Haupt/Masterstudiengang Physik
Methoden moderner Röntgenphysik II:
Streuung und Abbildung
SS 2014

Biology II: MX – The Method

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1/7/2014
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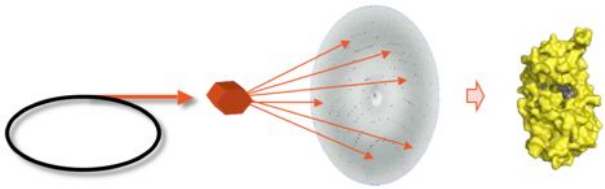
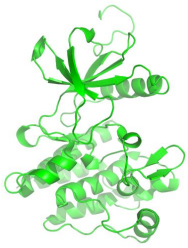
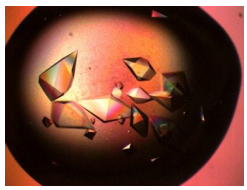
EMBL

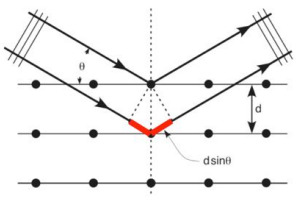
Recap

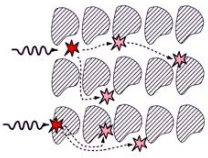
- MX
- Proteins
- Protein crystals
- Recombinant production of proteins
- Radiation damage and cryogenic cooling
- Bragg's law

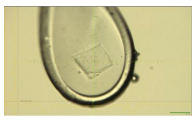












$2\,d\,\sin\Theta = n\,\lambda$

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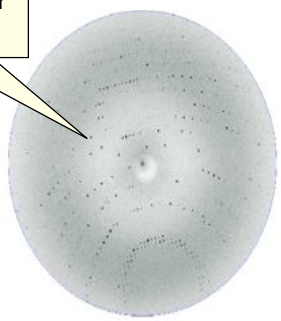
Today

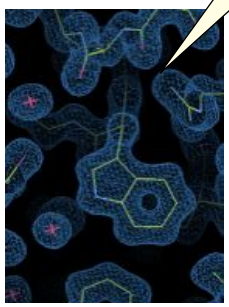
- Electron densities and structure factors / real space and reciprocal space
  - Fourier transforms
  - Argand diagrams
- The crystallographic phase problem
- Symmetry in real and reciprocal space

- Solving the crystallographic phase problem
- Anomalous scattering from a crystallographer's point of view

Calculating Electron density

Intensity I_{hkl} for some set of planes with Miller indices hkl





Electron density for all points in the unit cell

$$\rho_{xyz} = \sum_{hkl} |F_{hkl}| e^{-i\varphi_{hkl}} e^{-2\pi i(hx+ky+lz)}$$

Electron density at some point *xyz* in space

Sum over all Bragg reflections

Structure Factor Amplitude for each reflection hkl. $F_{hkl} \approx \sqrt{I_{hkl}}$

Structure Factor Phase for each reflection hkl.

Phase shift depending on hkl and position in space

Fourier Transforms

Fourier Transform

- Any periodic function can be considered as a sum of sinusoidal waves.

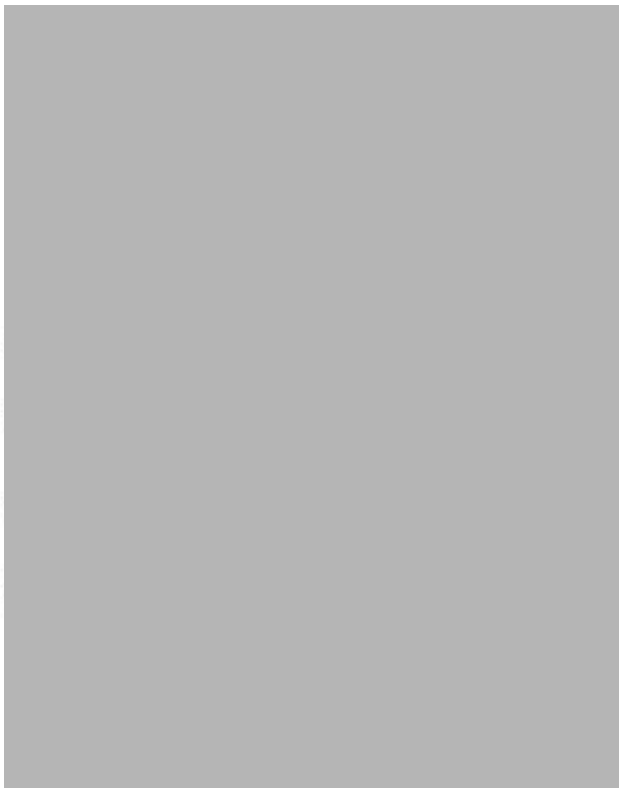
$$f(x) = \sum_{n=0}^{\infty} A_n \cos(nx) + B_n \sin(nx)$$

