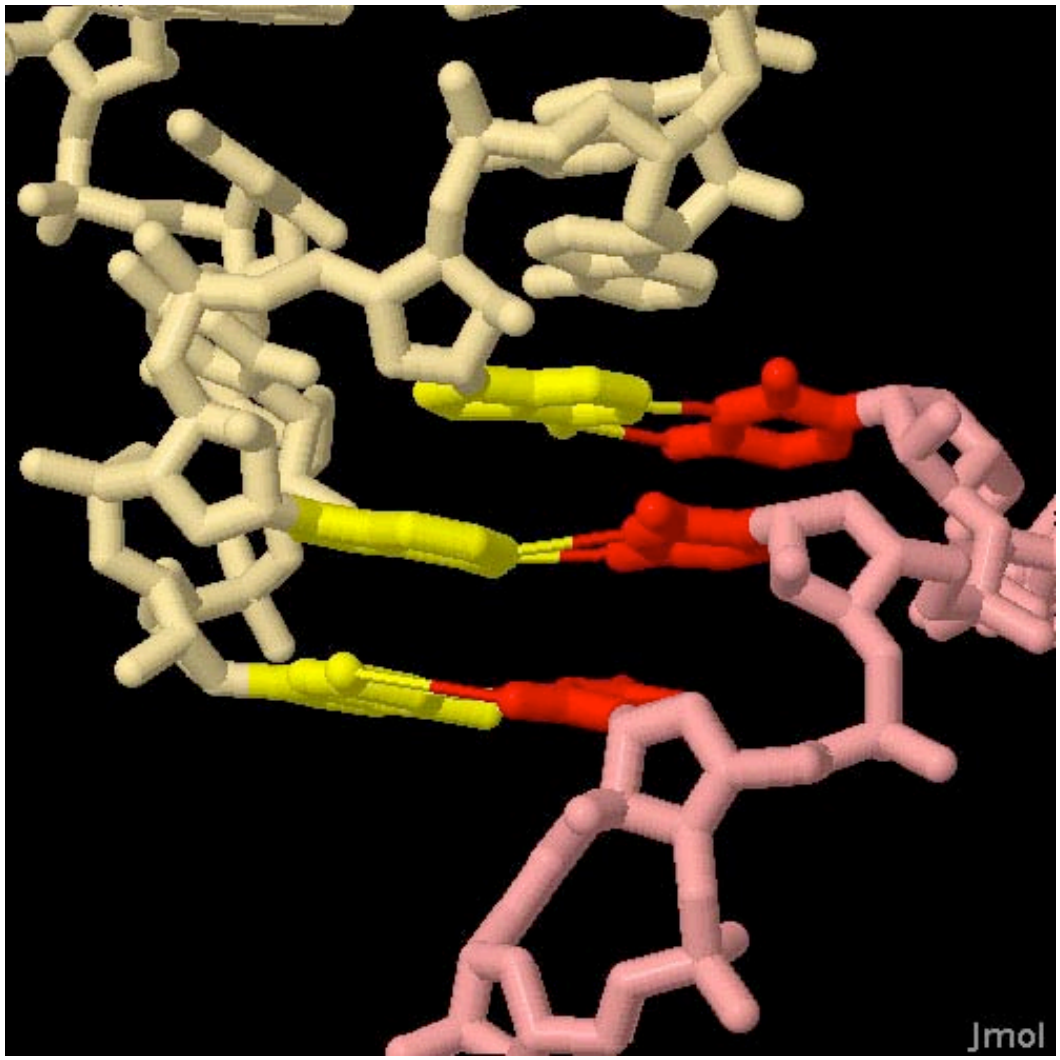


Kinesin *1bg2, 2kin*





Alpha Crystallin *3l1e,3l1g*



Ribosome decoding center *2wdg*

Acknowledgements

RCSB PDB PROTEIN DATA BANK

RUTGERS UCSD

www.pdb.org
info@rcsb.org

Funding Agencies for all Projects:
NSF, NIGMS, DOE, NLM, NCI,
NINDS, NIDDK
Wellcome Trust, EU,
CCP4, BBSRC, MRC, EMBL
BIRD-JST, MEXT

WORLDWIDE PDB PROTEIN DATA BANK

RCSB PDB PROTEIN DATA BANK

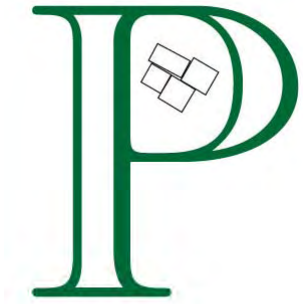
PDBe PROTEIN DATA BANK EUROPE

PDBj Protein Data Bank Japan

BMRB

Understanding Everyday Materials using X-ray Powder Diffraction

James A. Kaduk
Poly Crystallography Inc.
Naperville IL 60540
kaduk@polycrystallography.com

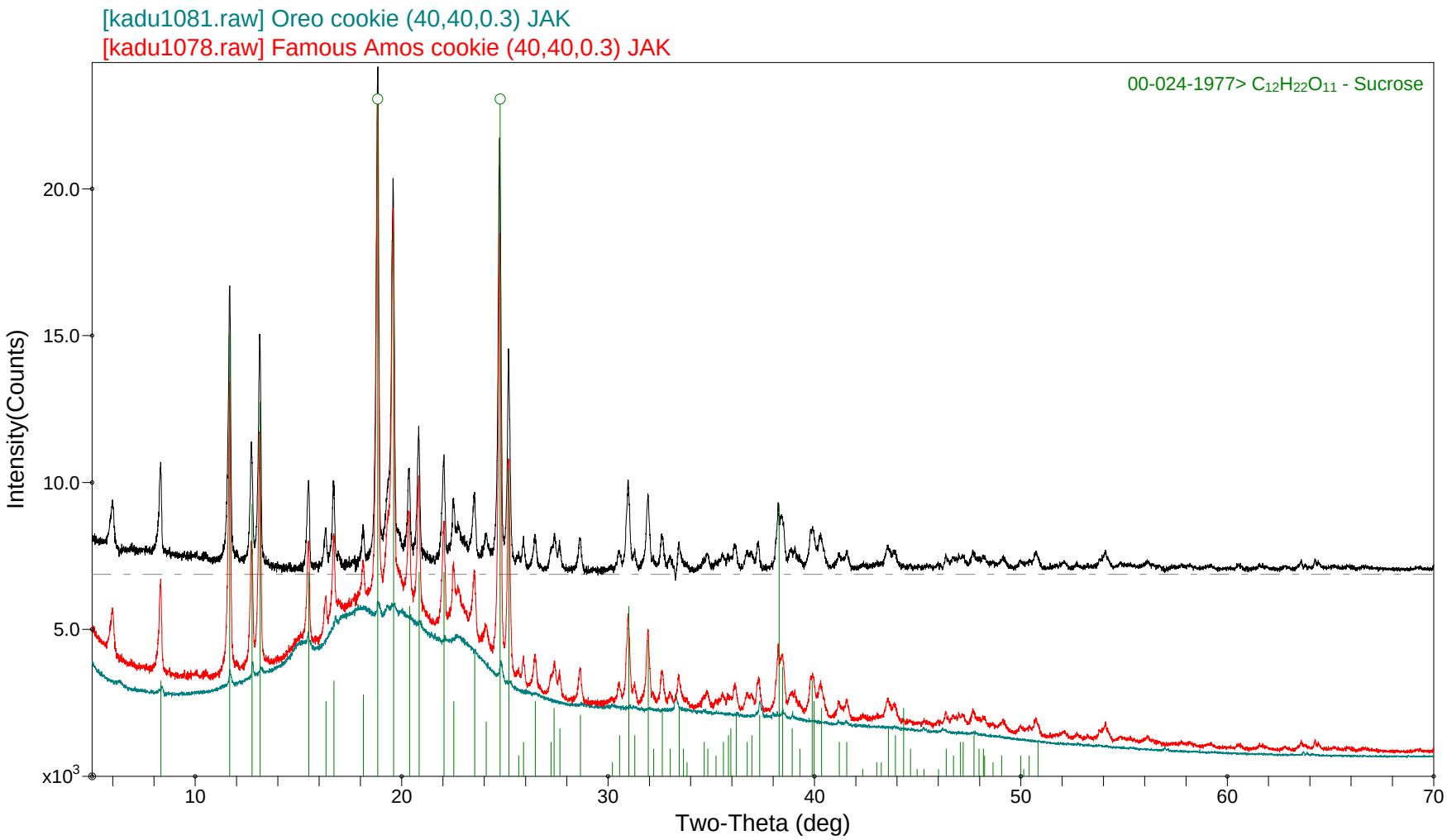


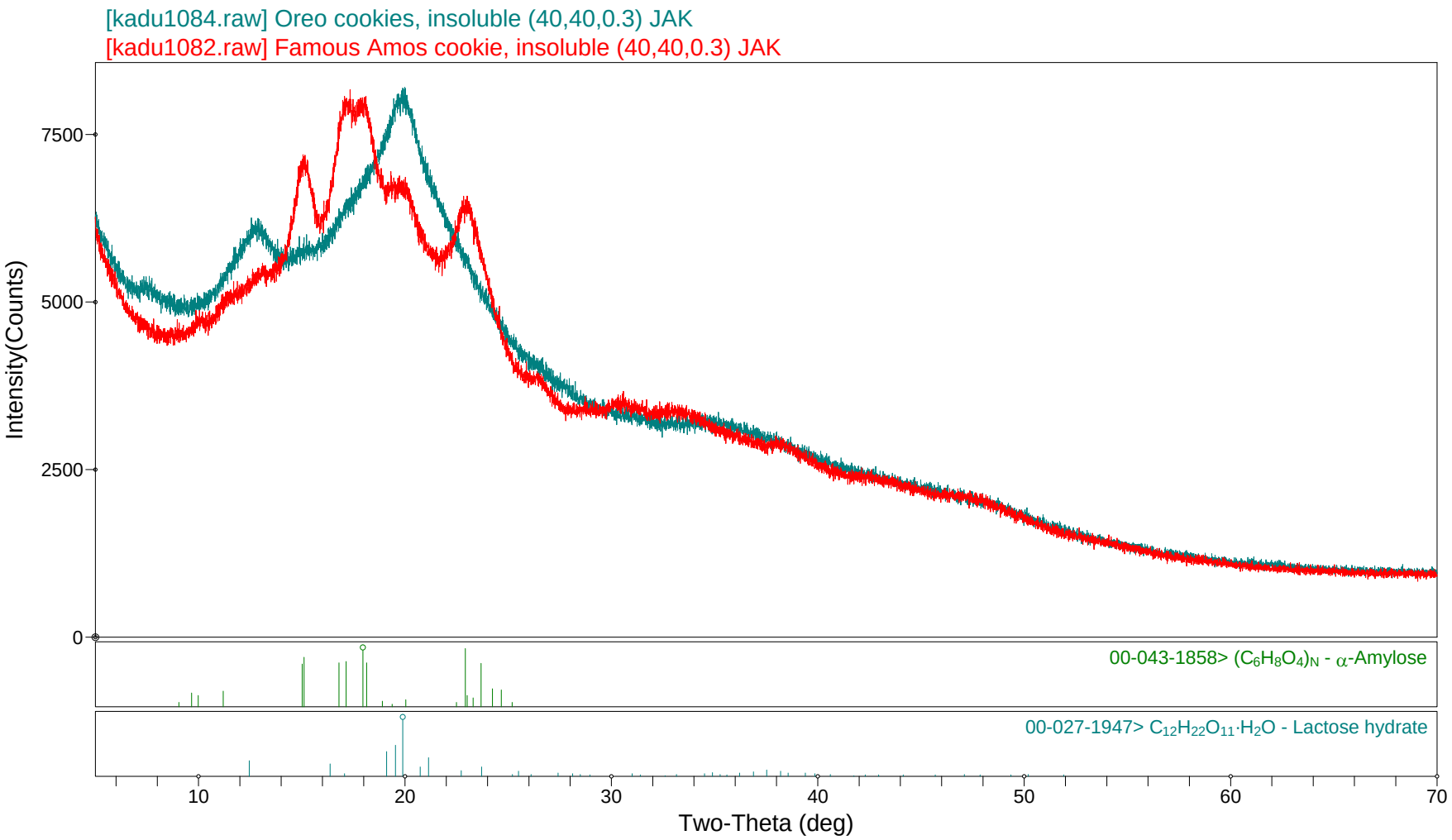
ILLINOIS INSTITUTE OF TECHNOLOGY

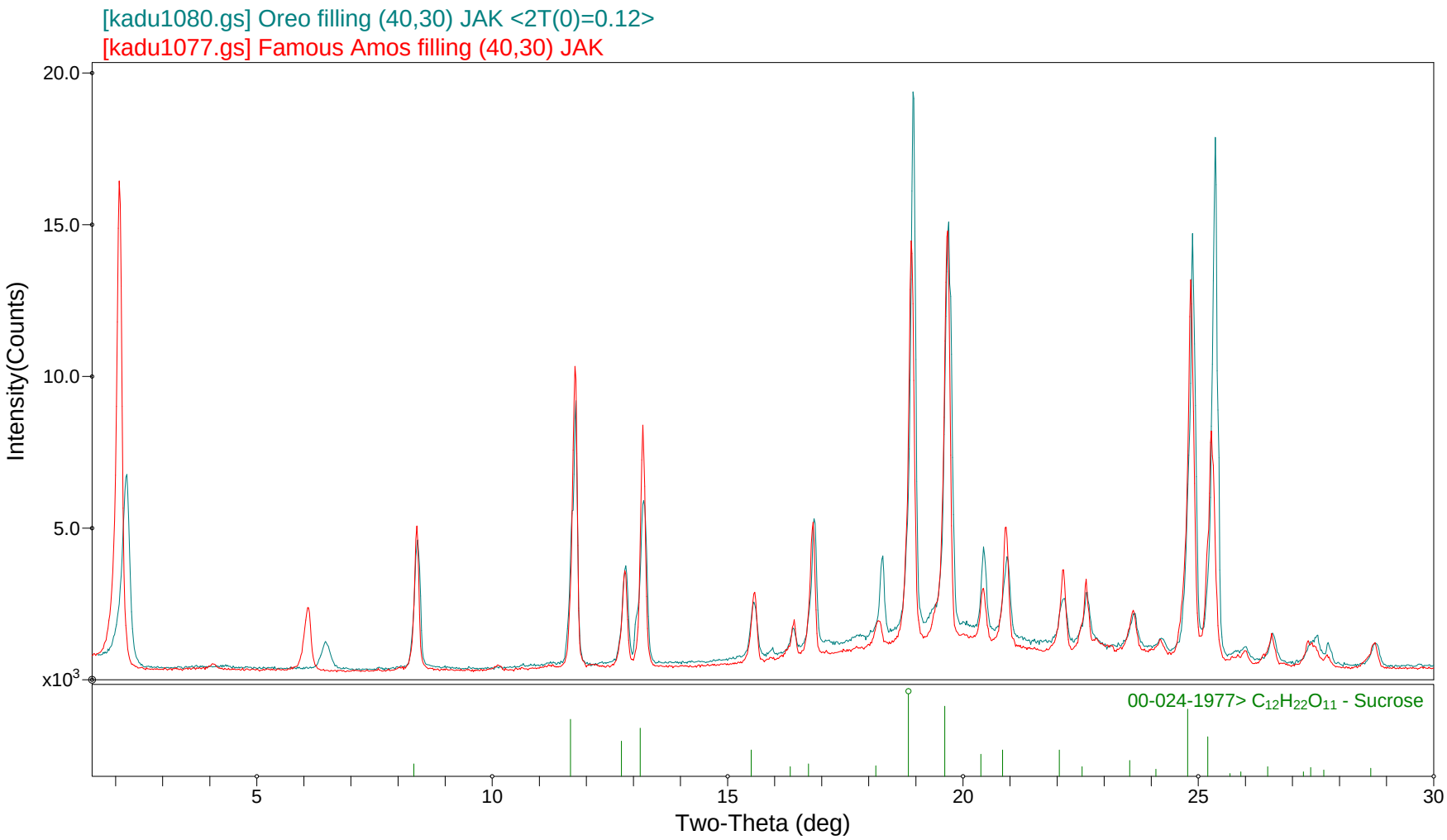
X-ray Diffraction

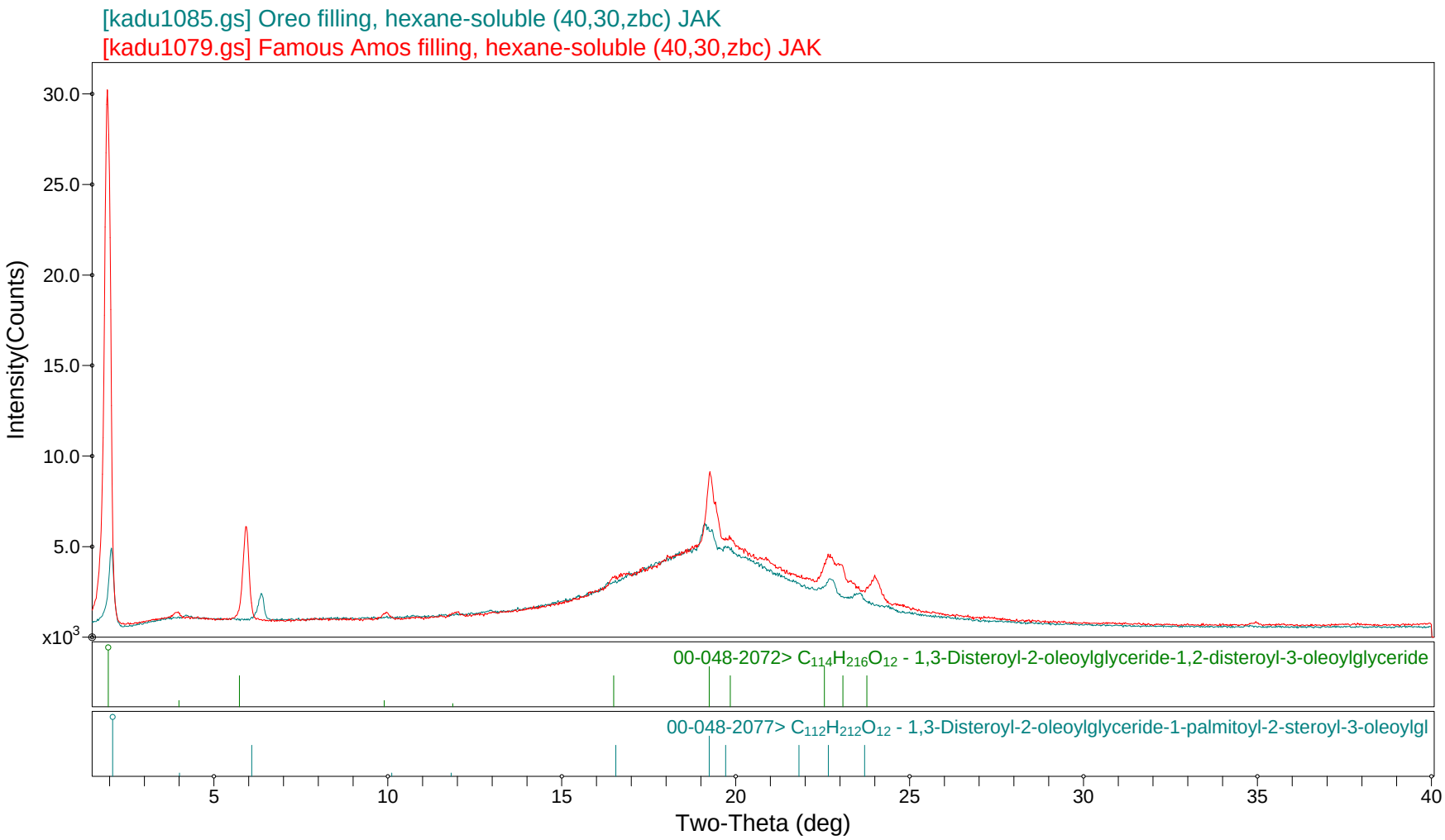

BRUKER
D2 PHASER
BELKIN

Chocolate Sandwich Cookies

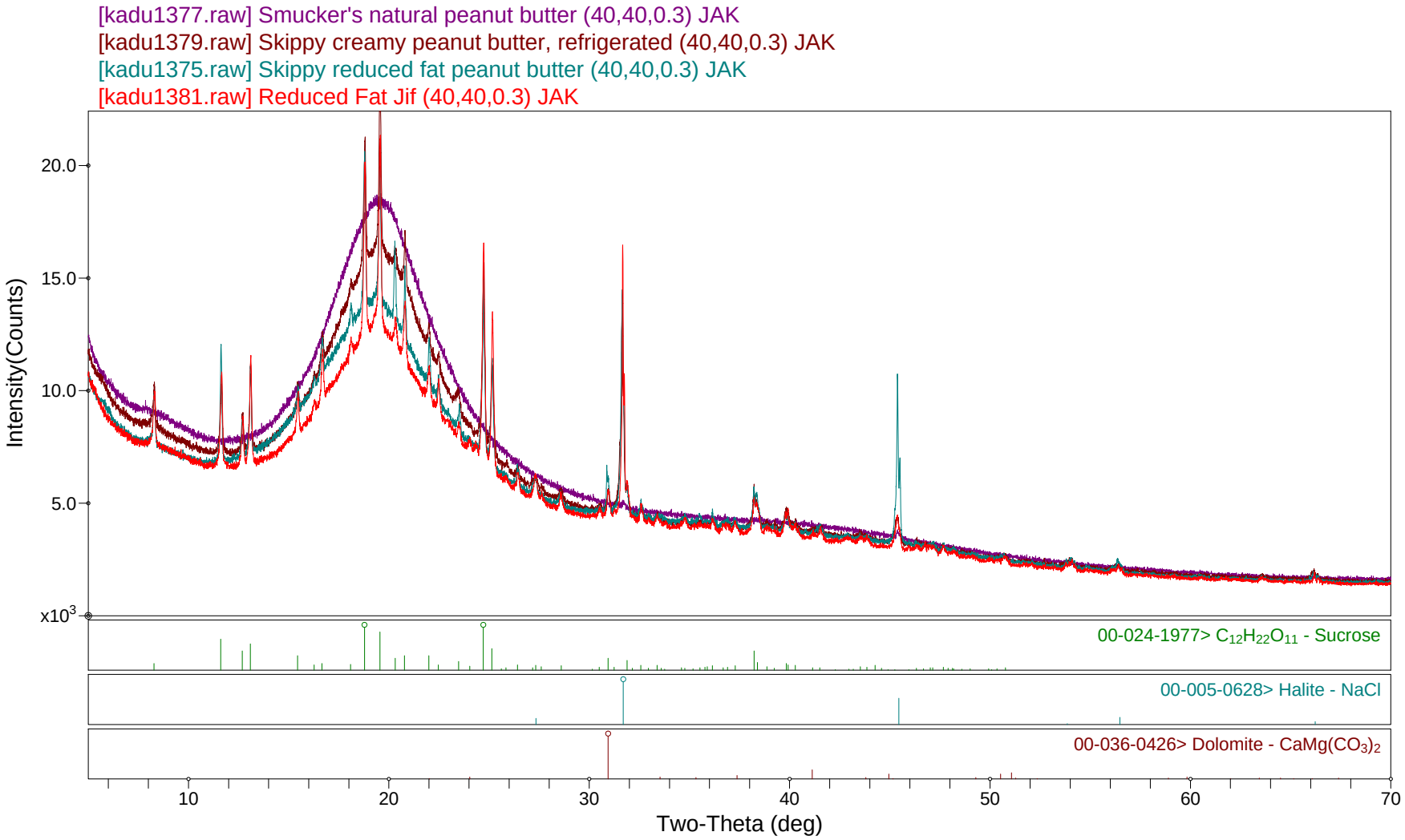

[kadu1081.raw] Oreo cookie (40,40,0.3) JAK
[kadu1078.raw] Famous Amos cookie (40,40,0.3) JAK
00-024-1977> C12H22O11 - Sucrose
Intensity(Counts)
20.0
15.0
10.0
5.0
x10^3
10
20
30
40
50
60
70
Two-Theta (deg)


[kadu1084.raw] Oreo cookies, insoluble (40,40,0.3) JAK
[kadu1082.raw] Famous Amos cookie, insoluble (40,40,0.3) JAK
Intensity(Counts)
7500
5000
2500
0
00-043-1858> (C6H8O4)N - α-Amylose
00-027-1947> C12H22O11·H2O - Lactose hydrate
10
20
30
40
50
60
70
Two-Theta (deg)


[kadu1080.gs] Oreo filling (40,30) JAK <2T(0)=0.12>
[kadu1077.gs] Famous Amos filling (40,30) JAK
Intensity(Counts)
20.0
15.0
10.0
5.0
x10^3
00-024-1977> C12H22O11 - Sucrose
5
10
15
20
25
30
Two-Theta (deg)

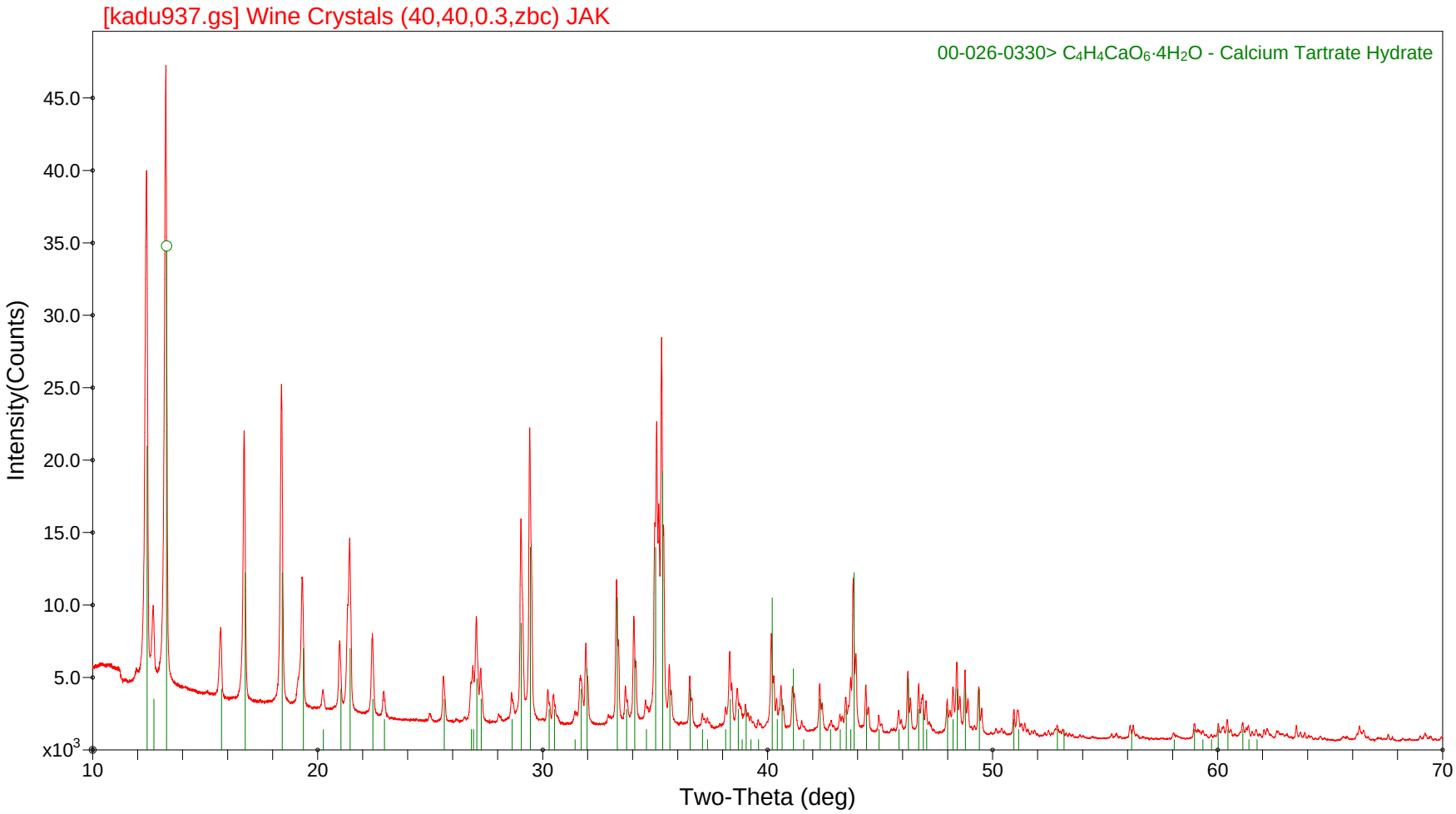

[kadu1085.gs] Oreo filling, hexane-soluble (40,30,zbc) JAK
[kadu1079.gs] Famous Amos filling, hexane-soluble (40,30,zbc) JAK
Intensity(Counts)
30.0
25.0
20.0
15.0
10.0
5.0
x10^3
00-048-2072> C114H216O12 - 1,3-Disteroyl-2-oleoylglyceride-1,2-disteroyl-3-oleoylglyceride
00-048-2077> C112H212O12 - 1,3-Disteroyl-2-oleoylglyceride-1-palmitoyl-2-steroyl-3-oleoylgl
5
10
15
20
25
30
35
40
Two-Theta (deg)

Peanut Butter


[kadu1377.raw] Smucker's natural peanut butter (40,40,0.3) JAK
[kadu1379.raw] Skippy creamy peanut butter, refrigerated (40,40,0.3) JAK
[kadu1375.raw] Skippy reduced fat peanut butter (40,40,0.3) JAK
[kadu1381.raw] Reduced Fat Jif (40,40,0.3) JAK
Intensity(Counts)
20.0
15.0
10.0
5.0
x10^3
00-024-1977> C12H22O11 - Sucrose
00-005-0628> Halite - NaCl
00-036-0426> Dolomite - CaMg(CO3)2
10
20
30
40
50
60
70
Two-Theta (deg)

Sample	Skippy creamy	Skippy reduced fat	Jif reduced fat	Smucker's Natural
Ingredients	peanuts sugar salt hydr. veg. oils	peanuts corn syrup sol. sugar soy protein salt hydr. veg. oils mono/diglyc. minerals vitamins	peanuts corn syrup sol. sugar soy protein salt hydr. veg. oils molasses minerals vitamins	peanuts salt
sucrose, $C_{12}H_{22}O_{11}$ wt% NI sugars, wt%	 9.4 9.4	 10.0 11.4	 12.2 11.1	- 3.1
halite, NaCl wt% NI NaCl, wt%	 ~1 1.2	 ~2? 1.2	 ~1 1.6	 Small 1.0
dolomite, $CaMg(CO_3)_2$ wt%	-	0.2	-	-
β_2 fat, SSS wt%	-	-	1.4	-

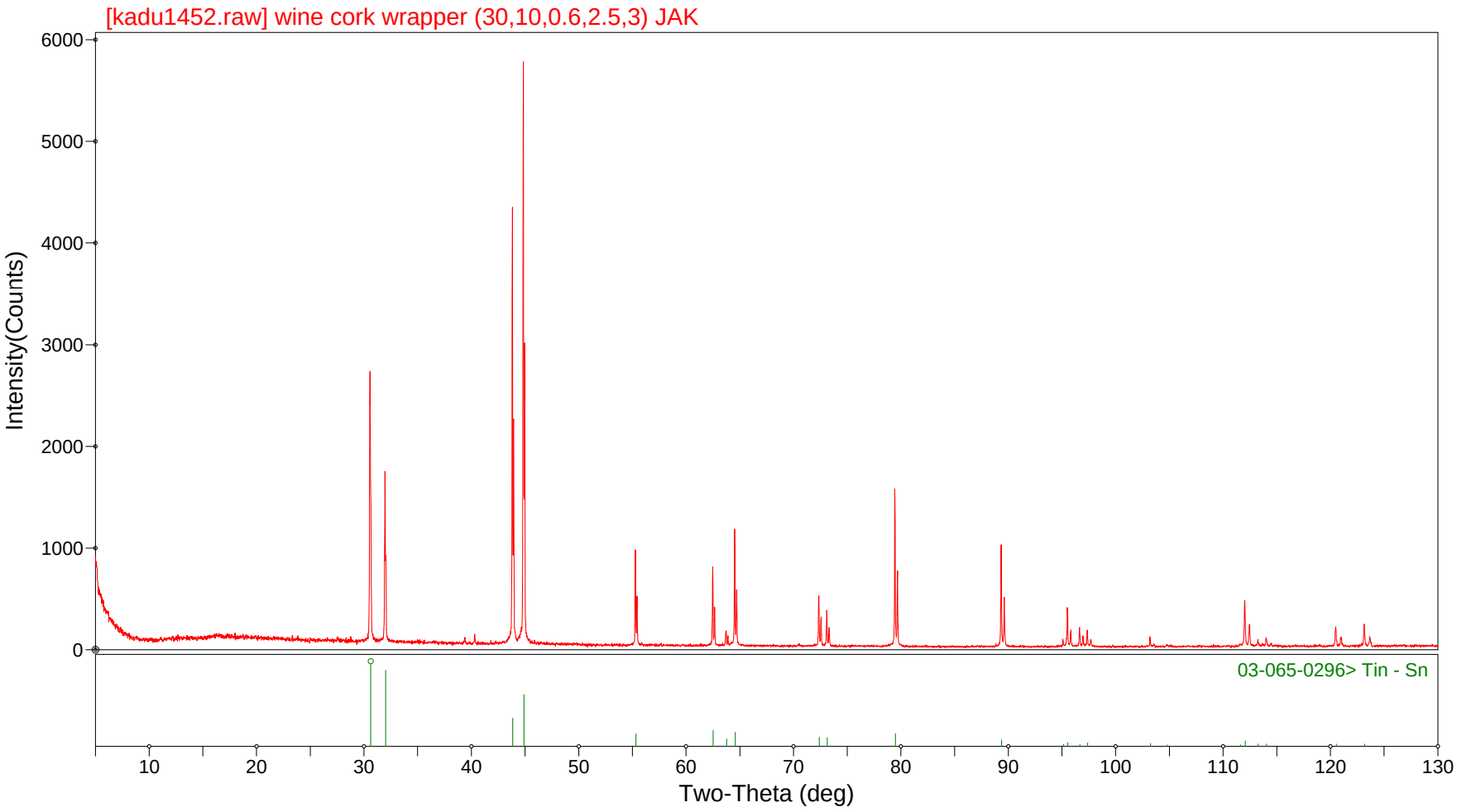
Wine Sediment

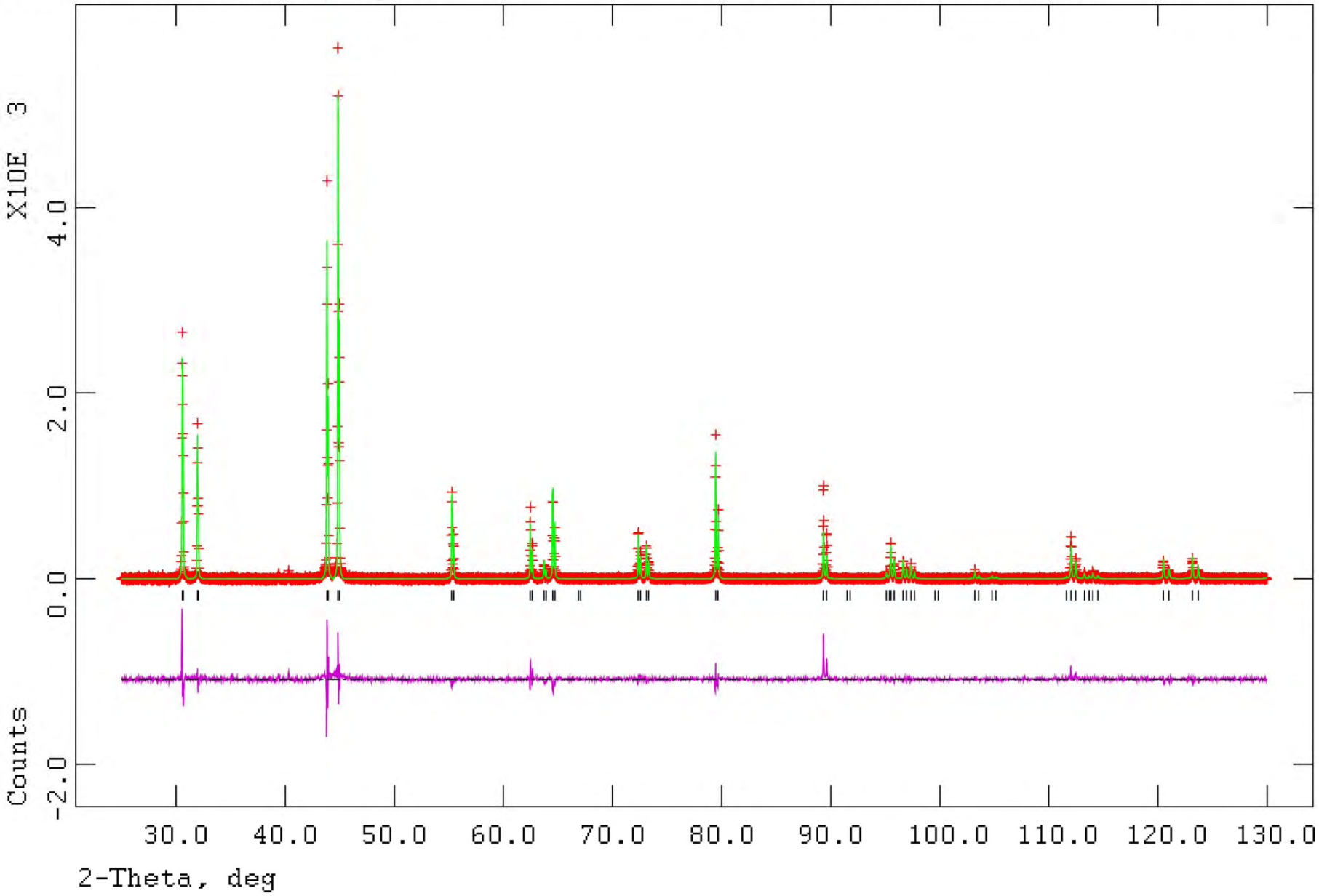

[kadu937.gs] Wine Crystals (40,40,0.3,zbc) JAK
00-026-0330> C4H4CaO6·4H2O - Calcium Tartrate Hydrate
Intensity(Counts)
x10^3
45.0
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
10
20
30
40
50
60
70
Two-Theta (deg)

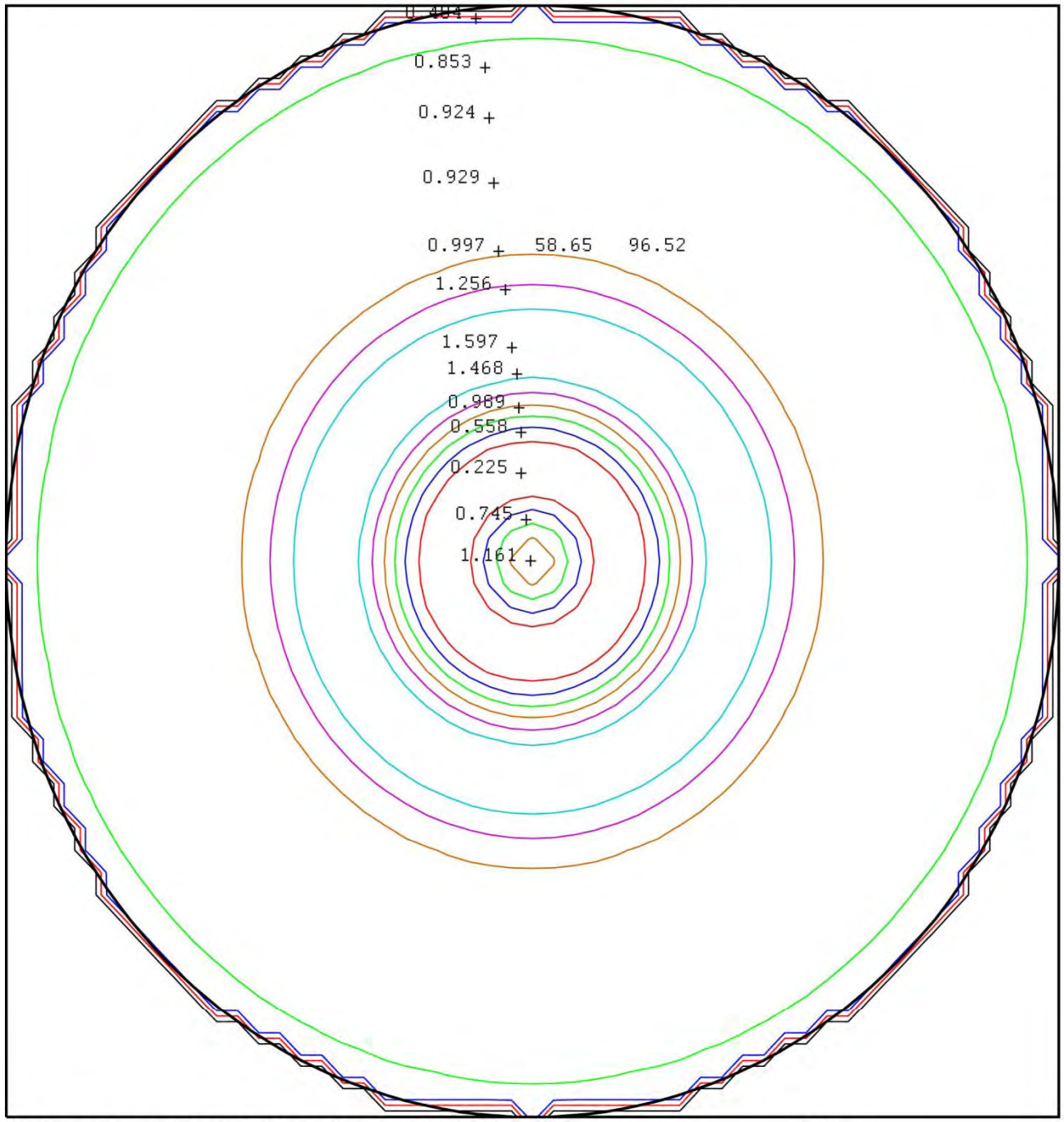
Bond Distances in Calcium Tartrate Tetrahydrate

Bond	This Work	CATART 1968	CATART01 1982	CATART02 1993	MOGUL 1.1
C2-C3 C4-C5	1.535(4) 1.536(4)	1.50 1.50	1.528 1.532	1.529 1.532	1.53(2)
C3-C4	1.559(4)	1.52	1.529	1.522	1.53(2)
C2-O6 C2-O7 C5-O10 C5-O11	1.230(4) 1.263(5) 1.231(5) 1.287(4)	1.27 1.26 1.24 1.31	1.267 1.242 1.269 1.241	1.266 1.248 1.269 1.242	1.25(2)
C3-O8 C4-O9	1.436(4) 1.424(4)	1.44 1.43	1.423 1.432	1.433 1.428	1.42(2)
Ca1-O6 Ca1-O11 Ca1-O7 Ca1-O10 Ca1-O8 Ca1-O9 Ca1-O12 Ca1-O13	2.350(3) 2.391(3) 2.437(3) 2.507(3) 2.481(3) 2.533(3) 2.482(3) 2.387(3)	2.39 2.43 2.42 2.52 2.51 2.54 2.48 2.47	2.377(3) 2.416(3) 2.425(3) 2.506(2) 2.474(3) 2.518(3) 2.494(3) 2.432(3)	2.374 2.419 2.403 2.483 2.458 2.510 2.488 2.438	2.42(10) CSD 5.27 2.48 bond valence

The foil from the wine bottle


[kadu1452.raw] wine cork wrapper (30,10,0.6,2.5,3) JAK
Intensity(Counts)
6000
5000
4000
3000
2000
1000
0
03-065-0296> Tin - Sn
10
20
30
40
50
60
70
80
90
100
110
120
130
Two-Theta (deg)


Kenwood wine bottle foil (KADU1452)
Hist 1
Lambda 1.5406 A, L-S cycle 69
Obsd. and Diff. Profiles
X10E 3
4.0
2.0
0.0
-2.0
Counts
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
2-Theta, deg



Sample X axis - horizontal to right

Sample Y axis - vertical

Pole figure psi 0 at center,
90 at rim

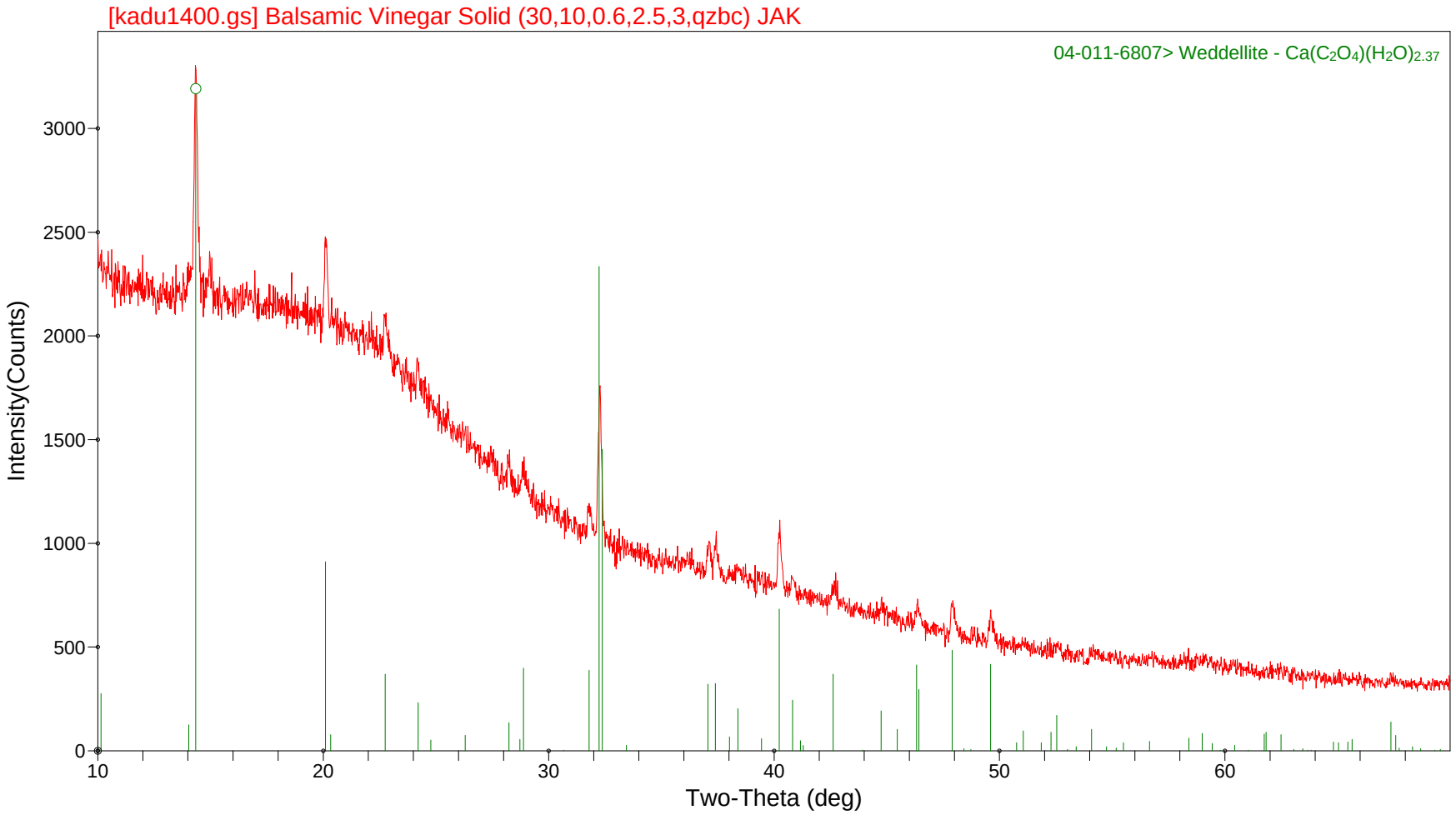
Pole figure gamma 0 at right,
90 at top

Contours are drawn at

0.217 0.419 0.621
0.823 1.025 1.226
1.428

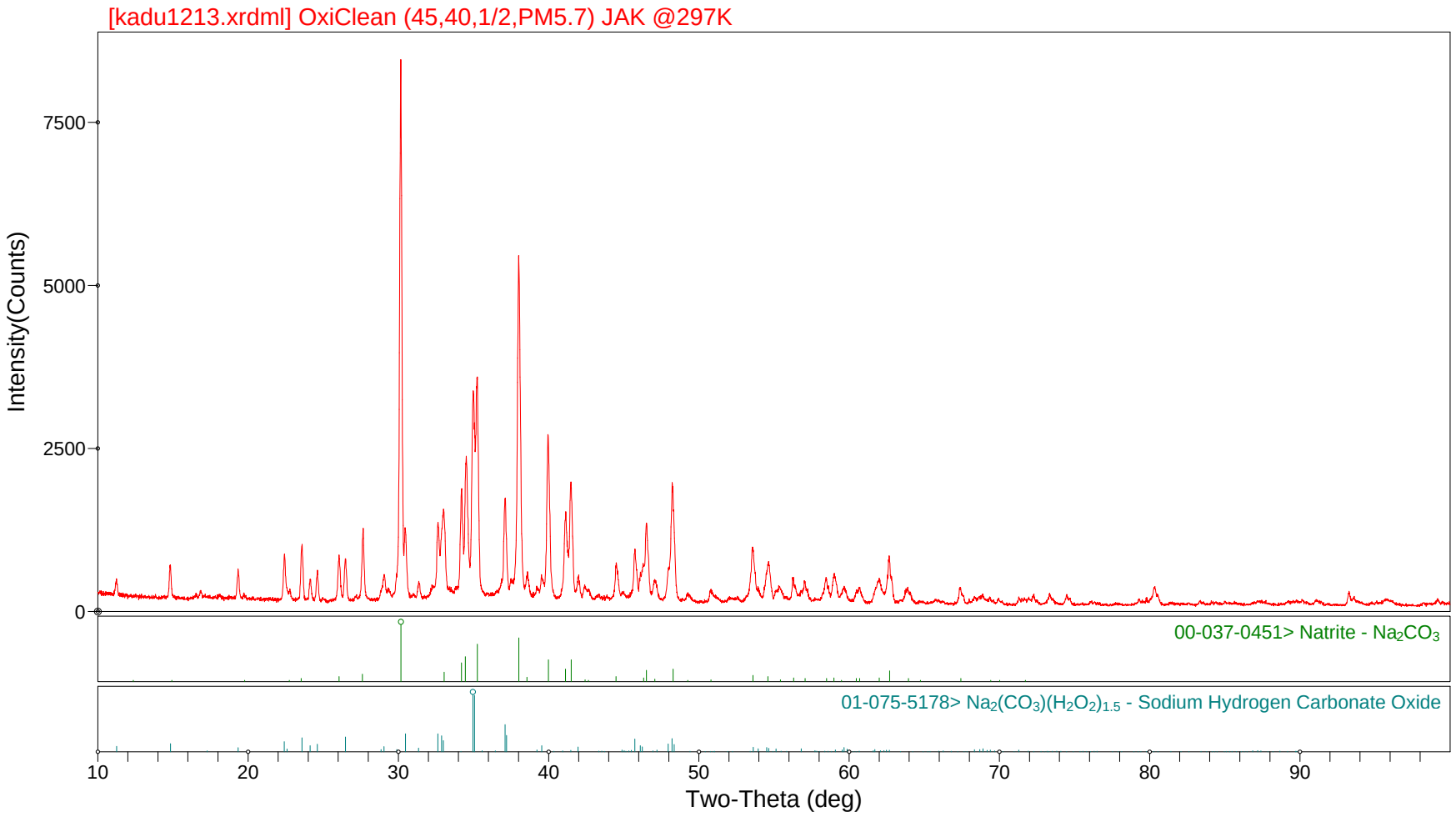
Kenwood wine bottle foil (KADU1452) Tin,
0 0 1 Pole Figure - Stereographic Proj.