- Or:

$$f(x) = \sum_{n=0}^{\infty} C_n \cos(nx - \varphi_n)$$

- The above sum is called a 'Fourier series'
- For higher n (i.e. shorter period) finer features are generated.

http://mathworld.wolfram.com/FourierSeries.html



Fourier Transform

- Any periodic function can be considered as a sum of sinusoidal waves.

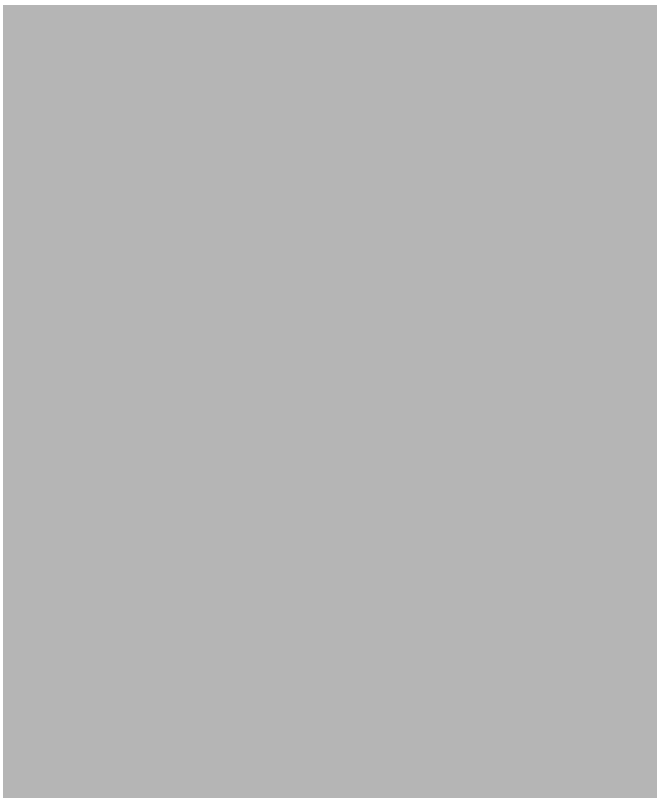
$$f(x) = \sum_n A_n \cos(nx) + B_n \sin(nx)$$

- To calculate the coefficients:

$$A_n = \int_{period} f(x)\cos(nx)dx$$

$$B_n = \int_{period} f(x)\sin(nx)dx$$

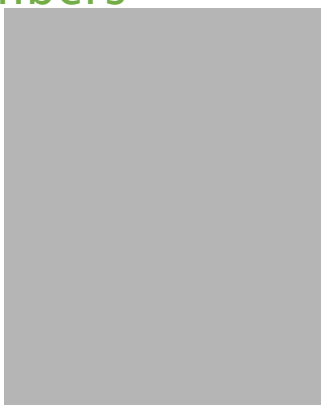
- The integral can be seen as the projection of f(x) onto cos(nx) – 'scalar product'



Fourier Transform with complex numbers

- Fourier series using real numbers / function:

$$W(x) = \sum_{n=0}^{\infty} A_n \cos(nx) + B_n \sin(nx) = \sum_{n=0}^{\infty} C_n \cos(nx - \varphi_n)$$



- Periodic functions can be written more elegantly using complex numbers instead of sine and cosine functions

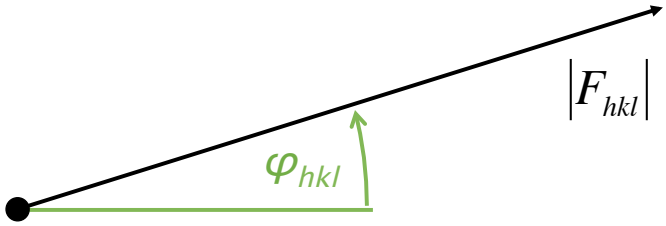
$$W(x) = \sum_{n=0}^{\infty} A_n \exp\left[i(2\pi nx + \varphi_n)\right] = \sum_{n=0}^{\infty} F_n \exp\left[i(2\pi nx)\right] = \sum_{n=0}^{\infty} |F_n| \exp\left[i\varphi_n\right] \exp\left[i(2\pi nx)\right]$$

Complex number

Electron density from Diffraction Data

- To evaluate the electron density in the crystal, we need to know the 'phase' φ_{hkl} for each reflection / complex structure factor

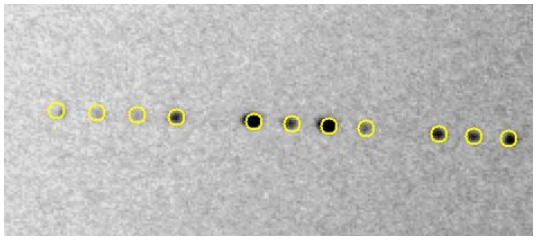
$$\rho_{xyz} = \sum_{hkl} |F_{hkl}| \exp(-i\varphi_{hkl}) \exp(-2\pi i(hx + ky + lz))$$

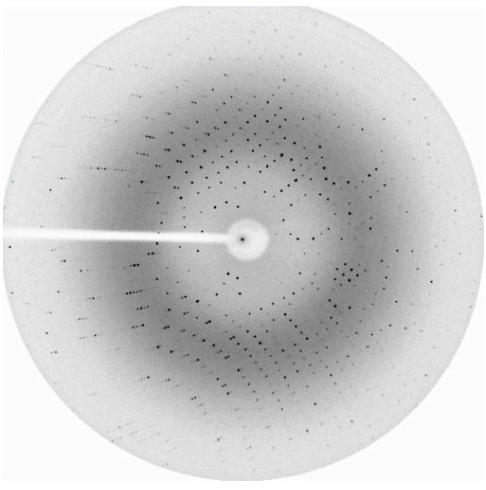


The Crystal as a Fourier-Analyzer

$$\rho_{xyz} = \sum_{hkl} |F_{hkl}| \exp(-i\varphi_{hkl}) \exp(-2\pi i(hx + ky + lz))$$

$$|F_{hkl}| = \sqrt{I}_{hkl}$$





Electron density from Diffraction Data

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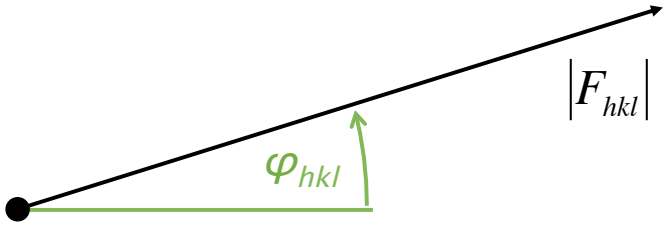

The Phase Problem - graphically

$$F_{hkl} = |F_{hkl}| e^{i\varphi}$$

h	k	l	$\lvert F_{hkl} \rvert$	φ_{hkl}
0	20	35	4980.5	???
0	20	36	6906.6	???
0	20	37	8302.3	???
0	20	38	63209.5	???
0	20	39	459.6	???
0	20	40	1017.4	???
0	20	41	5.6	???
0	20	42	33.8	???
0	20	43	4545.7	???
0	20	44	0.2	???
0	20	45	808.8	???
0	20	46	72.2	???
0	20	47	792.3	???
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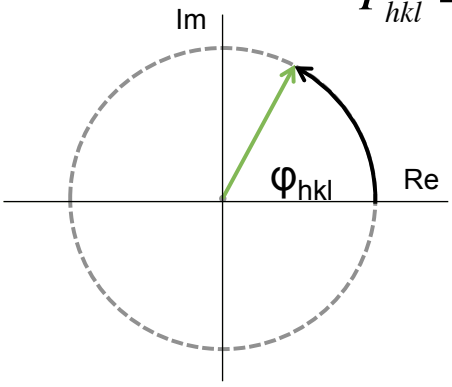


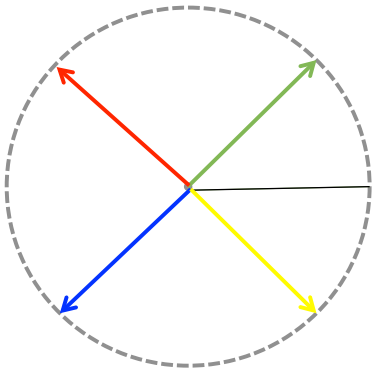
Argand Diagram

- The 'structure factor amplitude' |F| can be anything positive
- The 'phase angle' φ varies between 0 and 360°.
- The pair of |F| and phi can be collected in a complex number

http://demonstrations.wolfram.com/ArgandDiagram/

Let me do it …

- Knowing the phase within 90 deg. is good enough
- 4 possibilities: 45, 135, 225, 315
- A piece of cake for our 1000 CPU cluster