Balsamic Vinegar Sediment

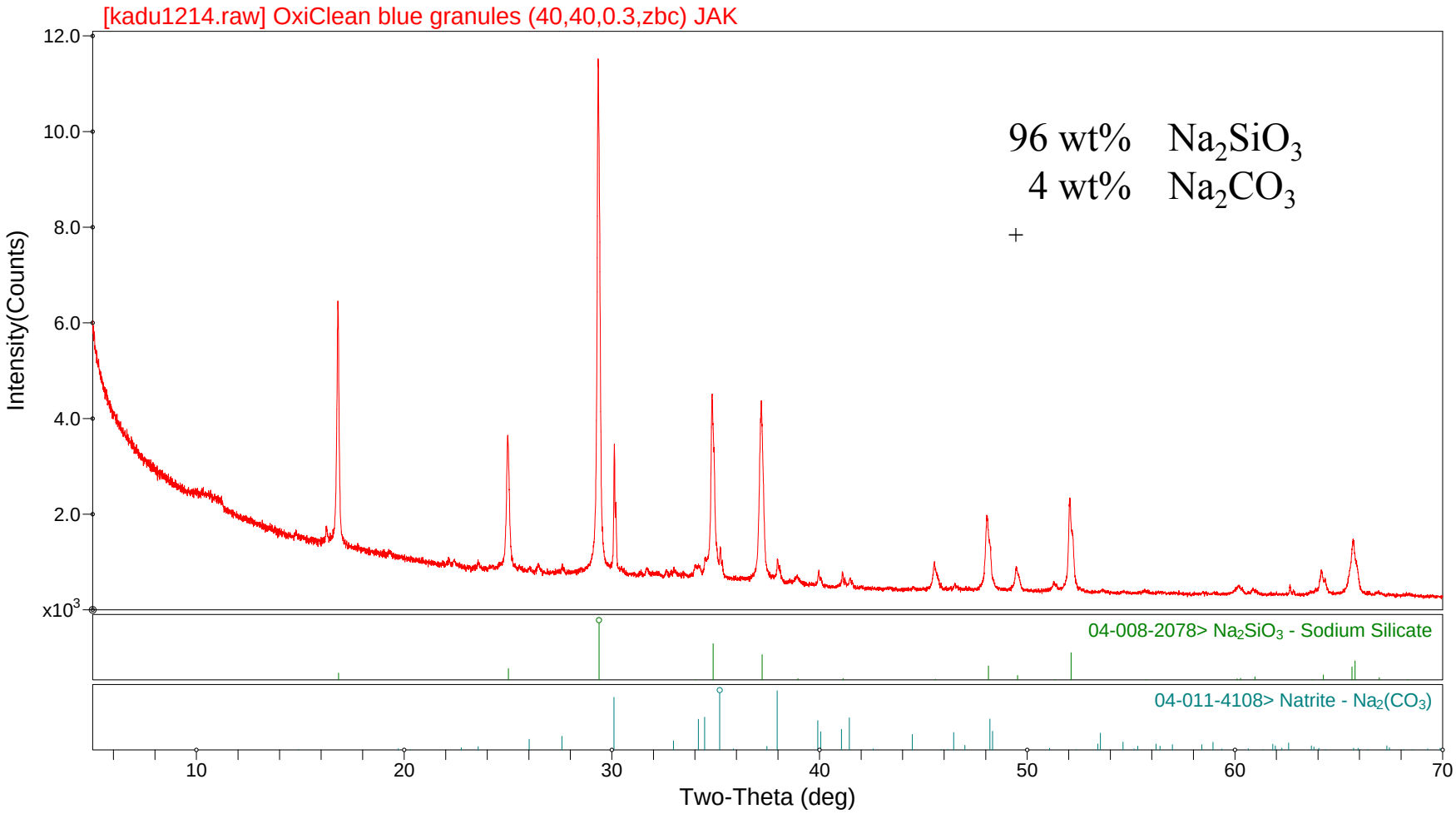

[kadu1400.gs] Balsamic Vinegar Solid (30,10,0.6,2.5,3,qzbc) JAK
04-011-6807> Weddellite - Ca(C2O4)(H2O)2.37
Intensity(Counts)
3000
2500
2000
1500
1000
500
0
10
20
30
40
50
60
Two-Theta (deg)

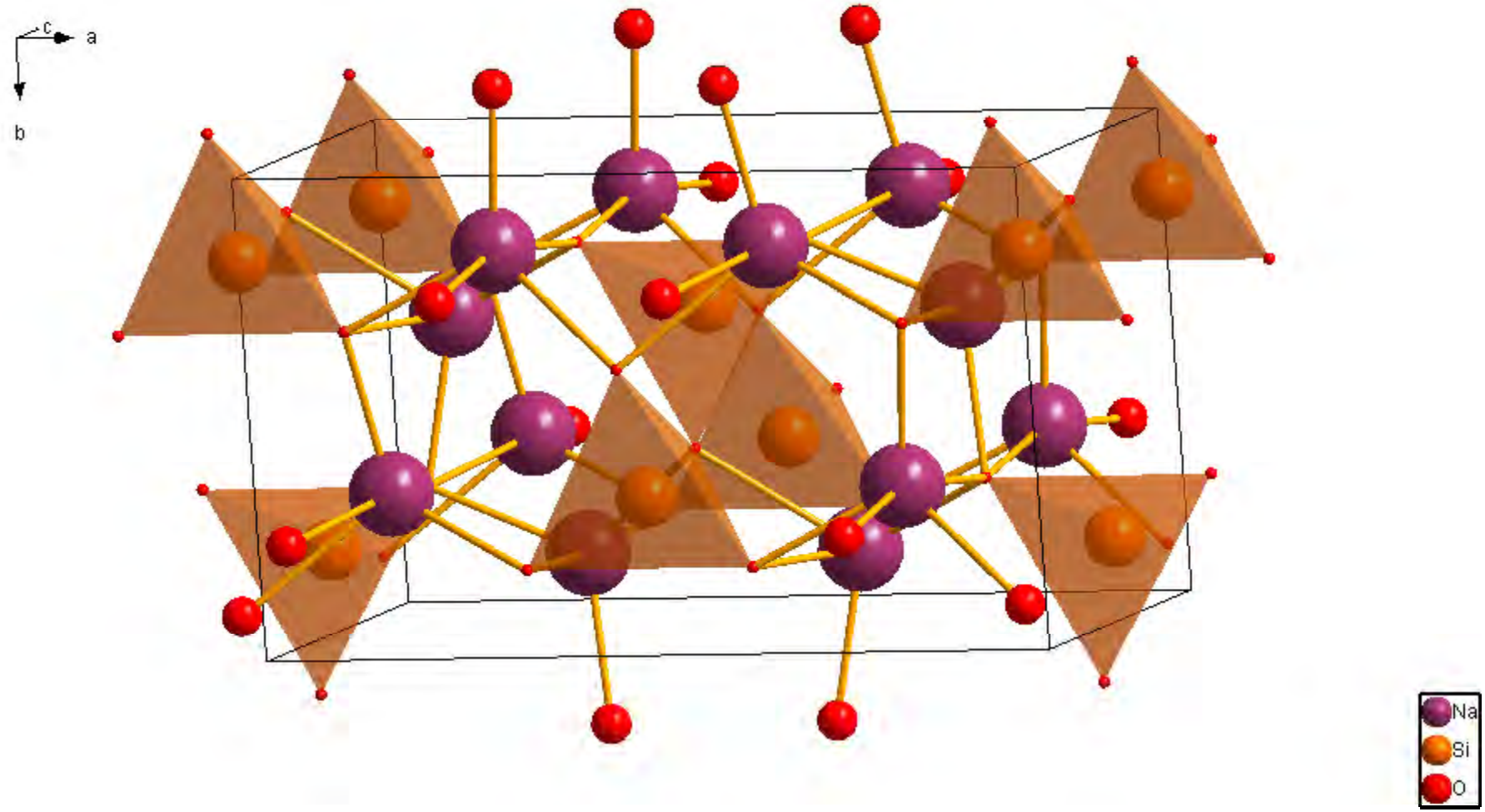
Cleaning Products

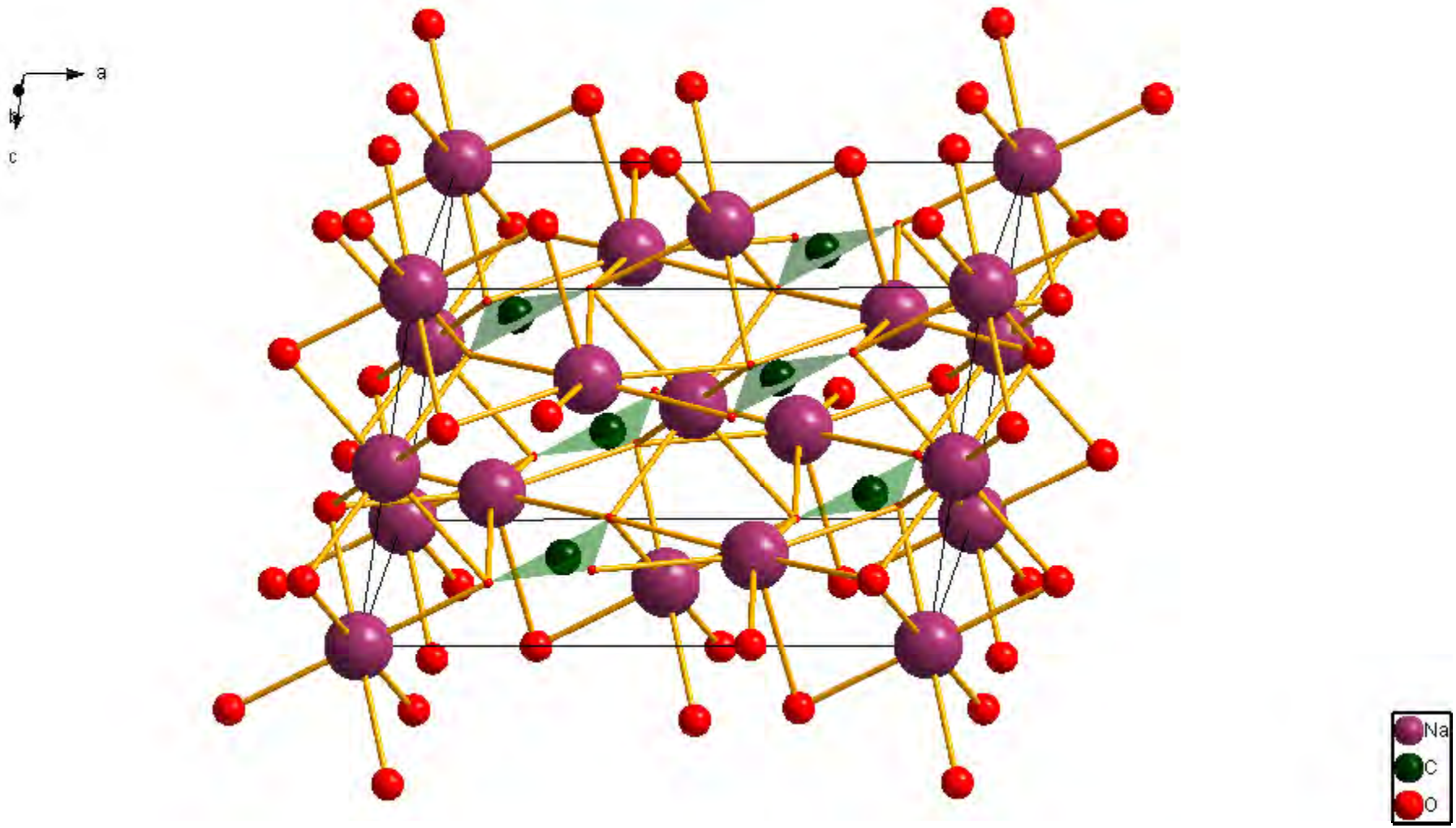
OxiClean Bulk



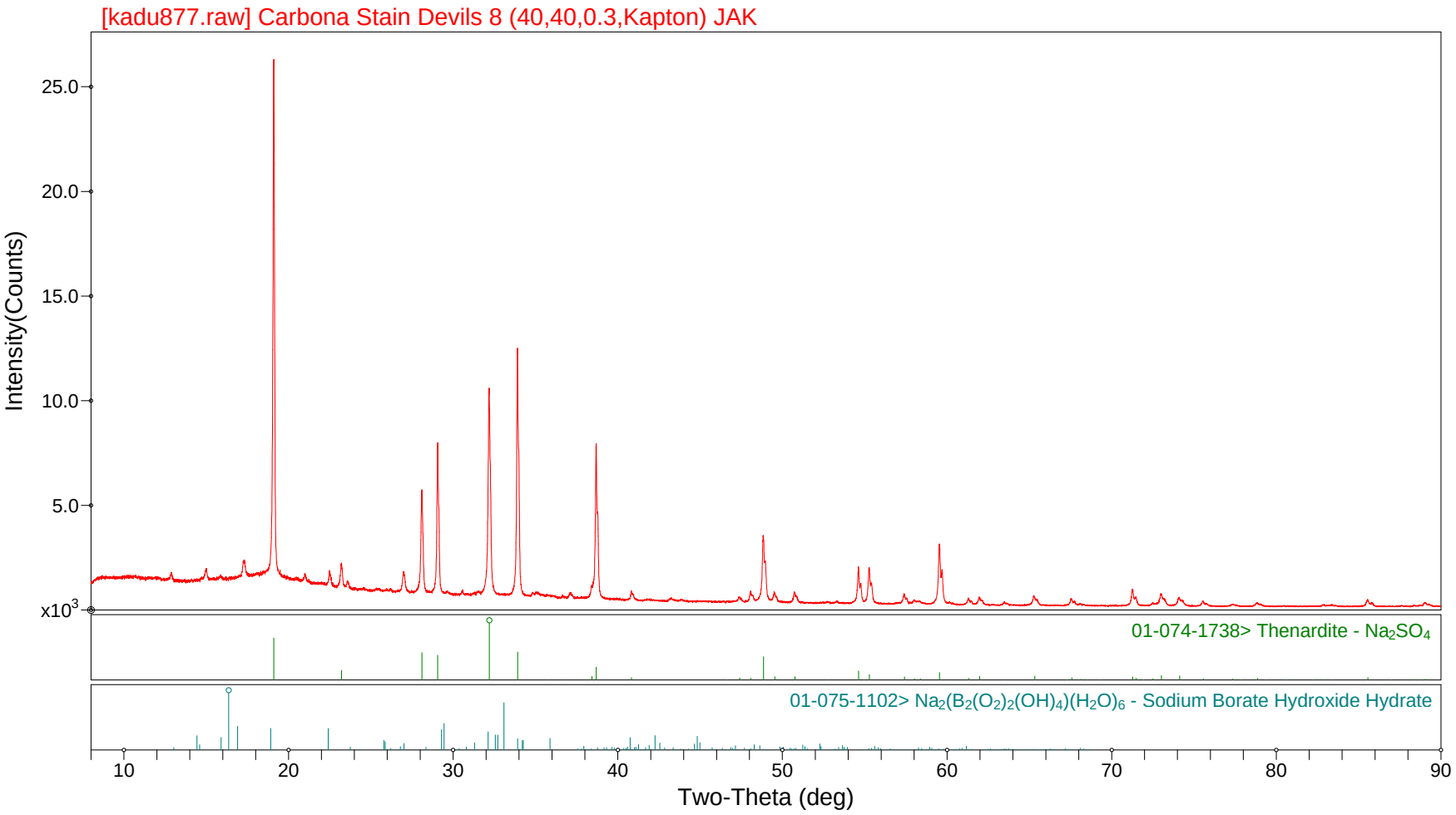
OxiClean Blue Granules




c
a
b
Na
Si
O


a
b
c
Na
C
O

Carbona Stain Devils 8

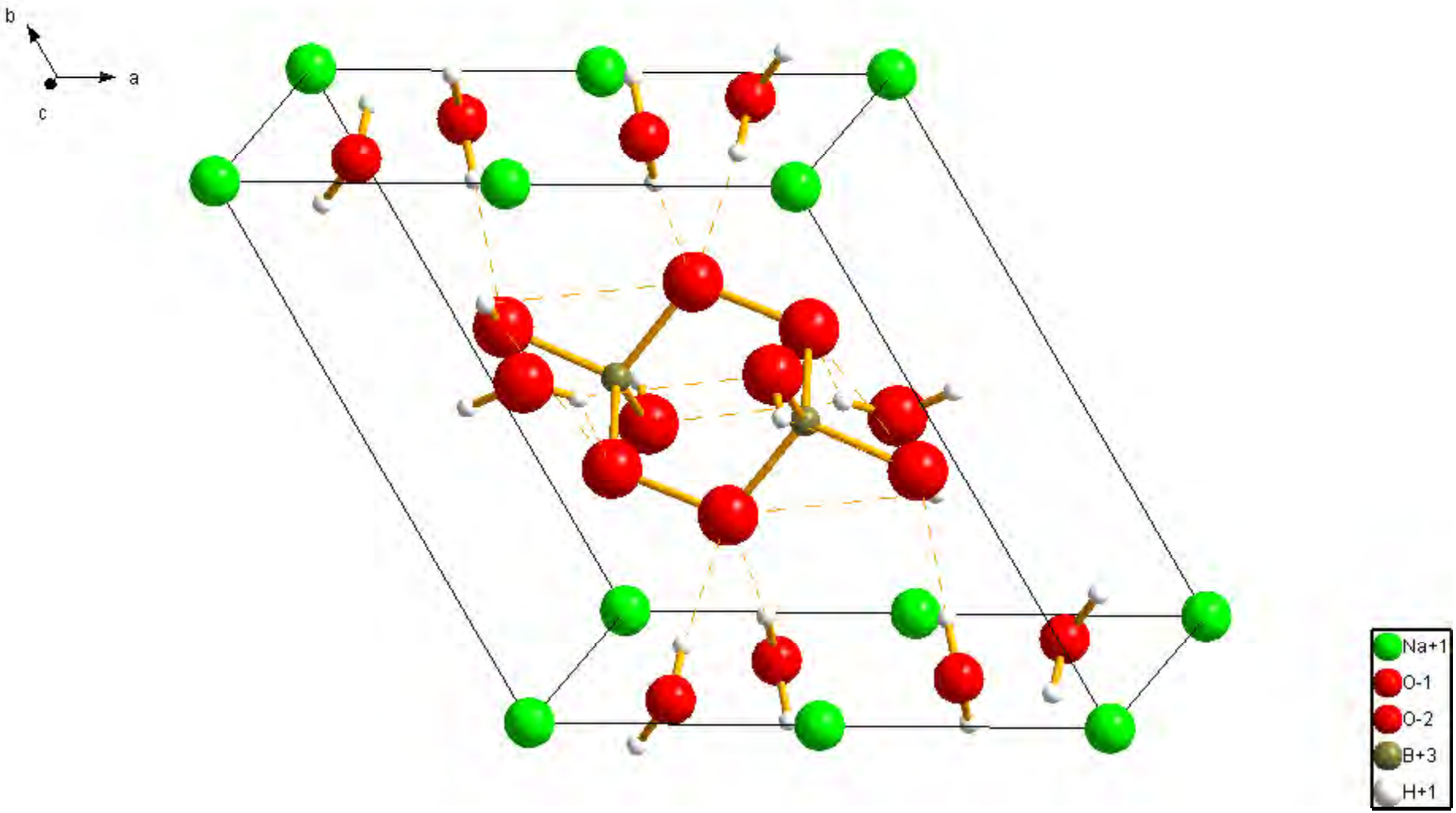


Sodium Peroxyborates

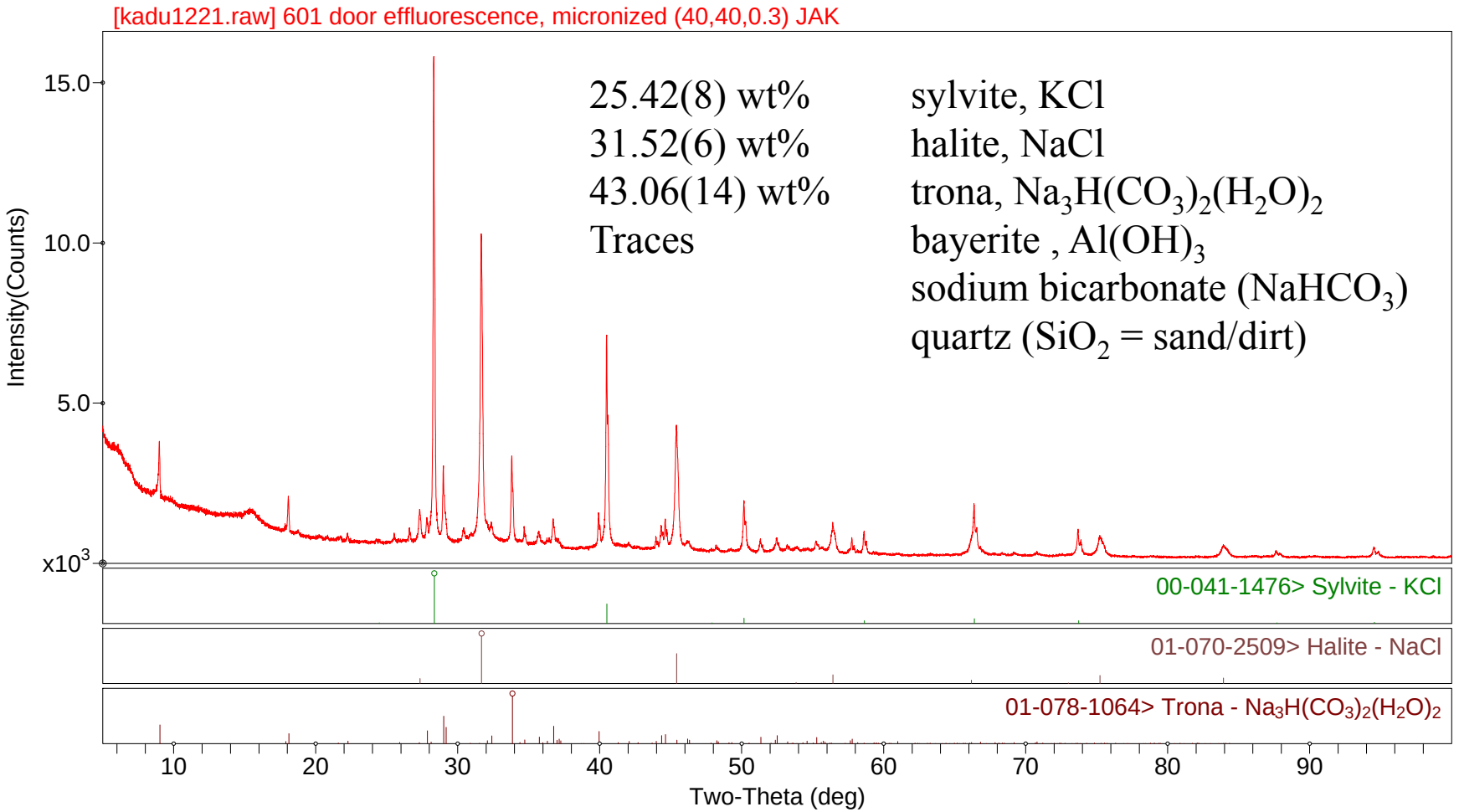
$Na_2[B_2(O_2)_2(OH)_4](H_2O)_6$

$Na_2[B_2(O_2)_2(OH)_4](H_2O)_4$

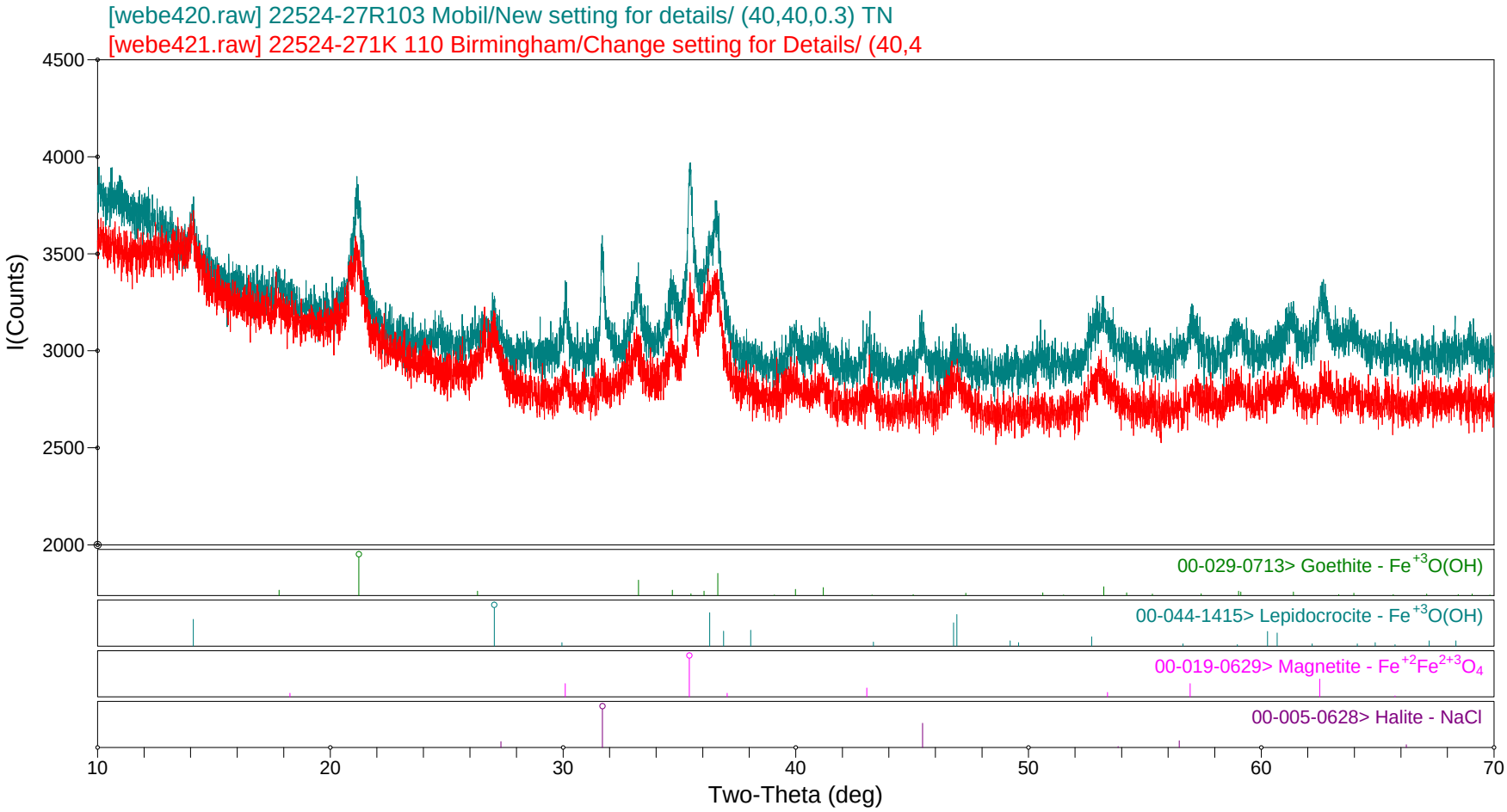
$Na_2[B_2(O_2)_2(OH)_4]$


b
a
c
Na+1
O-1
O-2
B+3
H+1

Door Crud


[kadu1221.raw] 601 door effluorescence, micronized (40,40,0.3) JAK
25.42(8) wt%
31.52(6) wt%
43.06(14) wt%
Traces
sylvite, KCl
halite, NaCl
trona, Na3H(CO3)2(H2O)2
bayerite , Al(OH)3
sodium bicarbonate (NaHCO3)
quartz (SiO2 = sand/dirt)
Intensity(Counts)
15.0
10.0
5.0
x10^3
00-041-1476> Sylvite - KCl
01-070-2509> Halite - NaCl
01-078-1064> Trona - Na3H(CO3)2(H2O)2
10
20
30
40
50
60
70
80
90
Two-Theta (deg)

Rust


[webe420.raw] 22524-27R103 Mobil/New setting for details/ (40,40,0.3) TN
[webe421.raw] 22524-271K 110 Birmingham/Change setting for Details/ (40,4
I(Counts)
4500
4000
3500
3000
2500
2000
00-029-0713> Goethite - Fe+3O(OH)
00-044-1415> Lepidocrocite - Fe+3O(OH)
00-019-0629> Magnetite - Fe+2Fe2+3O4
00-005-0628> Halite - NaCl
10
20
30
40
50
60
70
Two-Theta (deg)

Crystalline Phases in Tank Sludges

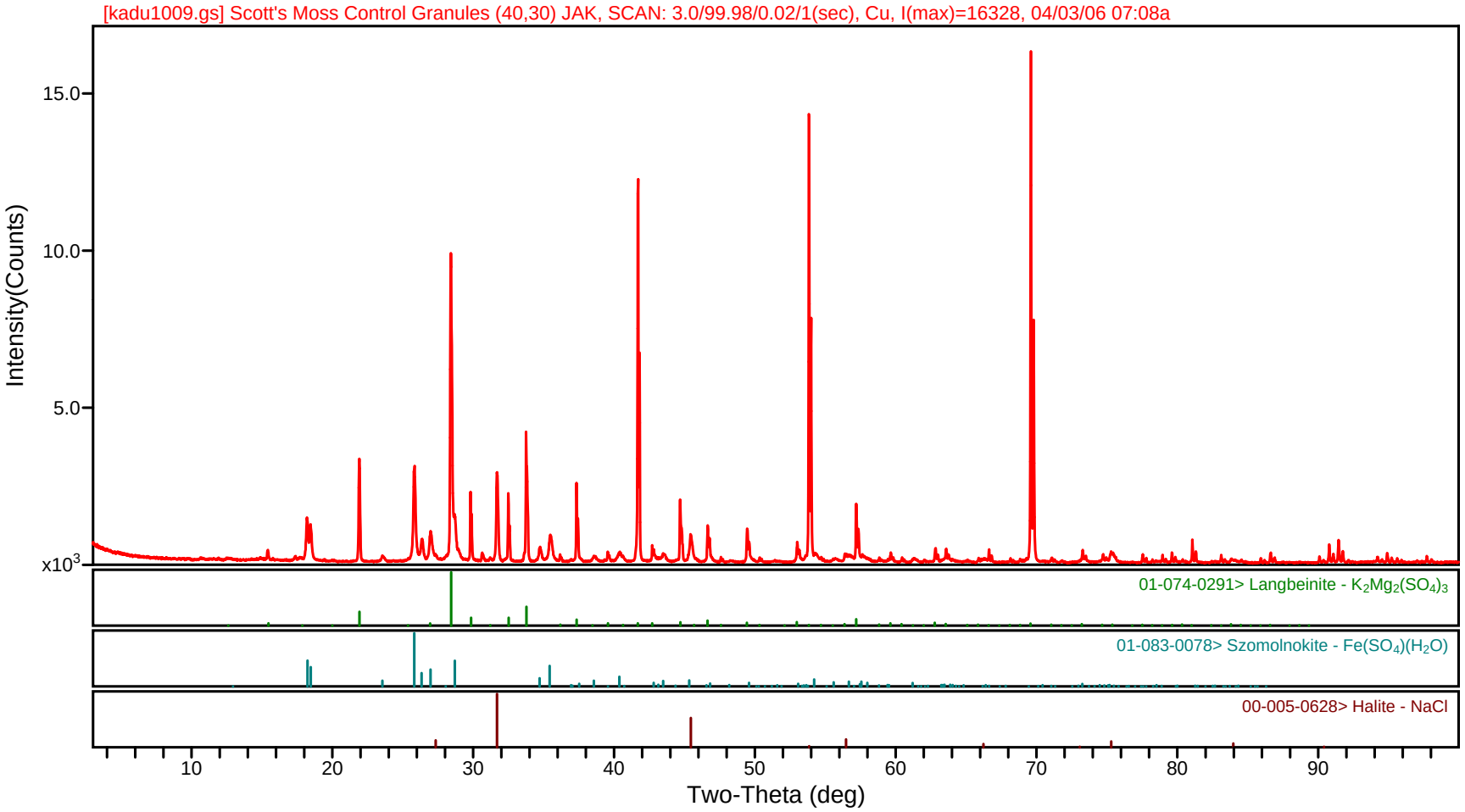
Sample	Mobile	Birmingham
Goethite, α-FeOOH, wt% Size, Å	57.7(3) 200 × 120 × 120	64.7(3) 160 × 80 × 80
Lepidocrocite, γ-FeOOH, wt% Size, Å	10.3(4) 110 × 580 × 580	26.8(5) 70 × 270 × 270
Magnetite, Fe_3O_4, wt% Size, Å	23.3(3) 190	8.6(3) 280
Halite, NaCl, wt% Size, Å	8.7(2) 480	-

Scott's Moss Control Granules

0-0-16 (N-P-K oxides)
double sulfate of K and Mg
17.5% $FeSO_4(H_2O)$

K_2O	16%
Mg	8%
S	20%
Fe	5%

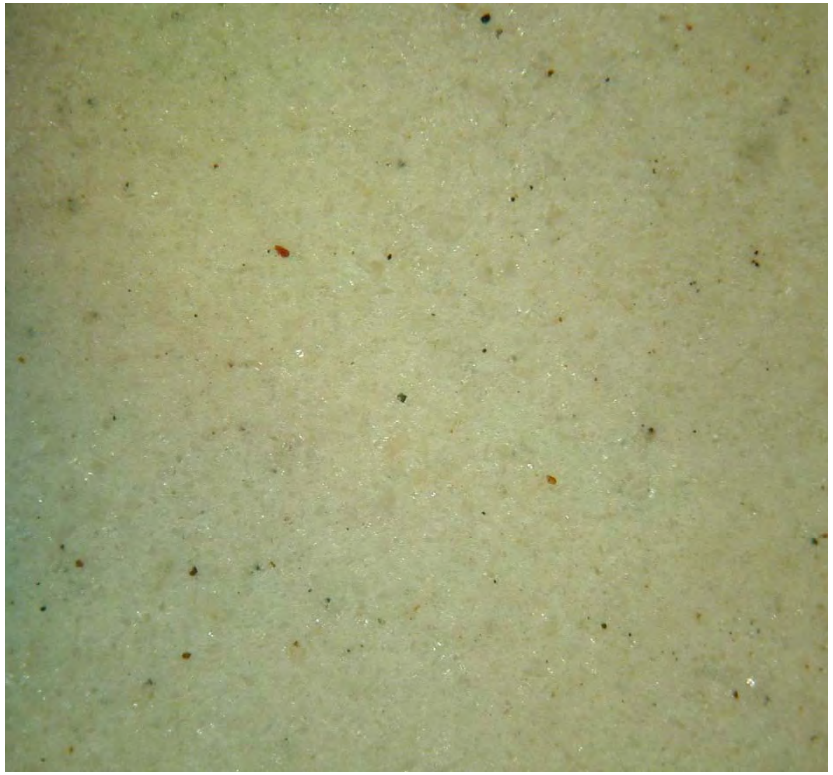
Grind in a mortar and pestle, and measure from a static specimen


[kadu1009.gs] Scott's Moss Control Granules (40,30) JAK, SCAN: 3.0/99.98/0.02/1(sec), Cu, I(max)=16328, 04/03/06 07:08a
Intensity(Counts)
15.0
10.0
5.0
x10^3
01-074-0291> Langbeinite - K2Mg2(SO4)3
01-083-0078> Szomolnokite - Fe(SO4)(H2O)
00-005-0628> Halite - NaCl
10
20
30
40
50
60
70
80
90
Two-Theta (deg)

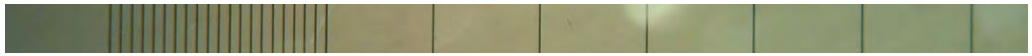
Micronize (corundum/hexane) and re-measure a rotating specimen

Pictures of the specimen surfaces

Hand Ground

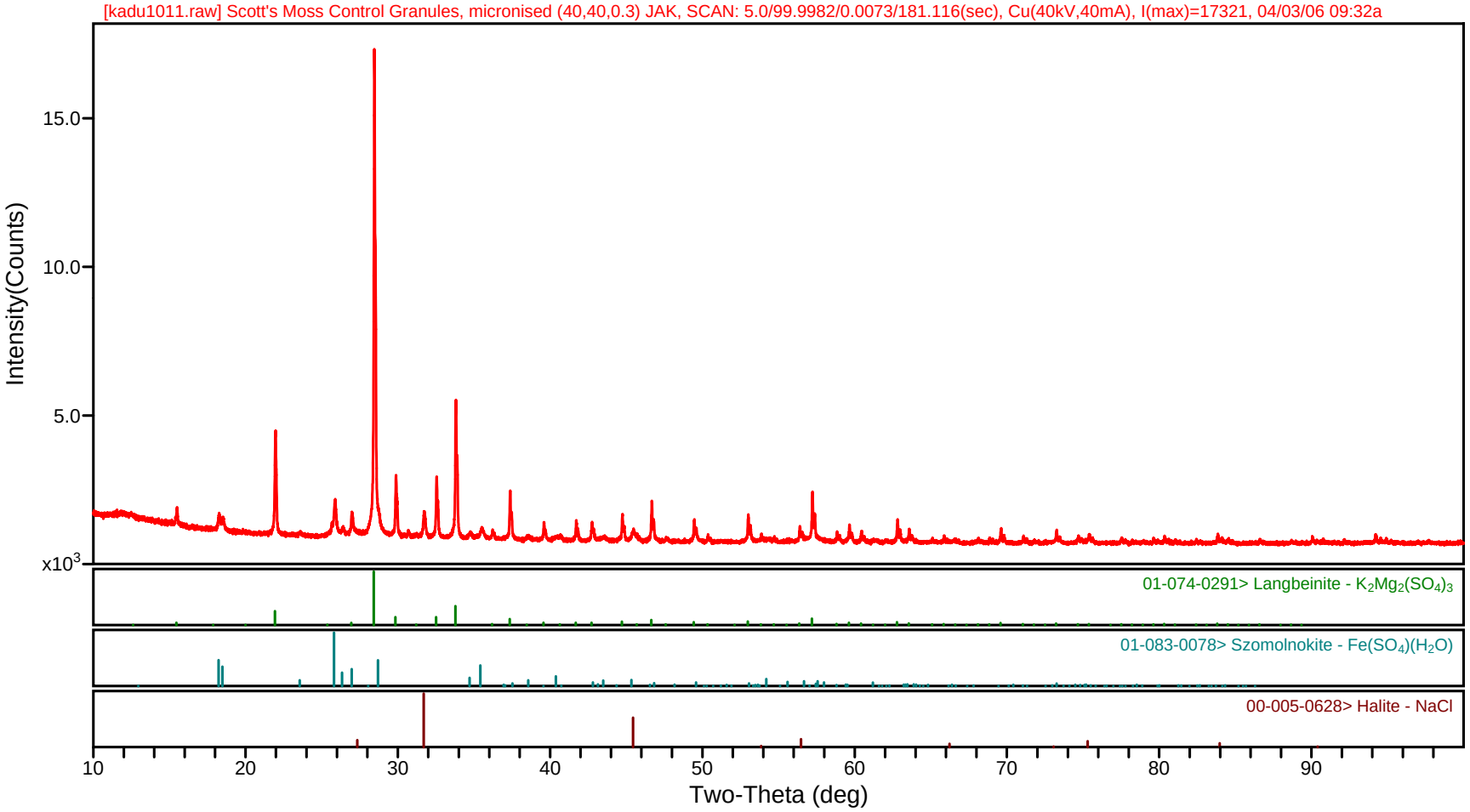


Micronised

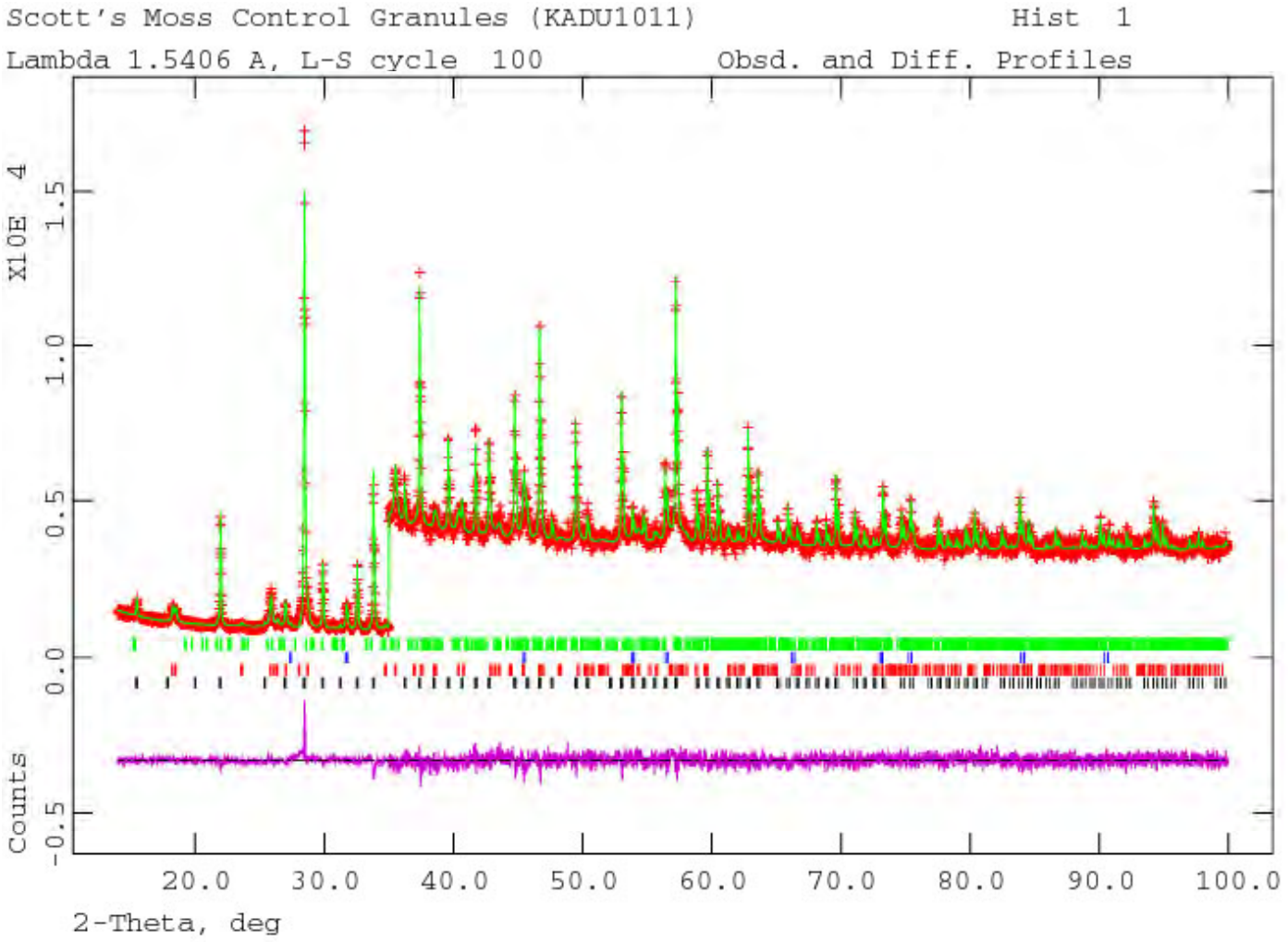


100 μm

Pictures by B. J. Huggins, BP Analytical


[kadu1011.raw] Scott's Moss Control Granules, micronised (40,40,0.3) JAK, SCAN: 5.0/99.9982/0.0073/181.116(sec), Cu(40kV,40mA), I(max)=17321, 04/03/06 09:32a
Intensity(Counts)
15.0
10.0
5.0
x10^3
01-074-0291> Langbeinite - K2Mg2(SO4)3
01-083-0078> Szomolnokite - Fe(SO4)(H2O)
00-005-0628> Halite - NaCl
10
20
30
40
50
60
70
80
90
Two-Theta (deg)

Look up the structures and carry out a Rietveld refinement



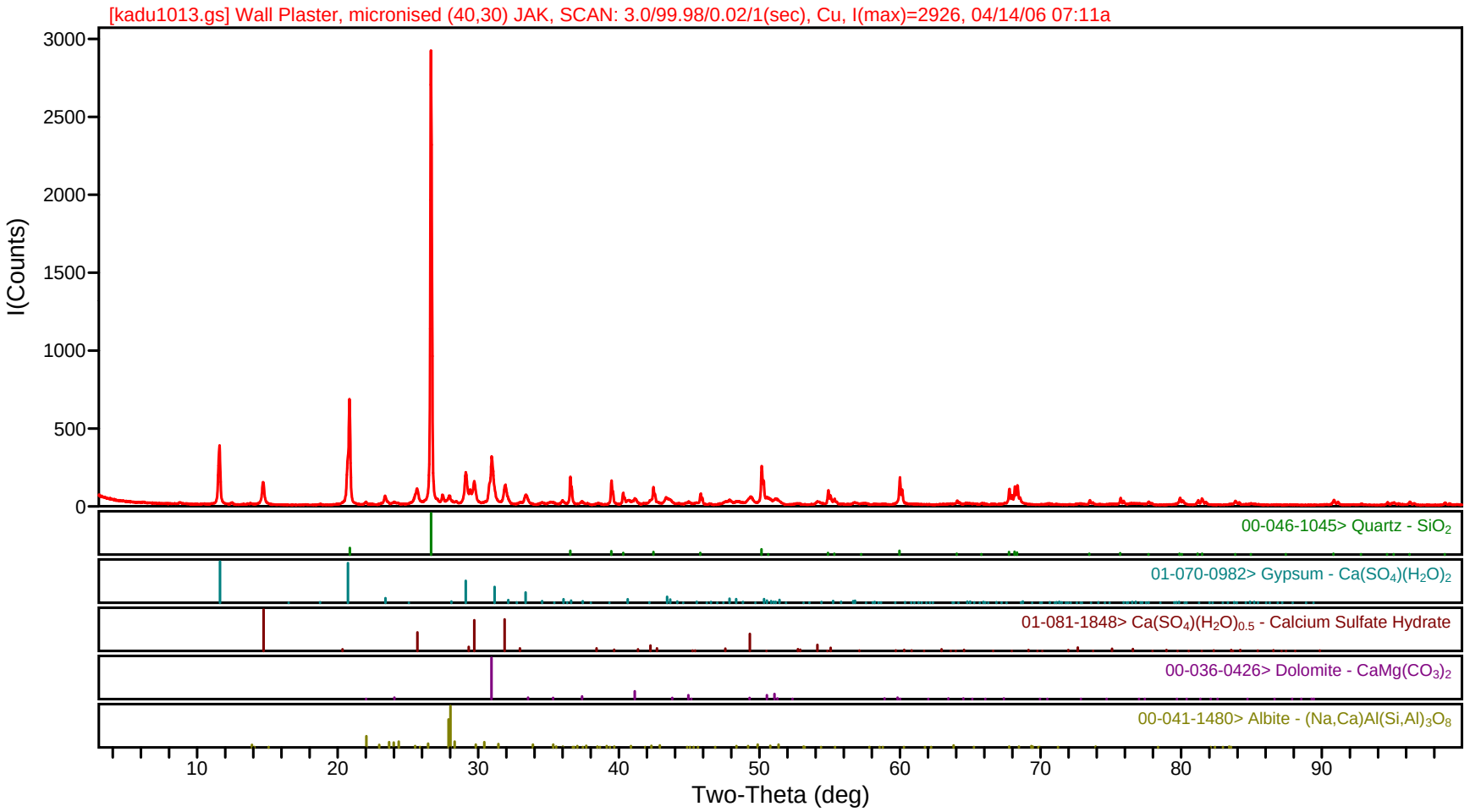
Quantitative Phase Analysis

Langbeinite	$K_2Mg_2(SO_4)_3$	80.49(4) wt%
Szomolnokite	$FeSO_4(H_2O)$	15.6(1) wt%
Halite	NaCl	3.74(6) wt%
Vanthoffite??	$Na_6Mg(SO_4)_4$	0.2(2) wt%