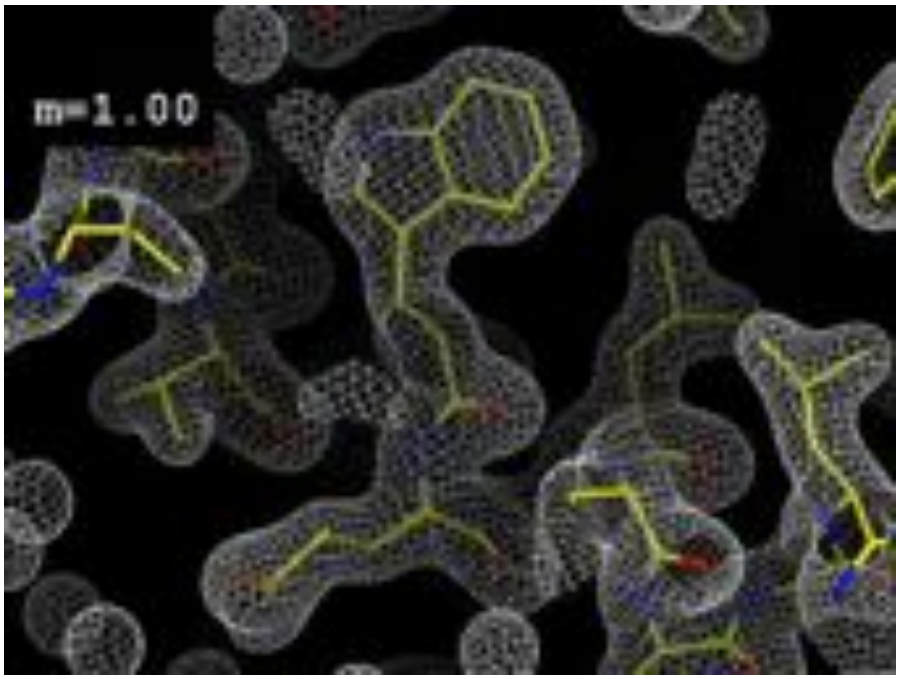


- 200 reflections
- $4^{200} = 2.5 * 10^{120}$ possibilities

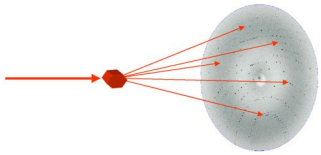
- Number of atoms in the universe 10^{80}
- Time since big bang $4.3*10^{17}$ sec
- 1 set per CPU cycle of 3 GHz = $12.9*10^{26}$ trials since the beginning

Phases

http://ucxray.berkeley.edu/~jamesh/movies



Diffraction from a Crystal



$$F_{hkl} = \sum_{j=1}^{N} f_j e^{2\pi i(hx_j + ky_j + lz_j)}$$

Complex Structure Factor for reflection hkl

Sum over all atoms in the unit cell

Scattering Factor of atom j. Depends on Z, B (and hkl)

Phase difference for the set of Bragg planes corresponding to reflection hkl

$$I_{hkl} = F_{hkl}^2$$

Real number

Source for Structure Factors: Stout and Jensen

Fourier Transforms

- Structure factors and electron density are related by FT and FT^{-1}

Fourier Transform

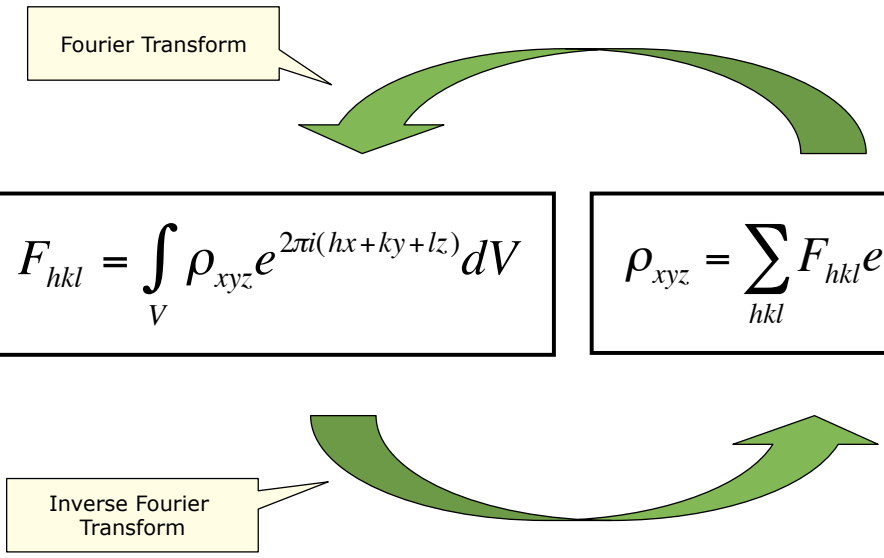
$$F_{hkl} = \sum_{j=1}^{N} f_j e^{2\pi i(hx_j + ky_j + lz_j)}$$

$$\rho_{xyz} = \sum_{hkl} F_{hkl} e^{-2\pi i(hx + ky + lz)}$$

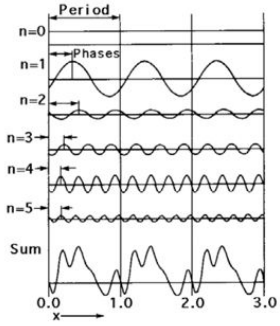
Inverse Fourier Transform

Fourier Transforms

- Equation for deriving diffraction from a crystal can be rewritten in terms of electron density



Electron density and the Fourier transform



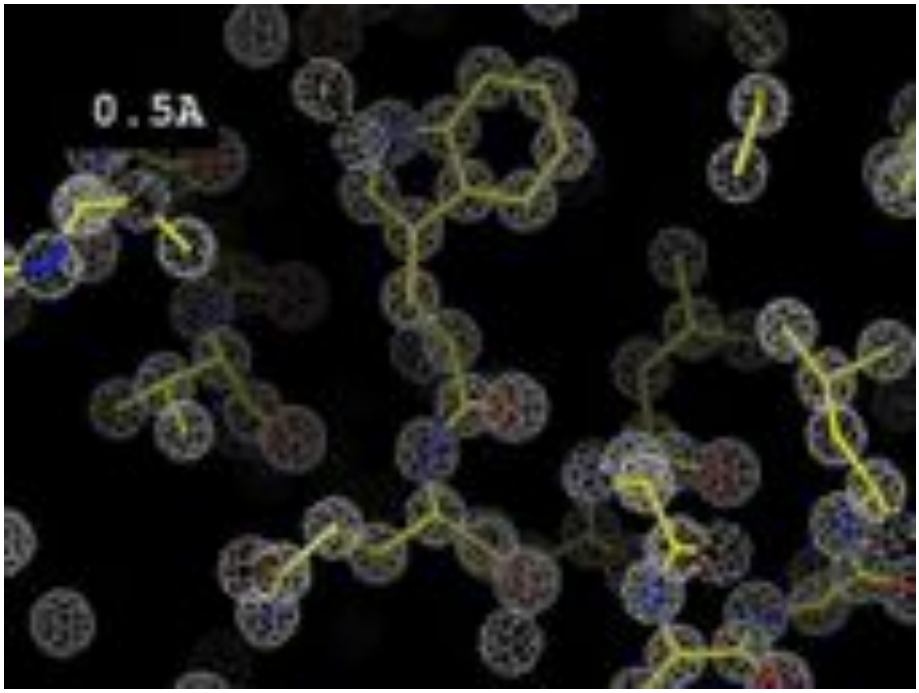
- The calculation of the electron density can be considered as adding 'base waves' with specified amplitude (structure factor amplitude) and phase:

$$\rho_{xyz} = \sum_{hkl} F_{hkl} e^{-2\pi i(hx+ky+lz)} = \sum_{hkl} |F_{hkl}| e^{-i\varphi_{hkl}} e^{-2\pi i(hx+ky+lz)}$$

- Technically, this operation corresponds to an ‘inverse’ Fourier transform.
- The higher the resolution / higher the diffraction angle / smaller the lattice plane distance, the shorter is the wavelength of the corresponding hkl-term. Shorter wavelength correspond to finer features.