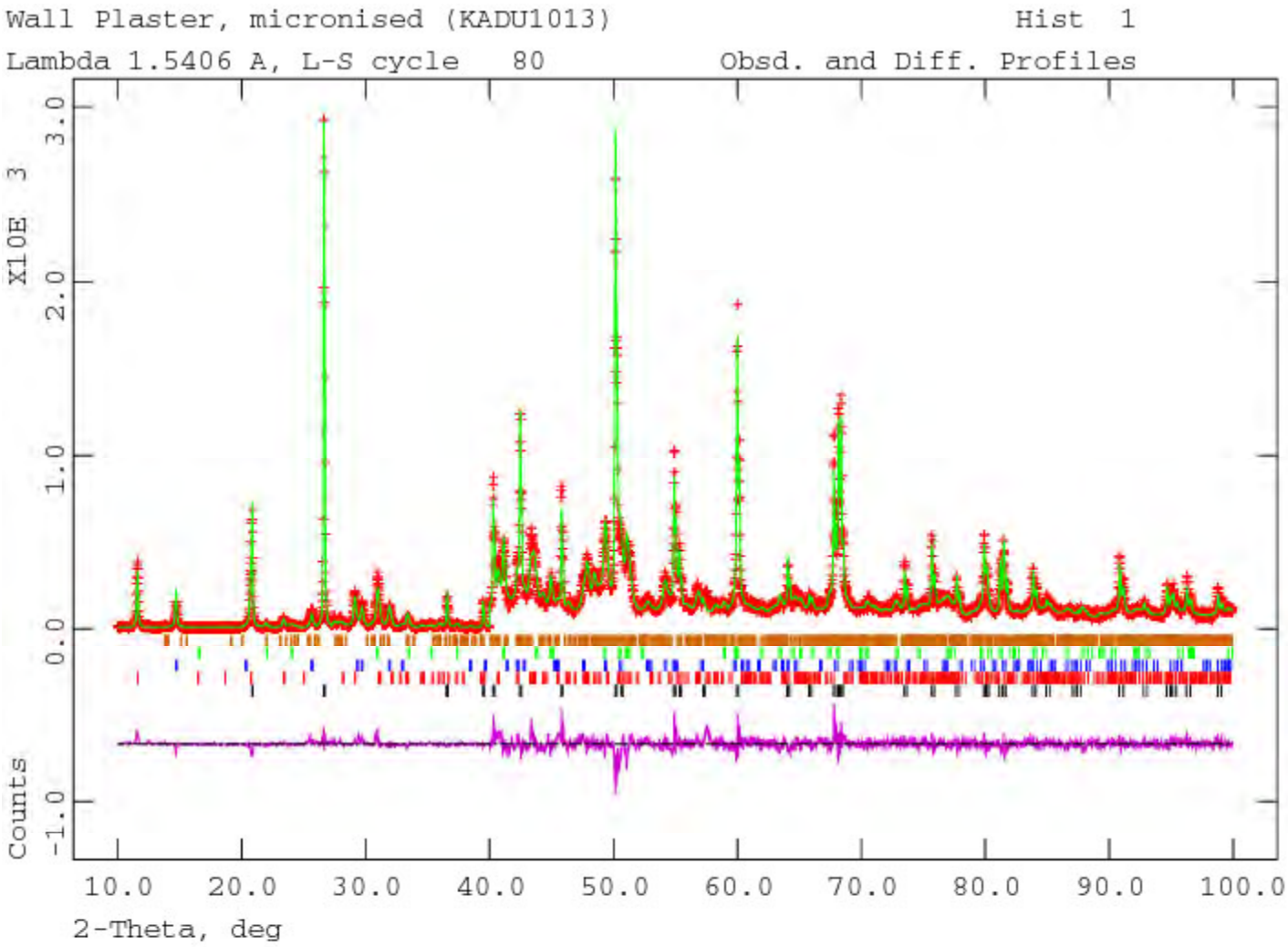
Observed and Expected Composition

	Observed, wt%	Bag, wt%
$FeSO_4(H_2O)$	15.6(1)	17.5
Fe	5.1	5
K_2O	18.2	16
Mg	9.4	8
S	21.5	20

Plaster


[kadu1013.gs] Wall Plaster, micronised (40,30) JAK, SCAN: 3.0/99.98/0.02/1(sec), Cu, I(max)=2926, 04/14/06 07:11a
I(Counts)
3000
2500
2000
1500
1000
500
0
00-046-1045> Quartz - SiO2
01-070-0982> Gypsum - Ca(SO4)(H2O)2
01-081-1848> Ca(SO4)(H2O)0.5 - Calcium Sulfate Hydrate
00-036-0426> Dolomite - CaMg(CO3)2
00-041-1480> Albite - (Na,Ca)Al(Si,Al)3O8
10
20
30
40
50
60
70
80
90
Two-Theta (deg)

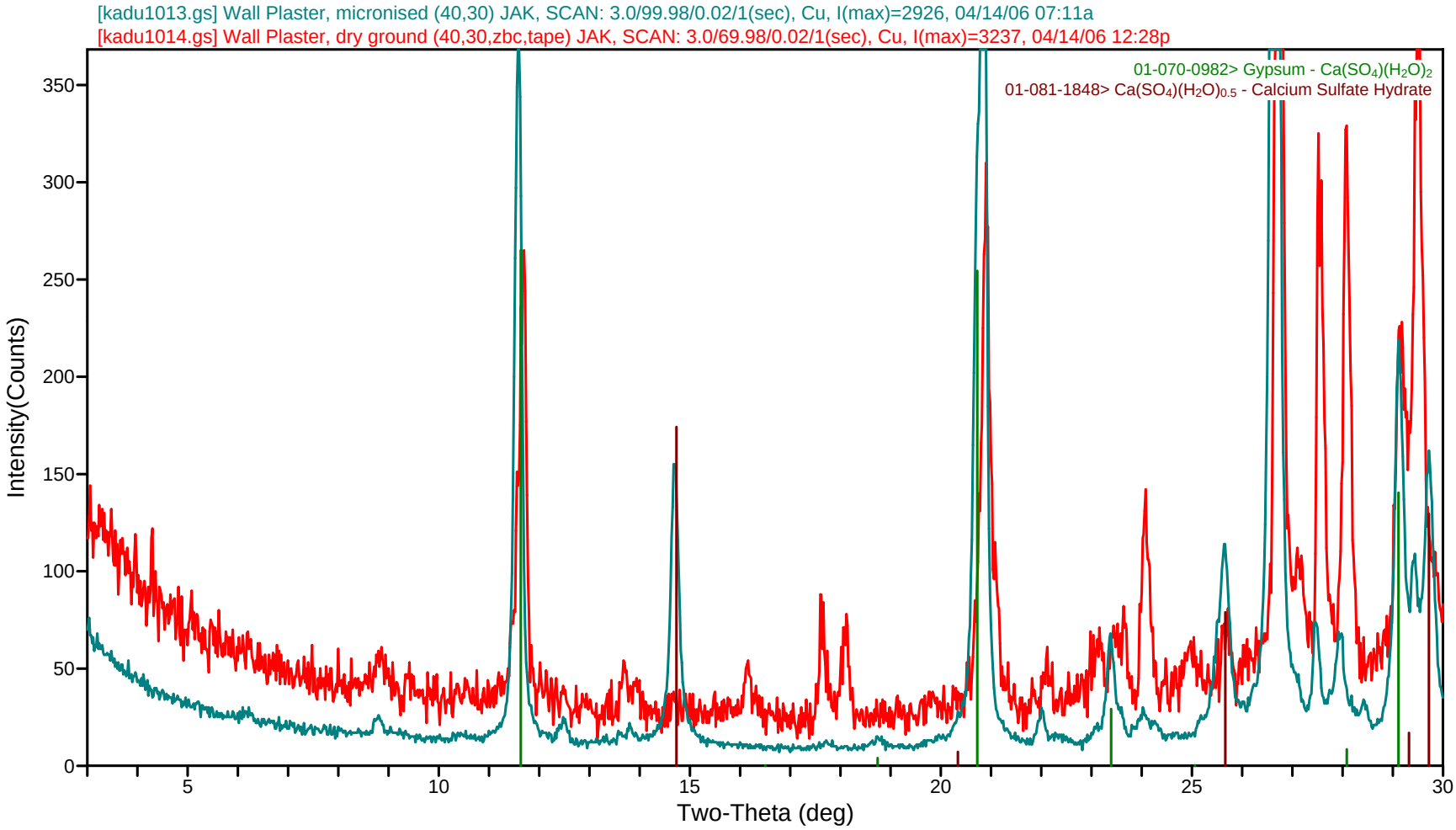
Carry out a Rietveld refinement



Quantitative Phase Analysis

Name	Formula	Concentration, wt%
Quartz	SiO_2	48.0(2)
Gypsum	$CaSO_4(H_2O)_2$	22.8(2)
Bassanite	$CaSO_4(H_2O)_{0.5}$	12.0(1)
Dolomite	$CaMg(CO_3)_2$	9.3(2)
Albite	$(Na,Ca)(Si,Al)_4O_8$	7.9(2)

But the bassanite was not present in the original sample!


[kadu1013.gs] Wall Plaster, micronised (40,30) JAK, SCAN: 3.0/99.98/0.02/1(sec), Cu, I(max)=2926, 04/14/06 07:11a
[kadu1014.gs] Wall Plaster, dry ground (40,30,zbc,tape) JAK, SCAN: 3.0/69.98/0.02/1(sec), Cu, I(max)=3237, 04/14/06 12:28p
01-070-0982> Gypsum - Ca(SO4)(H2O)2
01-081-1848> Ca(SO4)(H2O)0.5 - Calcium Sulfate Hydrate
Intensity(Counts)
0
50
100
150
200
250
300
350
5
10
15
20
25
30
Two-Theta (deg)

When heated in air, gypsum is converted slowly to the (metastable) hemihydrate at about 70°C or below, and rapidly at 90°C and above…

W. A. Deere, R. A. Howie, and J. Zussman,

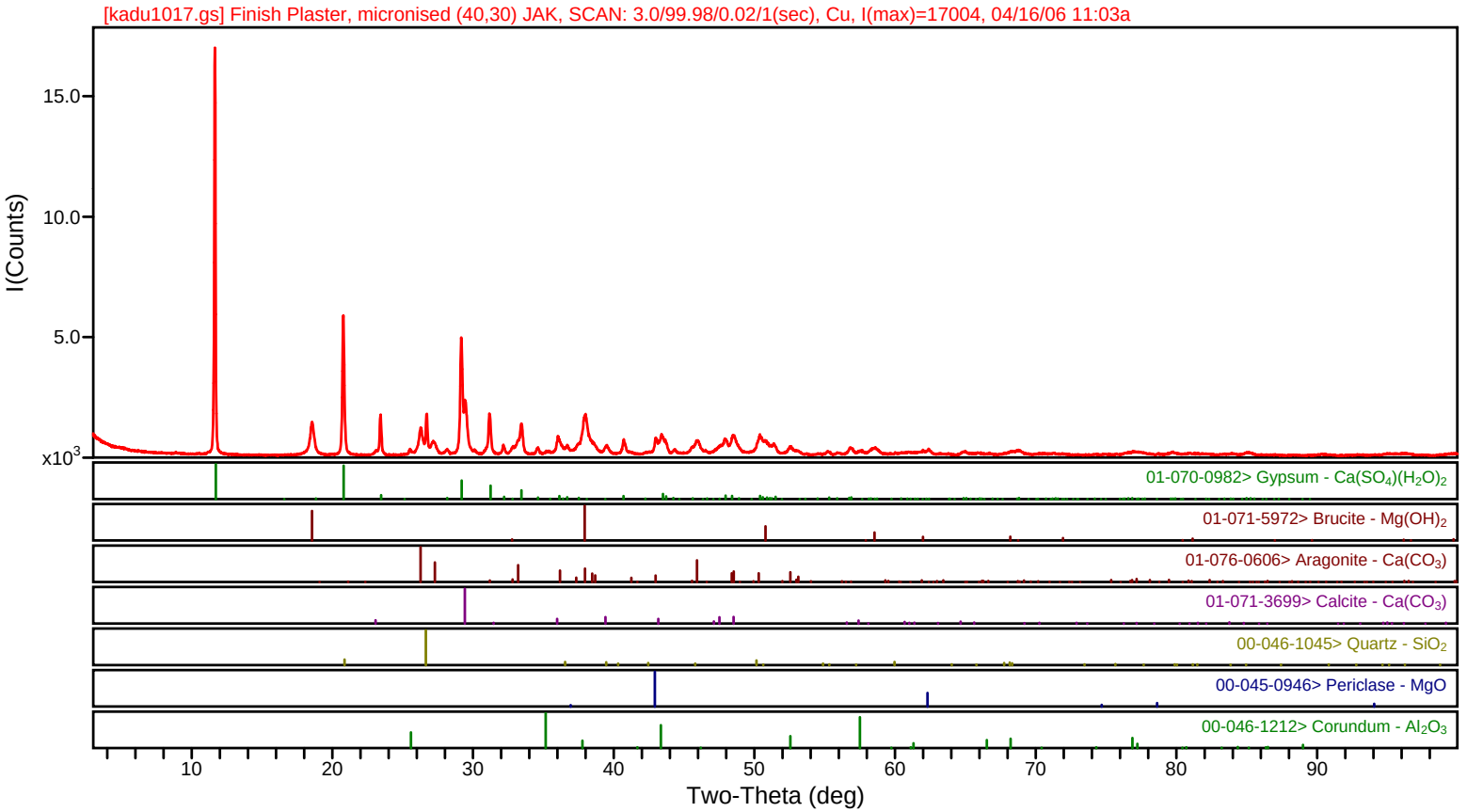
An Introduction to the Rock-Forming Minerals, 2nd Edition (1992), p. 614.

Renormalize the concentrations:

Name	Formula	Concentration, wt%
Quartz	SiO_2	47.0(2)
Gypsum	$CaSO_4(H_2O)_2$	36.2(3)
Dolomite	$CaMg(CO_3)_2$	9.1(2)
Albite	$(Na,Ca)(Si,Al)_4O_8$	7.7(2)

The finish coat plaster is different

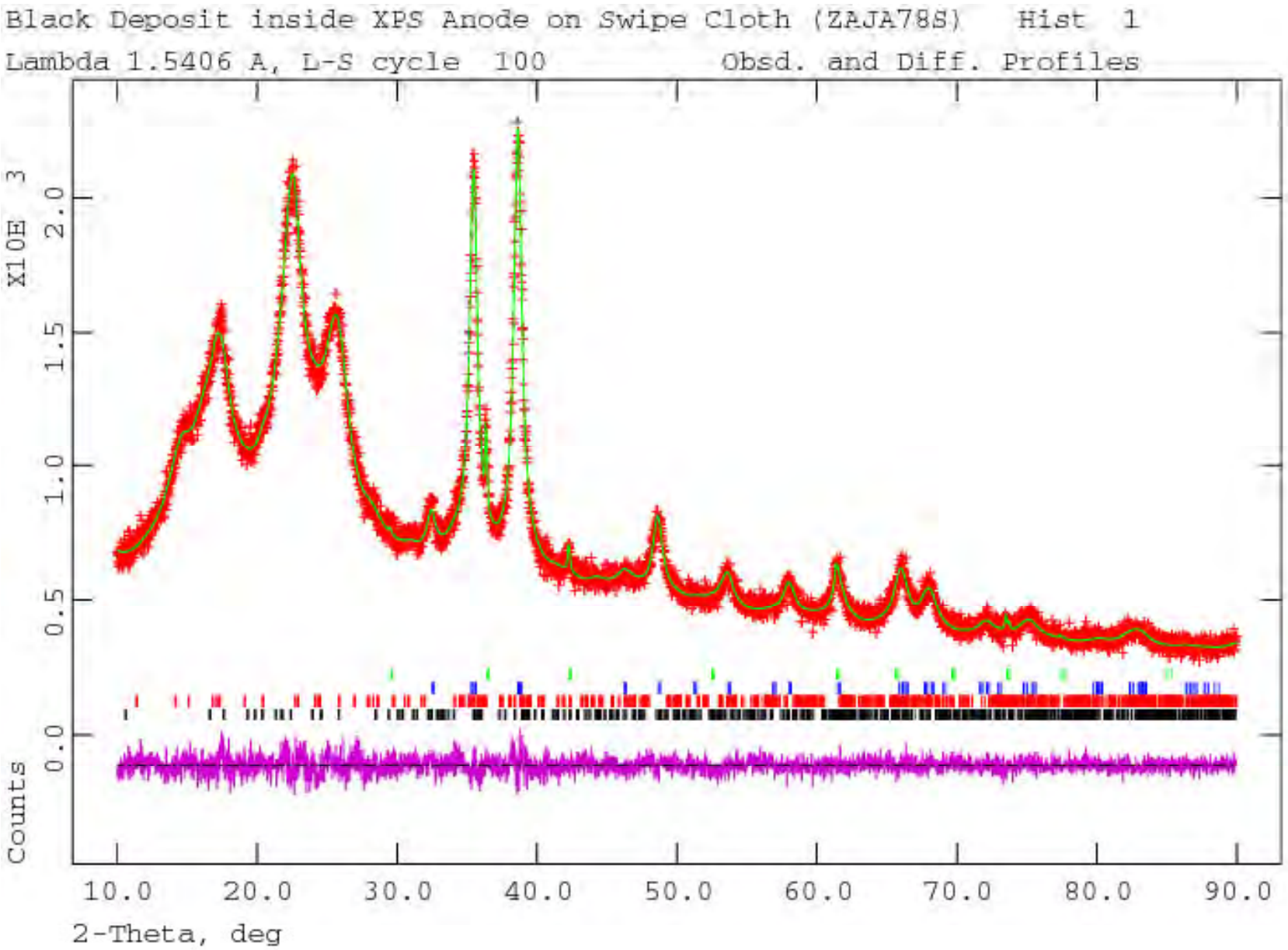
(dry the slurry at ambient conditions!)



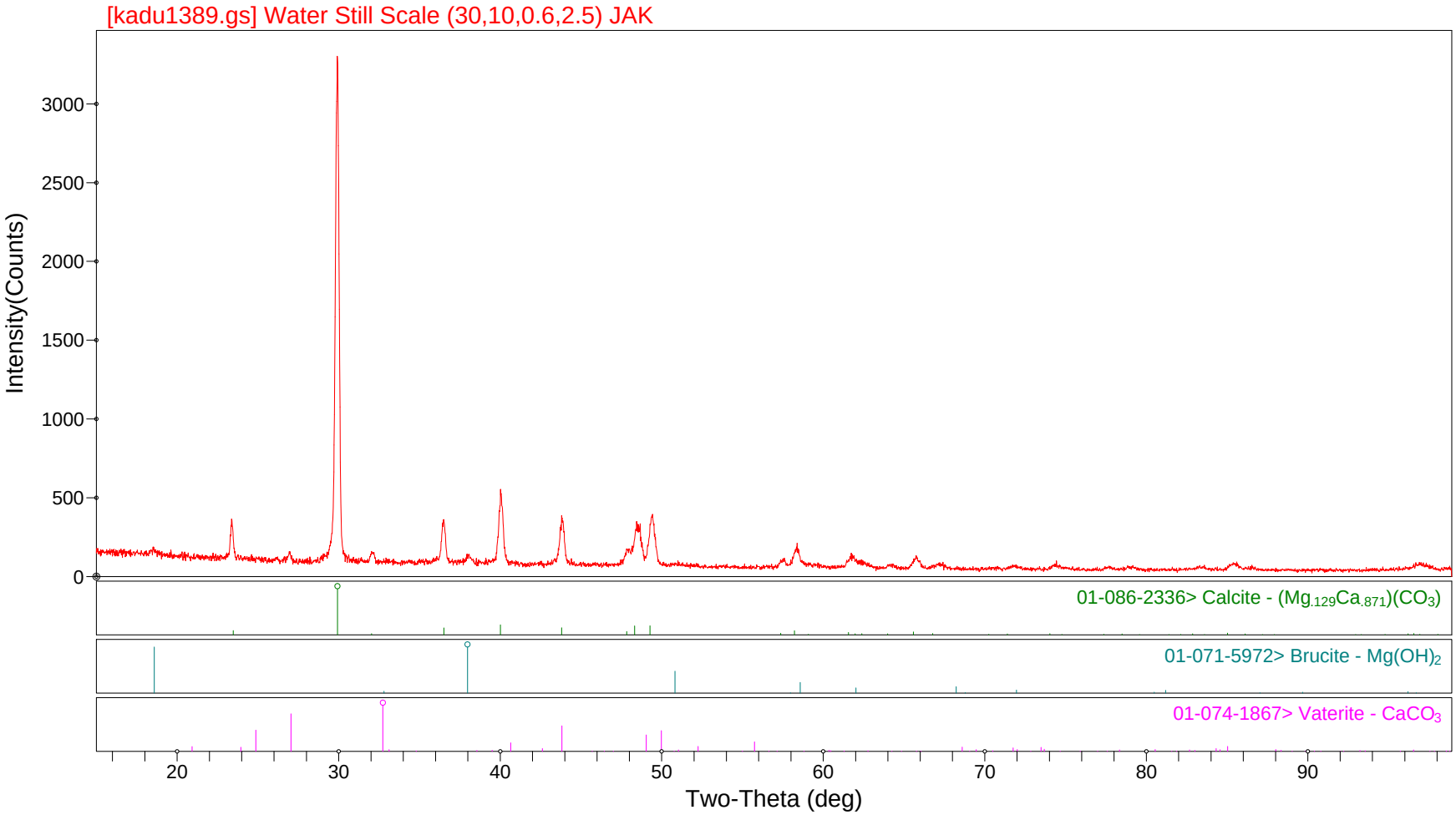
QPA of Finish Coat Plaster

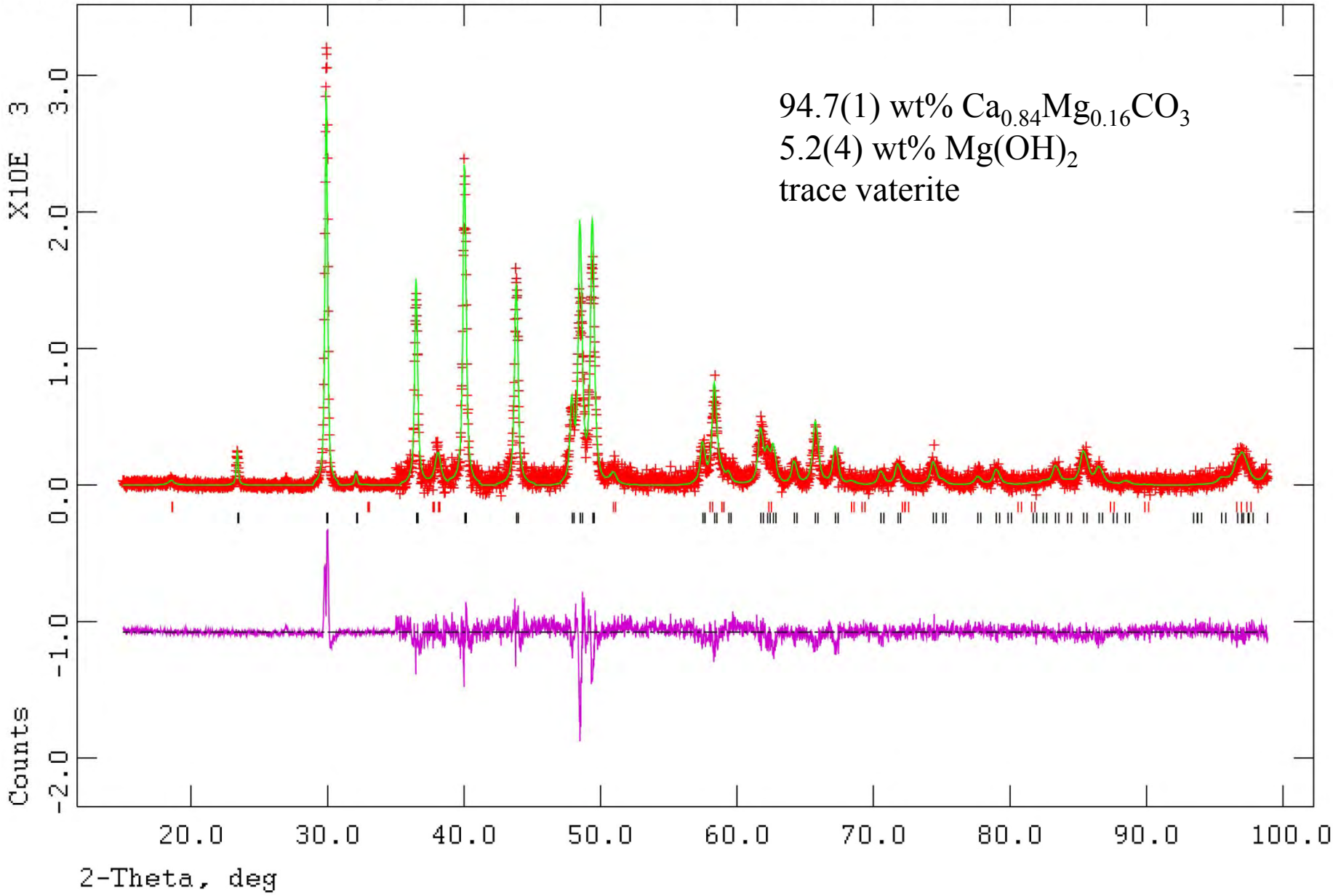
Mineral	Formula	Concentration, wt%
Gypsum	$CaSO_4(H_2O)_2$	34.2(2)
Brucite	$Mg(OH)_2$	19.3(1)
Aragonite	$CaCO_3$	26.0(2)
Calcite	$CaCO_3$	13.0(1)
Quartz	SiO_2	3.09(6)
Periclase	MgO	2.12(8)
Corundum (!)	Al_2O_3	2.3(1)

Crud on Cloth


Black Deposit inside XPS Anode on Swipe Cloth (ZAJA78S) Hist 1
Lambda 1.5406 A, L-S cycle 100 Obsd. and Diff. Profiles
X10E 3
2.0
1.5
1.0
0.5
0.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
2-Theta, deg

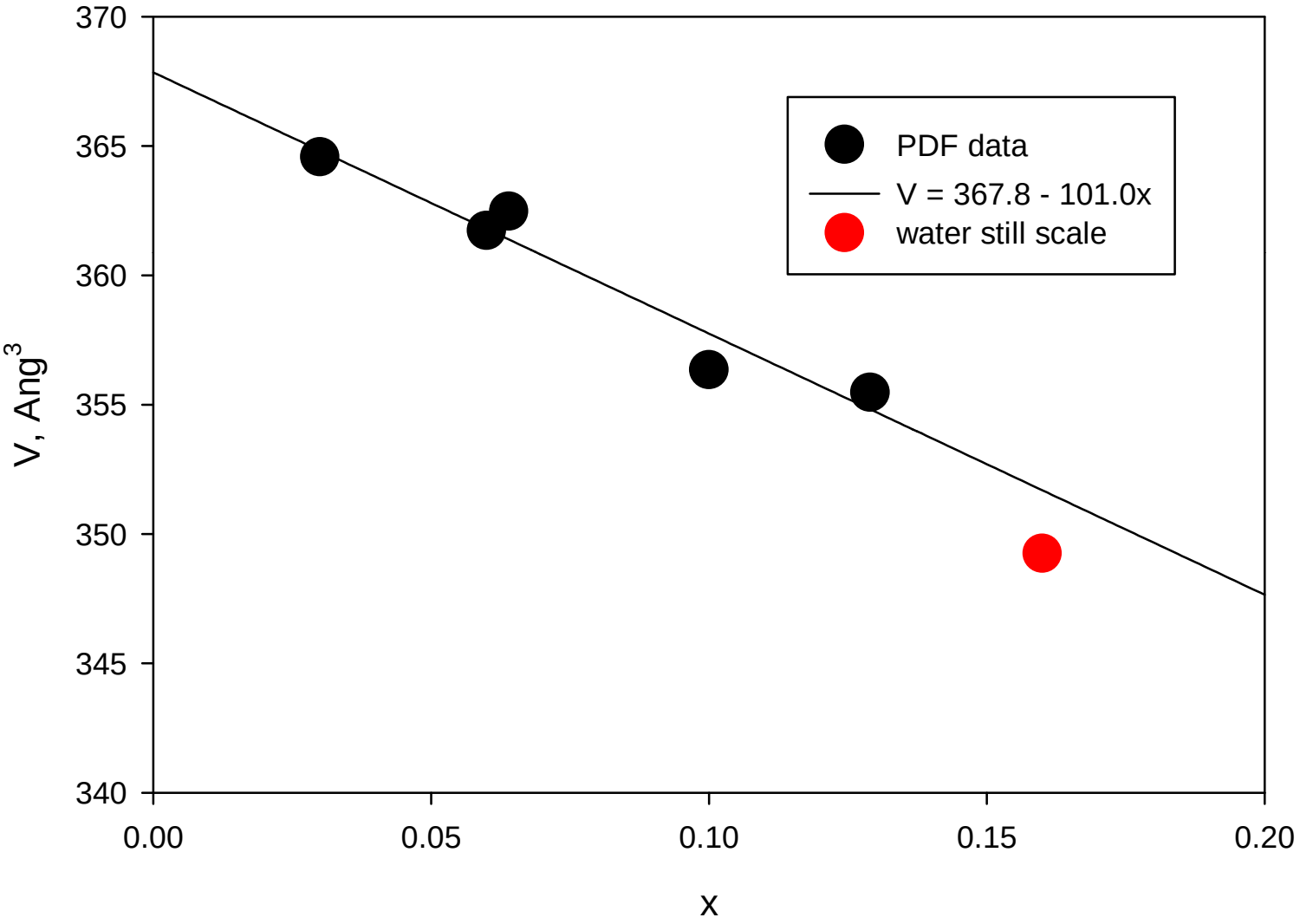
Water Still Scale


[kadu1389.gs] Water Still Scale (30,10,0.6,2.5) JAK
Intensity(Counts)
3000
2500
2000
1500
1000
500
0
01-086-2336> Calcite - (Mg.129Ca.871)(CO3)
01-071-5972> Brucite - Mg(OH)2
01-074-1867> Vaterite - CaCO3
20
30
40
50
60
70
80
90
Two-Theta (deg)



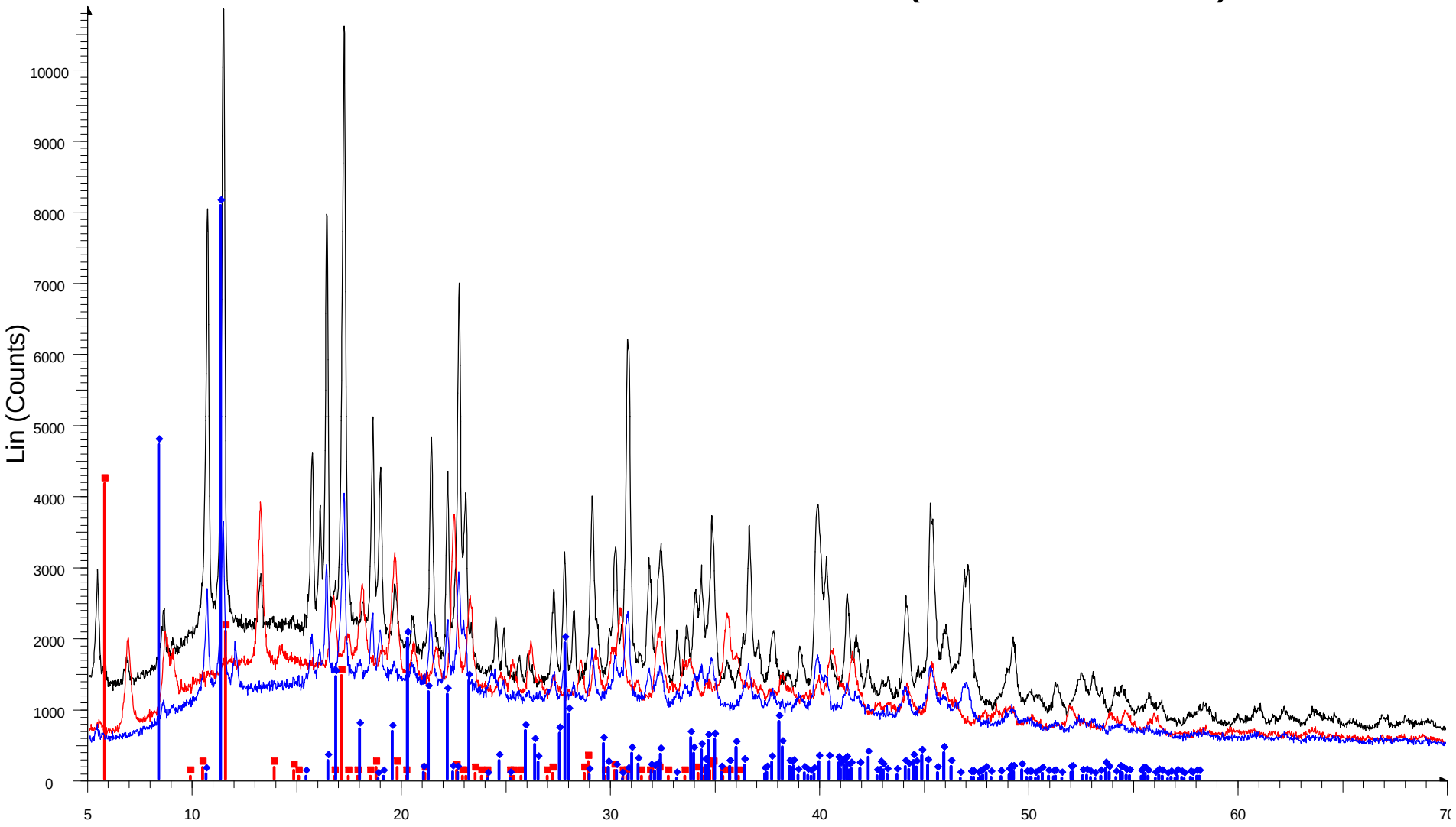
Scaling: 35.0(5.0X)

Unit Cell Volume of Magnesian Calcite, $Ca_{1-x}Mg_xCO_3$



Clean the still with citric acid solution

20307-1-4 citrate solution 3rd solid (30,10,0.6,2.5) JAK



20307-1-4 citrate solution 3rd solid (30,10,0.6,2.5) JAK - File: kadu1401.raw - Type: 2Th/Th locked - Start: 4.998 ° - End: 70.007 ° - Step: 0.020 ° - Step time: 192. s - Temp.: 25 °C (Room) - Time Started:
Operations: Import

Commander Sample ID - File: kadu1397.raw - Type: 2Th/Th locked - Start: 4.998 ° - End: 70.007 ° - Step: 0.020 ° - Step time: 96. s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 4.998 ° - Theta: 2.
Operations: Import

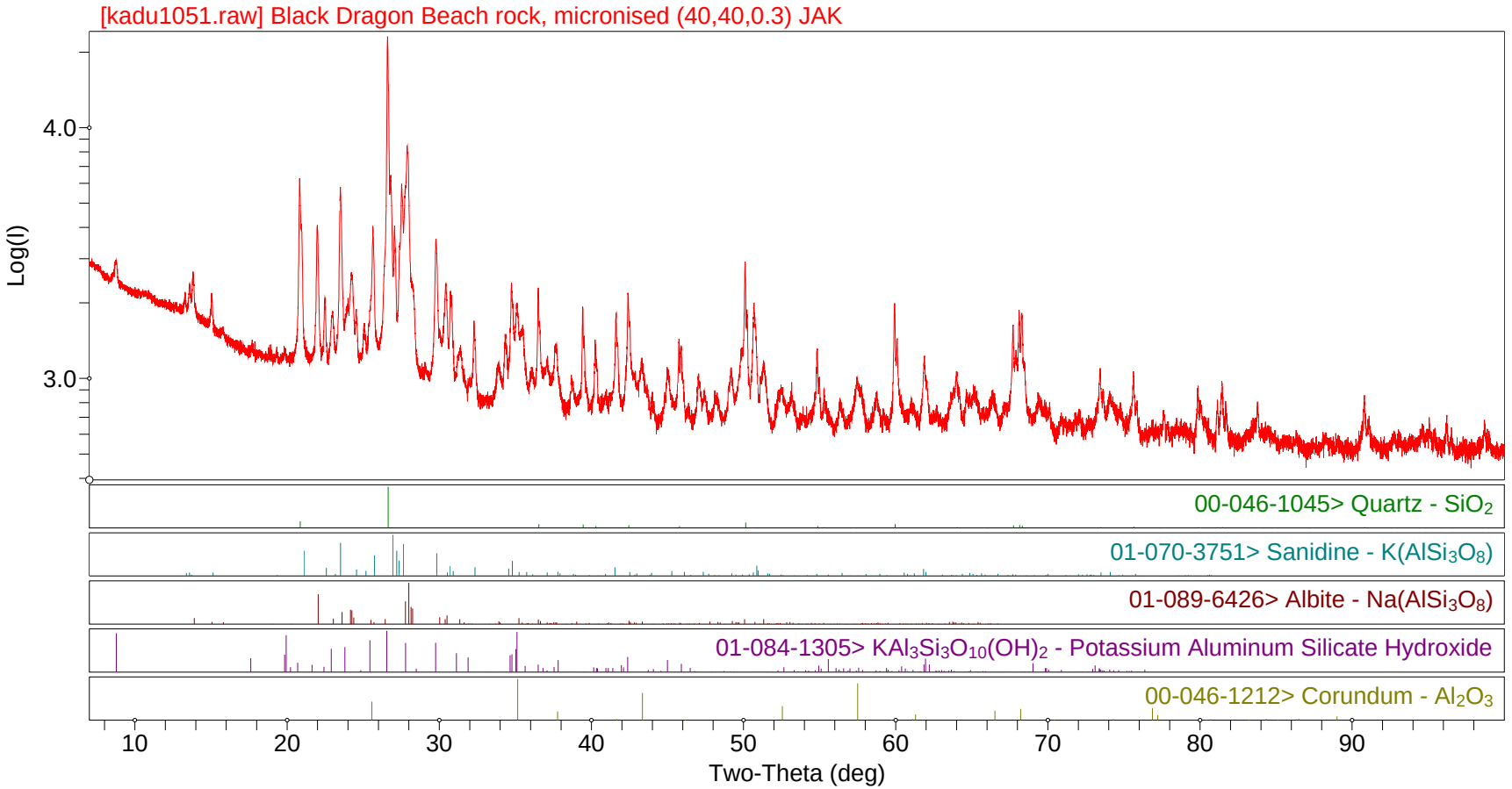
Commander Sample ID - File: kadu1393.raw - Type: 2Th/Th locked - Start: 4.998 ° - End: 70.007 ° - Step: 0.020 ° - Step time: 82.5 s - Temp.: 25 °C (Room) - Time Started: 0 s - 2-Theta: 4.998 ° - Theta: 2
Operations: Import

00-028-2003 (N) - Earlandite, syn - $Ca_3(C_6H_5O_7)_2 \cdot 4H_2O$ - Y: 38.37 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 30.94000 - b 5.93000 - c 10.56000 - alpha 90.000 - beta 93.740 - gamma 90.000 - Base-c

02-060-8946 (N) - Calcium hydrogen citrate trihydrate - $C_6H_6O_7 \cdot Ca \cdot 3(H_2O)$ - Y: 74.45 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 8.79550 - b 5.58910 - c 23.81800 - alpha 90.000 - beta 116.770 - gamm

Rocks


Puto Shan
Zhujia Jian
Image © 2006 DigitalGlobe
© 2006 Europa Technologies
Image © 2006 TerraMetrics
©2006 Google™
Pointer 29°56'23.58" N 122°22'27.33" E elev 7 ft
Streaming 100%
Eye alt 15.72 mi

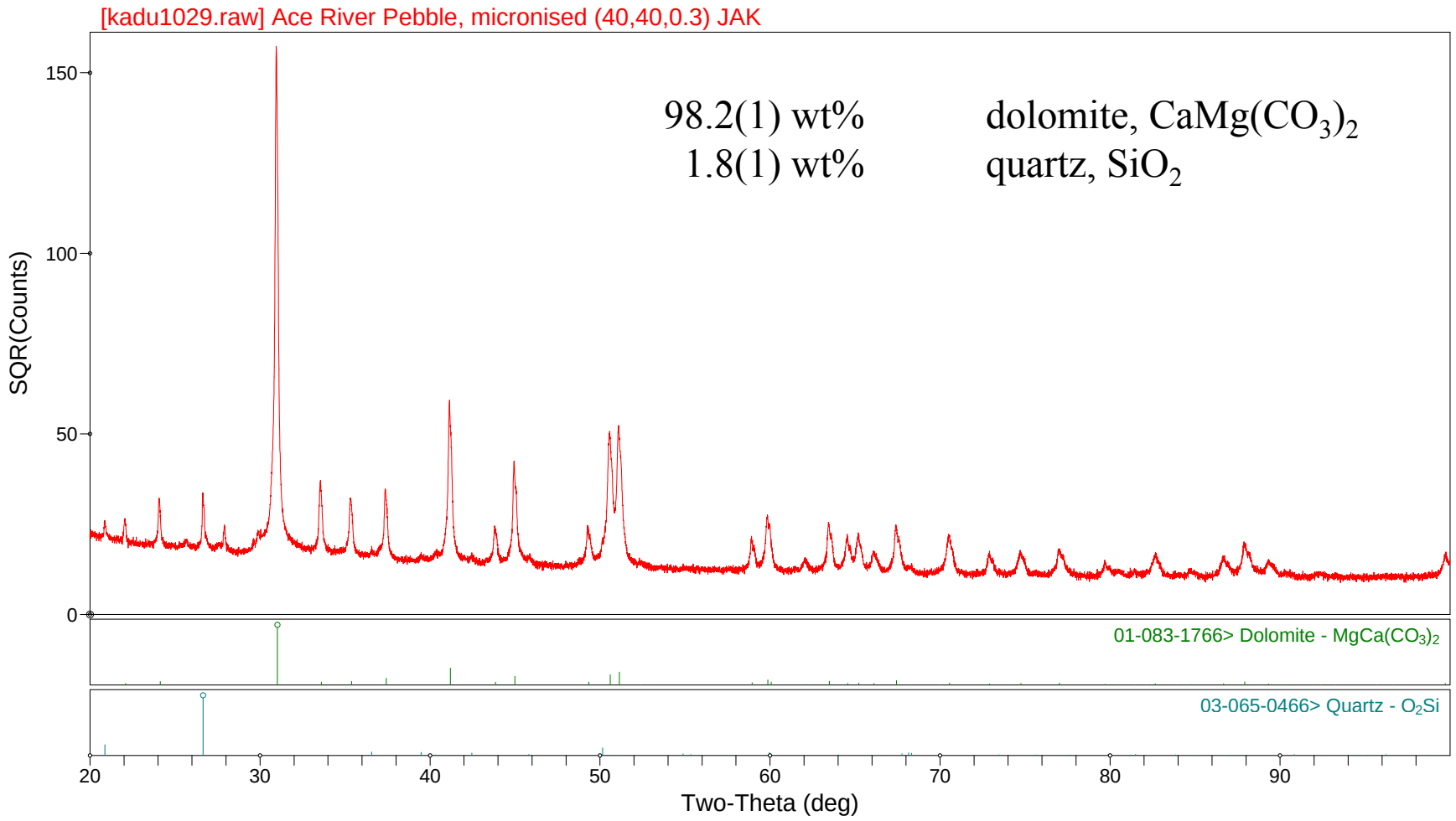

[kadu1051.raw] Black Dragon Beach rock, micronised (40,40,0.3) JAK
Log(I)
4.0
3.0
00-046-1045> Quartz - SiO2
01-070-3751> Sanidine - K(AlSi3O8)
01-089-6426> Albite - Na(AlSi3O8)
01-084-1305> KAl3Si3O10(OH)2 - Potassium Aluminum Silicate Hydroxide
00-046-1212> Corundum - Al2O3
10
20
30
40
50
60
70
80
90
Two-Theta (deg)

Quantitative Phase Analysis

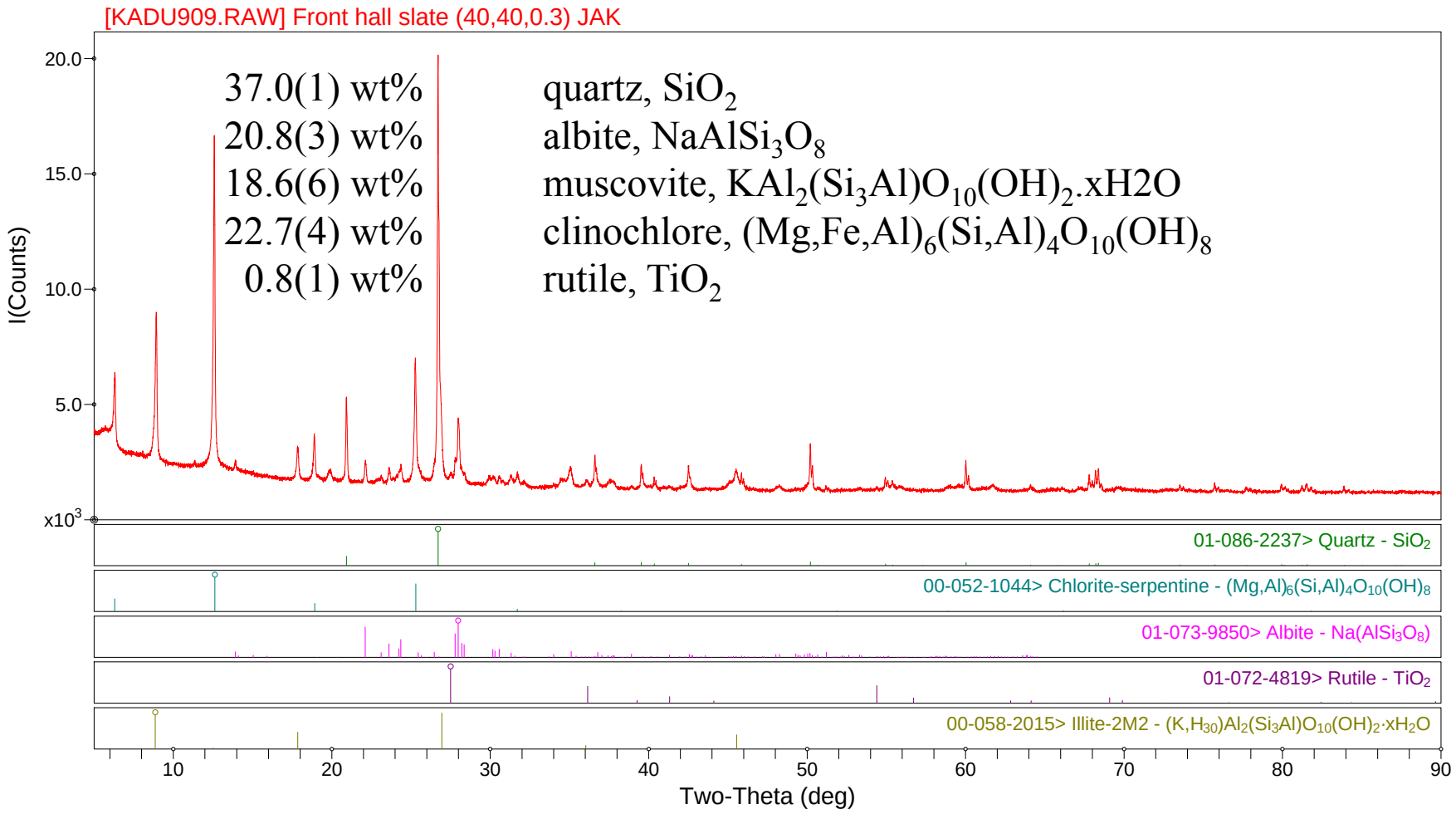
Black Beach Rock from Black Dragon Bay, Zhujiajian Island

20.8(1) wt%	quartz, SiO_2
28.0(1) wt%	orthoclase/sanidine, $KAlSi_3O_8$
40.2(1) wt%	albite, $NaAlSi_3O_8$
10.9(1) wt%	muscovite-2M1, $KAl_3Si_3O_{10}(OH)_2$, or related mica