Resolution

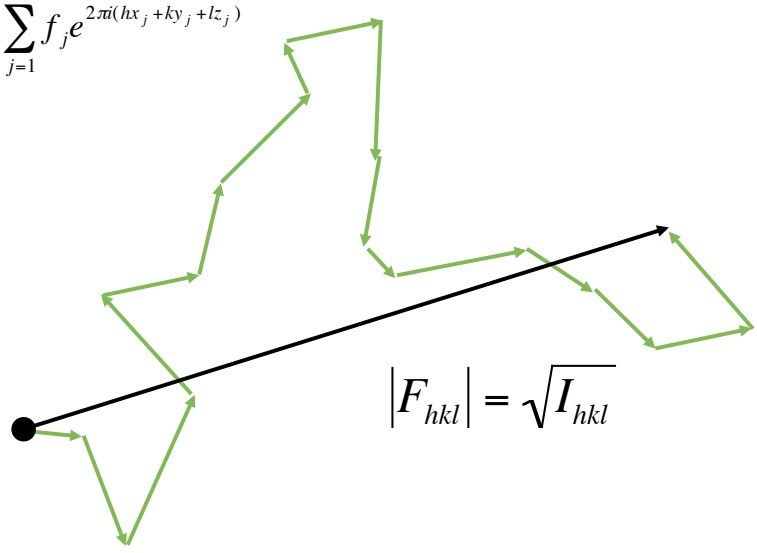
http://ucxray.berkeley.edu/~jamesh/movies



Diffraction from a protein (in vectors)

- To calculate the structure factor for a certain hkl based on the knowledge of the positions of the atoms in the unit cell, we sum over all atoms (here: 17 atoms):

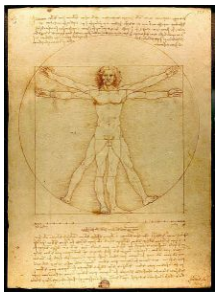
$$F_{hkl} = \sum_{j=1}^{N} f_j e^{2\pi i(hx_j + ky_j + lz_j)}$$

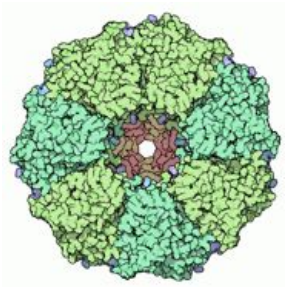


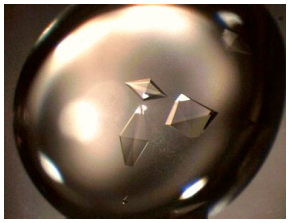
$$\left|F_{hkl}\right| = \sqrt{I_{hkl}}$$

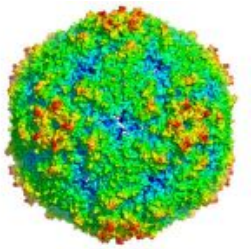
Symmetry

- An object is symmetric if, after some operation has been carried out, the result is indistinguishable from the original object [Blow2007].







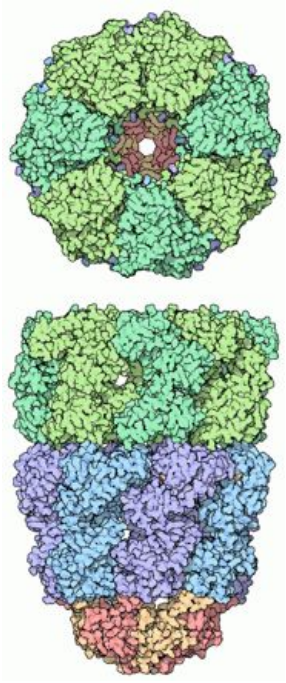


Rotational Symmetry

- After a rotation of 60 deg. about its centre, the picture of the snowflake is indistinguishable from the picture before the operation.
- The snowflake possesses 6-fold symmetry