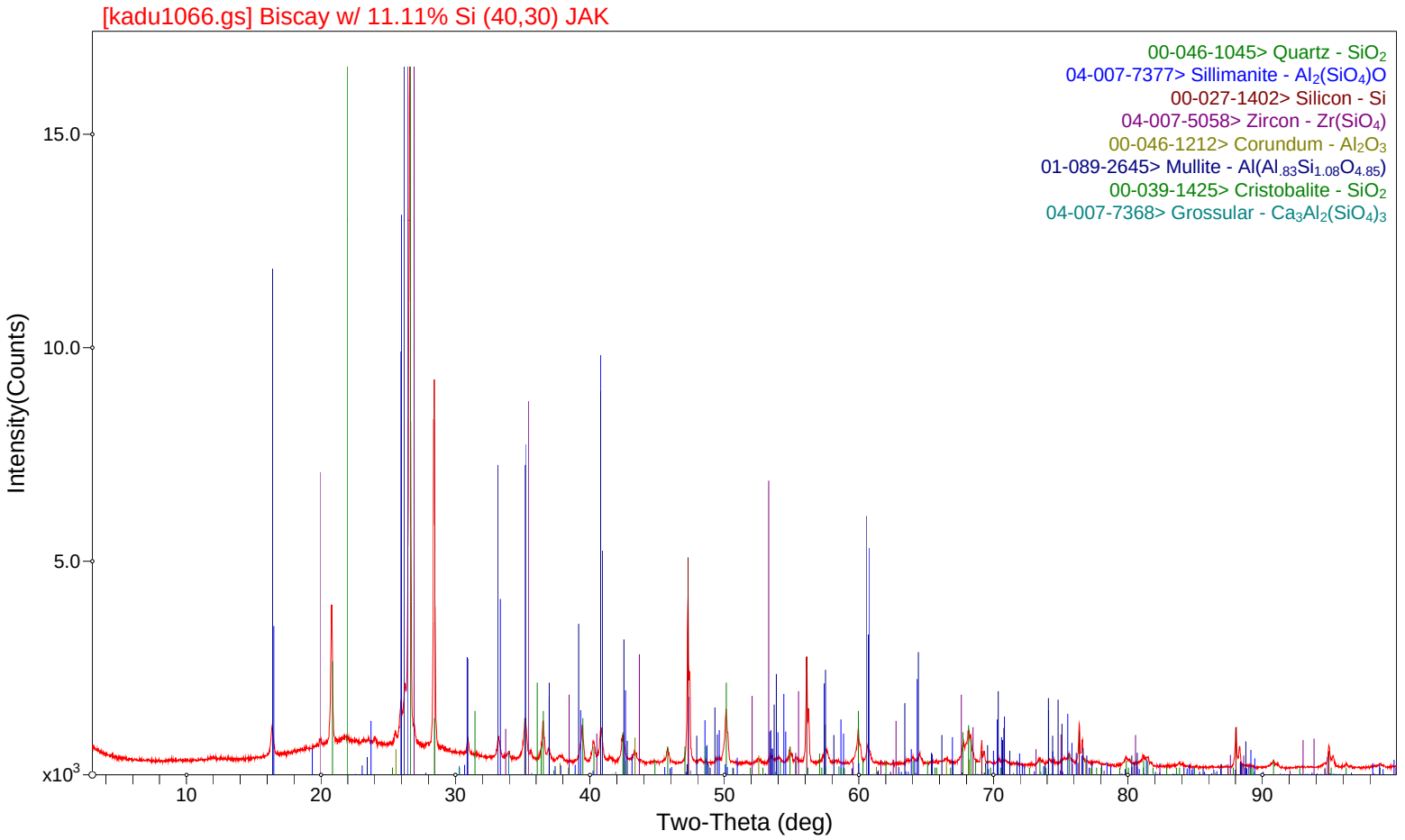
Ace River Pebble

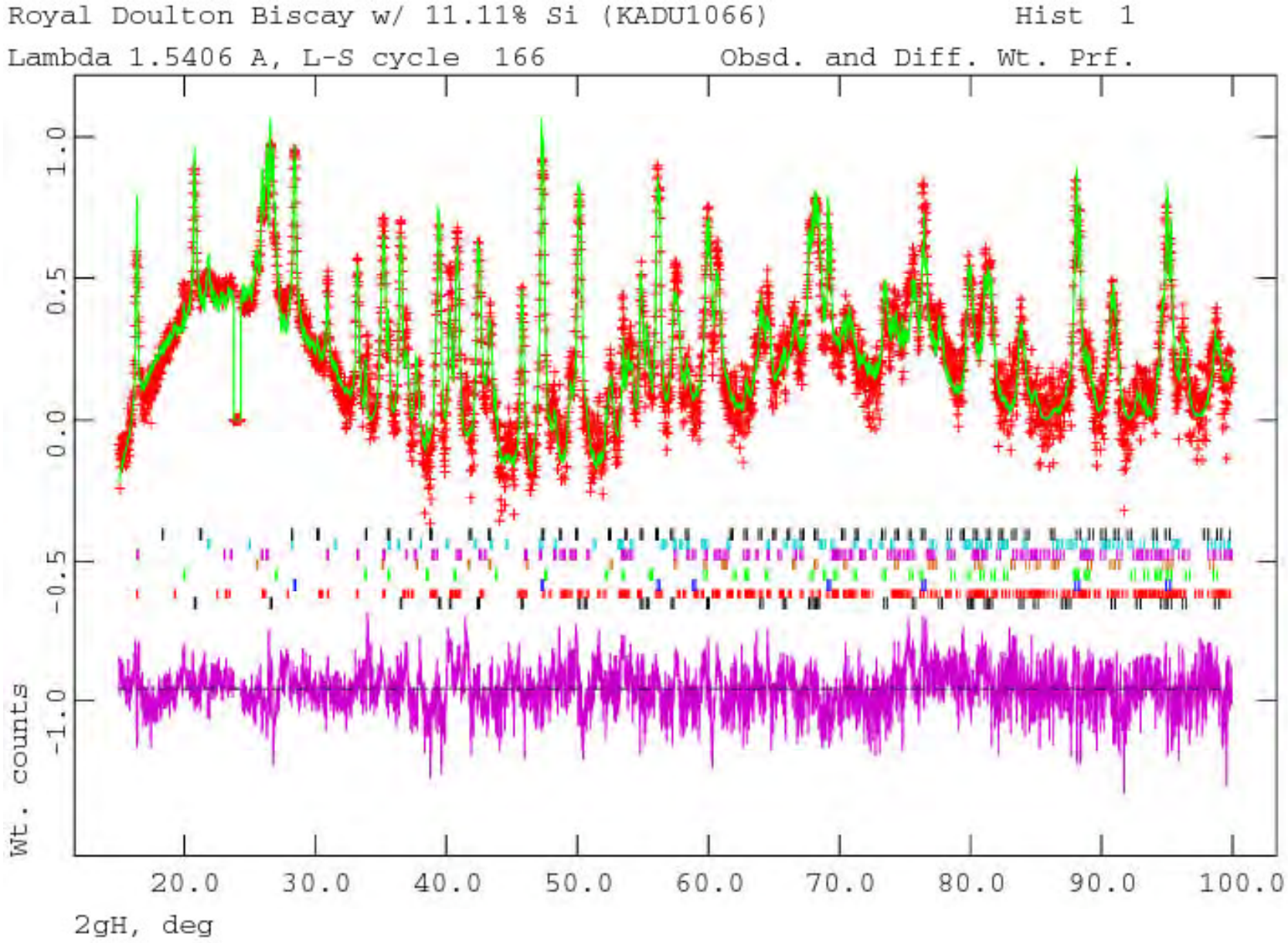


Front Hall Slate



A Broken Soup Bowl

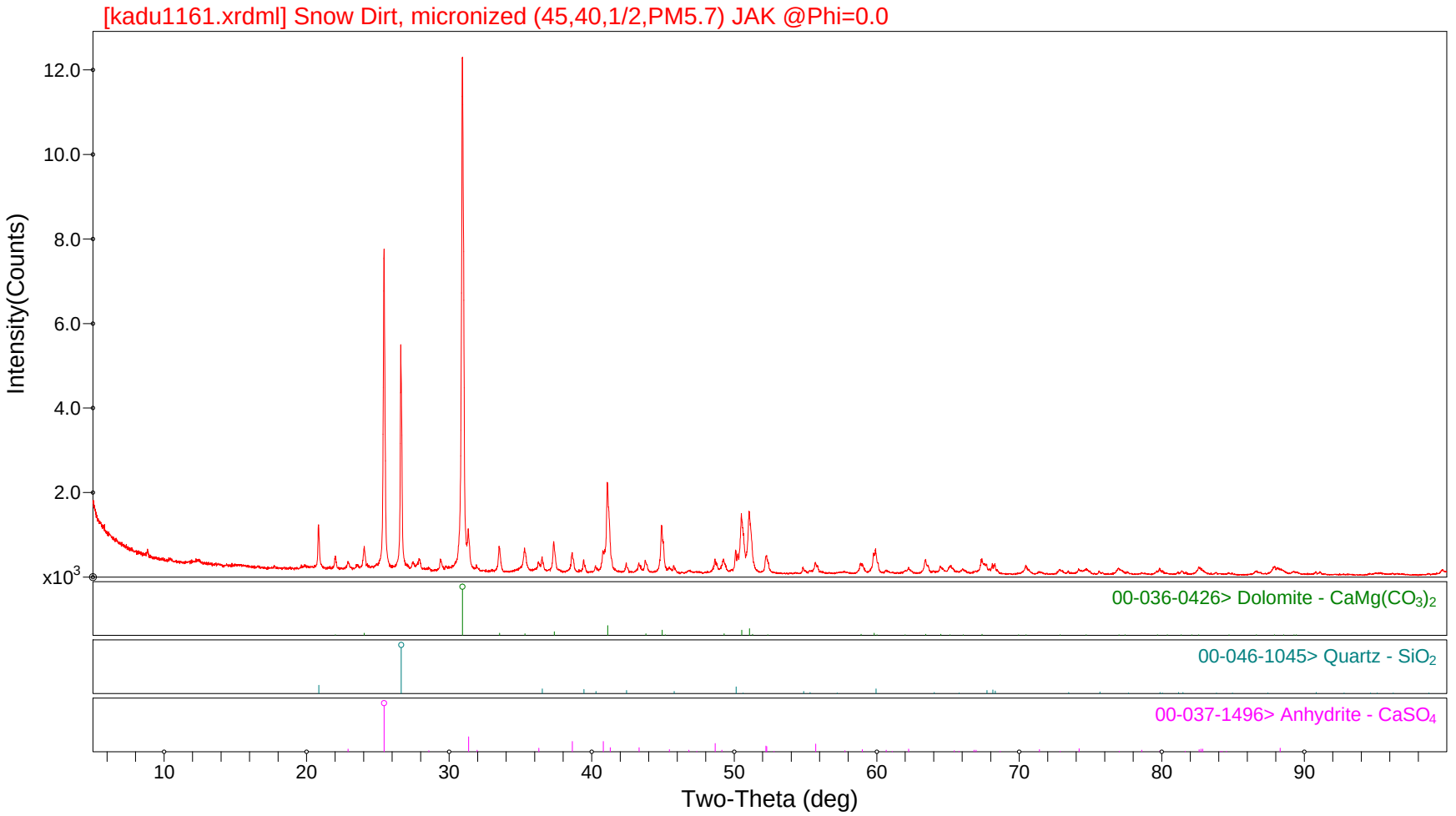


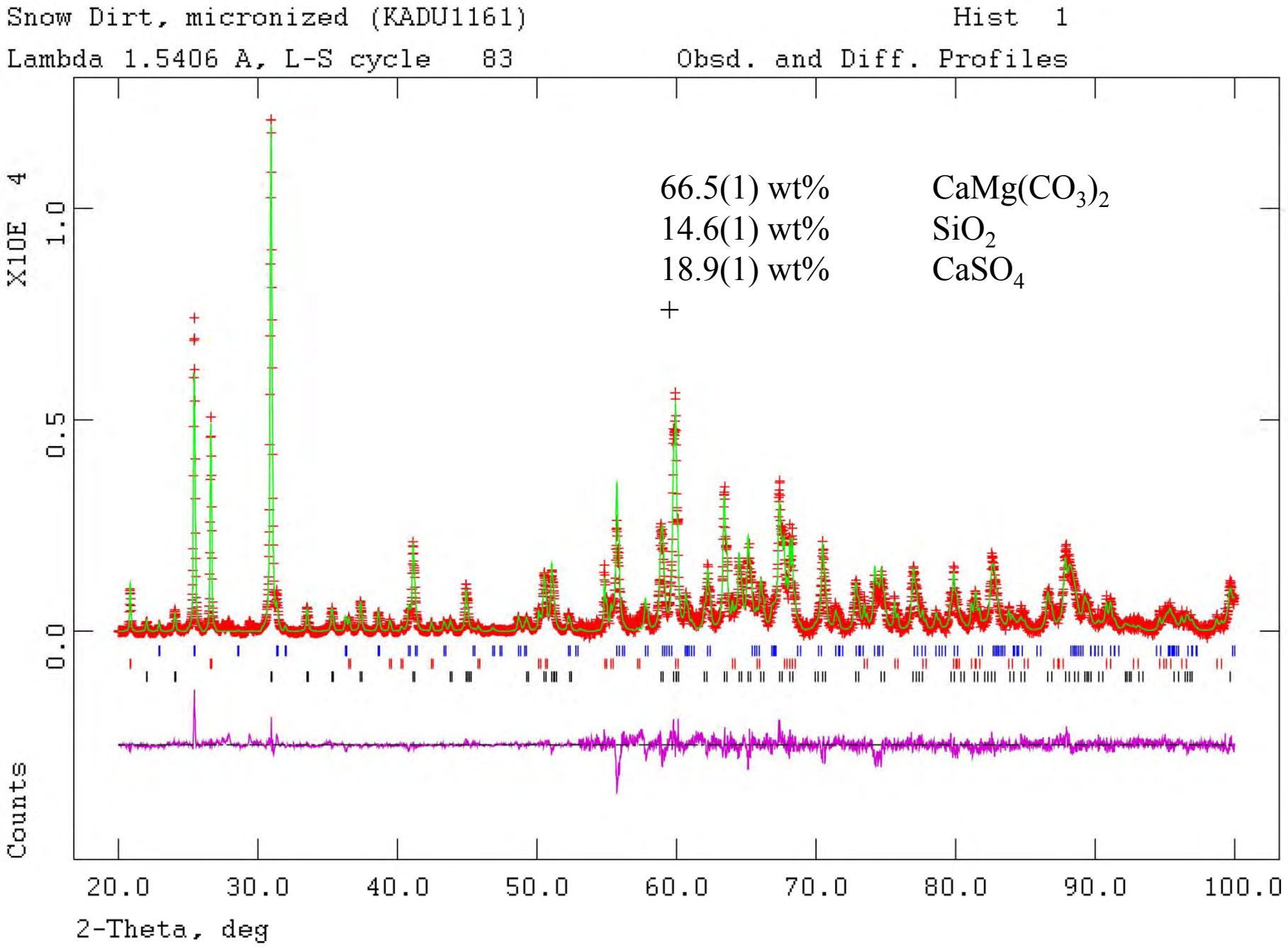

Royal Doulton Biscay w/ 11.11% Si (KADU1066) Hist 1
Lambda 1.5406 A, L-S cycle 166 Obsd. and Diff. Wt. Prf.
1.0
0.5
0.0
-0.5
-1.0
Wt. counts
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
2gH, deg

Stoneware QPA

Name	Formula	Wt%
Quartz	SiO_2	33.5(2)
Cristobalite	SiO_2	0.98(9)
Mullite	$Al_2(Al_{2.5}Si_{1.5})O_{9.75}$	16.8(2)
Sillimanite	Al_2SiO_5	2.6(5)
Zircon	$ZrSiO_4$	0.31(3)
Grossular	$Ca_3Al_2(SiO_4)_3$	0.22(6)
Glass		45.6

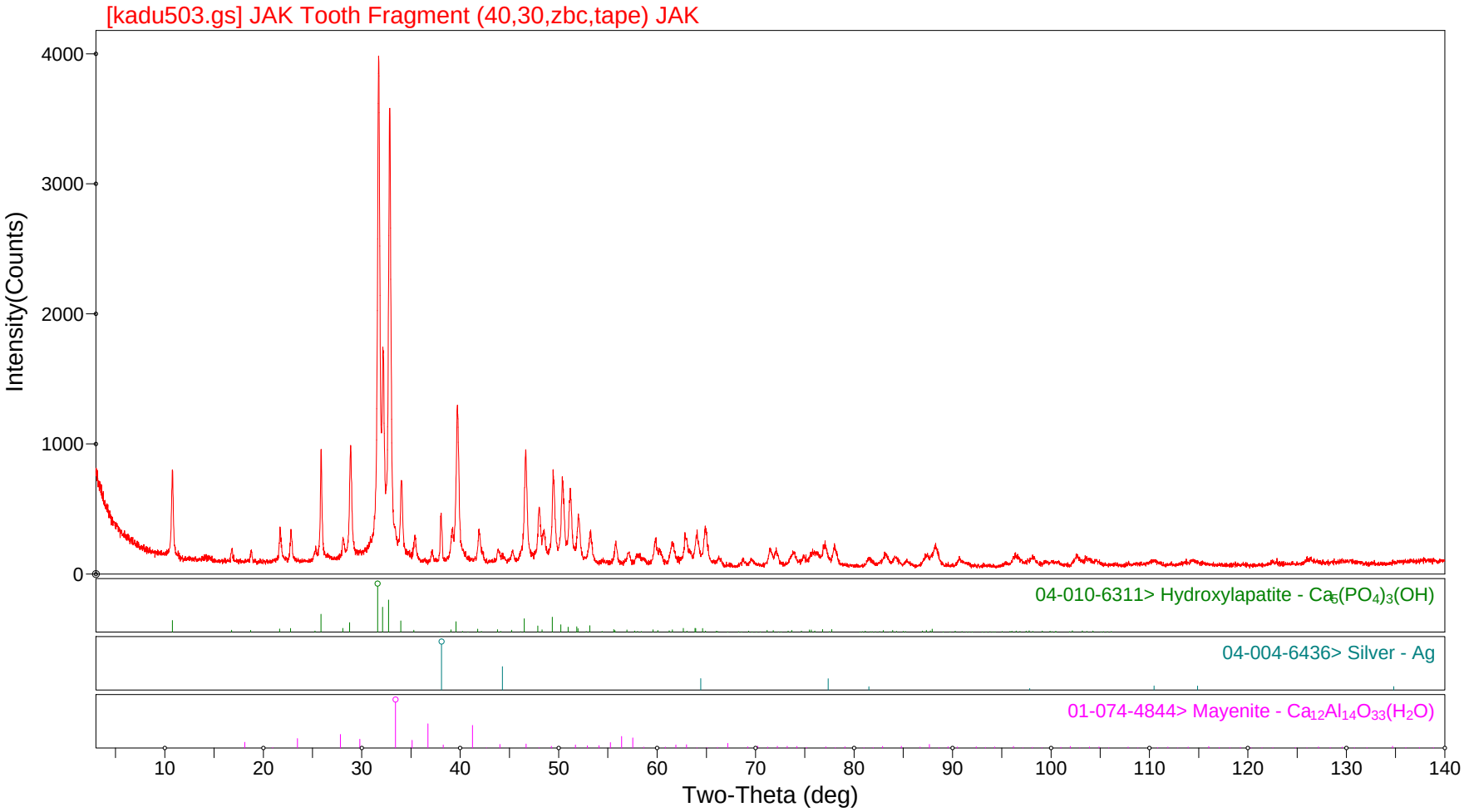
Snow Dirt

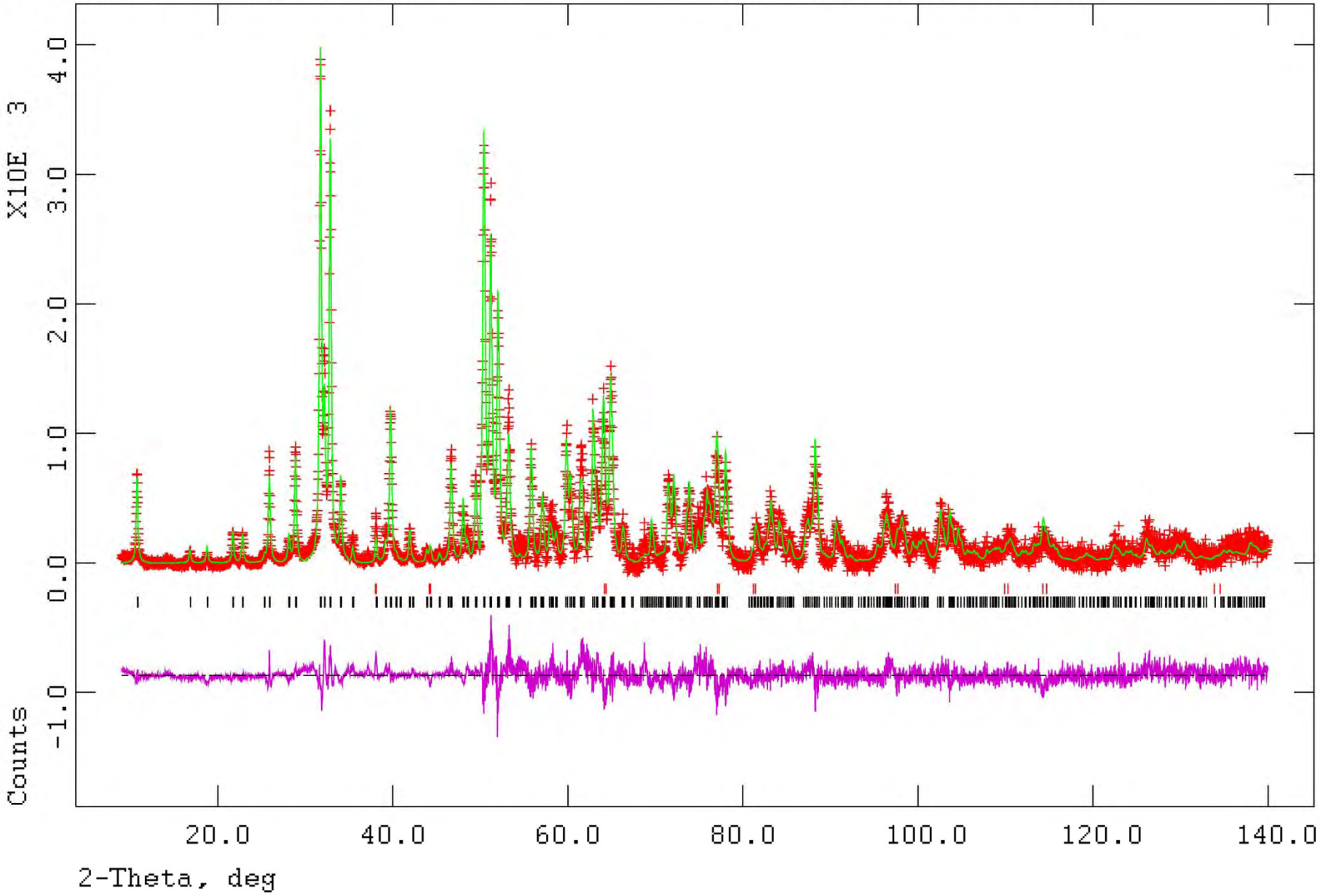

[kadu1161.xrdml] Snow Dirt, micronized (45,40,1/2,PM5.7) JAK @Phi=0.0
Intensity(Counts)
12.0
10.0
8.0
6.0
4.0
2.0
x10^3
00-036-0426> Dolomite - CaMg(CO3)2
00-046-1045> Quartz - SiO2
00-037-1496> Anhydrite - CaSO4
10
20
30
40
50
60
70
80
90
Two-Theta (deg)



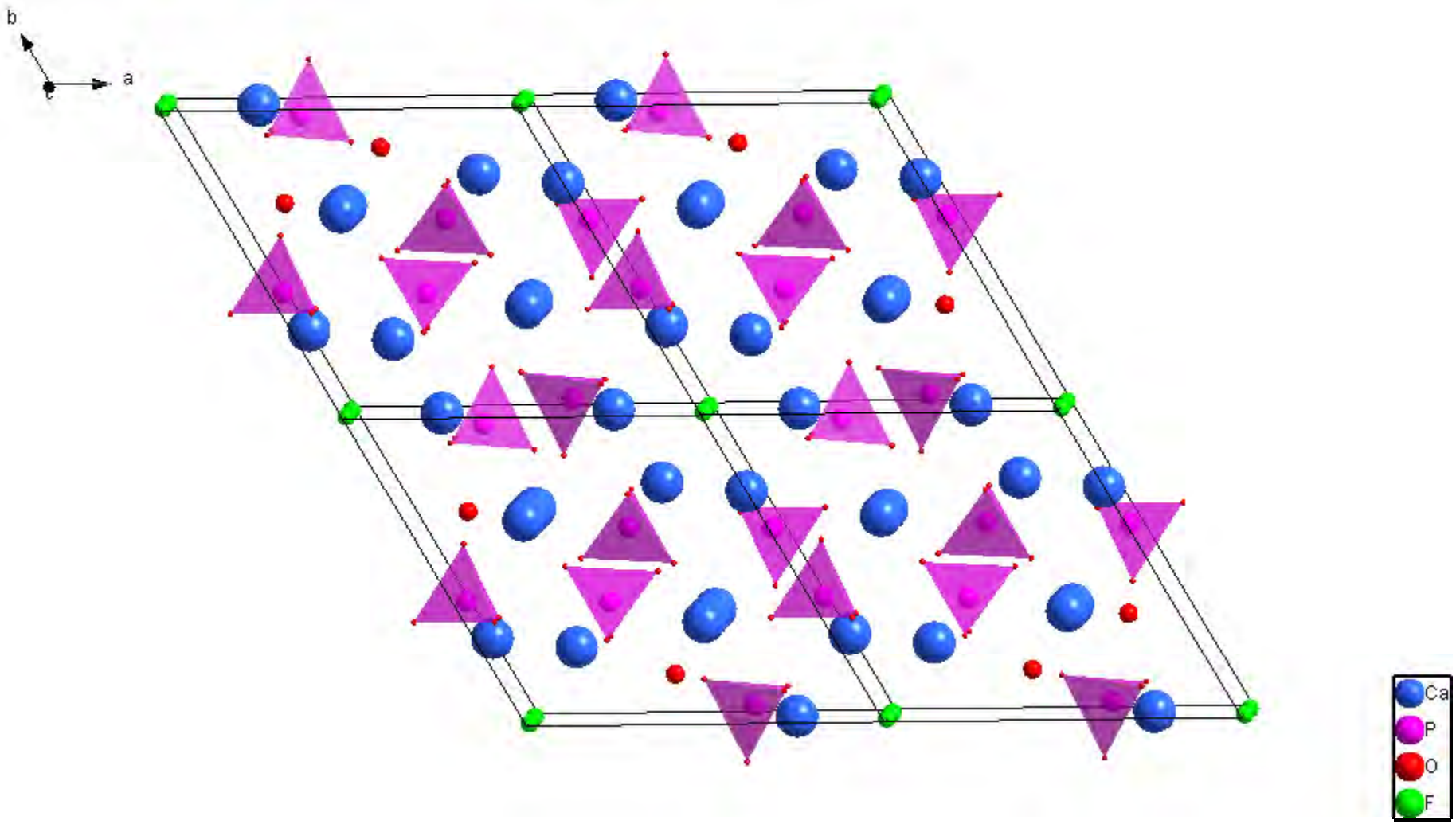
Scaling: 53.0(10.0X)

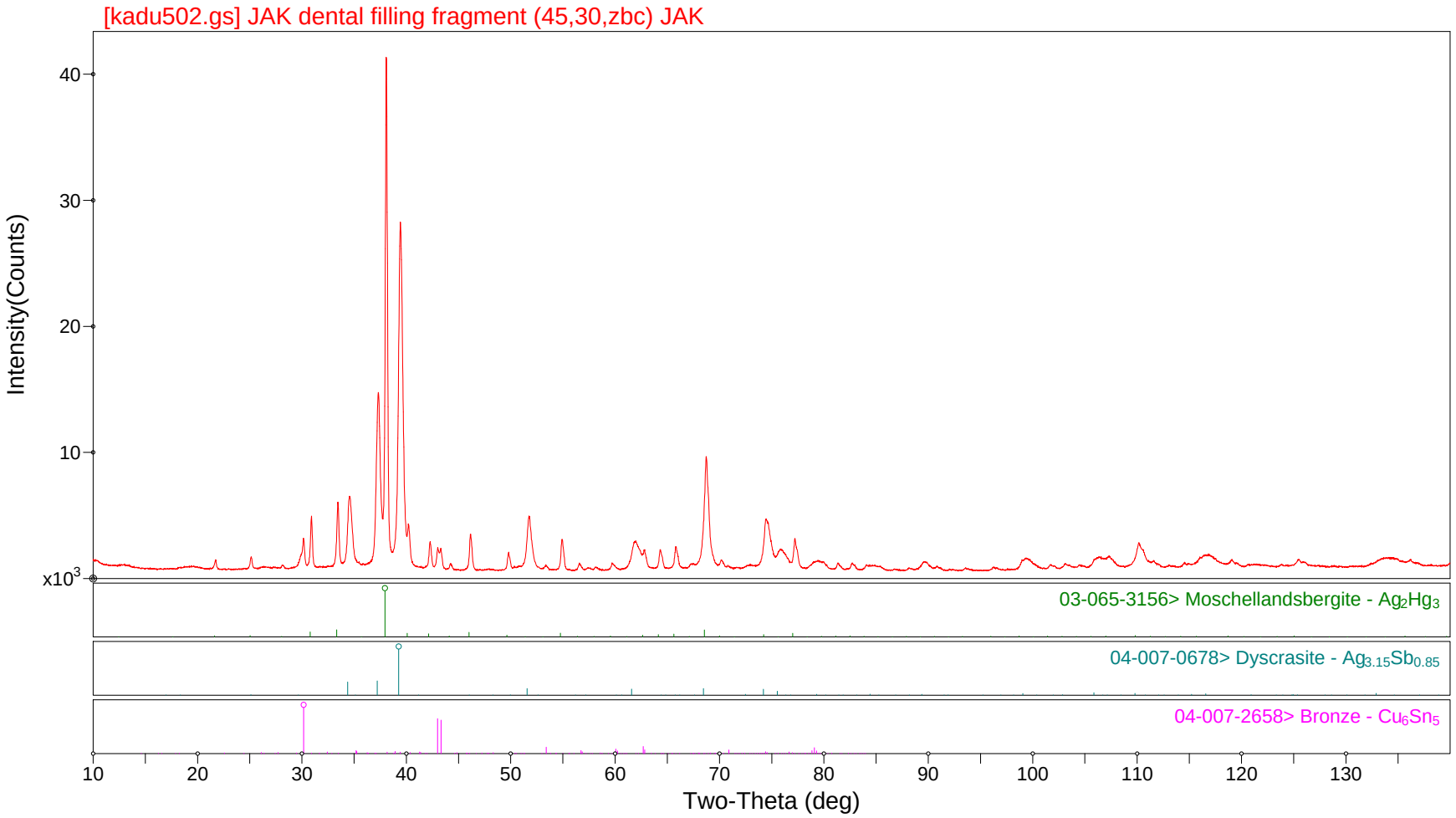
Tooth and Filling

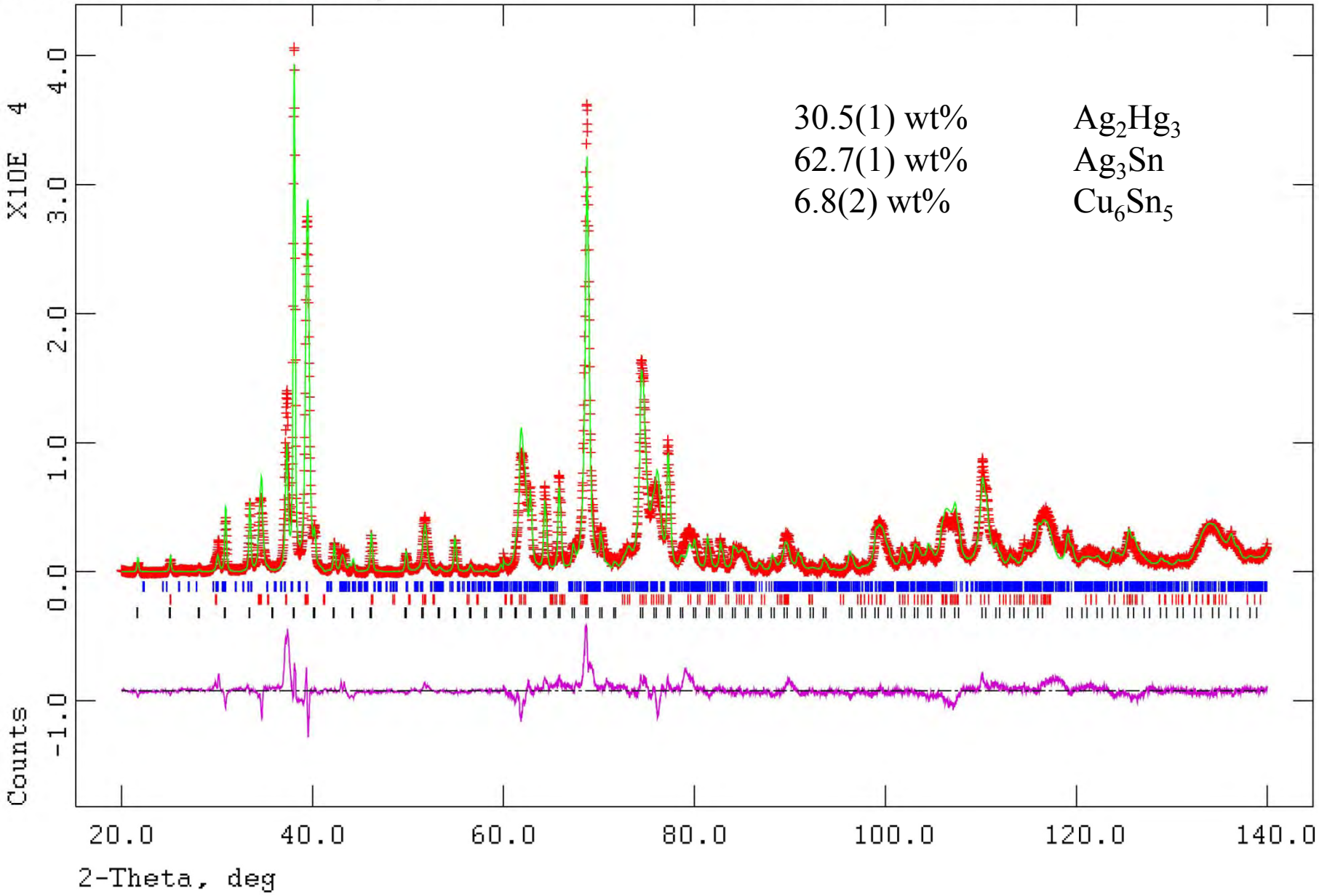

[kadu503.gs] JAK Tooth Fragment (40,30,zbc,tape) JAK
Intensity(Counts)
4000
3000
2000
1000
0
04-010-6311> Hydroxylapatite - Ca5(PO4)3(OH)
04-004-6436> Silver - Ag
01-074-4844> Mayenite - Ca12Al14O33(H2O)
10
20
30
40
50
60
70
80
90
100
110
120
130
140
Two-Theta (deg)



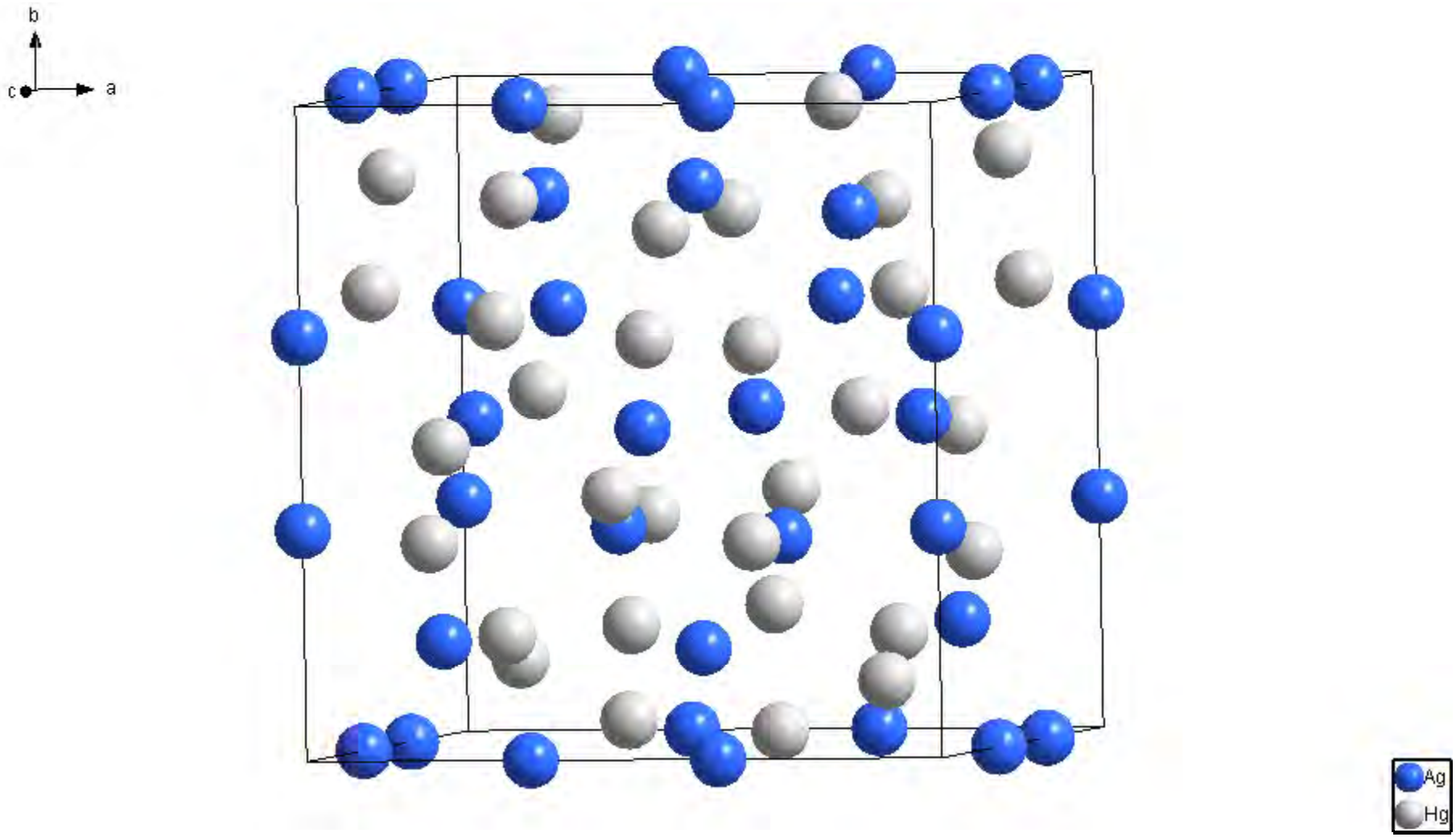
Scaling: 50.0(5.0X)

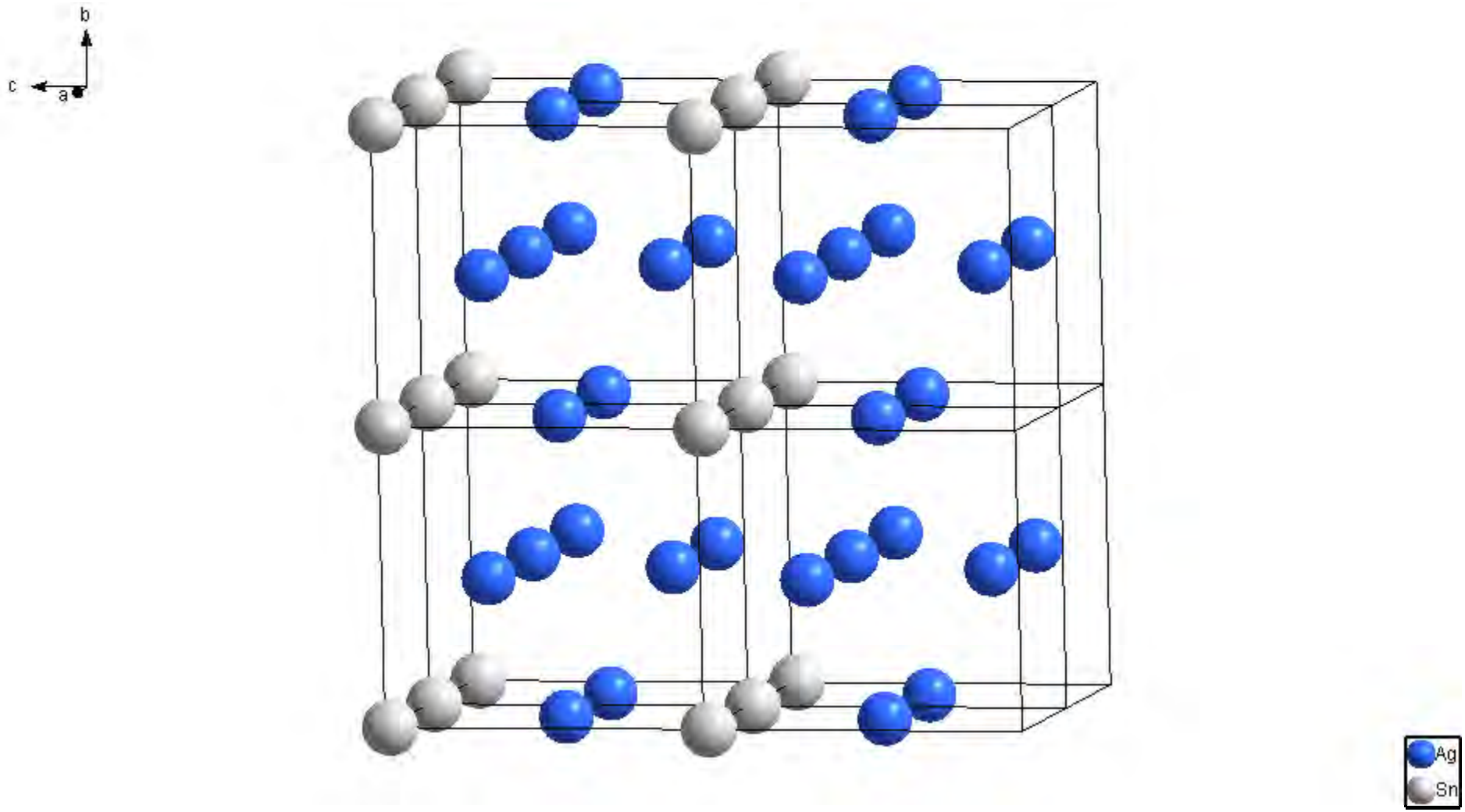

b
a
c
Ca
P
O
F

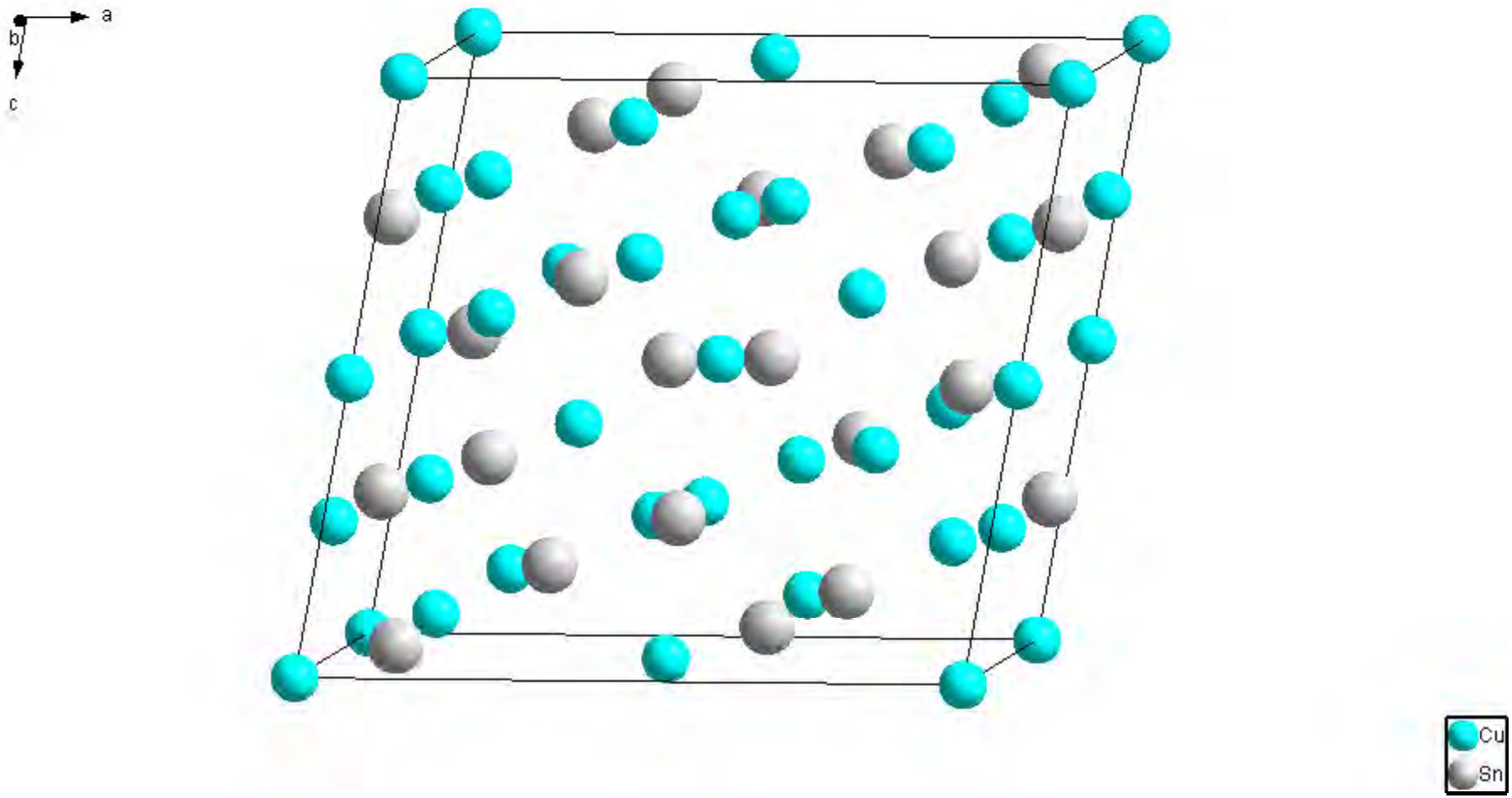

[kadu502.gs] JAK dental filling fragment (45,30,zbc) JAK
Intensity(Counts)
x10^3
40
30
20
10
03-065-3156> Moschellandsbergite - Ag2Hg3
04-007-0678> Dyscrasite - Ag3.15Sb0.85
04-007-2658> Bronze - Cu6Sn5
10
20
30
40
50
60
70
80
90
100
110
120
130
Two-Theta (deg)



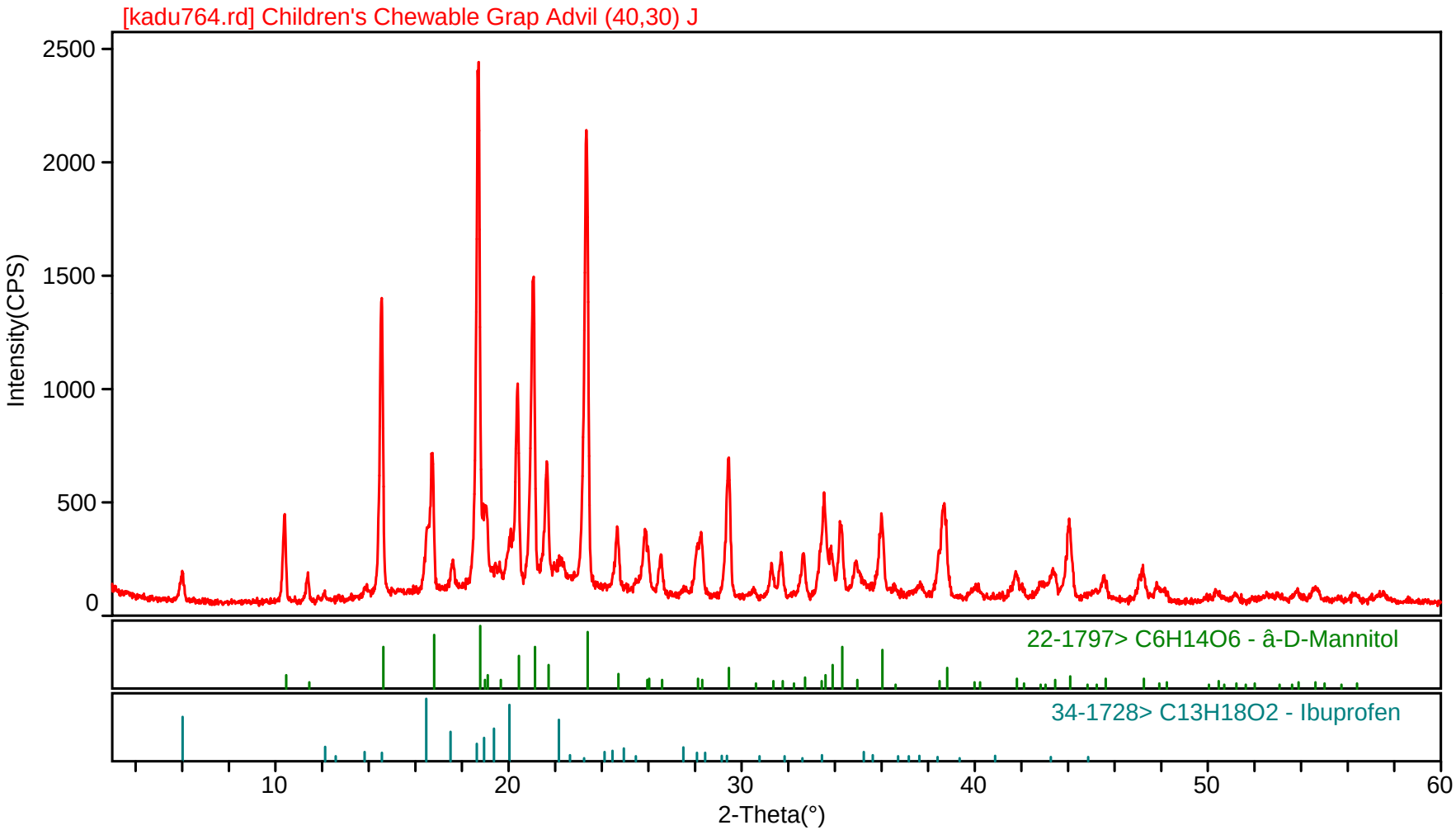
Scaling: 60.0(4.0X)

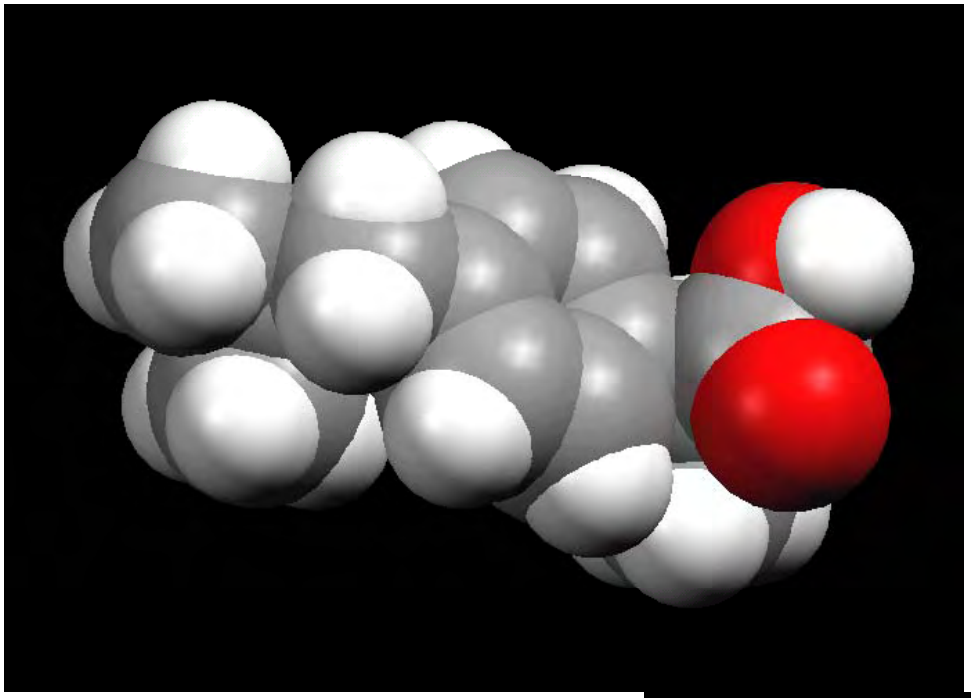

b
c
a
Ag
Hg


b
c
a
Ag
Sn


a
b
c
Cu
Sn

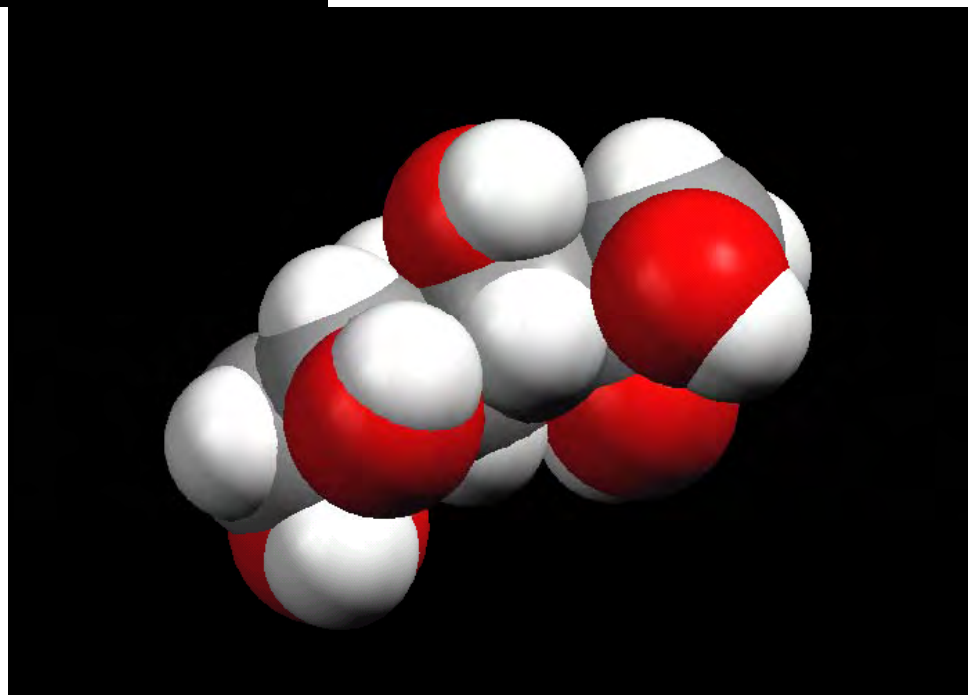
Childrens' Grape Advil

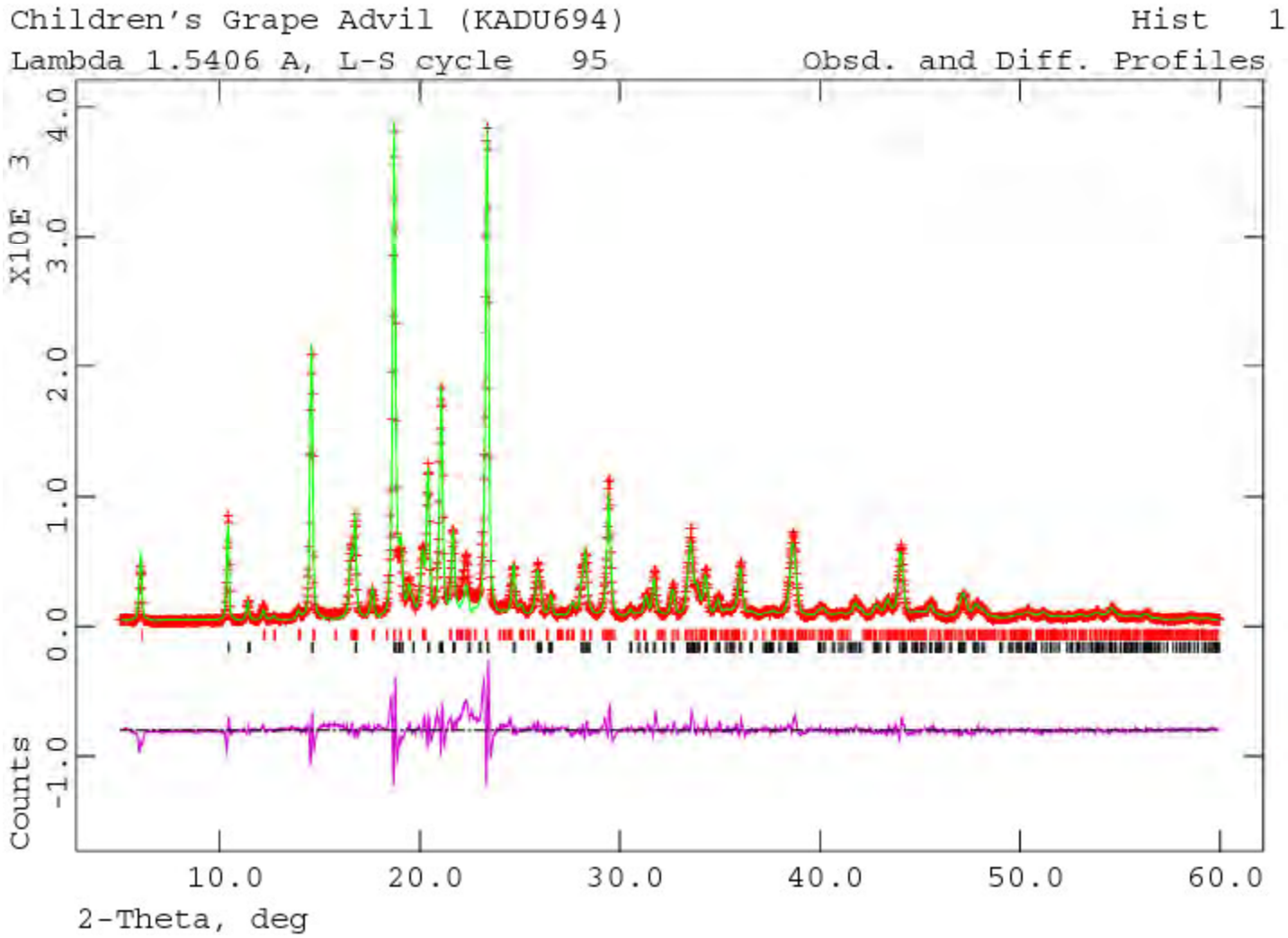

[kadu764.rd] Children's Chewable Grap Advil (40,30) J
Intensity(CPS)
2500
2000
1500
1000
500
0
22-1797> C6H14O6 - â-D-Mannitol
34-1728> C13H18O2 - Ibuprofen
10
20
30
40
50
60
2-Theta(°)



Ibuprofen

β-D-mannitol




Children's Grape Advil (KADU694)
Hist 1
Lambda 1.5406 A, L-S cycle 95
Obsd. and Diff. Profiles
X10E 3
4.0
3.0
2.0
1.0
0.0
-1.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
2-Theta, deg

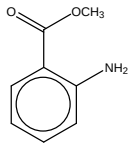
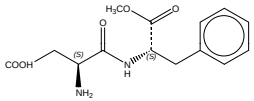
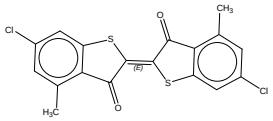
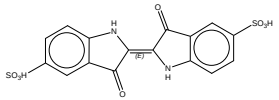
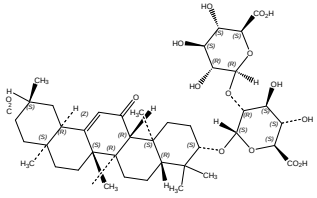
Concentrations

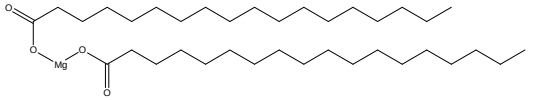
Observed	Expected
• Ibuprofen = 17.3	• 13.07 wt%
• Mannitol = 82.7	• ?

Concentrations

- Ibuprofen = 13.5(2) wt% (13.07)
- Mannitol = 69.6(2) wt%
- Cellulose = 16.8(6) wt%
- Plus small concentrations of many other compounds!

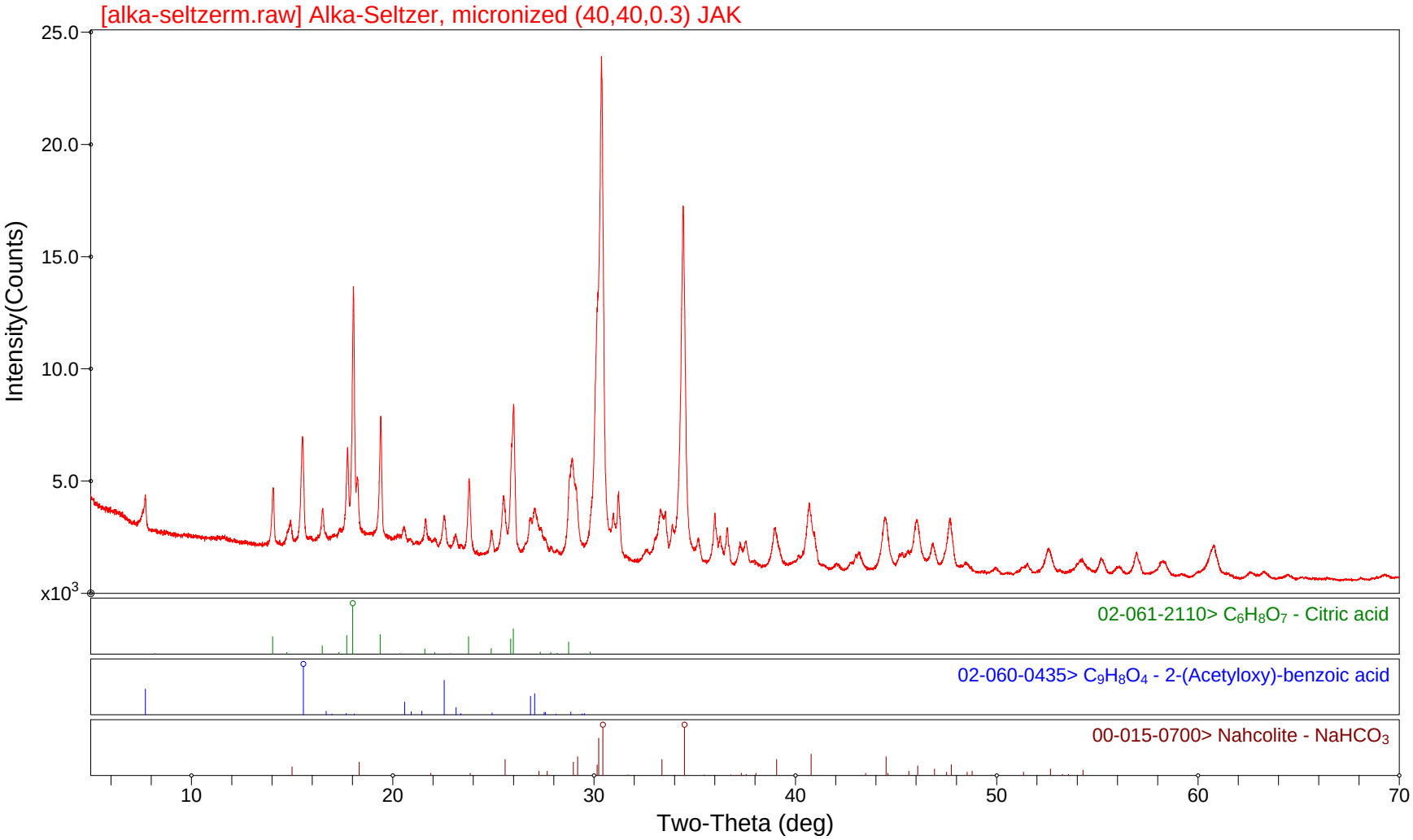
Children's Grape Advil

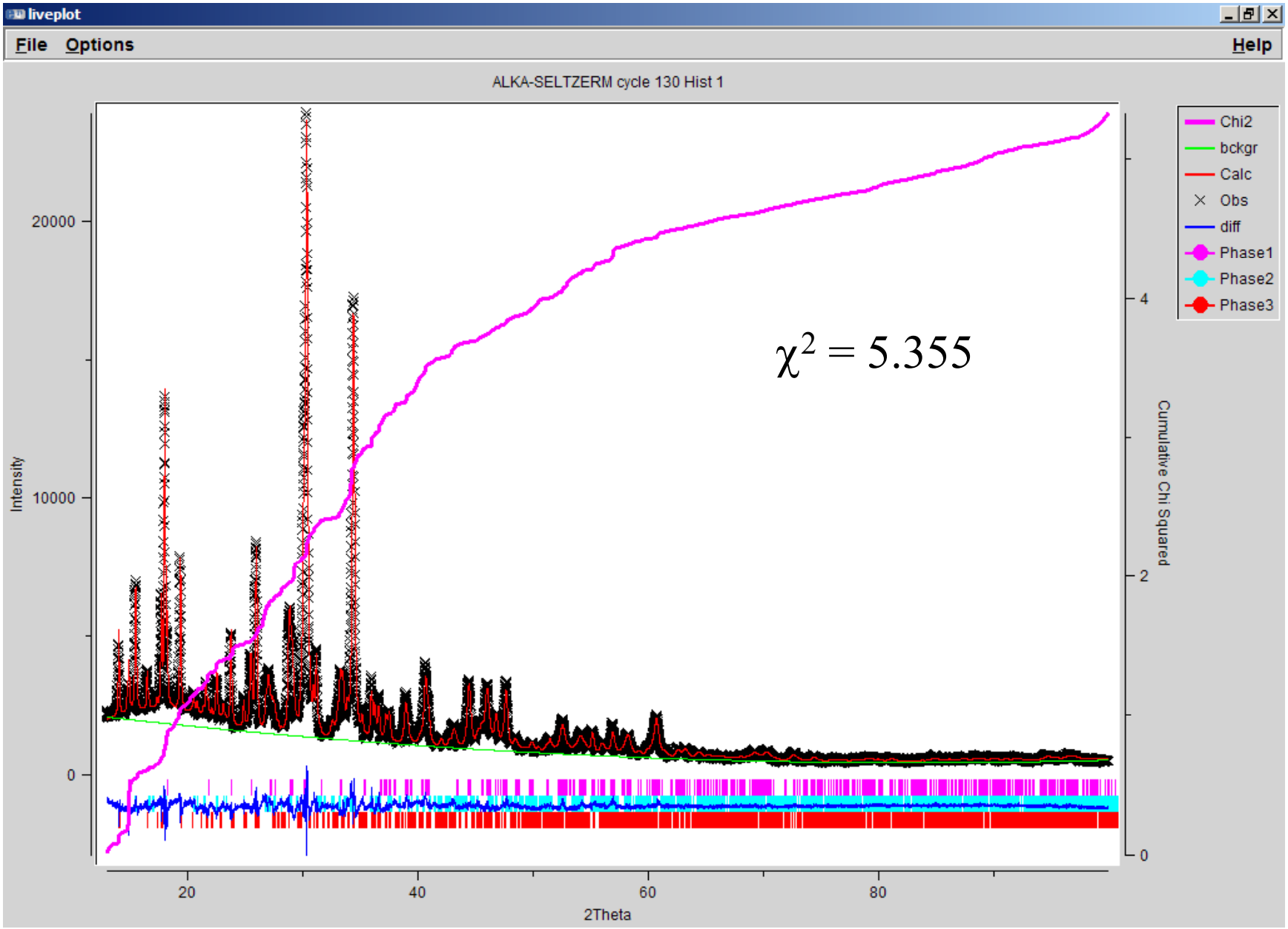
- Ibuprofen 
- Artificial flavor
- Aspartame 
- Cellulose acetate phthalate 
- D&C Red #30 lake
- FD&C Blue #2 lake
- Gelatin 
- Magnasweet 



- Magnesium stearate
- Mannitol
- Microcrystalline cellulose
- Silicon dioxide
- Sodium starch glycolate

Alka-Seltzer


[alka-seltzerm.raw] Alka-Seltzer, micronized (40,40,0.3) JAK
Intensity(Counts)
25.0
20.0
15.0
10.0
5.0
x10^3
02-061-2110> C6H8O7 - Citric acid
02-060-0435> C9H8O4 - 2-(Acetyloxy)-benzoic acid
00-015-0700> Nahcolite - NaHCO3
10
20
30
40
50
60
70
Two-Theta (deg)


liveplot
File
Options
Help
ALKA-SELTZERM cycle 130 Hist 1
Chi2
bckgr
Calc
Obs
diff
Phase1
Phase2
Phase3
$\chi^2 = 5.355$
Intensity
20000
10000
0
Cumulative Chi Squared
4
2
0
20
40
60
80
2Theta

Alka-Seltzer Analysis

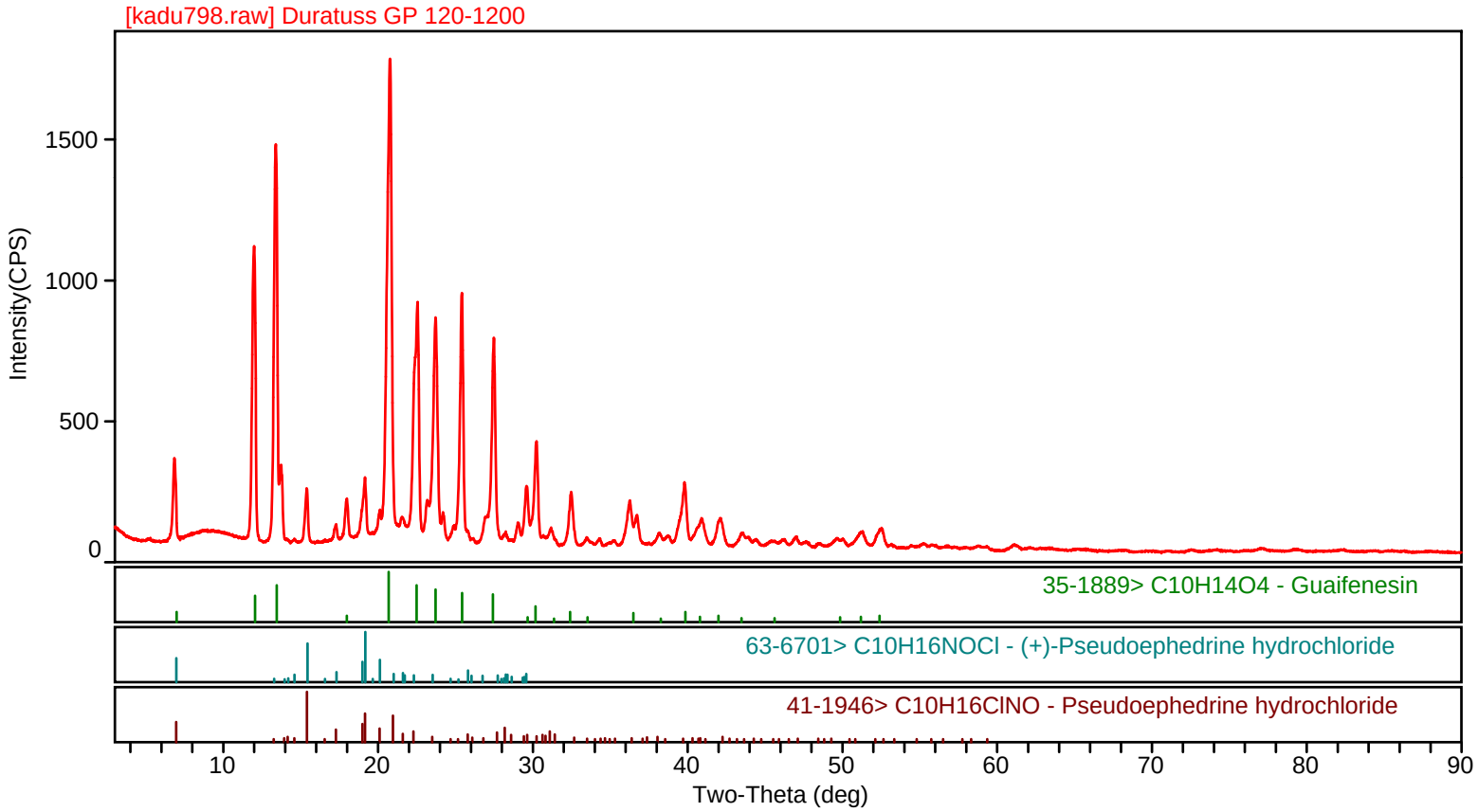
wt%	Expected	Refined
Sodium Bicarbonate	59.12	63.1(1)
Acetylsalicylic Acid	10.03	8.6(1)
Citric Acid	30.86	28.3(1)

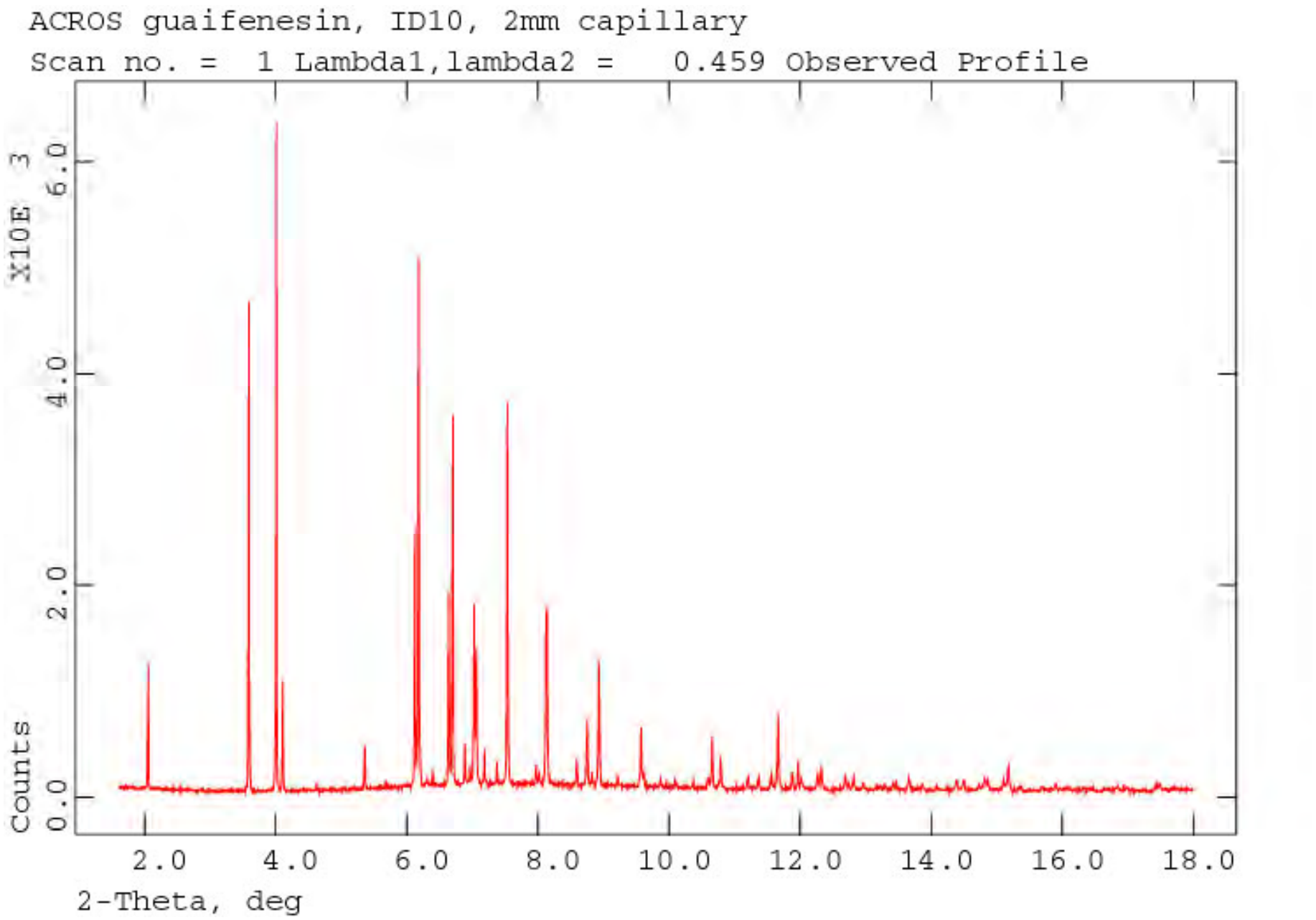
$AKLD = 0.080$

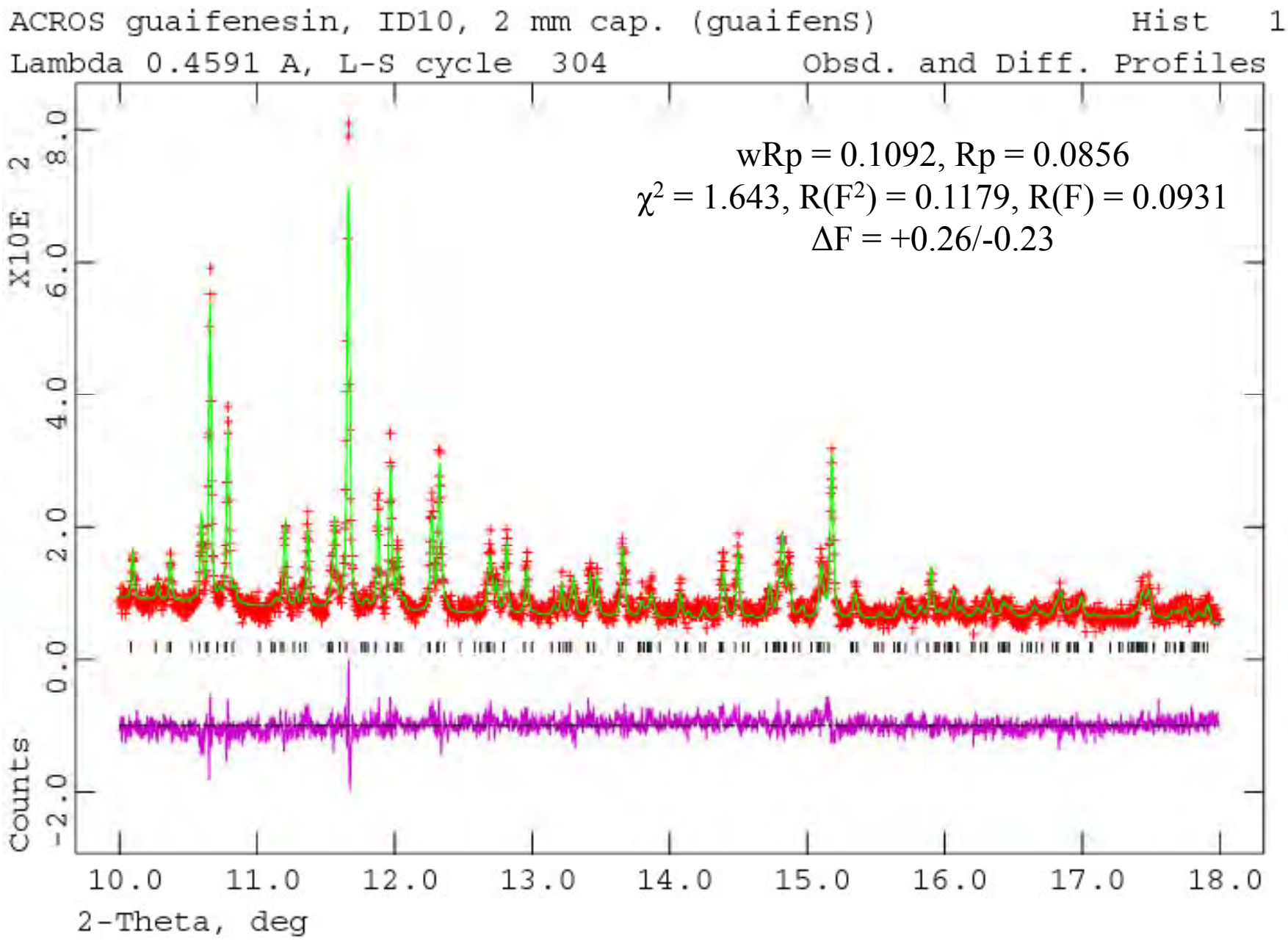
Decongestant

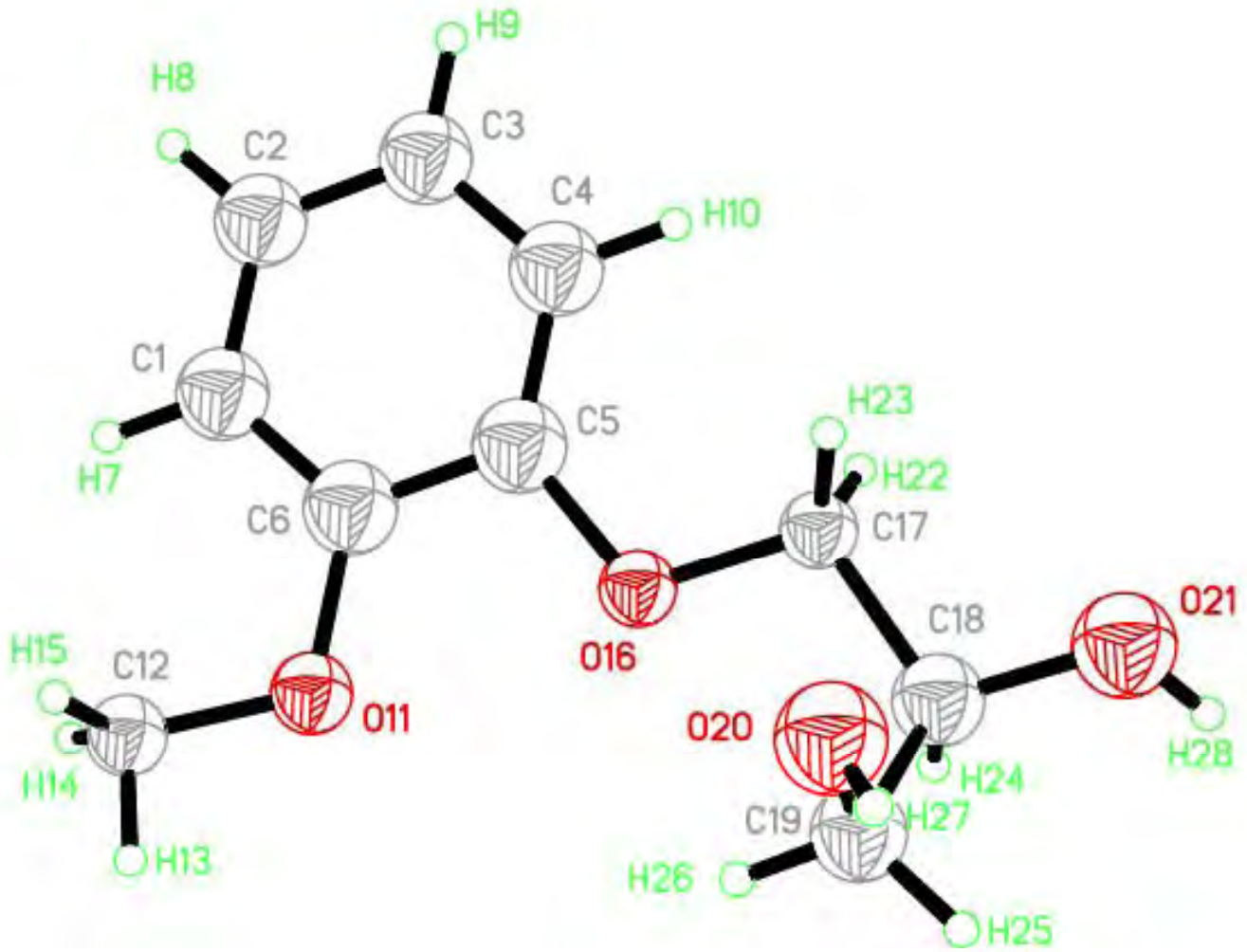
It was a bad trip home
from the conference …

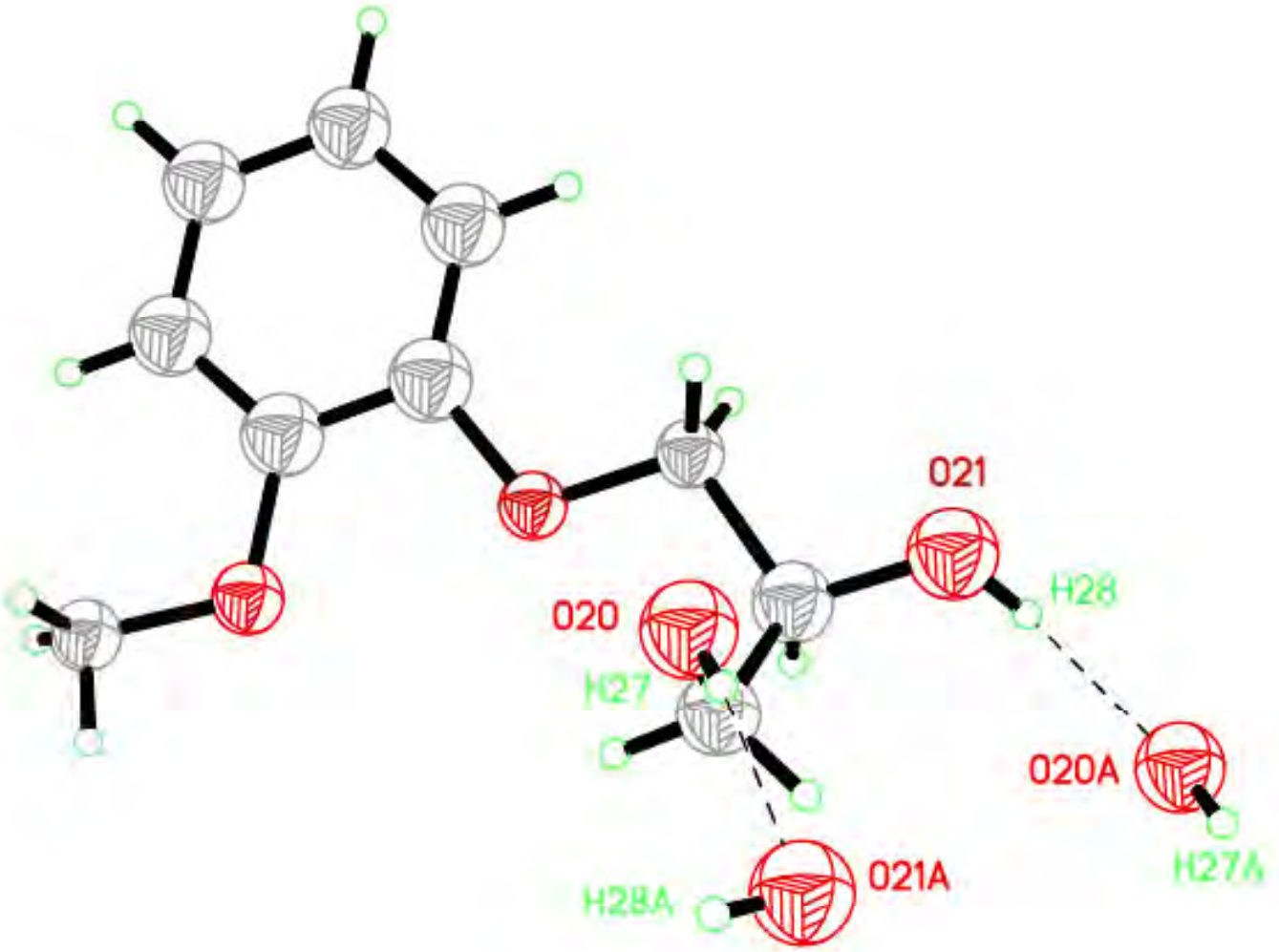
Duratuss GP 120-1200

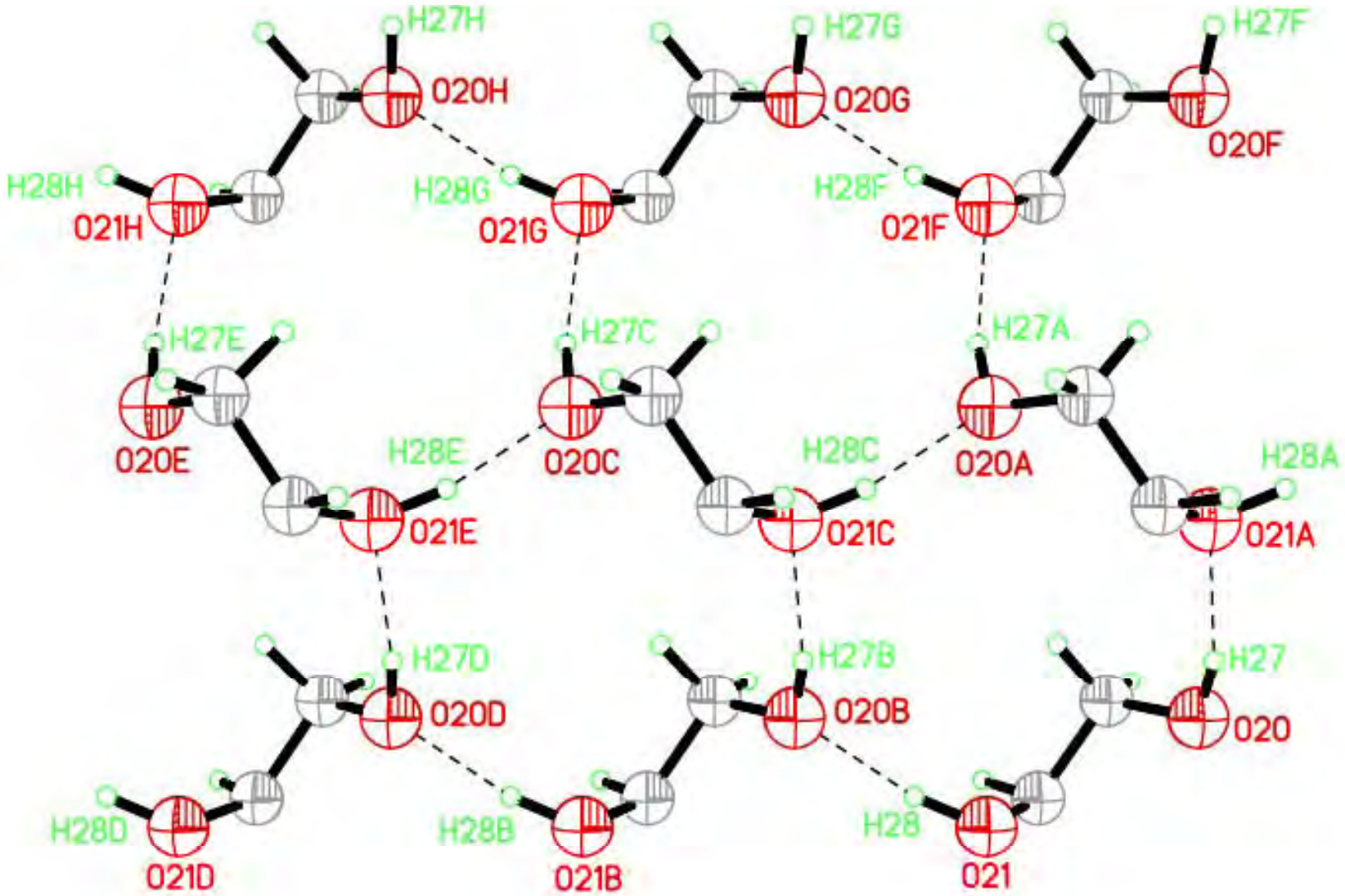



ACROS guaifenesin, ID10, 2mm capillary
Scan no. = 1 Lambda1,lambda2 = 0.459 Observed Profile
X10E 3
6.0
4.0
2.0
0.0
Counts
2.0
4.0
6.0
8.0
10.0
12.0
14.0
16.0
18.0
2-Theta, deg

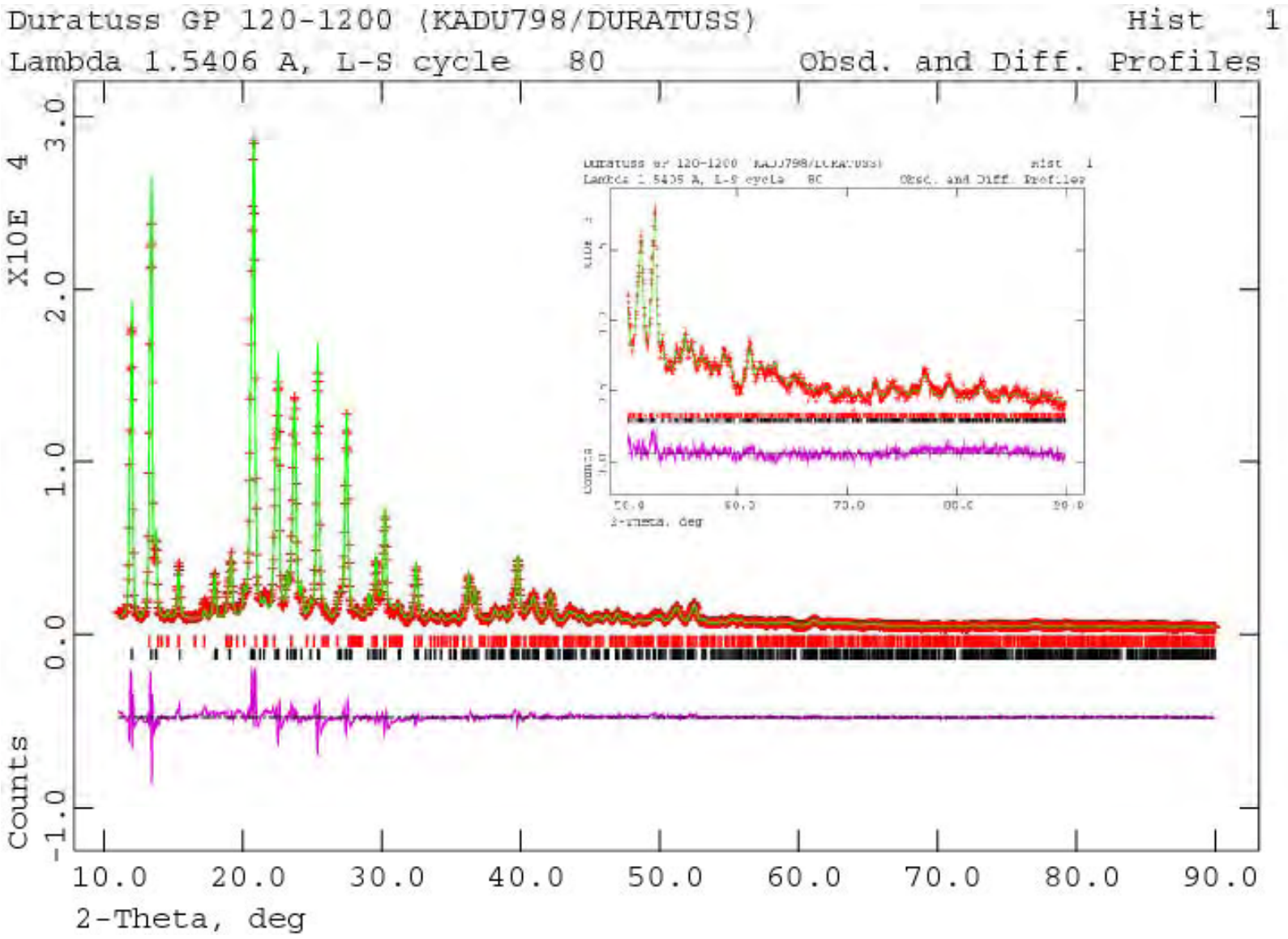

ACROS guaifenesin, ID10, 2 mm cap. (guaifenS) Hist 1
Lambda 0.4591 A, L-S cycle 304 Obsd. and Diff. Profiles
wRp = 0.1092, Rp = 0.0856
χ² = 1.643, R(F²) = 0.1179, R(F) = 0.0931
ΔF = +0.26/-0.23
X10E 2
8.0
6.0
4.0
2.0
0.0
-2.0
Counts
10.0
11.0
12.0
13.0
14.0
15.0
16.0
17.0
18.0
2-Theta, deg


H9
H8
C2
C3
C4
H10
C1
C5
H23
H7
H22
C6
C17
O16
C18
O21
H15
C12
O11
O20
H28
H14
H24
C19
H27
H13
H26
H25


021
H28
020
H27
020A
H27A
021A
H28A


H27H
O20H
H27G
O20G
H27F
O20F
H28H
O21H
H28G
O21G
H28F
O21F
H27E
O20E
H27C
O20C
H27A
O20A
H28E
O21E
H28C
O21C
H28A
O21A
H27D
O20D
H27B
O20B
H27
O20
H28D
O21D
H28B
O21B
H28
O21

Back to Duratuss…

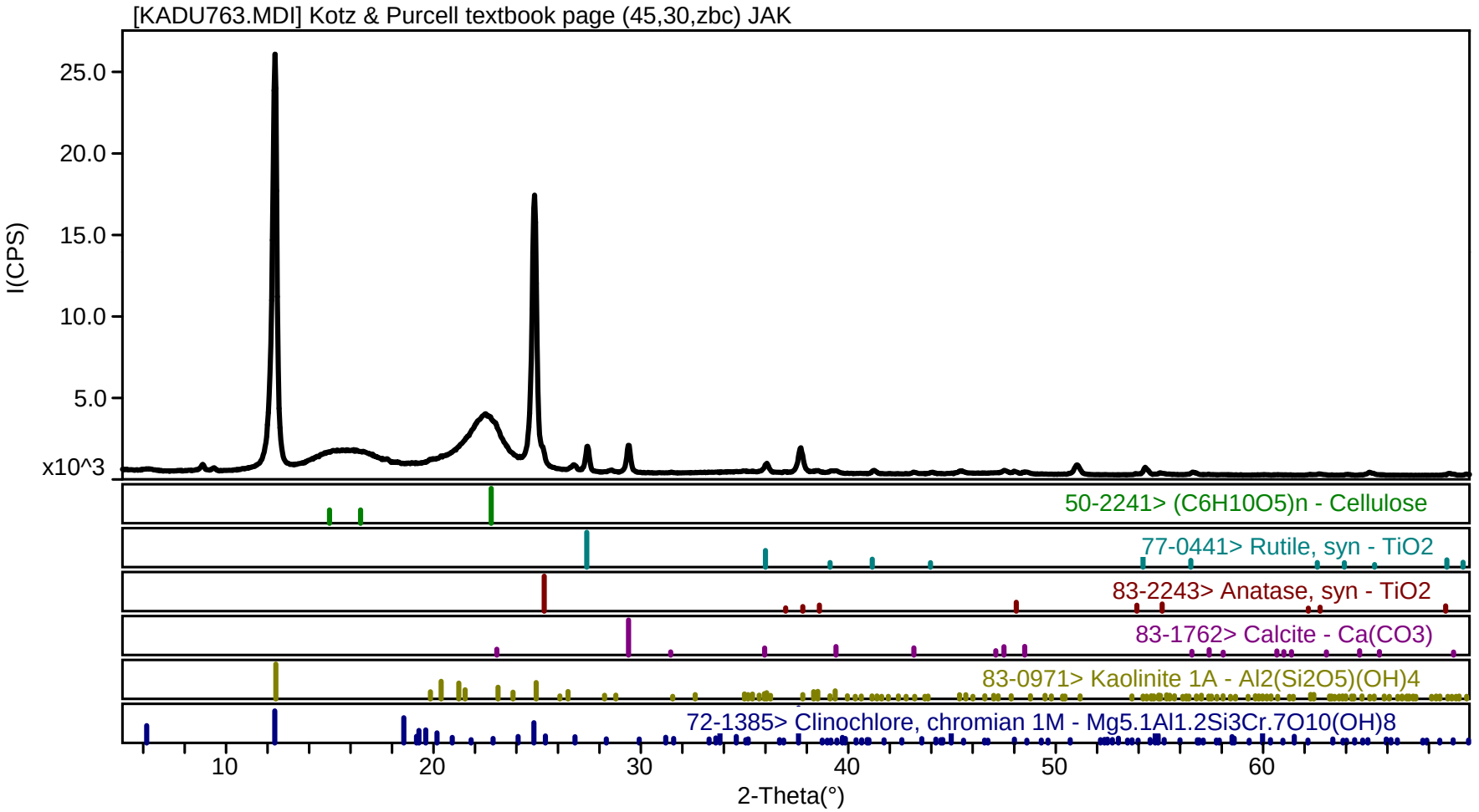


Quantitative Phase Analysis of Duratuss GP 120-1200

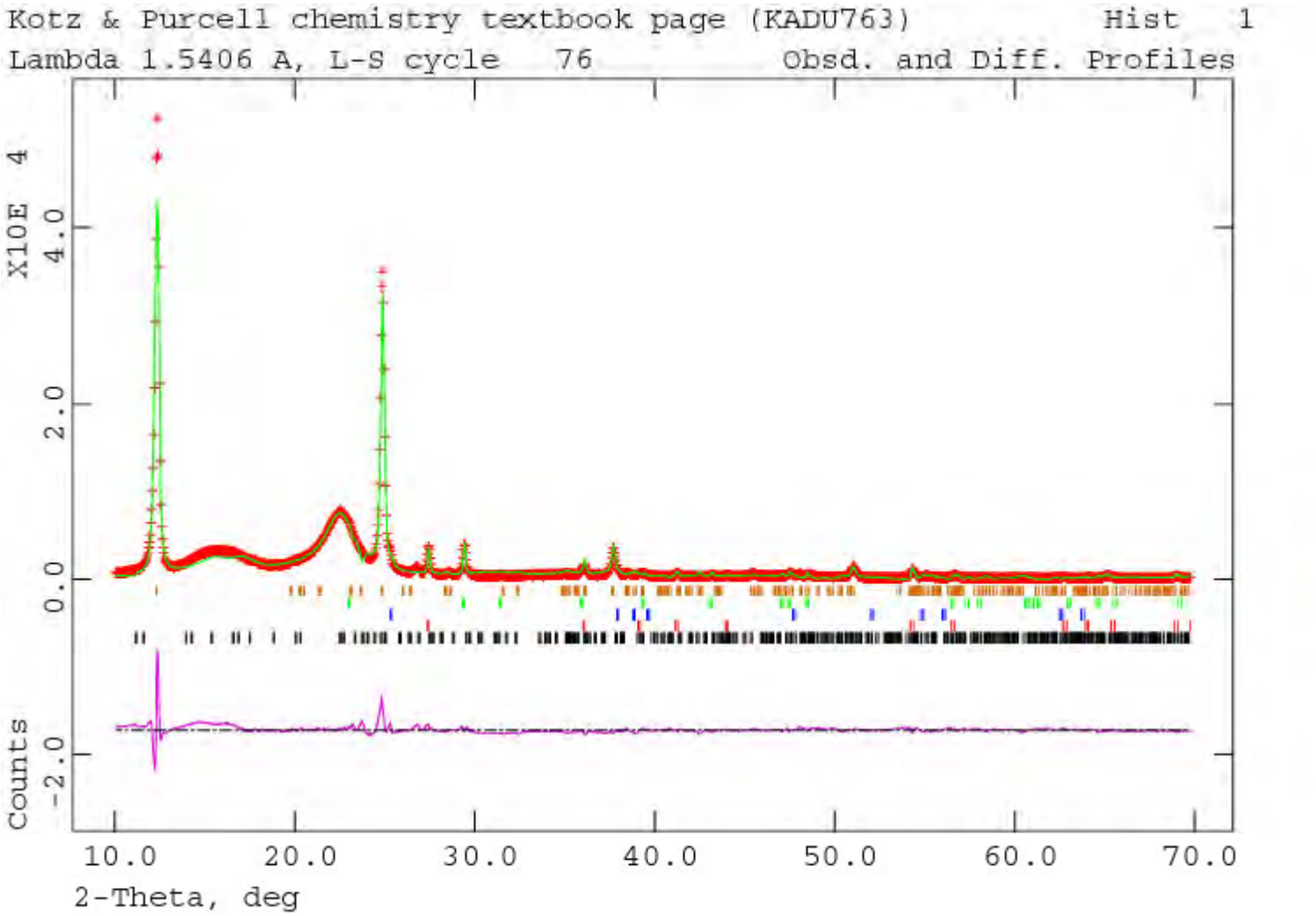
Phase	wt%	int. std. wt%	expected mg	wt%
guaifenesin	91.62(2)	90.4(4)	1200	90.9
pseudoephedrine hydrochloride	8.38(15)	7.7(4)	120	9.1
sum	100	98.1	1320	100