http://commons.wikimedia.org/wiki/File:Snowflake6.png

Other n-fold symmetries

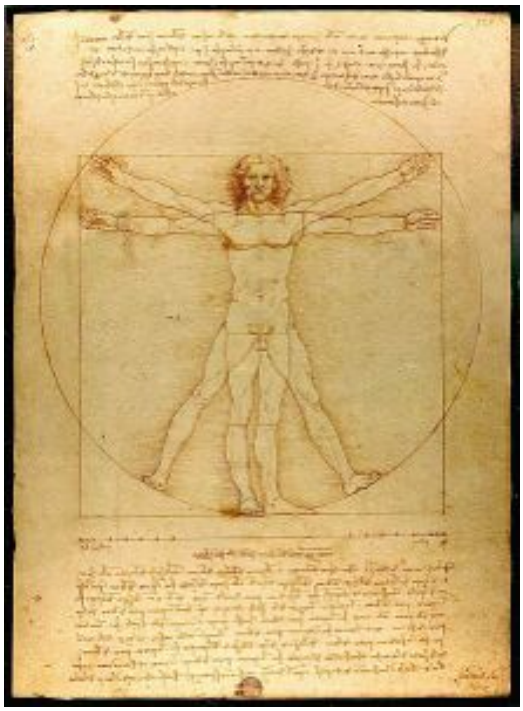


GROEL. http://www.rcsb.org/pdb/101/motm.do?momID=32

Mirror symmetry

- One version of the object is related to the other one by reflection through a plane (the mirror)

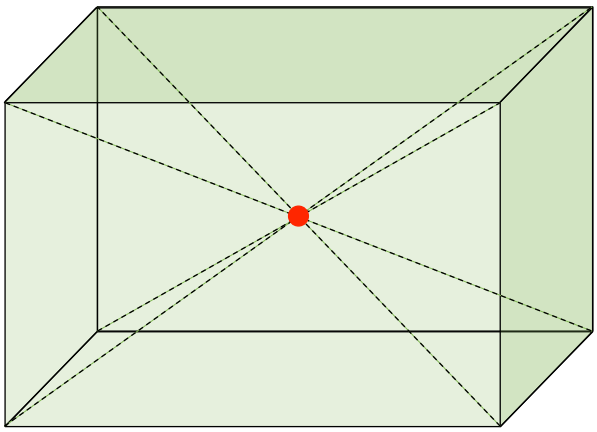
http://commons.wikimedia.org/wiki/File:Schmetterling_1a_neucc.jpg



http://commons.wikimedia.org/wiki/File:Da_Vinci_Vitruve_Luc_Viatour.jpg

Centrosymmetry

- When the object created by moving every point in the object along the line joining it to a chosen centre point, and continuing aloing this line unti it is equally far from the centre on the other side, is identical to the original object, the object is centrosymmetric, or symmetric under inversion in the point.
- Example: reactangular block



Centrosymmetry and Biology

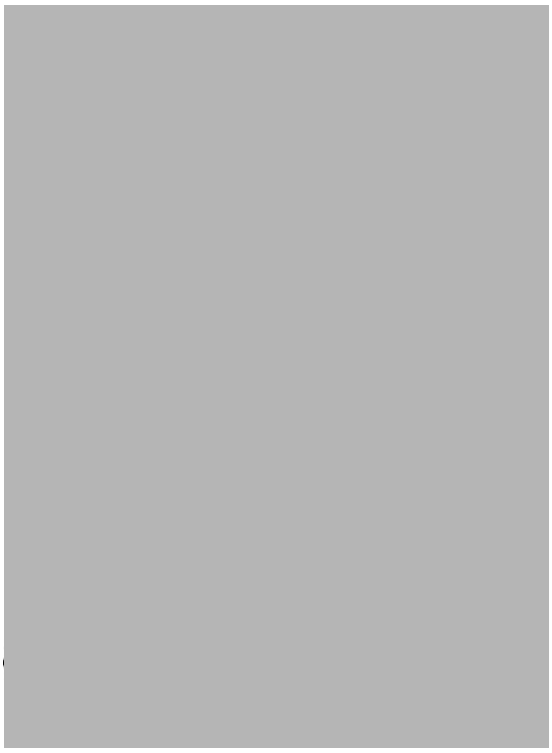
- **Would you expect centrosymmetric structures in biology?**

- **No, because natural proteins are made (almost exclusively) of L-amino acids. By inversion, one would obtain D-amino acids.**