Actual tablets weigh ~1540 mg

Chemistry Textbook Paper


[KADU763.MDI] Kotz & Purcell textbook page (45,30,zbc) JAK
I(CPS)
25.0
20.0
15.0
10.0
5.0
x10^3
50-2241> (C6H10O5)n - Cellulose
77-0441> Rutile, syn - TiO2
83-2243> Anatase, syn - TiO2
83-1762> Calcite - Ca(CO3)
83-0971> Kaolinite 1A - Al2(Si2O5)(OH)4
72-1385> Clinochlore, chromian 1M - Mg5.1Al1.2Si3Cr.7O10(OH)8
10
20
30
40
50
60
2-Theta(°)

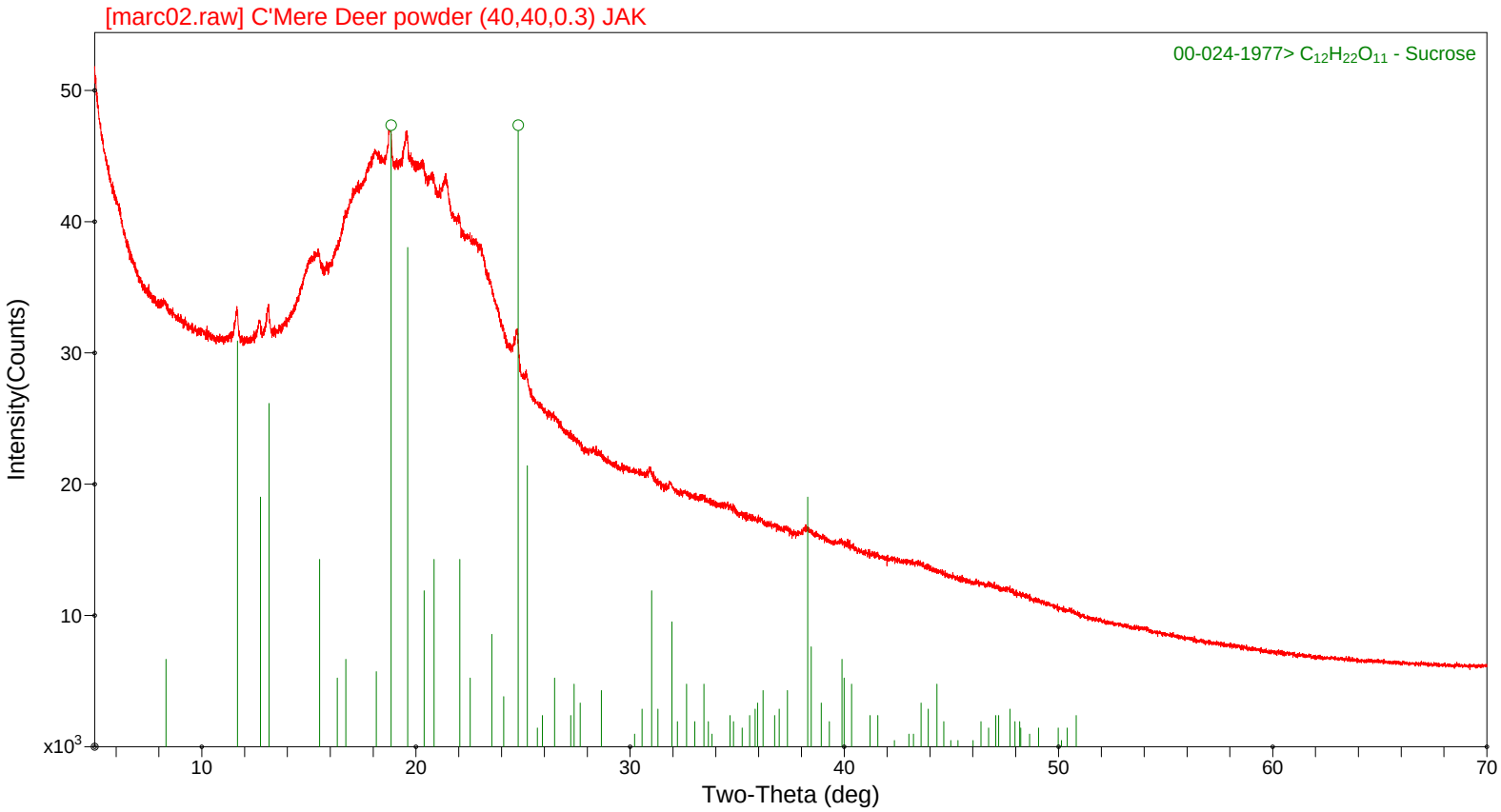
Rietveld Difference Plot



Quantitative Phase Analysis

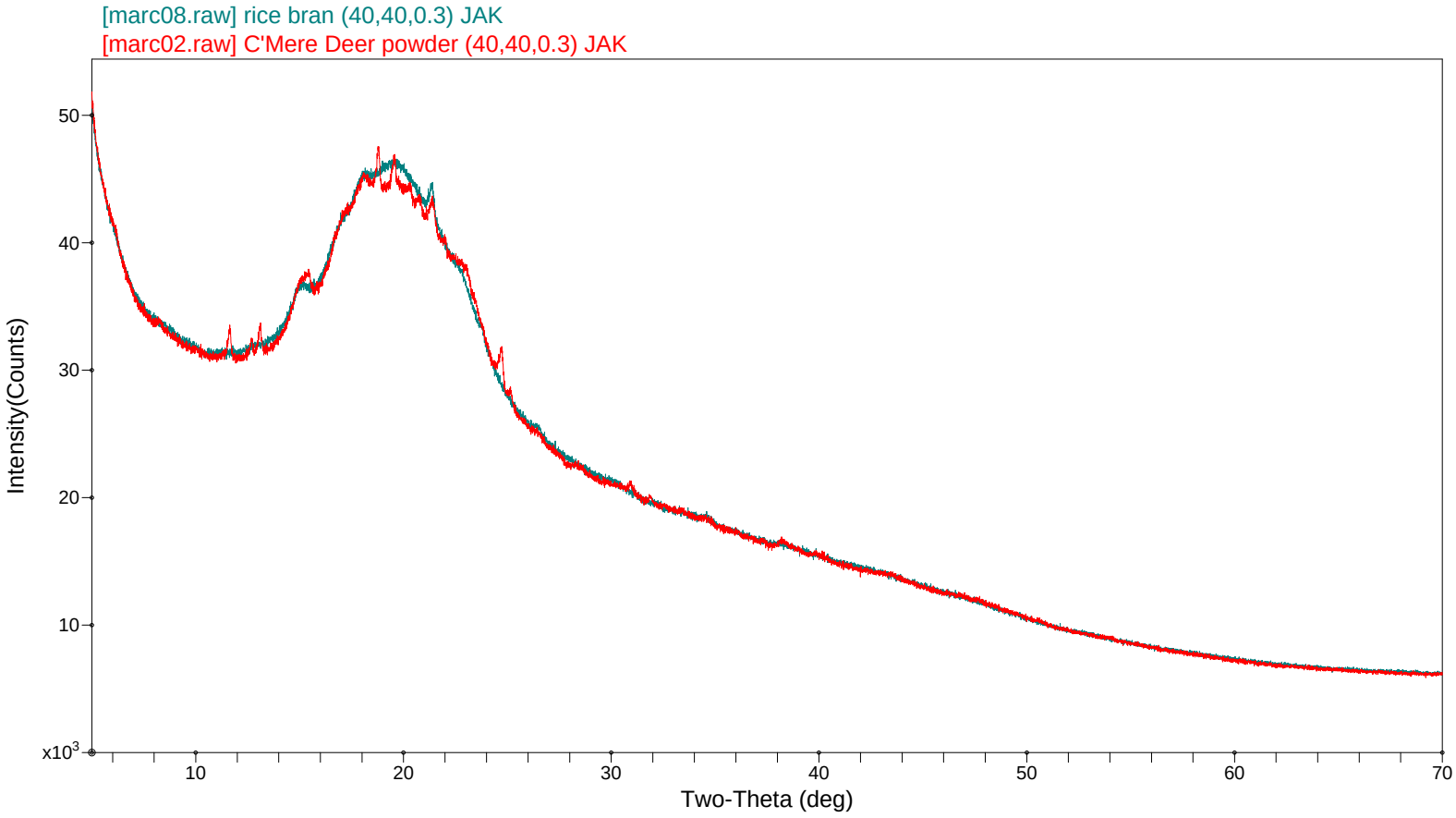
	Concentration
Cellulose	86.6 wt%
Rutile	1.1 wt%
Anatase	0.1 wt%
Calcite	1.6 wt%
Kaolinite	10.5 wt%
Chlorite	trace

Deer Attractant


[marc02.raw] C'Mere Deer powder (40,40,0.3) JAK
00-024-1977> C12H22O11 - Sucrose
Intensity(Counts)
x10^3
50
40
30
20
10
10
20
30
40
50
60
70
Two-Theta (deg)

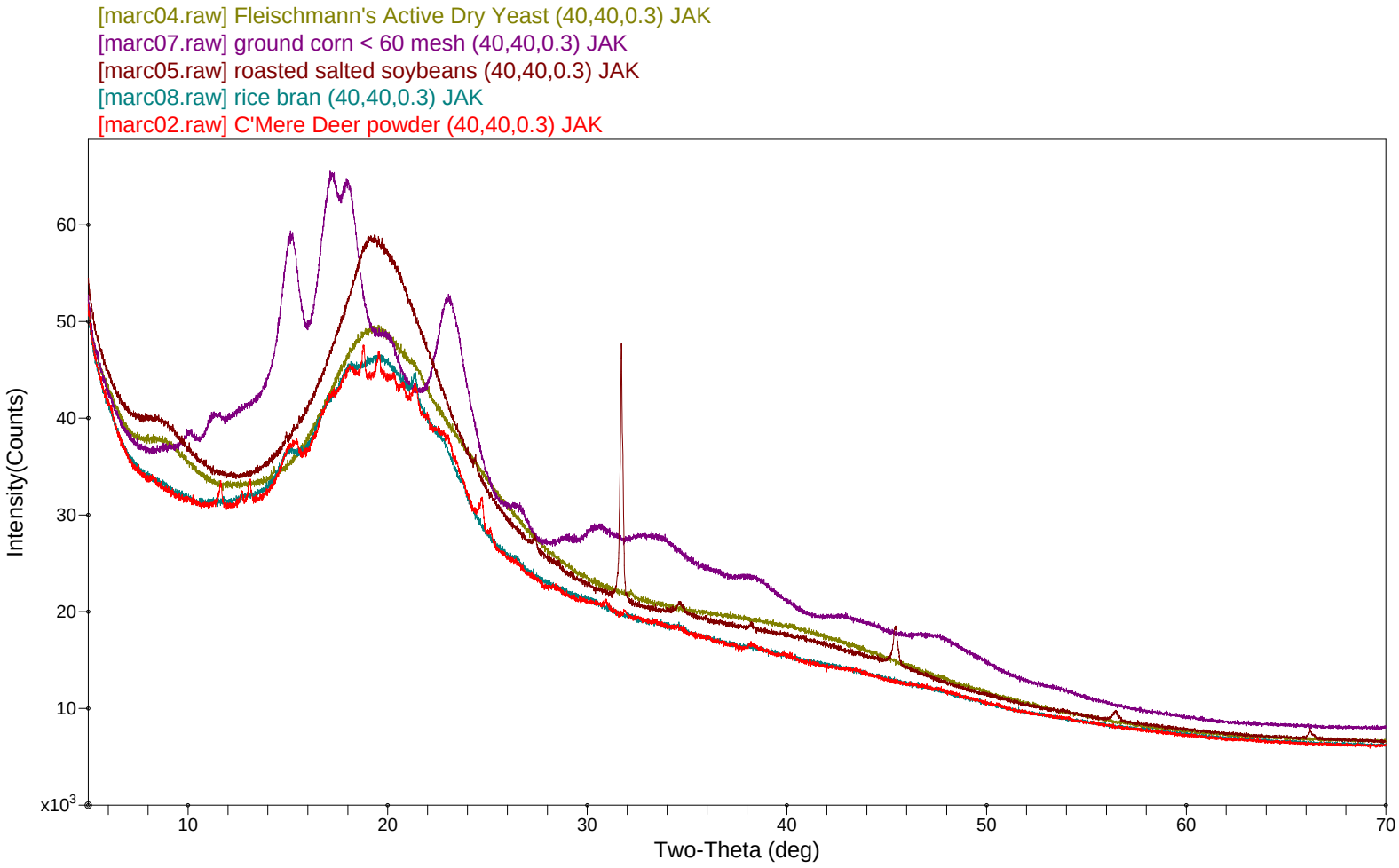
C'Mere Deer powder

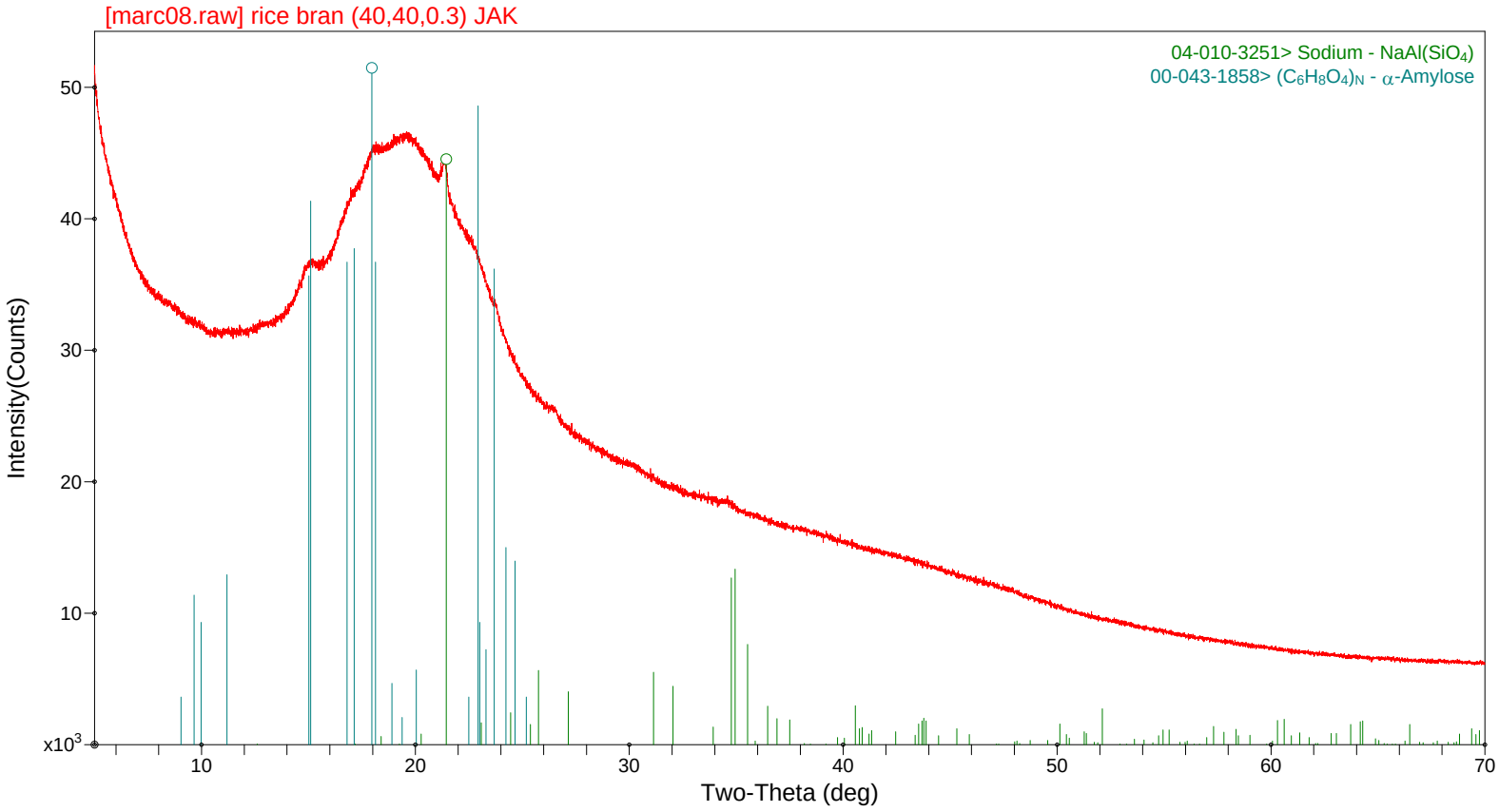
rice bran, soybeans, corn, yeast,
trace minerals (< 2%),
artificial and natural flavorings


[marc08.raw] rice bran (40,40,0.3) JAK
[marc02.raw] C'Mere Deer powder (40,40,0.3) JAK
Intensity(Counts)
x10^3
50
40
30
20
10
10
20
30
40
50
60
70
Two-Theta (deg)

Address on the label is:
EST, LLC
205 Fair Ave.
Winnsboro LA 71295

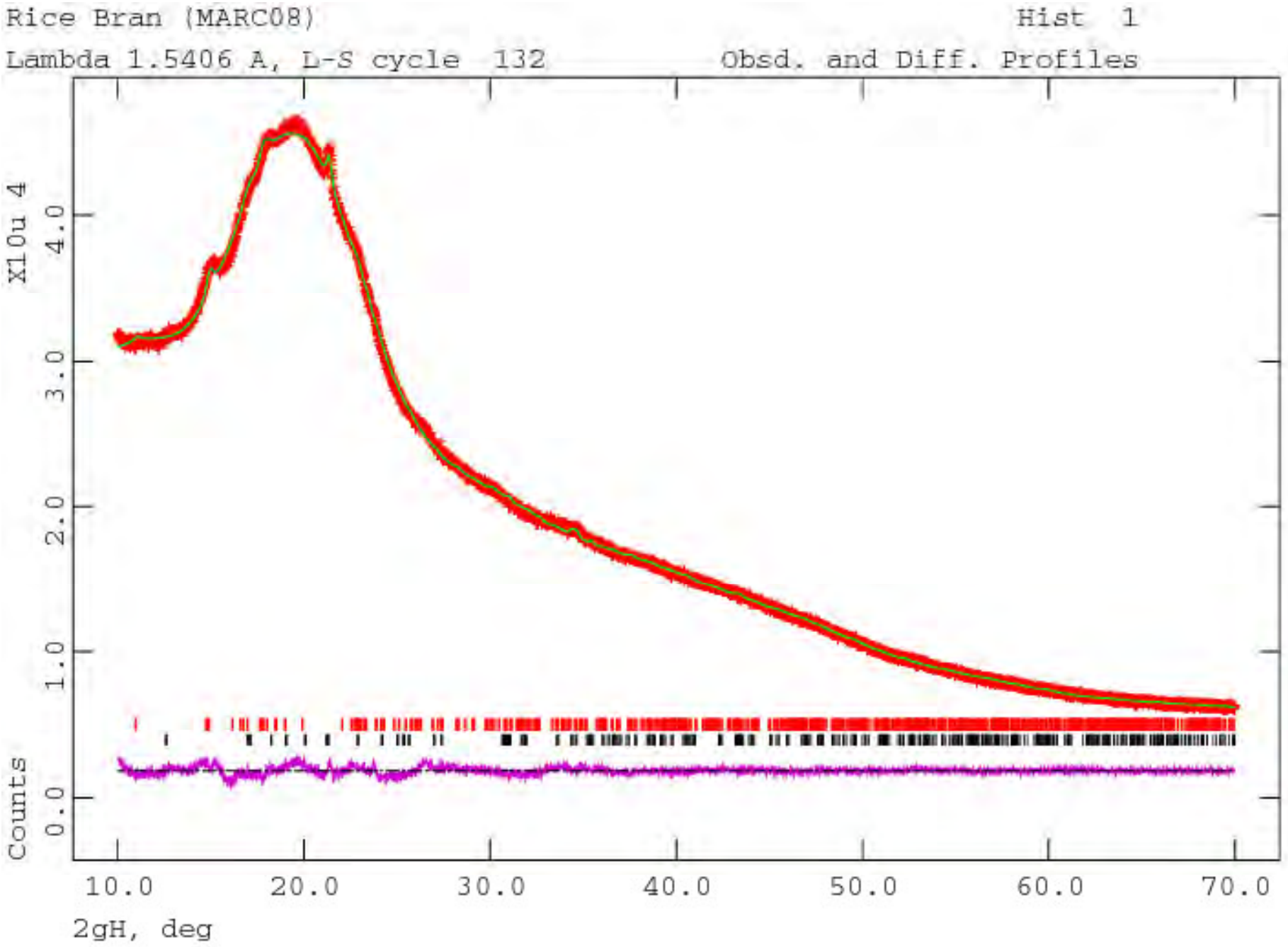
Most US rice is grown in LA, so perhaps rice bran is cheap!

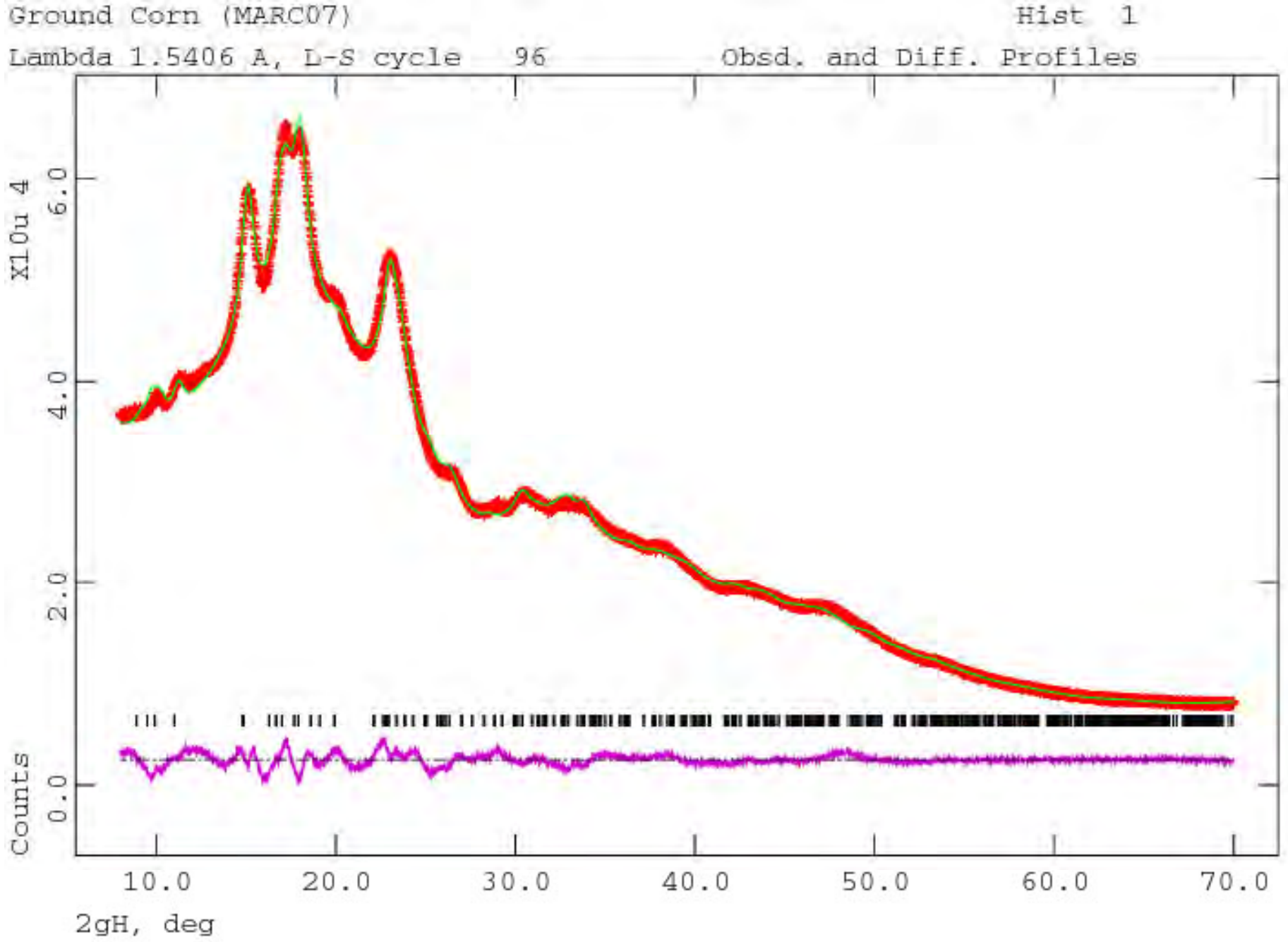

[marc04.raw] Fleischmann's Active Dry Yeast (40,40,0.3) JAK
[marc07.raw] ground corn < 60 mesh (40,40,0.3) JAK
[marc05.raw] roasted salted soybeans (40,40,0.3) JAK
[marc08.raw] rice bran (40,40,0.3) JAK
[marc02.raw] C'Mere Deer powder (40,40,0.3) JAK
Intensity(Counts)
x10^3
60
50
40
30
20
10
10
20
30
40
50
60
70
Two-Theta (deg)

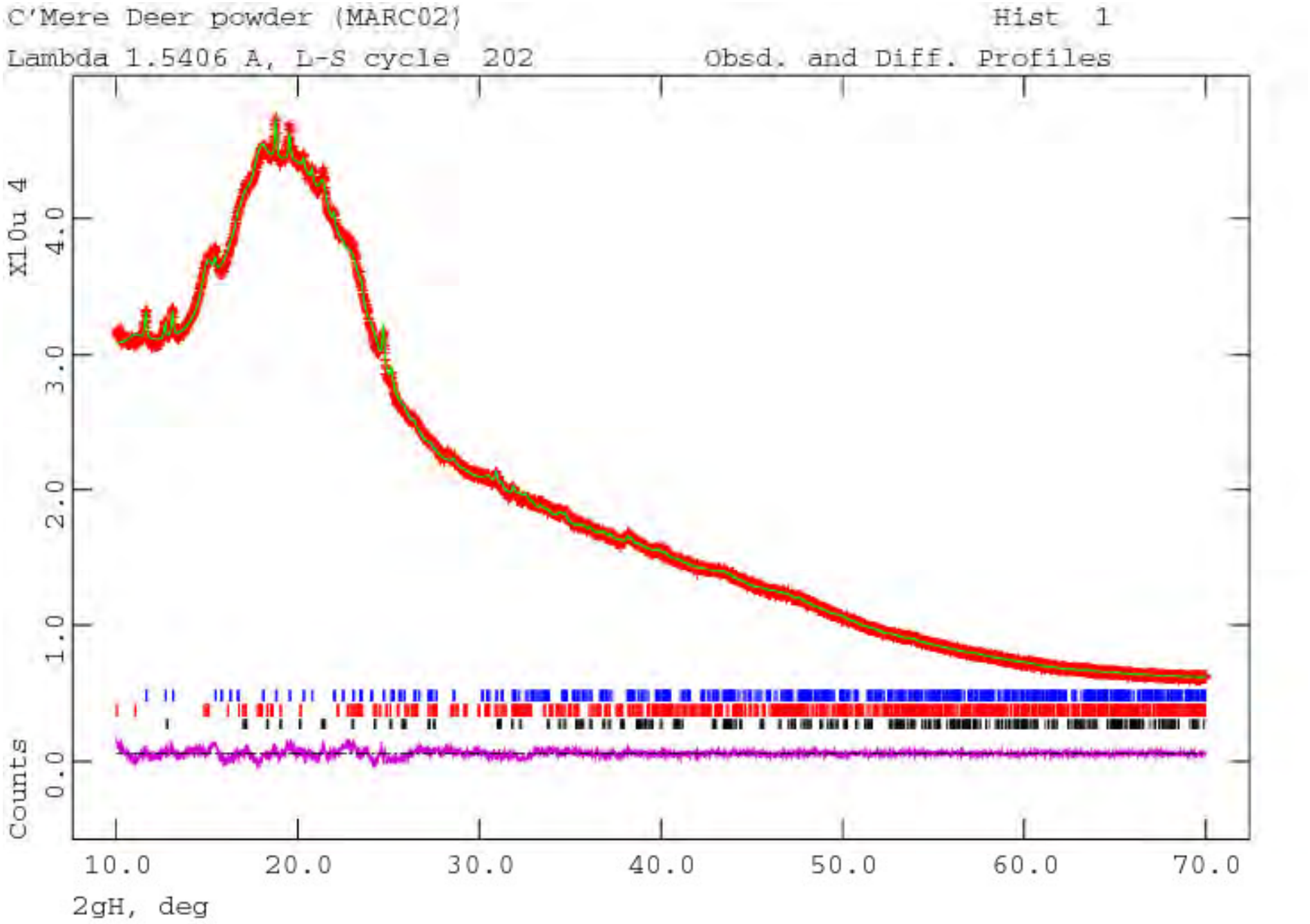

[marc08.raw] rice bran (40,40,0.3) JAK
04-010-3251> Sodium - NaAl(SiO4)
00-043-1858> (C6H8O4)N - α-Amylose
Intensity(Counts)
x10^3
50
40
30
20
10
10
20
30
40
50
60
70
Two-Theta (deg)

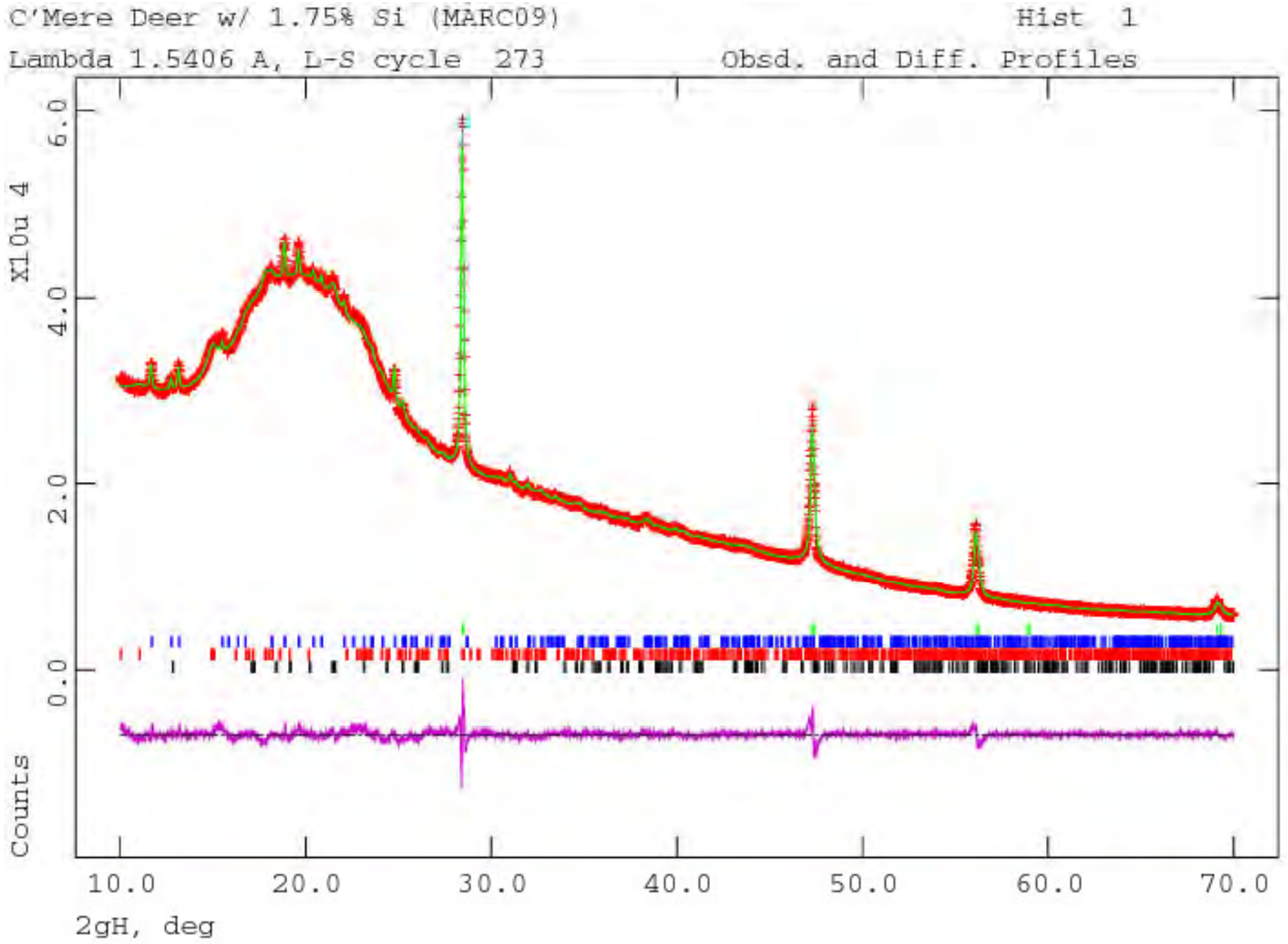
Rice is known to be good at extracting silica from the soil.

Maybe some quartz, too?


Rice Bran (MARC08)
Hist 1
Lambda 1.5406 A, L-S cycle 132
Obsd. and Diff. Profiles
X10u 4
4.0
3.0
2.0
1.0
0.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
70.0
2gH, deg


Ground Corn (MARC07)
Hist 1
Lambda 1.5406 A, L-S cycle 96
Obsd. and Diff. Profiles
X10u 4
6.0
4.0
2.0
0.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
70.0
2gH, deg


C'Mere Deer powder (MARC02)
Hist 1
Lambda 1.5406 A, L-S cycle 202
Obsd. and Diff. Profiles
X10u 4
4.0
3.0
2.0
1.0
0.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
70.0
2gH, deg


C'Mere Deer w/ 1.75% Si (MARC09)
Hist 1
Lambda 1.5406 A, L-S cycle 273
Obsd. and Diff. Profiles
X10u 4
6.0
4.0
2.0
0.0
Counts
10.0
20.0
30.0
40.0
50.0
60.0
70.0
2gH, deg

Quantitative Phase Analysis of C'Mere Deer Powder

Phase	Raw wt%	Abs. wt%	Real wt%
$NaAlSiO_4$	5.2(2)	0.42	0.4(1)
Amylose	56.9(4)	4.60	4.7(1)
Sucrose	16.3(2)	1.32	1.3(1)
Si	21.62(6)	1.75	-

The Merck Index says that corn is typically 27% amylose and 73% amylopectin, so this translates into ~17 wt% corn.

Scaling "Experiments"

Variable	Rice Bran	Corn	Soybeans	Yeast
(background-subtracted) raw patterns	70	10	10	10
Amylose scale factors		11		
Diffuse scattering amplitudes	83			
Best Guess	76	15	4	4

1.3% sucrose, and traces of minerals and flavors.

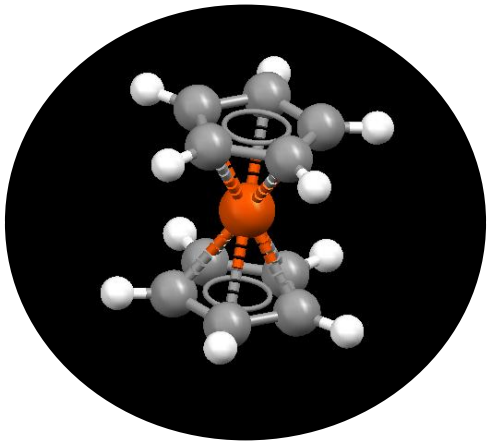
Teaching Chemistry with Experimentally Measured 3D Structures

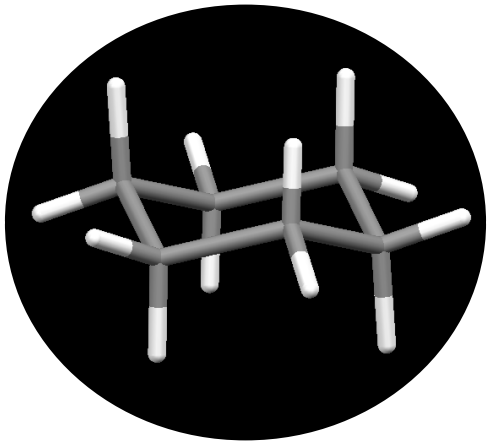
Colin Groom

Cambridge Crystallographic Data Centre

Cambridge, UK

teaching@ccdc.cam.ac.uk



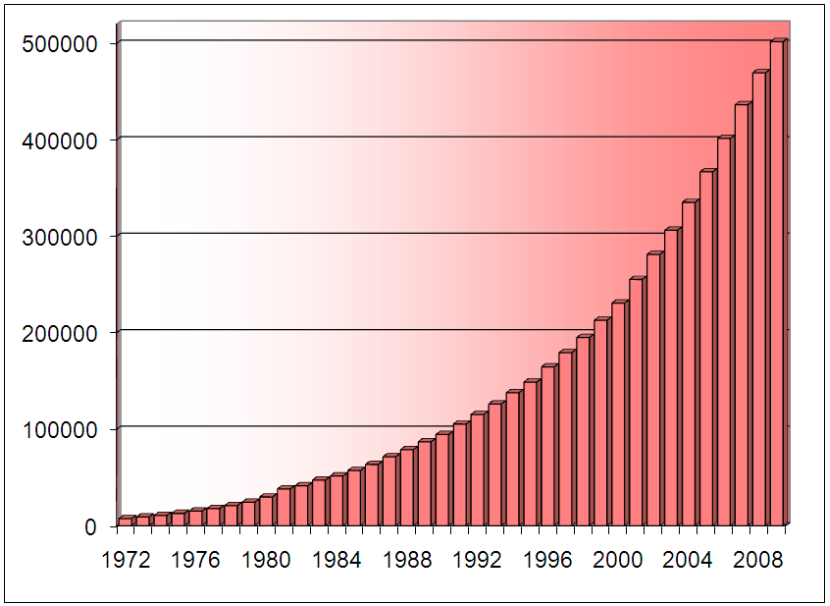


History of the CCDC

- Founded in 1965 with grant funding in the Department of Chemistry, University of Cambridge
- Self financing, self administering Institution since 1987
  - Not-for-profit, charitable, research institute
  - Recognized institute for postgraduate degrees of the University of Cambridge
- Objectives
  - “advancement and promotion of the science of chemistry and crystallography for the public benefit”

Cambridge Structural Database

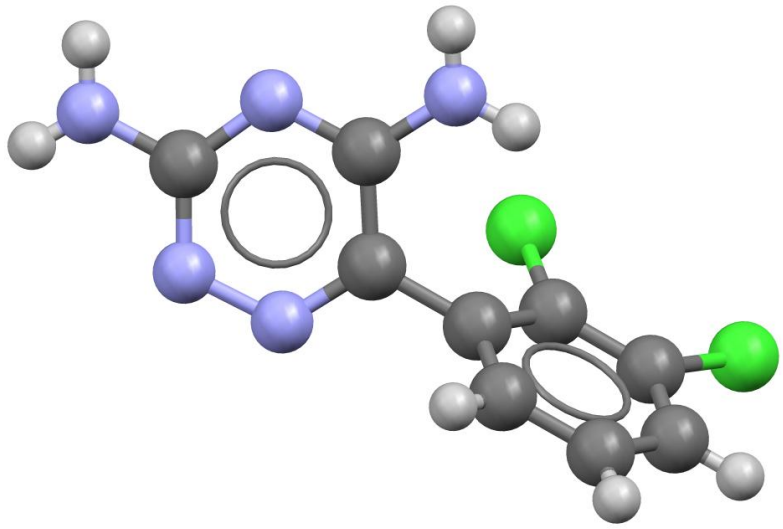
Worldwide repository of validated small-molecule crystal structures



CSD Growth 1970-2010

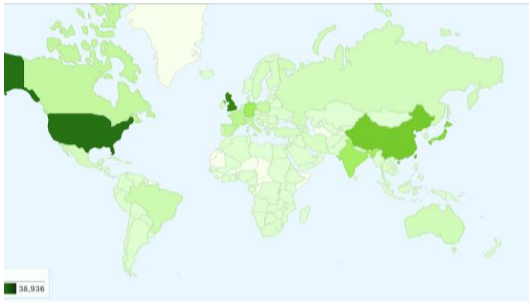
Dec 09 – 500,000th structure milestone reached

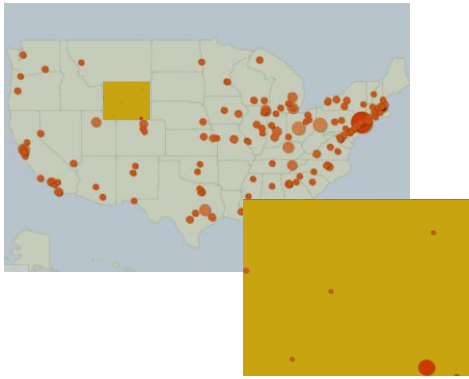
Lamotrigine
Acta Cryst., Sect.C:Cryst Struct. Commun. (2009), **65**, o460
Refcode: **EFEMUX01**



The Cambridge Crystallographic Data Centre

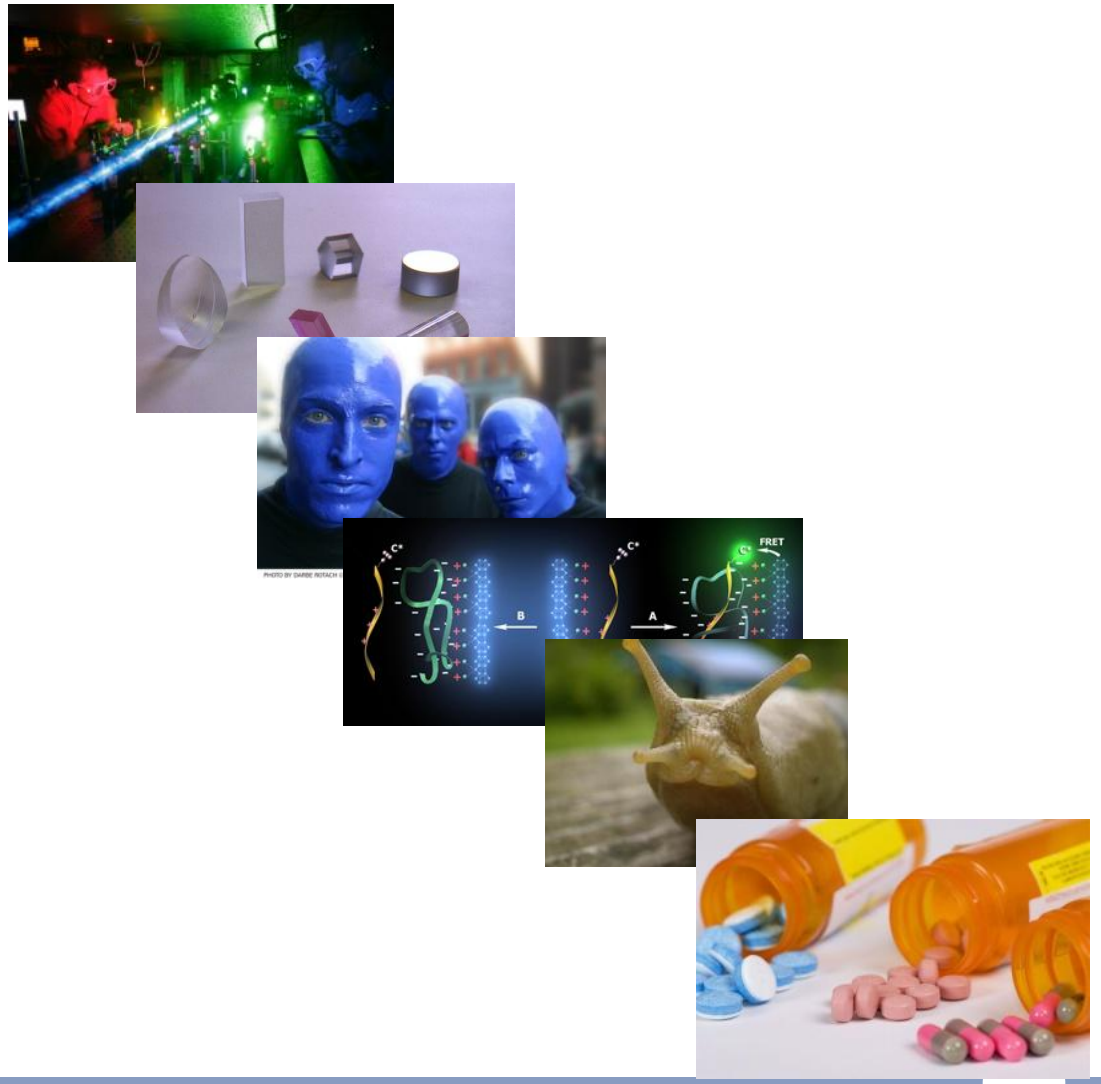
- Compiles the Cambridge Structural Database
- Develops scientific products and services
- Maximises worldwide accessibility to the CSD
  - Web access from 186 countries
  - Applications installed in 69 countries
- Performs and supports fundamental research
  - 50 PhD students since 1991
  - Over 500 publications
- Promotes and supports applications of crystal structure information
  - 15,000 users of Mercury





Why small molecule crystals?

- Organic non-linear optical materials
- Piezoelectric crystals
- Pigments
- Organic semiconductors
- Liquid crystals
- Pharmaceuticals

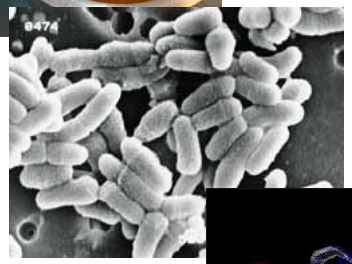
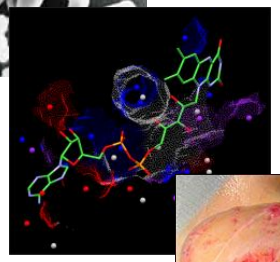
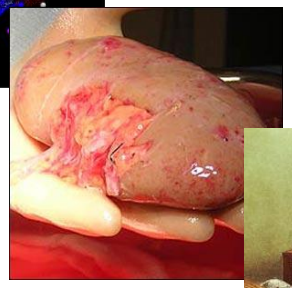


Why small molecule crystals?

- Biological processes
  - Pressure effects on amino acids
    - pressure cooking
    - piezophilic organisms
  - The binding of drug molecules
  - Cystals in the body
    - Kidney stones

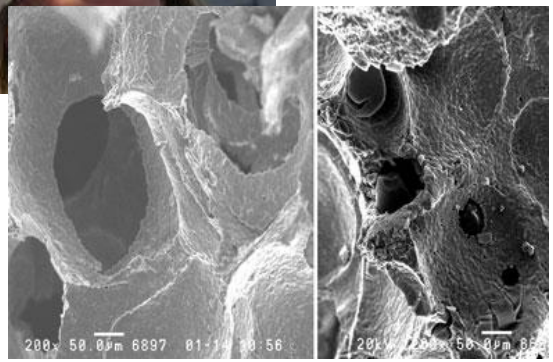
      calcium oxalate, uric acid or cystine
    - Gout

      Monosodium urate


Why small molecule crystals?

- Understanding of every-day physical processes
  - Chocolate
  - The setting of cement
  - The healing of bones



3D Visualisation

- Chemistry students need 3D visualisation proficiency
- 3D visualisation shown to enhance students conceptual understanding and spatial abilities

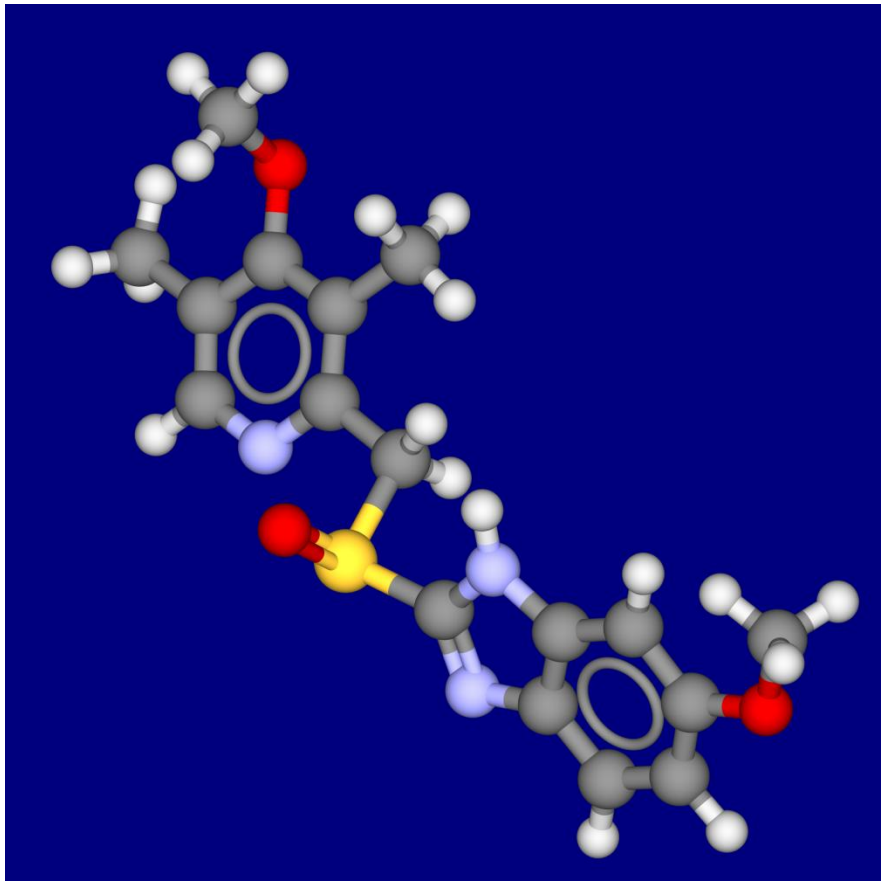
Williamson, V. M. *J. Chem. Educ*., **2008**, 85, 718-723
Wu, H. K. *Sci. Educ. Res. Pract*., **2004**, 8, 61-72
Bodner, G. M. *The Chemical Educator*, **1997**, 2

"Because basic 3D spatial relationships in molecules have systematic and profound causal significance, chemistry is an extraordinary fertile field for visual learning"

2001 NSF Workshop: Molecular Visualisation in Science Education

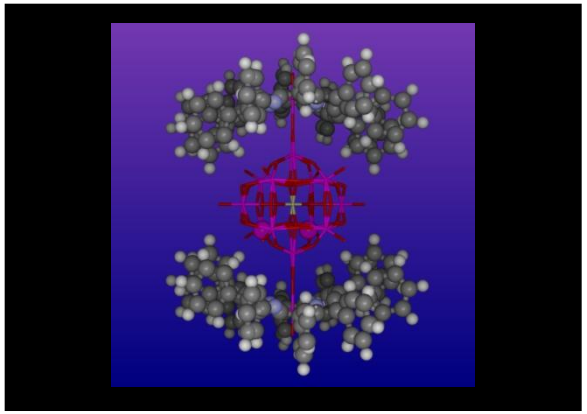
Molecules in the CSD

- Many drugs
  - Omeprazole

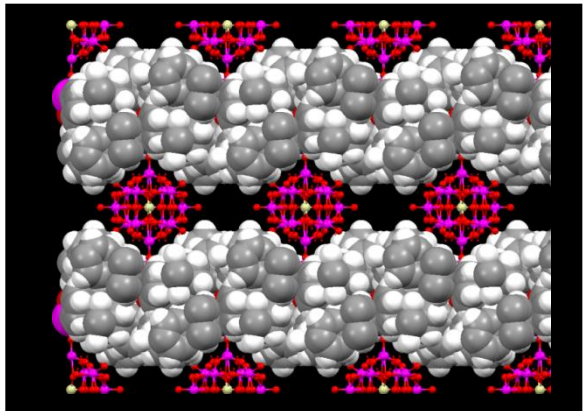


Molecules in the CSD: Porphyrin Hamburgers

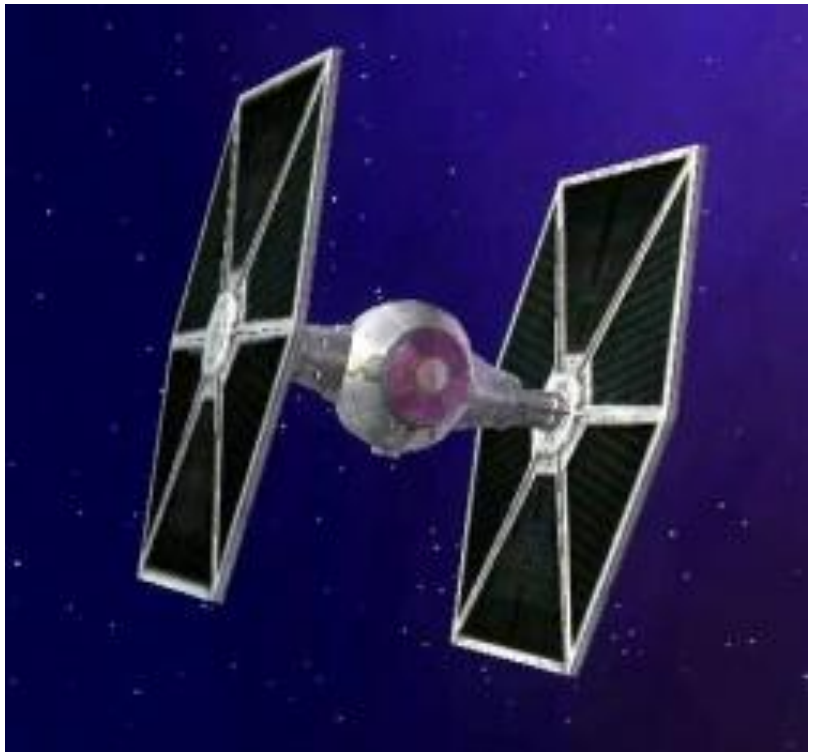
NIPBEU



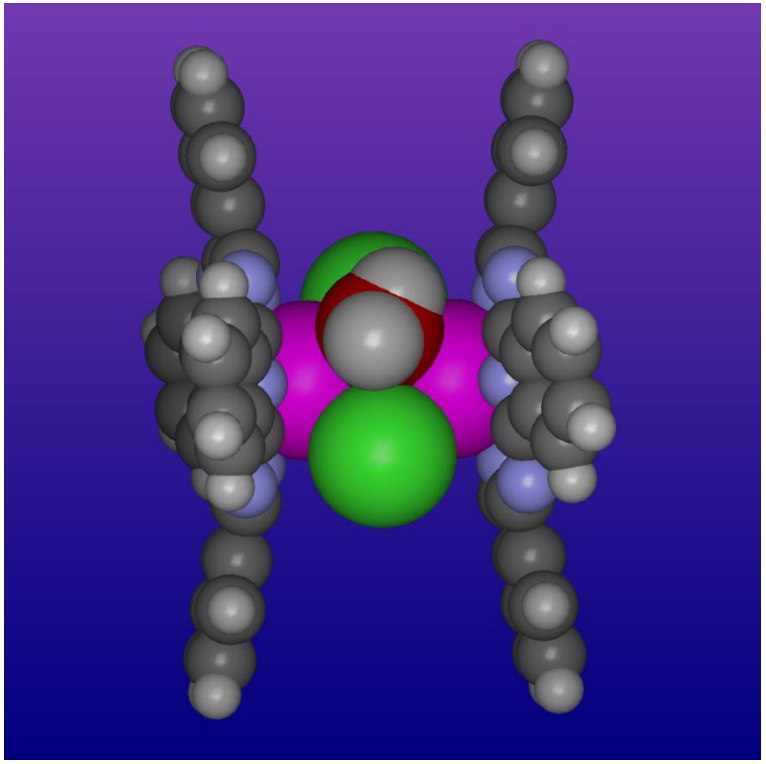
BURGER



Molecules in the CSD: Star Wars TIE Fighter

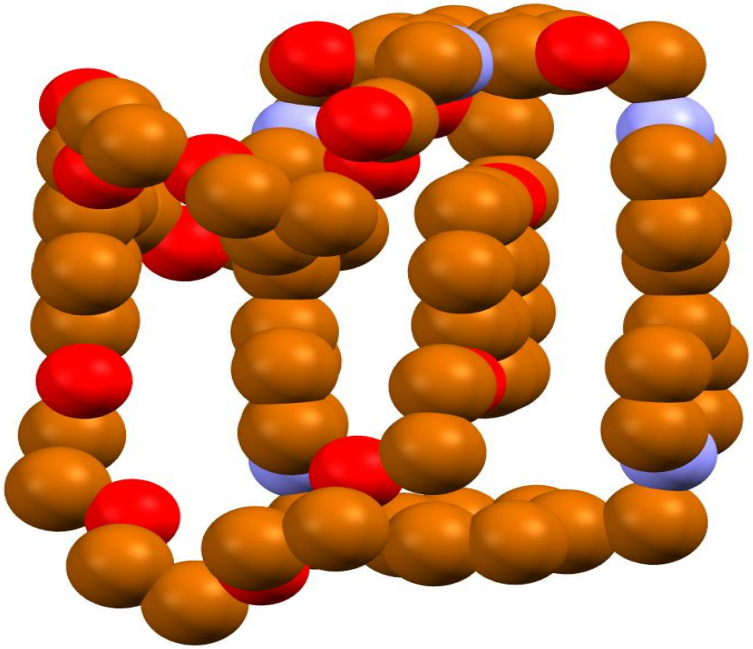


BOSQAB

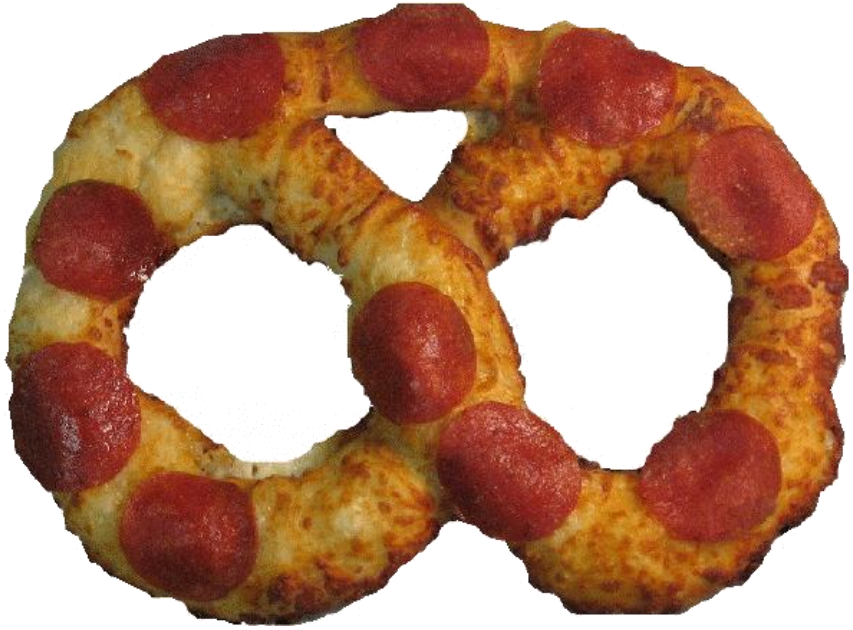


TIE FIGHTER

Molecules in the CSD: Pretzelanes

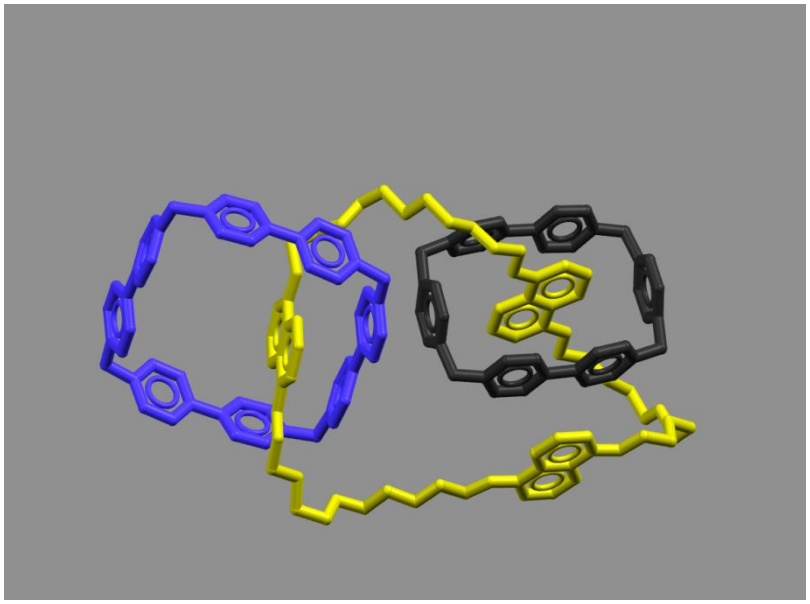


PRETZEL



SARFEX

Molecules in the CSD: Olympiadane

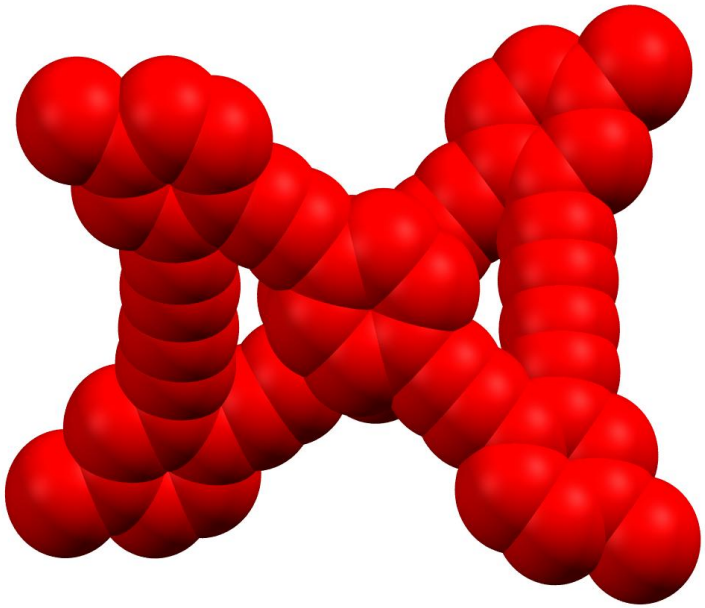


Olympic rings

SOHNEI

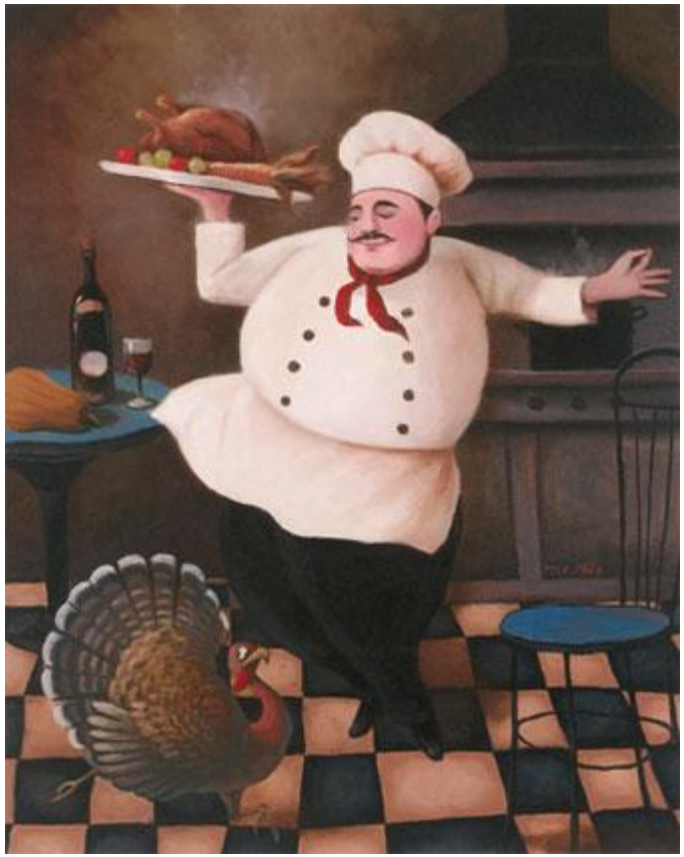
Molecules in the CSD:
Paracyclophane/Dehydrobenzoannulene Hybrids

QEQJAY

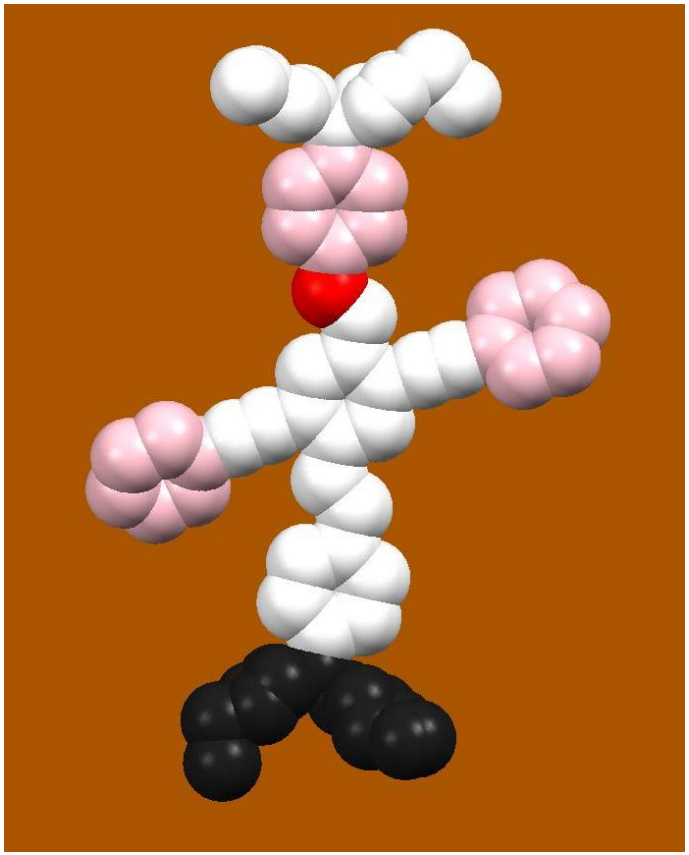


BOW TIE

Molecules in the CSD: Nanoputians



SAFKOA



CHEF

Experimentally Measured Data

- Use of experimentally measured data is of great pedagogical value
  - Inherent experimental error and statistical variation provides an opportunity to deal with the uncertainties of chemistry
  - Challenges students to think more critically about bonding and molecular structure
  - Encourages method and limitations of data collection to be considered
- Measured data shown to enhance student learning

DeHaan, R. L. *J. Sci. Educ. Technol*., **2005**, 14, 253-269
Handelsman, J. *Science* **2004**, 304, 521-522
Prince, M. *J. Eng. Educ.,* **2004**, 89, 1-9

Free Educational Resources

- Teaching subset of 500 CSD entries chosen to illustrate a wide range of 3D structural issues
- Web-based interface for browsing the teaching subset
- A downloadable version of the Mercury visualiser
- Tutorials and worksheets

http://www.ccdc.cam.ac.uk/free_services/teaching/

Teaching Subset of the CSD

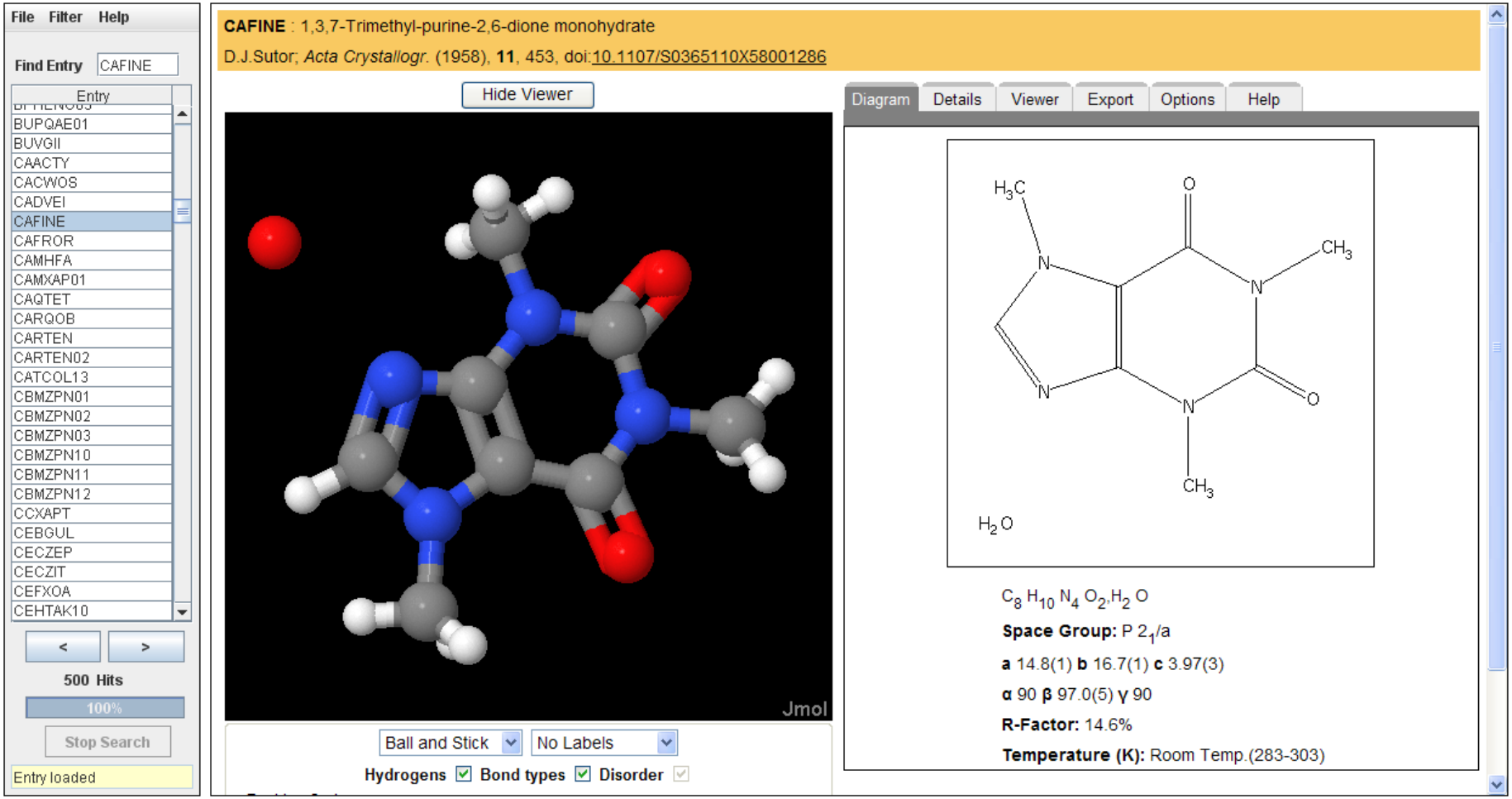
- 500 structures that have important applications in chemistry and chemical education selected from the full CSD of almost half a million crystal structures
  - Includes key molecules typically used in textbooks to exemplify core concepts and principles
  - Simple examples of all the main molecular geometry types
  - Chemically diverse; representative of full database

http://www.ccdc.cam.ac.uk/free_services/teaching/

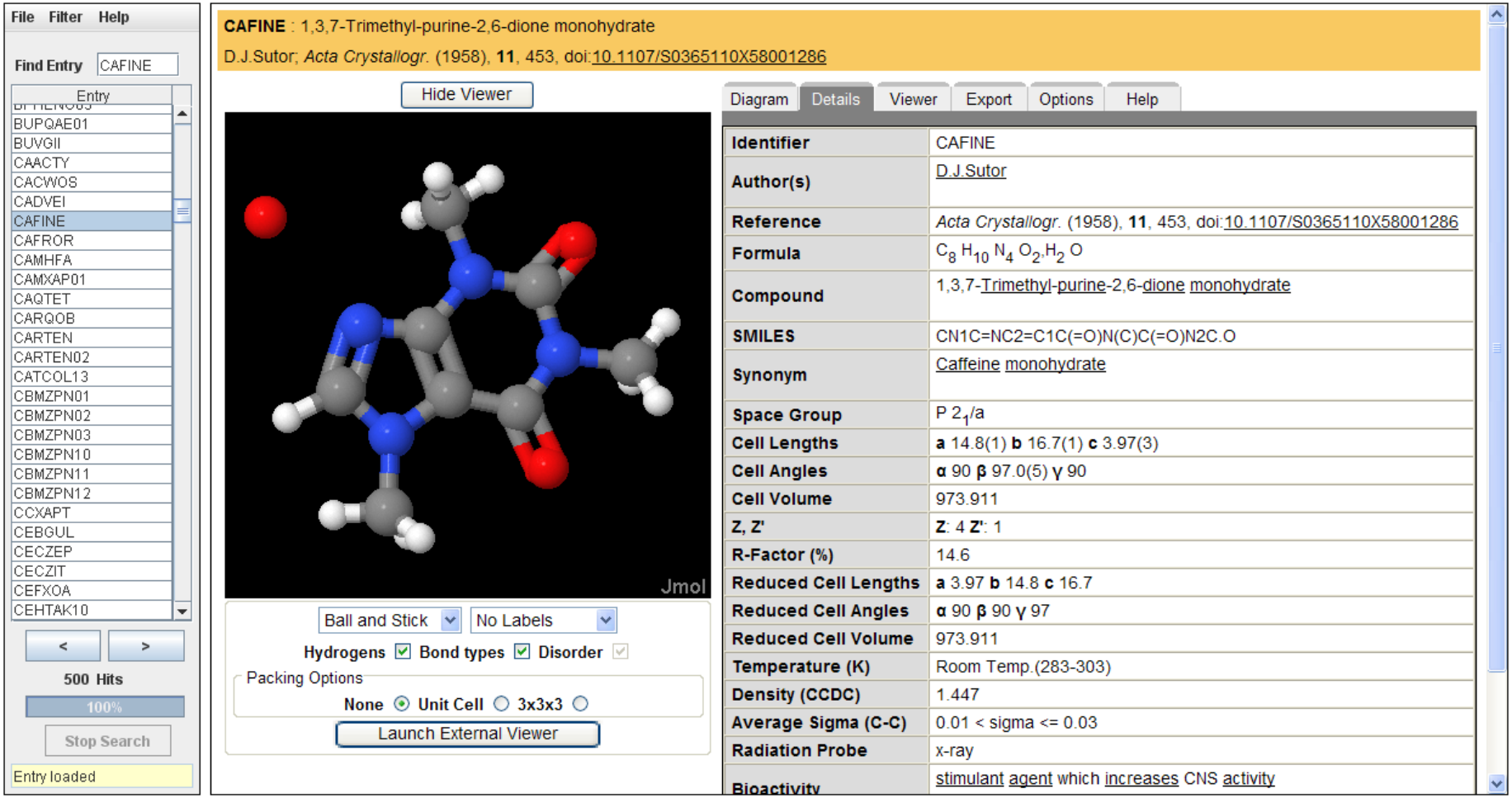
How Can I Access the Subset

- Mercury visualiser
  - Advanced crystal structure visualisation program by CCDC
  - Freely downloadable desktop application
  - Teaching subset embedded and available via Databases menu
- Interactive web-based interface
  - Uses WebCSD: new on-line search interface to the CSD
  - Not required to download, install or register any software

http://www.ccdc.cam.ac.uk/free_services/teaching/



http://www.ccdc.cam.ac.uk/free_services/teaching/



http://www.ccdc.cam.ac.uk/free_services/teaching/

Further Information

- Teaching 3D Structural Chemistry Using Crystal Structure Databases 1. An Interactive Web Accessible teaching Subset of the Cambridge Structural Database
  Gary M. Battle and Frank H. Allen, Gregory M. Ferrence
  J. Chem. Educ., Articles ASAP; DOI: 10.1021/ed100256k
- Teaching 3D Structural Chemistry Using Crystal Structure Databases 2. Teaching Units that Utilize an Interactive Web Accessible teaching Subset of the Cambridge Structural Database
  Gary M. Battle and Frank H. Allen, Gregory M. Ferrence
  J. Chem. Educ., Articles ASAP; DOI: 10.1021/ed100257t
- Applications of the Cambridge Structural Database in Chemical Education
  Gary M. Battle and Frank H. Allen, Gregory M. Ferrence
  J. Appl. Cryst. Special Teaching Edition, 2010 submitted

Crystallography World of Wonders

Claudia J. Rawn

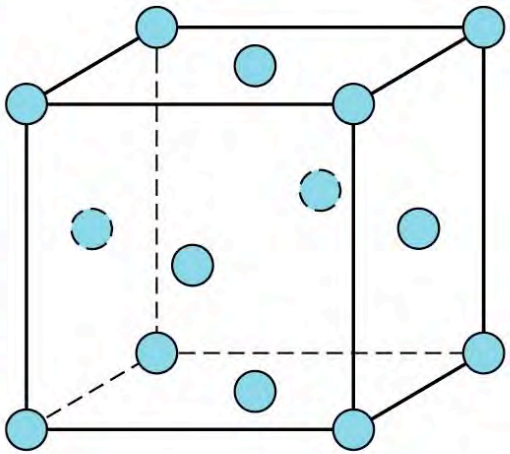
crawn@utk.edu

University of Tennessee/

Oak Ridge National Laboratory

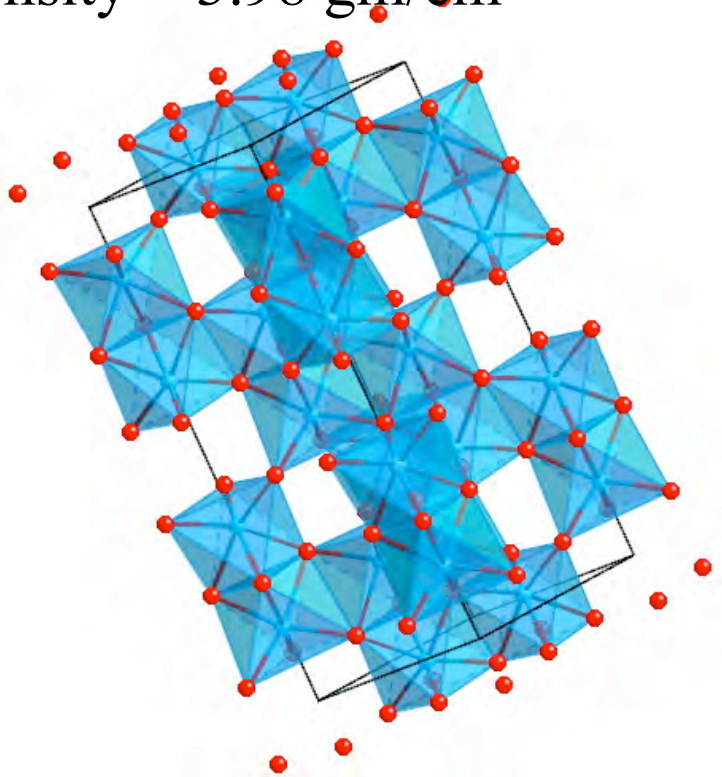
Al vs Al_2O_3

Al
Melts at 660 °C
FCC a = 4.0495 Å
Density = 2.71 gm/cm^3



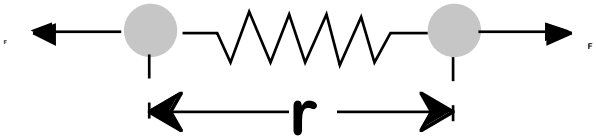
(b)

Al_2O_3
Melts at 2000 °C
Based on HCP
a = 4.7589 and c = 12.991 Å
Density = 3.98 gm/cm^3

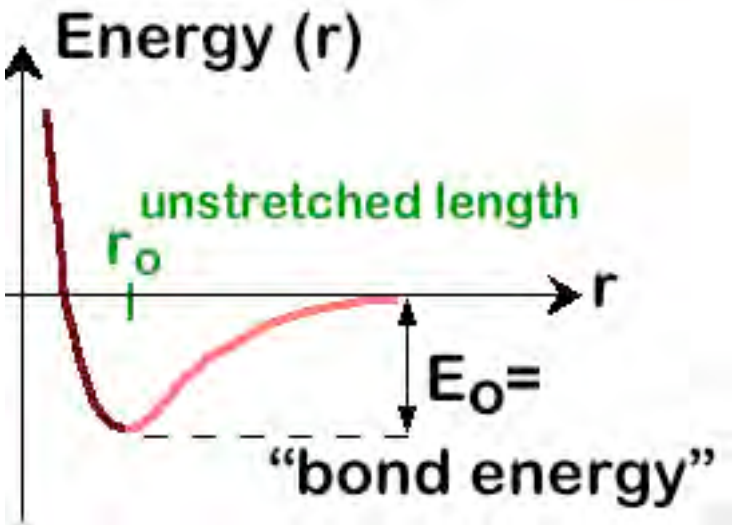


PROPERTIES FROM BONDING: T_M

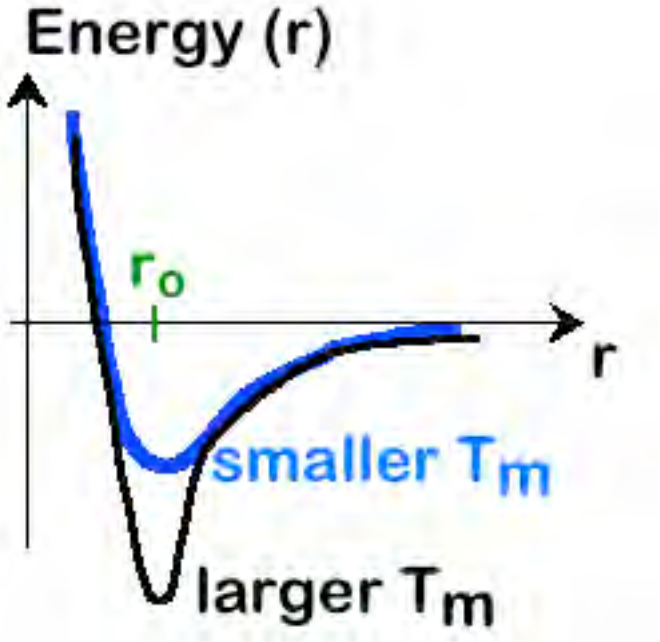
- **Bond length**, r



- **Bond energy**, E_o



- **Melting Temperature**, T_m



T_m is larger if E_o is larger.

From W.D. Callister: Materials Science and Engineering: An Introduction

DENSITIES OF MATERIAL CLASSES

$\rho_{metals} \geq \rho_{ceramics} \geq \rho_{pc}$

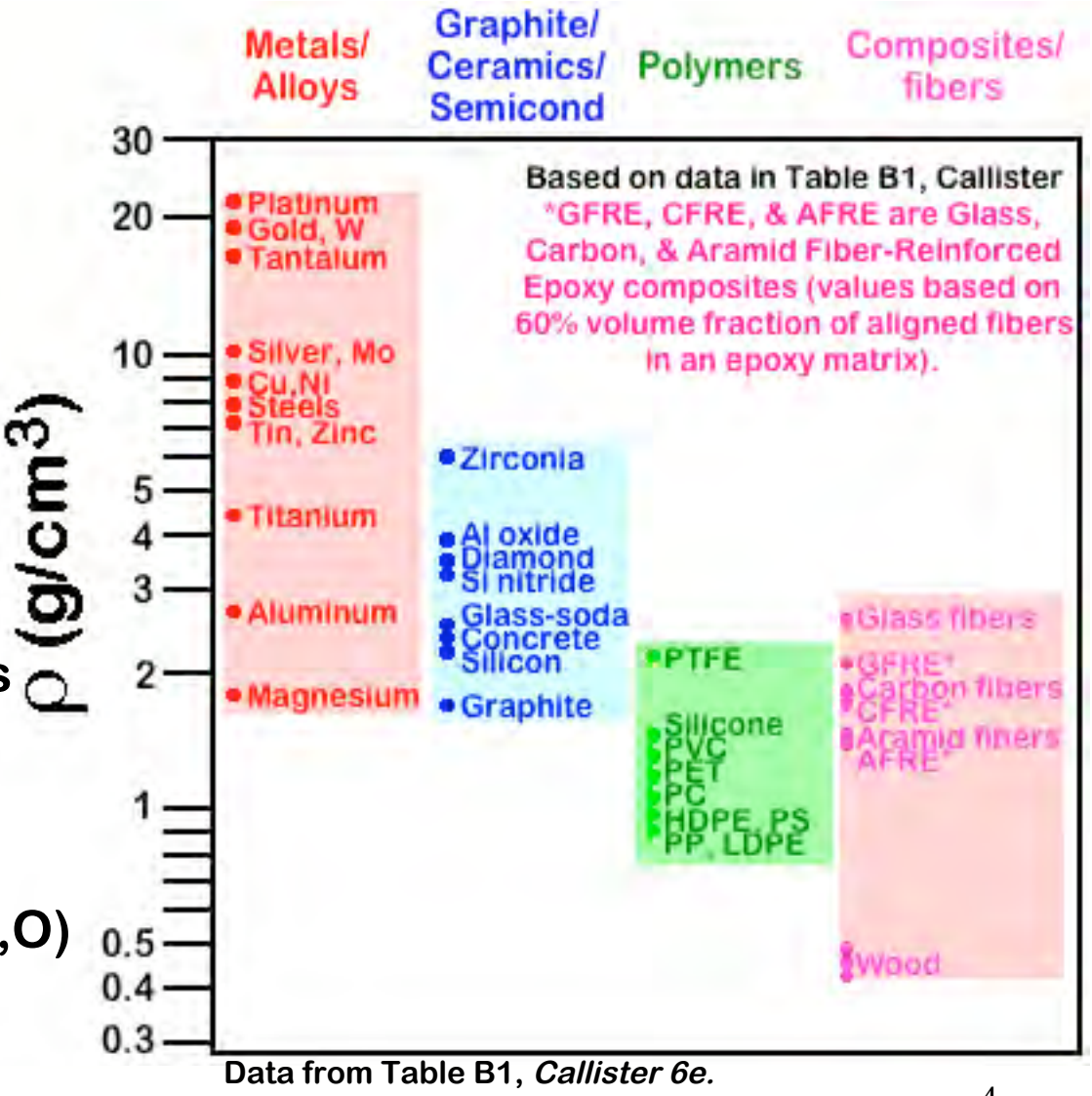
Why?

Metals have...
- close-packing (metallic bonding)
- large atomic mass

Ceramics have...
- less dense packing (covalent bonding)
- often lighter elements

Polymers have...
- poor packing (often amorphous)
- lighter elements (C,H,O)

Composites have...
- intermediate values

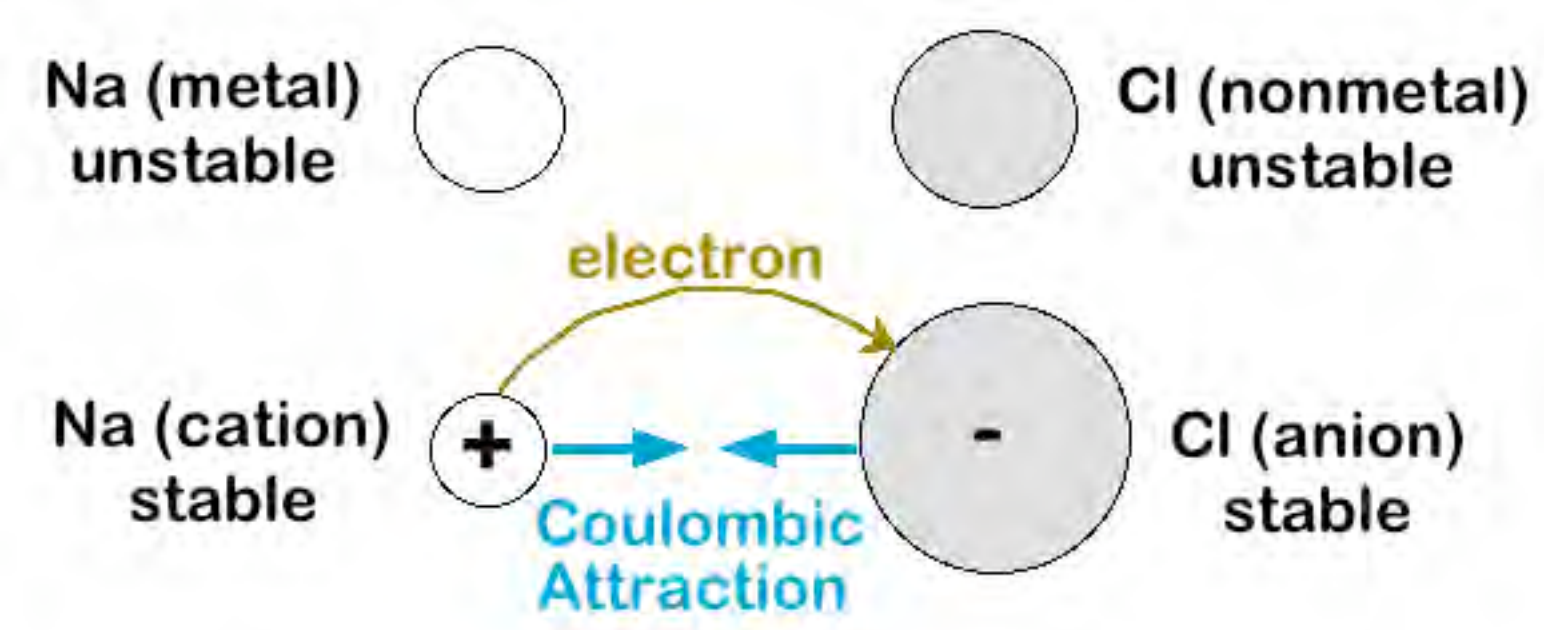


Data from Table B1, *Callister 6e.*

From W.D. Callister: Materials Science and Engineering: An Introduction

IONIC BONDING

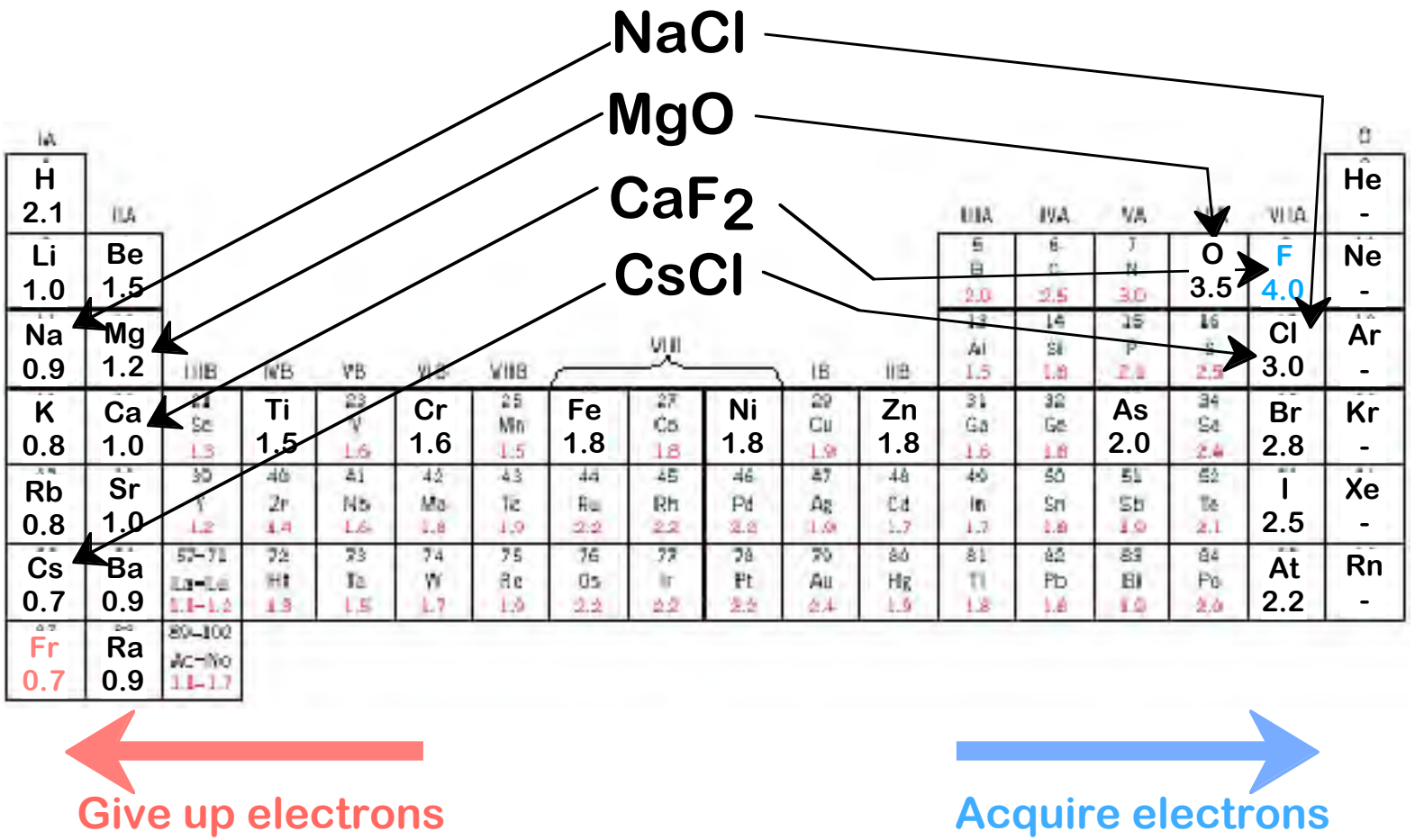
- **Occurs between + and - ions.**
- **Requires electron transfer.**
- **Large difference in electronegativity required.**
- **Example: NaCl**



From W.D. Callister: Materials Science and Engineering: An Introduction

EXAMPLES: IONIC BONDING

- **Predominant bonding in Ceramics**



Adapted from Fig. 2.7, *Callister 6e.* (Fig. 2.7 is adapted from Linus Pauling, *The Nature of the Chemical Bond*, 3rd edition, Copyright 1939 and 1940, 3rd edition. Copyright 1960 by Cornell University.

From W.D. Callister: Materials Science and Engineering: An Introduction

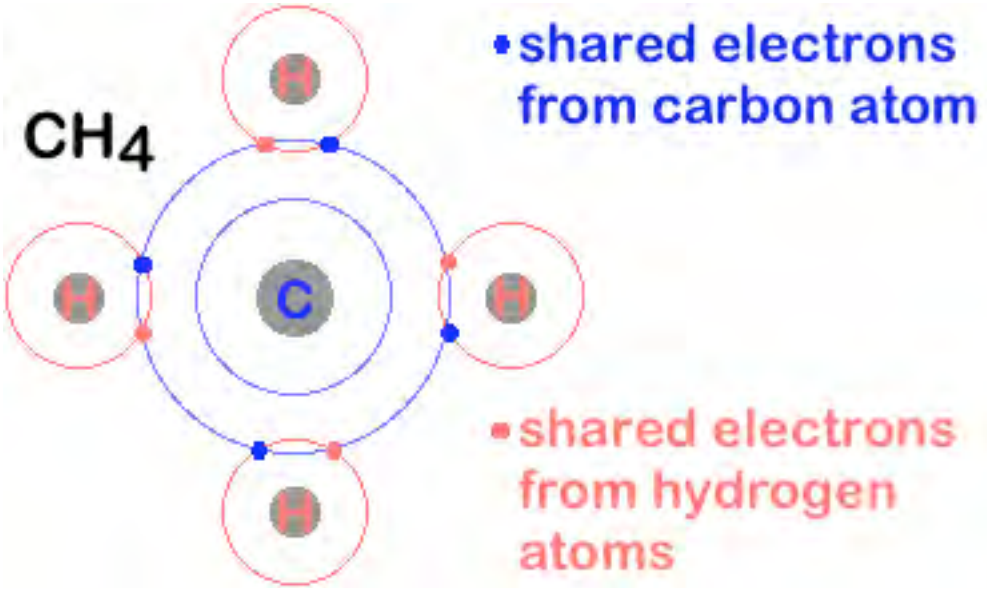
COVALENT BONDING

- **Requires shared electrons**
- **Example: CH_4**

  C: has 4 valence e, needs 4 more

  H: has 1 valence e, needs 1 more

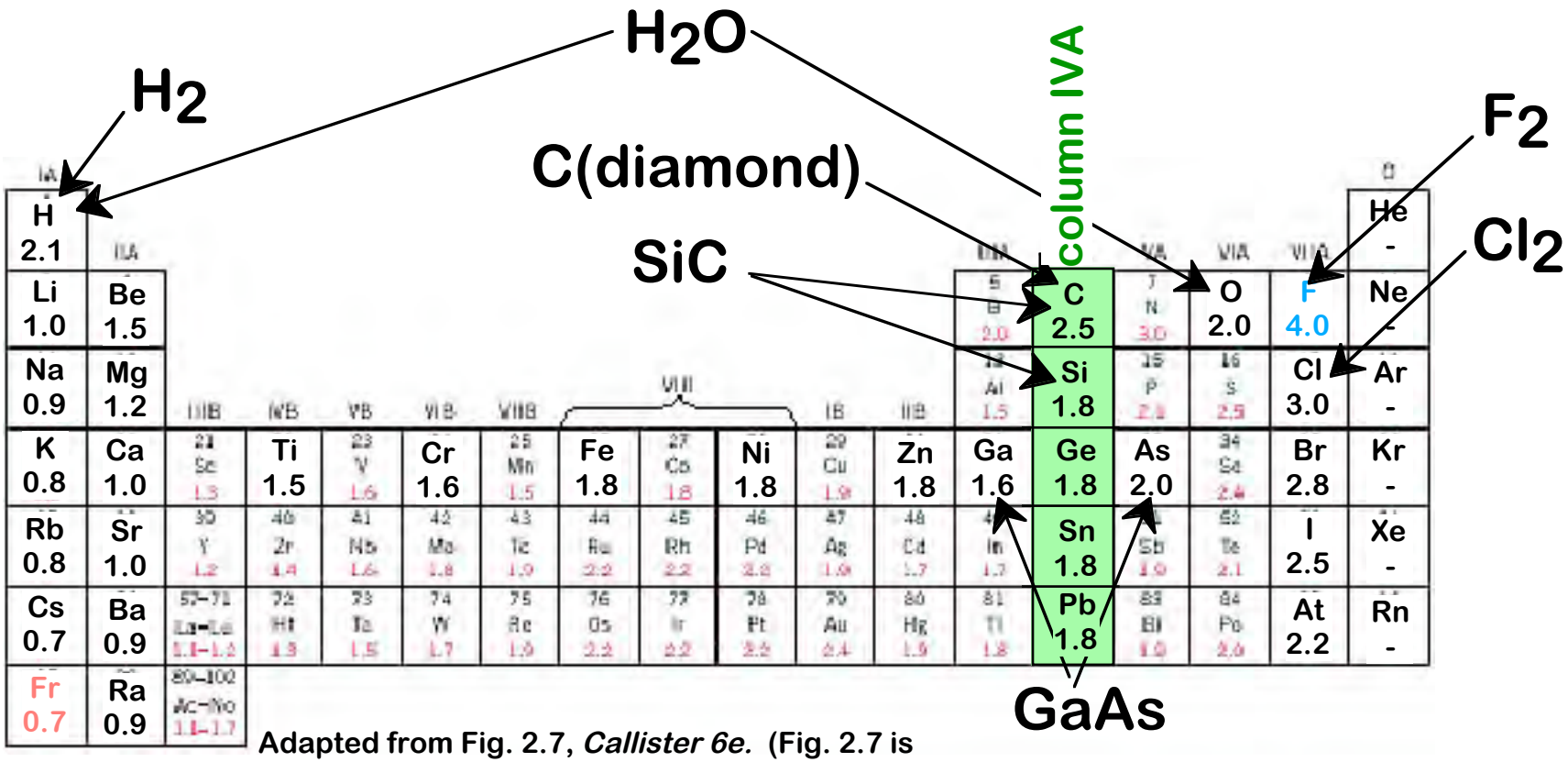
  Electronegativities are comparable.



Adapted from Fig. 2.10, *Callister 6e.*

H, atomic number 1, electronic configuration $1s^1$
C, atomic number 6, electronic configuration $1s^2 2s^2 2p^2$

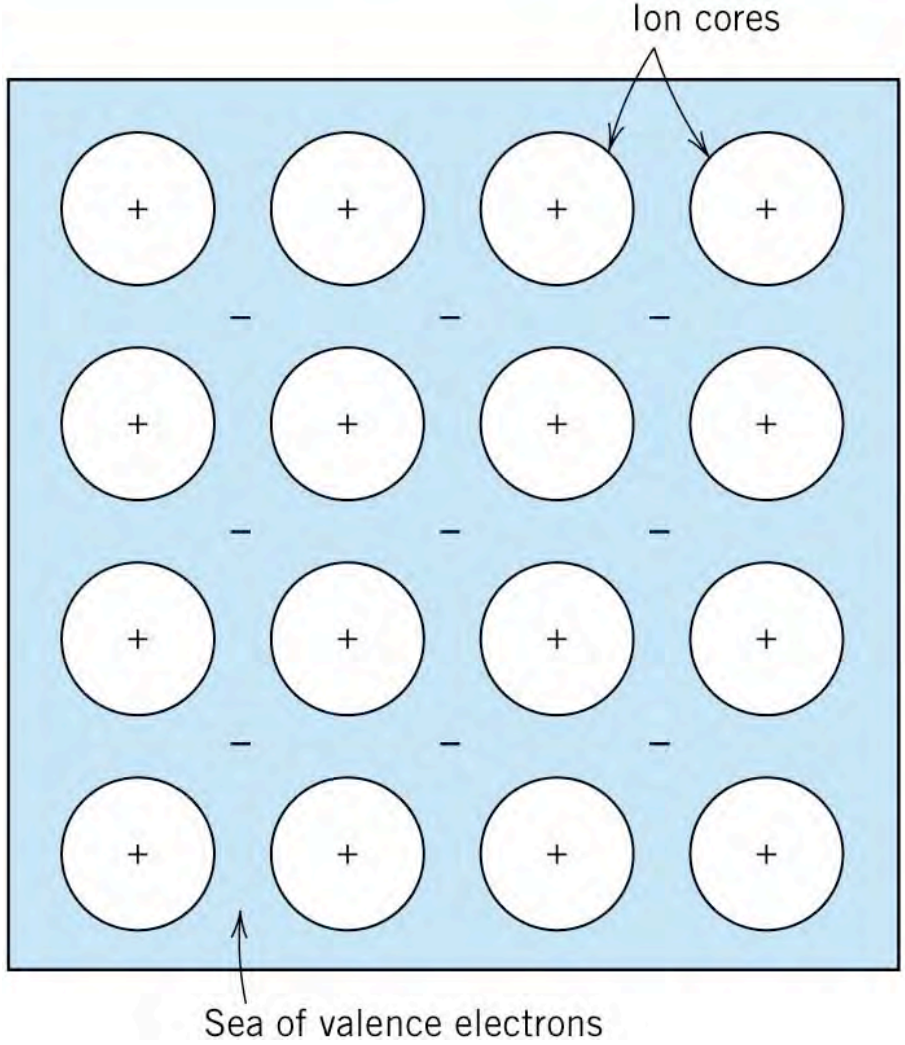
EXAMPLES: COVALENT BONDING



Adapted from Fig. 2.7, *Callister 6e.* (Fig. 2.7 is adapted from Linus Pauling, *The Nature of the Chemical Bond*, 3rd edition, Copyright 1939 and 1940, 3rd edition. Copyright 1960 by Cornell University.

- Molecules with nonmetals
- Elemental solids (RHS of Periodic Table)
- Molecules with metals and nonmetals
- Compound solids (about column IVA)

Metallic bonding



From W.D. Callister: Materials Science and Engineering: An Introduction

SUMMARY: PRIMARY BONDS

Ceramics

(Ionic & covalent bonding):

Large bond energy

large T_m
large E
small α

Metals

(Metallic bonding):

Variable bond energy

moderate T_m
moderate E
moderate α

Polymers

(Covalent & Secondary):

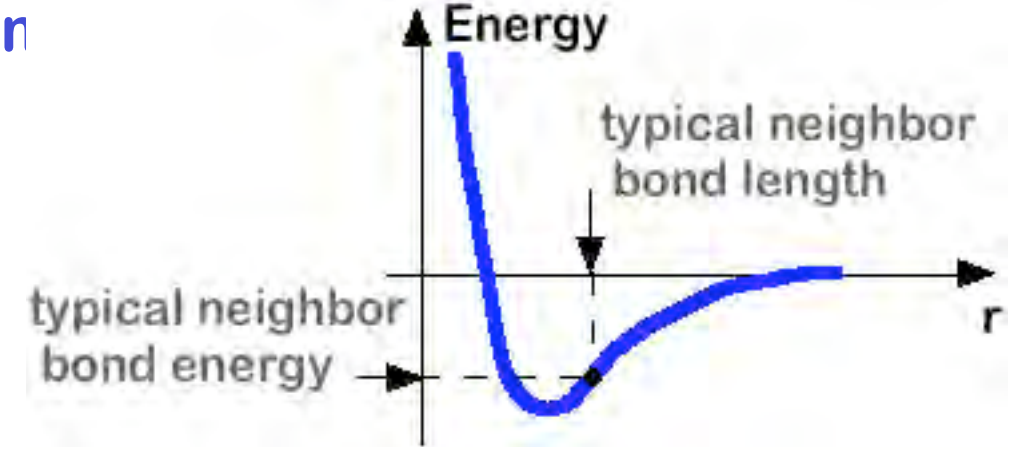
Directional Properties

Secondary bonding dominates
small T
small E
large α

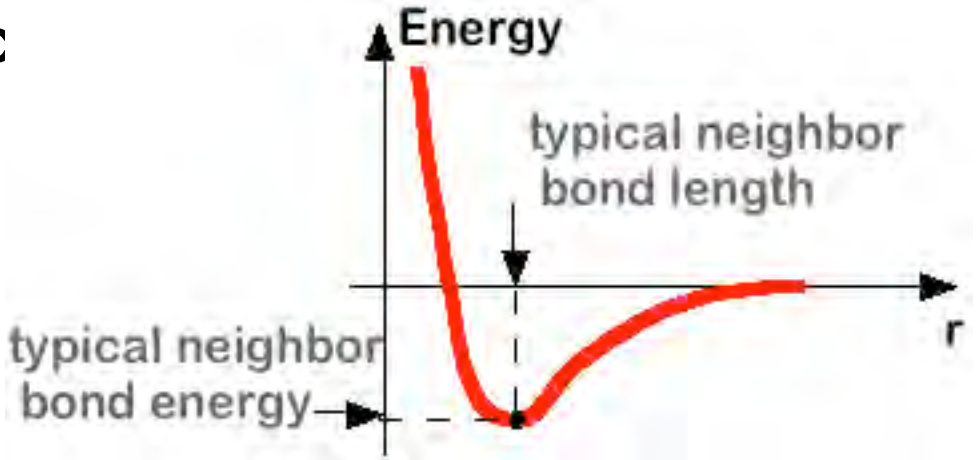
From W.D. Callister: Materials Science and Engineering: An Introduction

ENERGY AND PACKING

- Non dense, randon



- Dense, regular pac



Dense, regular-packed structures tend to have lower energy.

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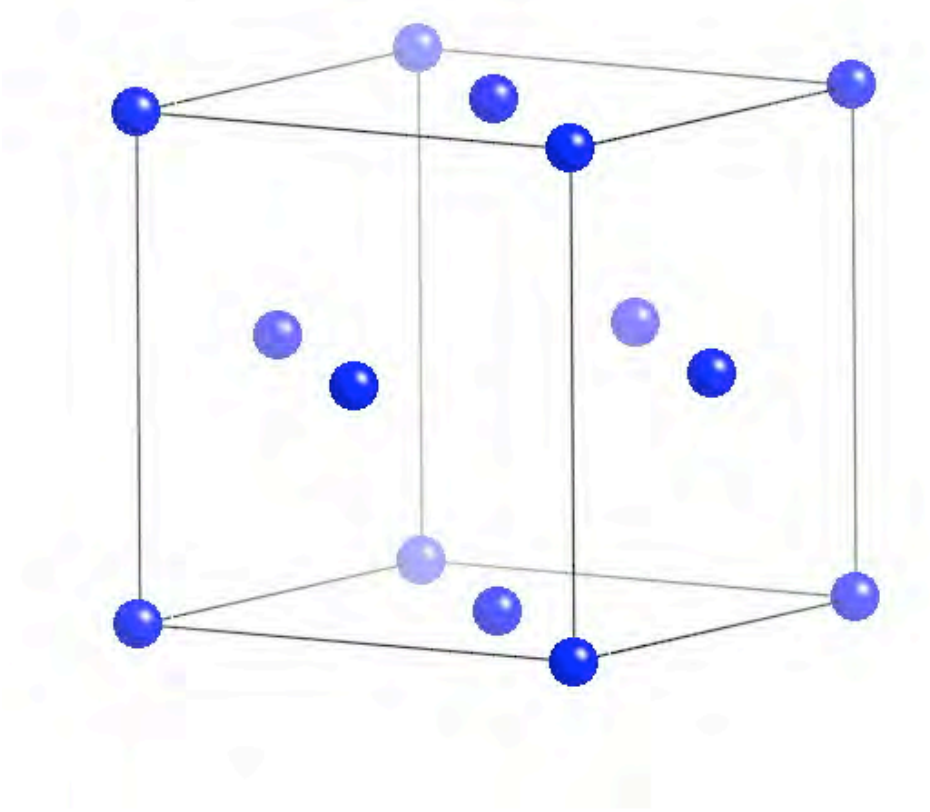
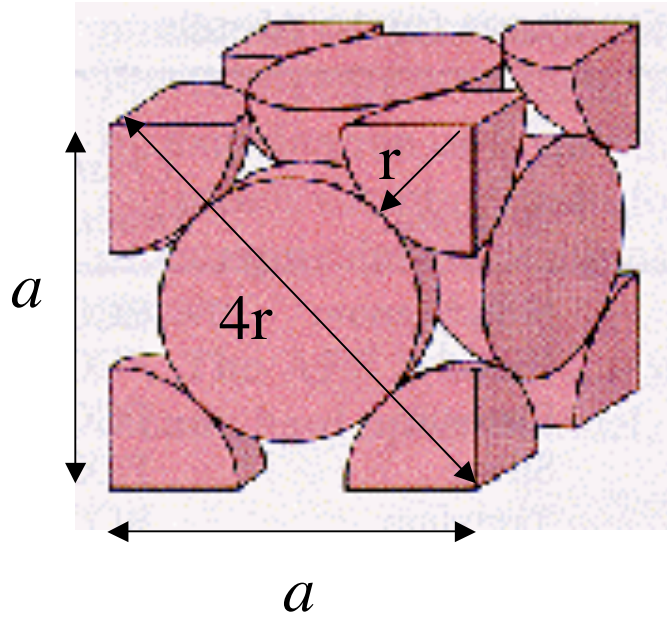
FCC

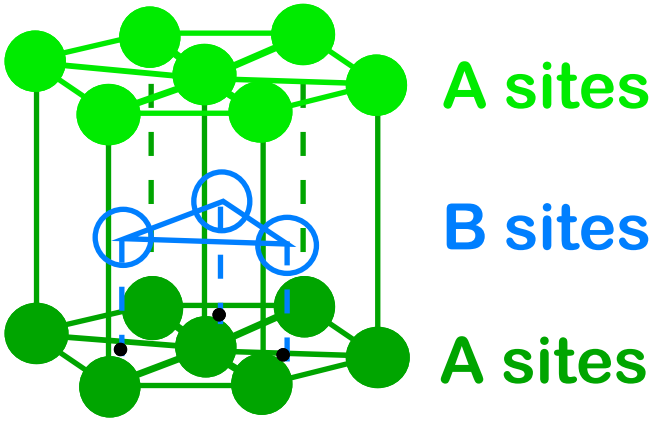




From W.D. Callister: Materials Science and Engineering: An Introduction

HEXAGONAL CLOSE-PACKED STRUCTURE (HCP)

- 3D Projection



Adapted from Fig. 3.3, *Callister 6e.*

- 2D Projection

- Coordination # = 12
- APF = 0.74

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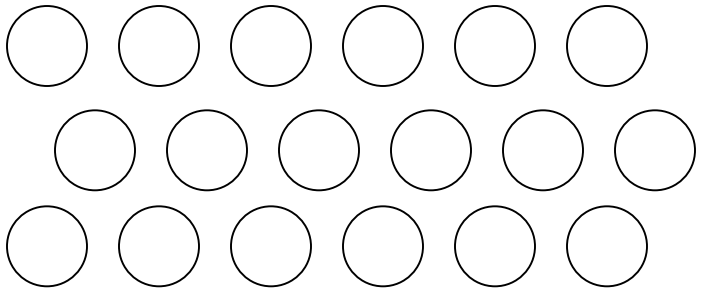
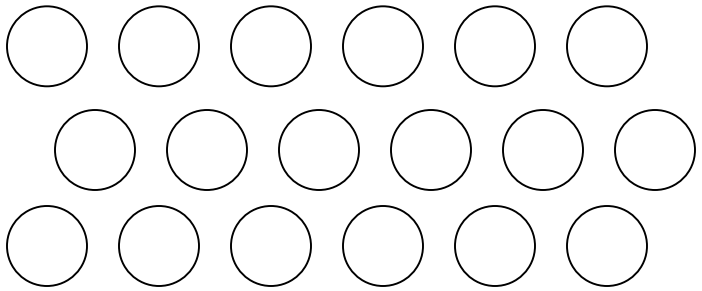
FCC and HCP close-packed lattices

- Both lattices are formed by a sequential stacking of planar layers of close packed atoms.
- Within each layer each atom has six nearest neighbors.



FCC and HCP close-packed lattices

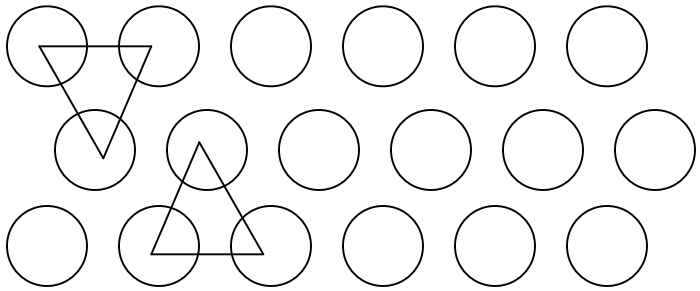


The “A” layer
all positions that are directly above the centers of the A atoms are referred to as “A” positions, whether they are occupied or not

FCC and HCP close-packed lattices

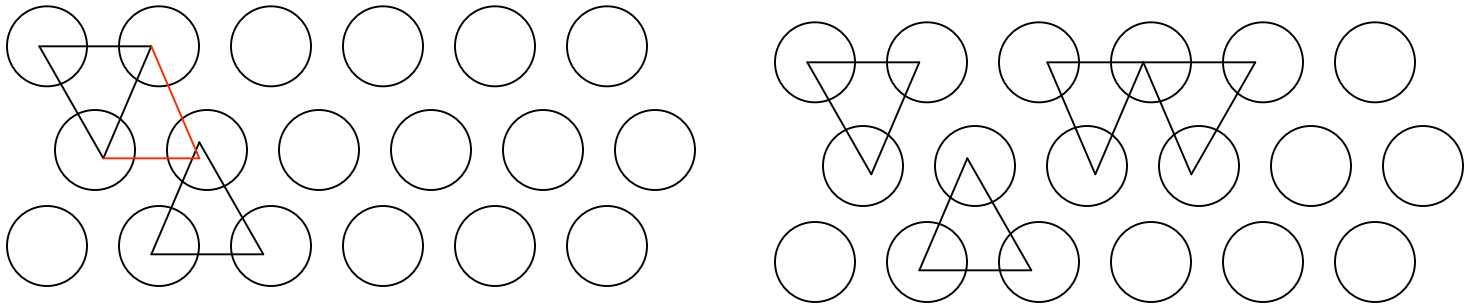
- Both FCC and HCP lattices are formed by stacking like layers on top of this first layer in a specific order to make a three dimensional lattice.
- These become close-packed in three dimensions as well as within each planar hexagonal layer.
- Close packing is achieved by positioning the atoms of the next layer in the troughs between the atoms in the “A” layer

FCC and HCP close-packed lattices



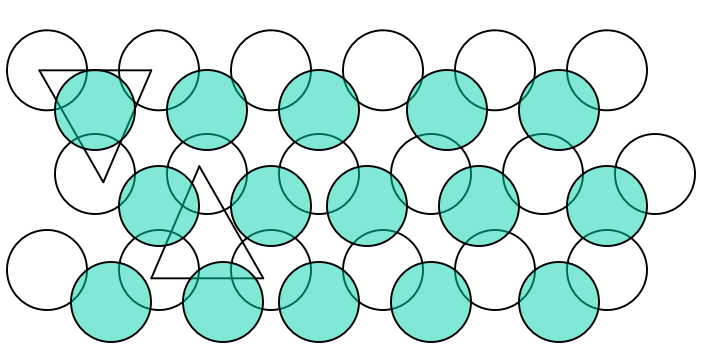
- Each one of these low positions occurs between a triangle of atoms. Some point towards the top of the page and some point towards the bottom of the page.

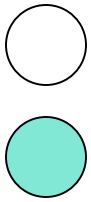
FCC and HCP close-packed lattices



- Any two of these immediately adjacent triangles are too close to be both occupied by the next layer of atoms.
- Instead the next close-packed “B” layer will fill every other triangle, which will all point in the same direction.

FCC and HCP close-packed lattices



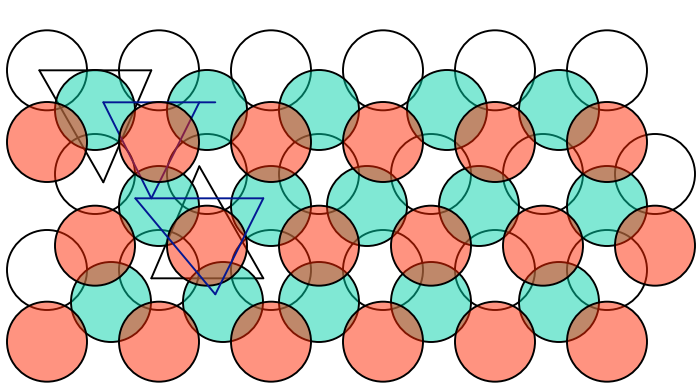


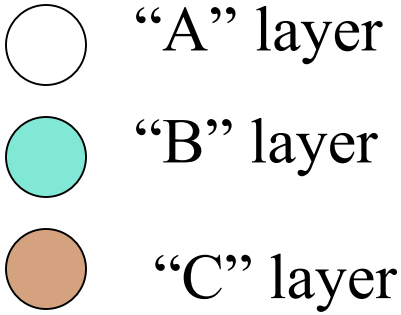
"A" layer

"B" layer

- The "B" layer is identical to the A-layer except for its slight off translation.
- Continued stacking of close-packed layers on top of the B-layers generates both the FCC and HCP lattices.

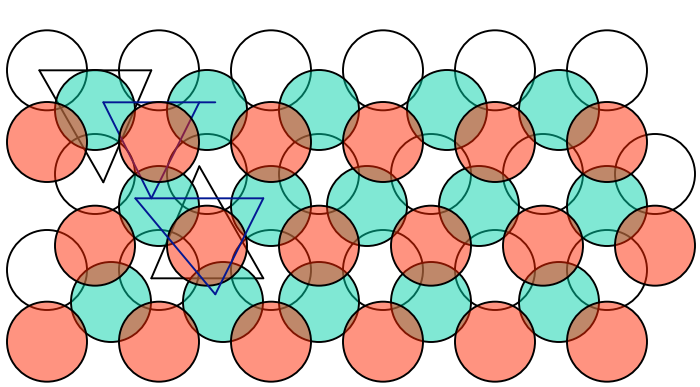
The FCC close-packed lattice

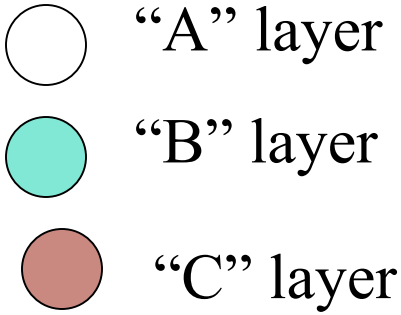




- The FCC lattice is formed when the third layer is stacked so that its atoms are positioned in downward-pointing triangles of oxygen atoms in the "B" layer.
- These positions do not lie directly over the atoms in either the A or B layers, so it is denoted as the "C" layer

The FCC close-packed lattice

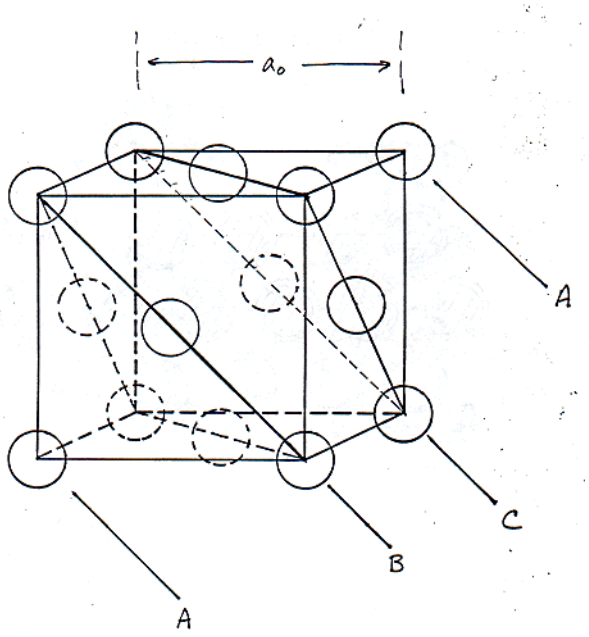




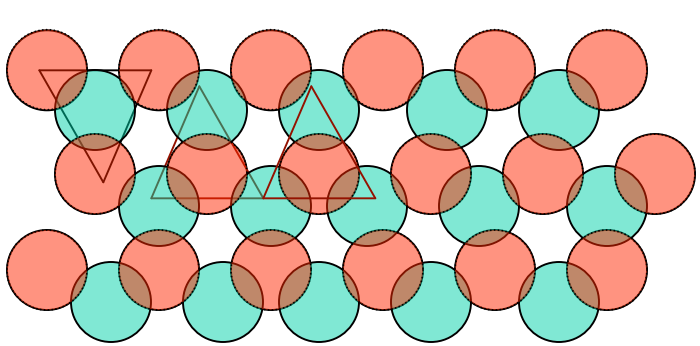
- The stacking sequence finally repeats itself when a fourth layer is added over the C atoms with its atoms directly over the A layer (the occupied triangles in the C layer again point downward) so it is another A layer.
- The FCC stacking sequence (ABCA) is repeated indefinitely to form the lattice:
- …ABCABCABCABC...

The FCC close-packed lattice

Even though this lattice is made by stacking hexagonal planar layers, in three dimensions its unit cell is cubic. A perspective showing the cubic FCC unit cell is shown below, where the body-diagonal planes of the atoms are the original A, B, C, and layers of oxygen atoms



The HCP close-packed lattice



"A" layer

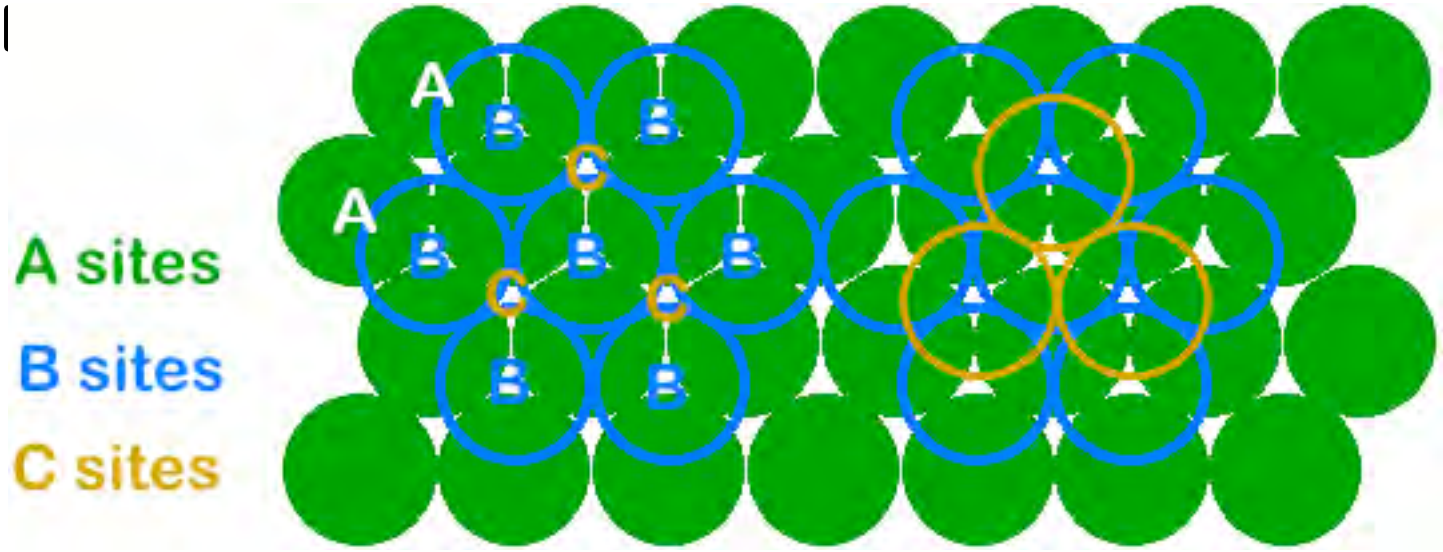
"B" layer

Repeat "A" layer

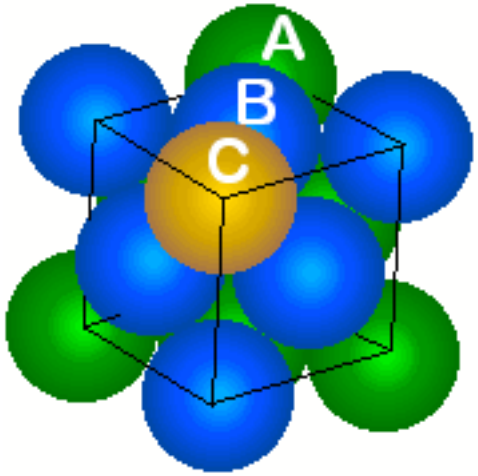
- The HCP lattice is formed when the third layer is stacked so that its atoms are positioned directly above the "A" layer (in the upward facing triangles of the "B" layer).
- The HCP stacking sequence (ABAB) is repeated indefinitely to form the lattice:
- …ABABABAB...

FCC STACKING SEQUENCE

- **ABCABC… Stacking Sequence**
- **2D I**



- **FCC Unit Cell**



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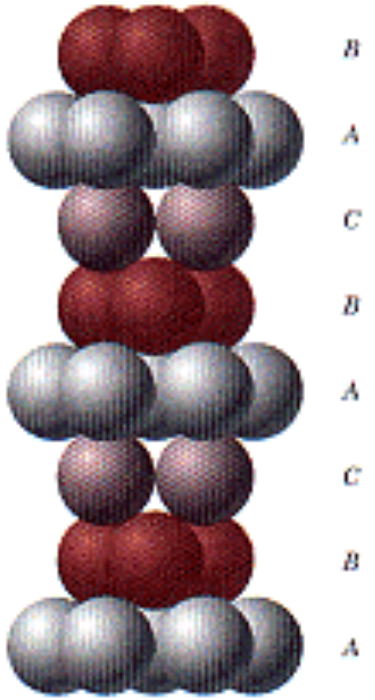
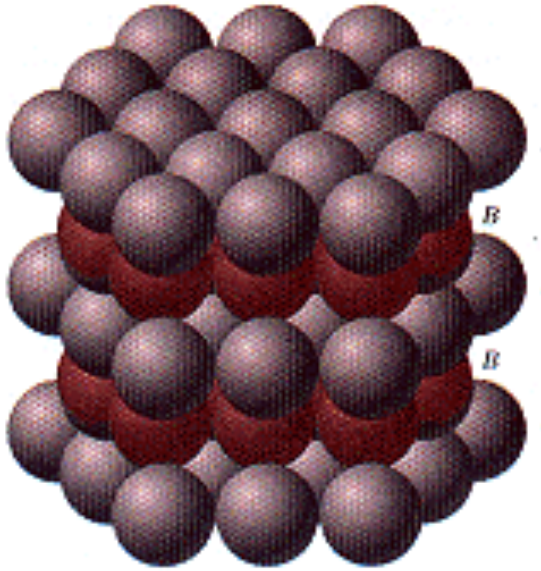
Perspective of FCC and HCP structures viewed parallel to close-packed planes





FCC (...ABCABCABC…) HCP (...ABABABAB…)

Ceramics

- Characteristics
  - Hard
  - Brittle
  - Heat- and corrosion-resistant
- Made by firing clay or other minerals together and consisting of one of more metals in combination with one or more nonmetals (usually oxygen)

Nomenclature

- The letter a is added to the end of an element name implies that the oxide of that element is being referred to:
- SiO_2 - silica $Si^{4+} + 2(O^{2-})$
- Al_2O_3 - alumina $2(Al^{3+}) + 3(O^{2-})$
- MgO - magnesia $Mg^{2+} + O^{2-}$

Charge balanced

Positively charged ions cations example: Si^{4+}, Al^{3+}, Mg^{2+}

Negatively charged ions - anions example: O^{2-}

Closed Packed Lattices

The Basis for Many Ceramic Crystal Structures

- Ionic crystal structures are primarily formed as derivatives of the two simple close packed lattices: face center cubic (FCC) and hexagonal close packed (HCP).
- Most ionic crystals are easily derived from these by substituting atoms into the interstitial sites in these structures.

Closed Packed Lattices

- The larger of the ions, generally the anion, forms the closed-packed structure, and the cations occupy the interstices.
  - We will often consider the anion to be oxygen (O^{2-}) for convenience since so many important ceramics are oxides. However, the anion could be a halogen or sulfur.
  - In the case of particularly heavy cations, such as zirconium and uranium, the cations are larger than the oxygen and the structure can be more easily represented as a closed packed arrangement of cations with oxygen inserted in the interstices.

Location and Density of Interstitial Sites

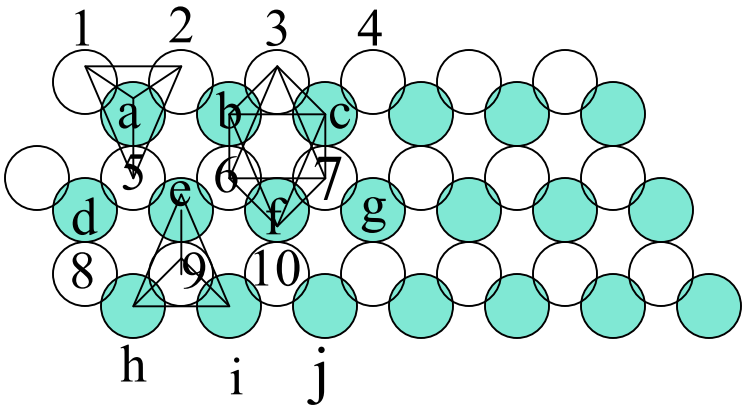
Interstitial Sites

- The interstitial sites exist between the layers in the close-packed structures
- There are two types of interstitial sites
  - tetrahedral
  - octahedral
- These are the common locations for cations in ceramic structures

Interstitial Sites

- Each site is defined by the local coordination shell formed between any two adjoining close-packed layers
  - the configuration of the third layer does not matter
  - the nearest neighbor configuration of oxygen atoms around the octahedral and tetrahedral cations is independent of whether the basic structure is derived from FCC or HCP
    - FCC and HCP have the same density of these sites

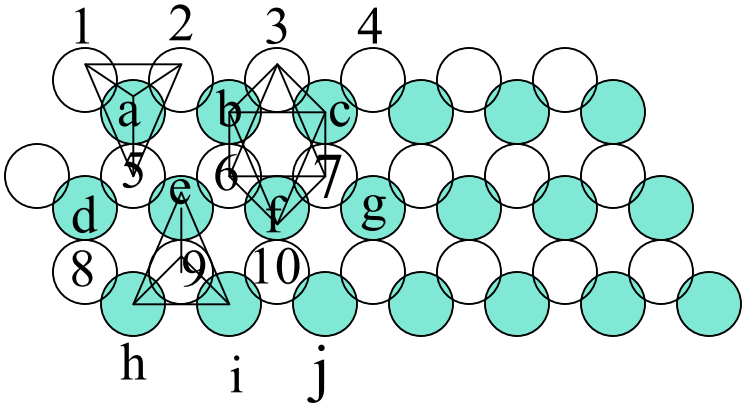
Interstitial sites



Numbers = A sites
lower case letters = B sites

- Octahedral: 3-6-7-b-c-f
  - 3 from the A layer and 3 from the B layer
  - an octahedron has eight sides and six vertices
  - the octahedron centered between these six atoms, equidistant from each - exactly half way between the two layers

Interstitial sites

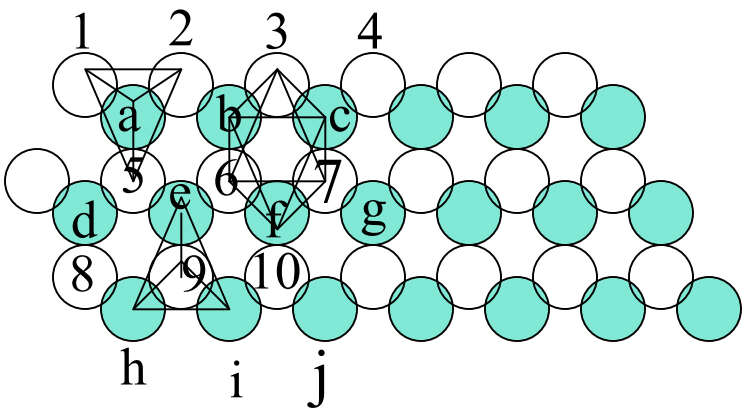


Numbers = A sites
lower case letters = B sites

The octahedral site neither directly above nor directly below any of the atoms of the A and B layers that surround the site

- The octahedral site will be directly above or below a C-layered atom (if it is FCC)
- These octahedral sites form a hexagonal array, centered exactly half-way between the close-packed layers

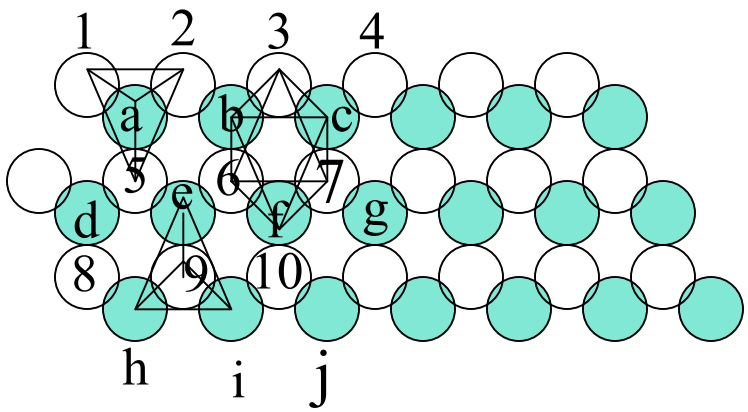
Interstitial sites



Numbers = A sites
lower case letters = B sites

- Tetrahedral: 1-2-5-a and e-h-i-9
  - 1 negative tetrahedron
  - 1 positive tetrahedron
  - three of one layer and one of the second layer
  - 3A and 1B – one apex pointing out of the plane of the board
  - 3B and 1 A – one apex pointing into the plane of the board

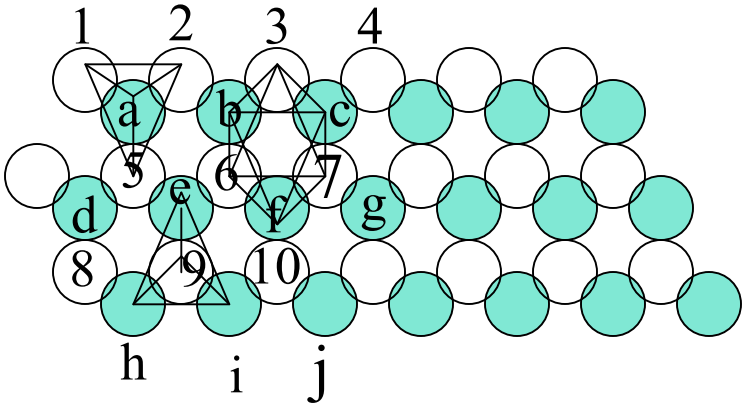
Interstitial sites



Numbers = A sites
lower case letters = B sites

- Tetrahedral: 1-2-5-a and e-h-i-9
  - For both tetrahedral sites the center of the tetrahedron is either directly above or below an atom in either the A or B layers
  - The geometric centers are not halfway between the adjacent oxygen planes but slightly closer to the plane that forms the base of the tetrahedron

Interstitial sites

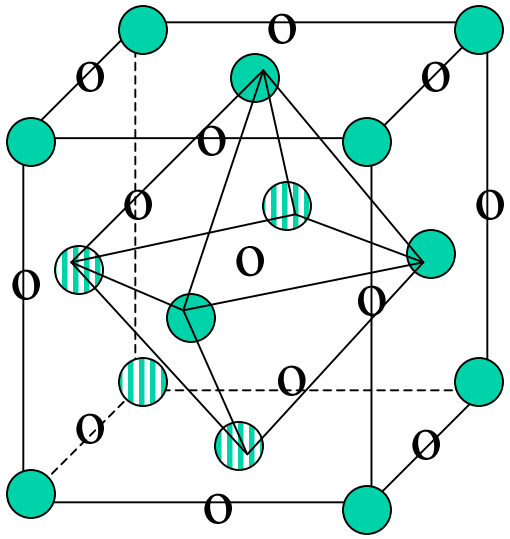


Numbers = A sites

lower case letters = B sites

Octahedral sites in the FCC Unit Cell

One octahedral site halfway along each edge and one at the cube center



The FCC cell contains four atoms

six faces that each contribute one half and atom

eight corners that each contribute one-eighth an atom

FCC cell contains four octahedral sites

12 edges each with one quarter of a site

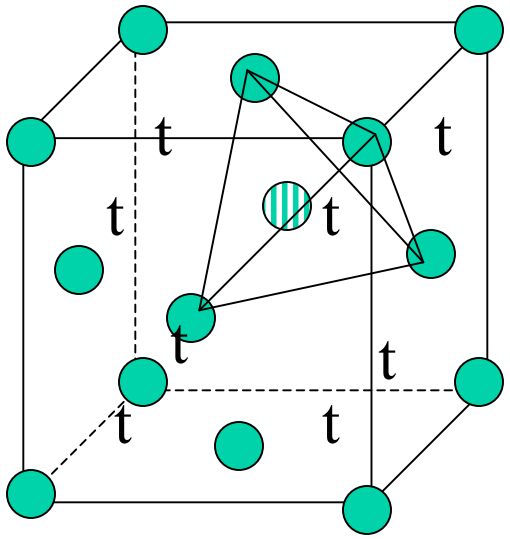
one site in the center

The ratio of octahedral sites to atoms
Is 1:1

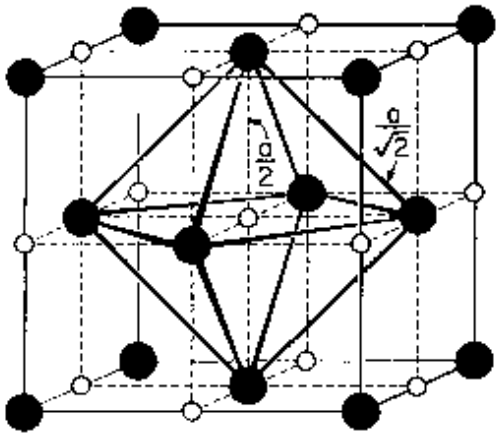
Tetrahedral sites in the FCC Unit Cell

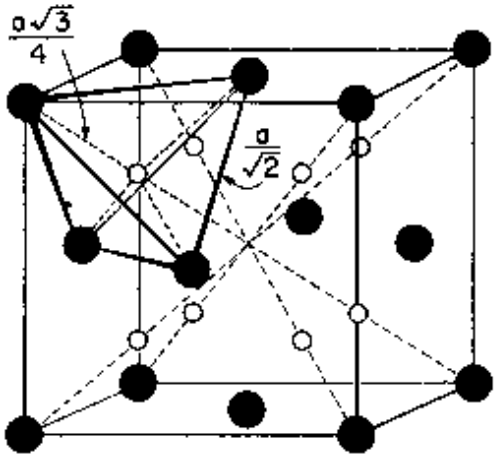
One tetrahedral site inside each corner

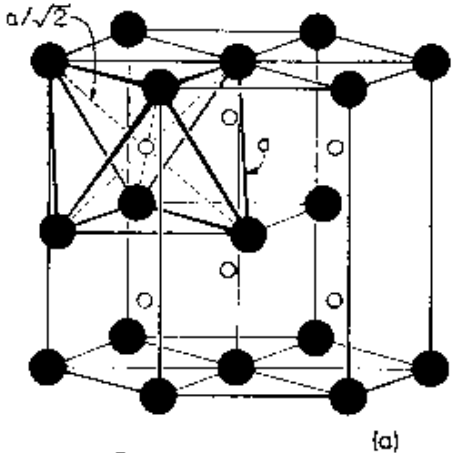
Eight tetrahedral sites

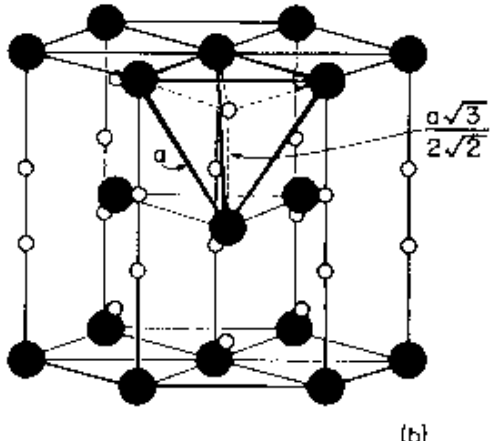


The ratio of tetrahedral sites to atoms Is 2:1


a/2
a/√2


a√3/4
a/√2


a/√2
a
(a)


a√3/2√2
a
(b)

General Structural formula for close-packed structures

- $T_{2n}O_nX_n$

T – Tetrahedral sites

O – Octahedral sites

X – Anions

Example:

$A_{2n}B_nX_n$
A = tetrahedral sites
B = octahedral sites
X = anions

$MgAl_2O_4$
If fully occupied $A_8B_4X_4$

Mg in tetrahedral sites - 1/8 of the sites occupied
Al in octahedral sites - 1/2 of the sites occupied

Linus Pauling

- Nobel Prize in Chemistry 1954
  - "for his research into the nature of the chemical bond and its application to the elucidation of the structure of complex substances"
- Nobel Peace Prize 1962
- Born in 1901 and died in 1994
- We may use Pauling's rules to predict the tendency for a specific compound to form a specific crystal structure

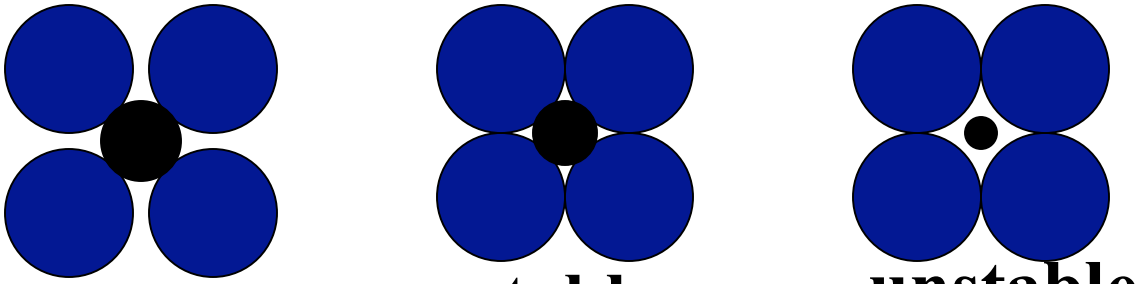
Pauling's Rules

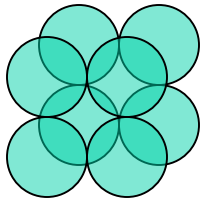
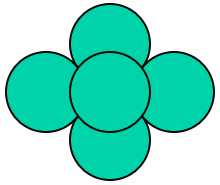
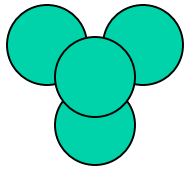
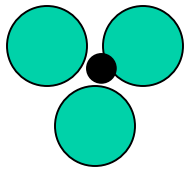
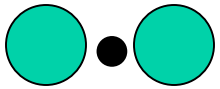
- Pauling's rules are based on the geometric stability of packing for ions of different sizes and simple electrostatic stability arguments.
  - These geometric arguments treat the ions as hard spheres which is an over implication

Ionic crystal radii

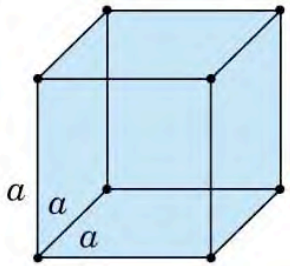
- Ionic radii (as defined by interatomic spacings) do vary from compound to compound
  - they tend to vary most strongly with the valance state of the ion and the number of nearest neighbor ions of the opposite charge
    - We may consider an ionic radius to be constant for a given valance state and nearest-neighbor coordination number

Pauling's Rule 1

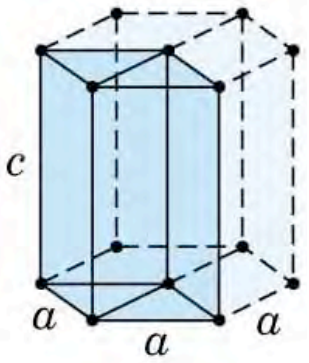


CN	Disposition of ions about central atom	r_c/r_a	
8	corners of a cube	≥ 0.732	
6	corners of an octahedron	≥ 0.414	
4	corners of a tetrahedron	≥ 0.225	
2	corners of a triangle	≥ 0.155	
1	linear	≥ 0	

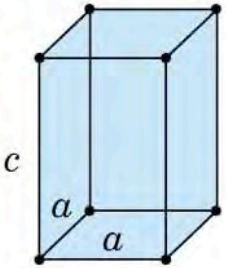
When the radius ratio is less than this geometrically determined critical value the next lower coordination is preferred



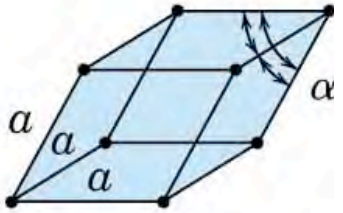
Cubic $a = b = c$ $\alpha = \beta = \gamma = 90^o$



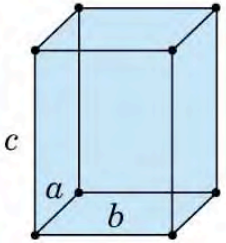
Hexagonal $a = b \neq c$ $\alpha = \beta = 90^o, \gamma = 120^o$



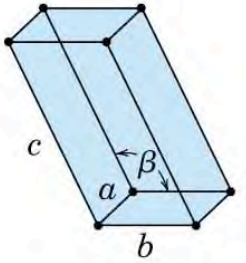
Tetragonal $a = b \neq c$ $\alpha = \beta = \gamma = 90^o$



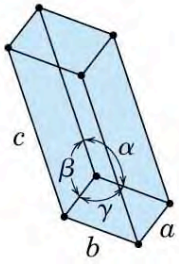
Rhombohedral $a = b = c$ $\alpha = \beta = \gamma \neq 90^o$



Orthorhombic $a \neq b \neq c$ $\alpha = \beta = \gamma = 90^o$



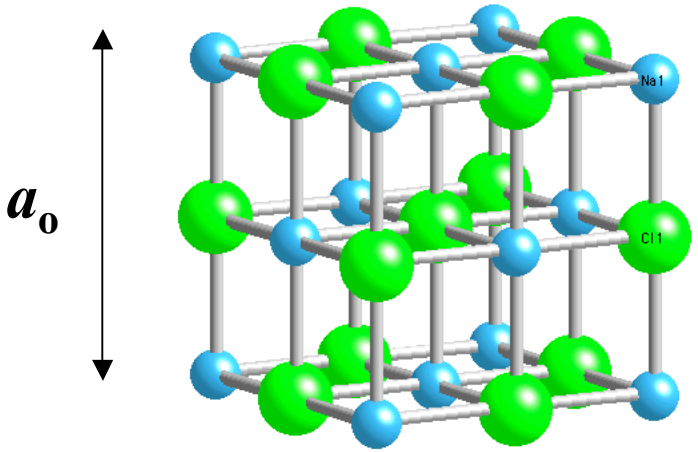
Monoclinic $a \neq b \neq c$ $\alpha = \gamma = 90^o, \neq \beta$



Triclinic $a \neq b \neq c$ $\alpha \neq \beta \neq \gamma$

Rocksalt

- NaCl, KCl, LiF, MgO, CaO, SrO, NiO, CoO, MnO, PbO
  - for all of these the anion is larger than cation and forms the basic FCC lattice



The lattice parameter of the cubic unit cell is "a_o" and each unit contains 4 formula units

Calculating density

- NiO -rocksalt structure

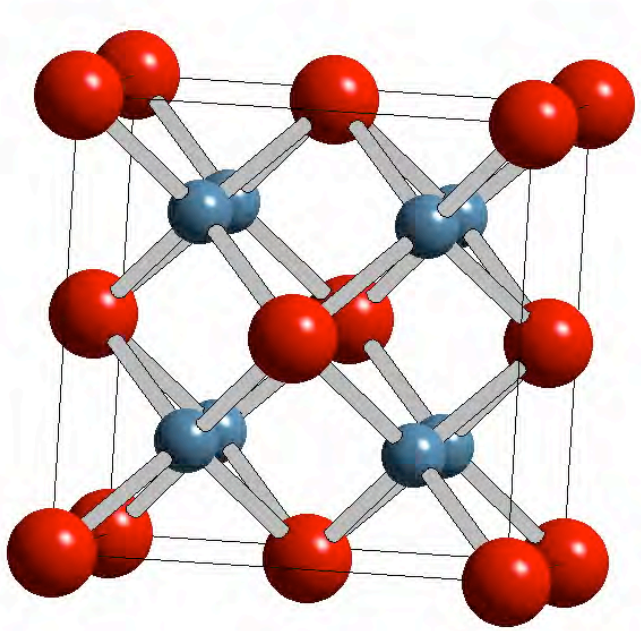
a = 4.1771 Å space group *Fm3m*

Atom	Ox	Wy	x	y	z
Ni	+2	4a	0	0	0
O	-2	4b	0.5	0.5	0.5

$$\rho = \frac{n'(\sum M_{Ni} + \sum M_O)}{V_{unit\ cell} N_{AV}} = \frac{4(58.69 + 15.999)\ \text{g/mol}}{((4.1771 \times 10^{-8}\ \text{cm})^3)(6.022 \times 10^{23}\ \text{atom/mol})}$$

$$\frac{\text{g/mol}}{\text{cm}^3 \times \text{molecules/mol}} = 6.81\ \text{g/cm}^3$$

Anti-fluorite



Li_2O
a = 4.619 Å
space group *Fm3m* (225)

atom	x	y	z	Wy
O	0	0	0	4a
Li	1/4	1/4	1/4	8c