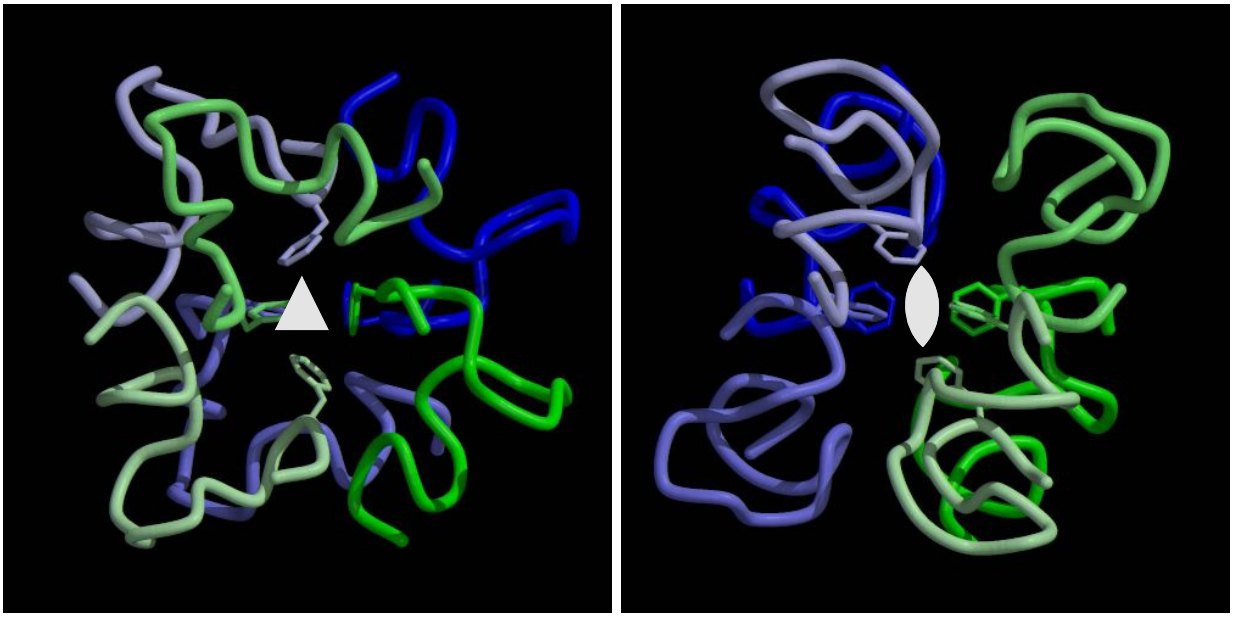
Point Groups

- groups of symmetries that leave one point fixed are called 'point groups'
- rotations and mirrors are possible
- There are a total of 32 crystallographic point groups
- Local assemblies of objects (oligomers of protein molecules) can follow point group symmetry
- Examples: 1, 4, 422, 2, 222, 6, 622, 3, 322, 23, 432 [Blow2007, p27]
- These are the 11 enantiomorphic point groups accessible for chiral objects (such as proteins)

Goodsell (2000) Annu. Rev. Biophys. Biom. 29:105-153



Mersacidin: NCS



Some objects ...

Symmetry of a cube

Figure from Glusker1994

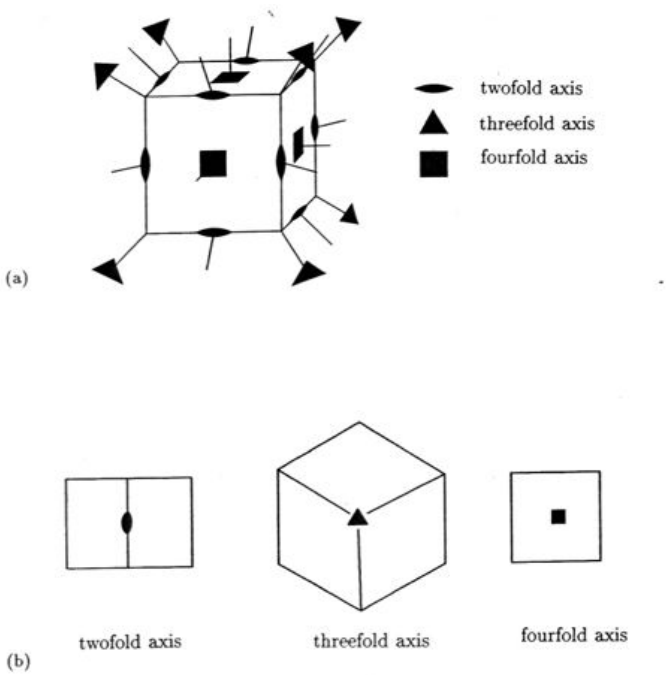


FIGURE 4.7. The symmetry of a cube. (a) The various rotation axes and (b) view down one of each type of rotation axis. The reader is encouraged to pick up a cube and examine its symmetry.

How can we build a crystal from these symmetric objects?

In 2D: Tiling

- Invariance of a system under translation
- Parallelogram is the general shape that can be used.

4 - square

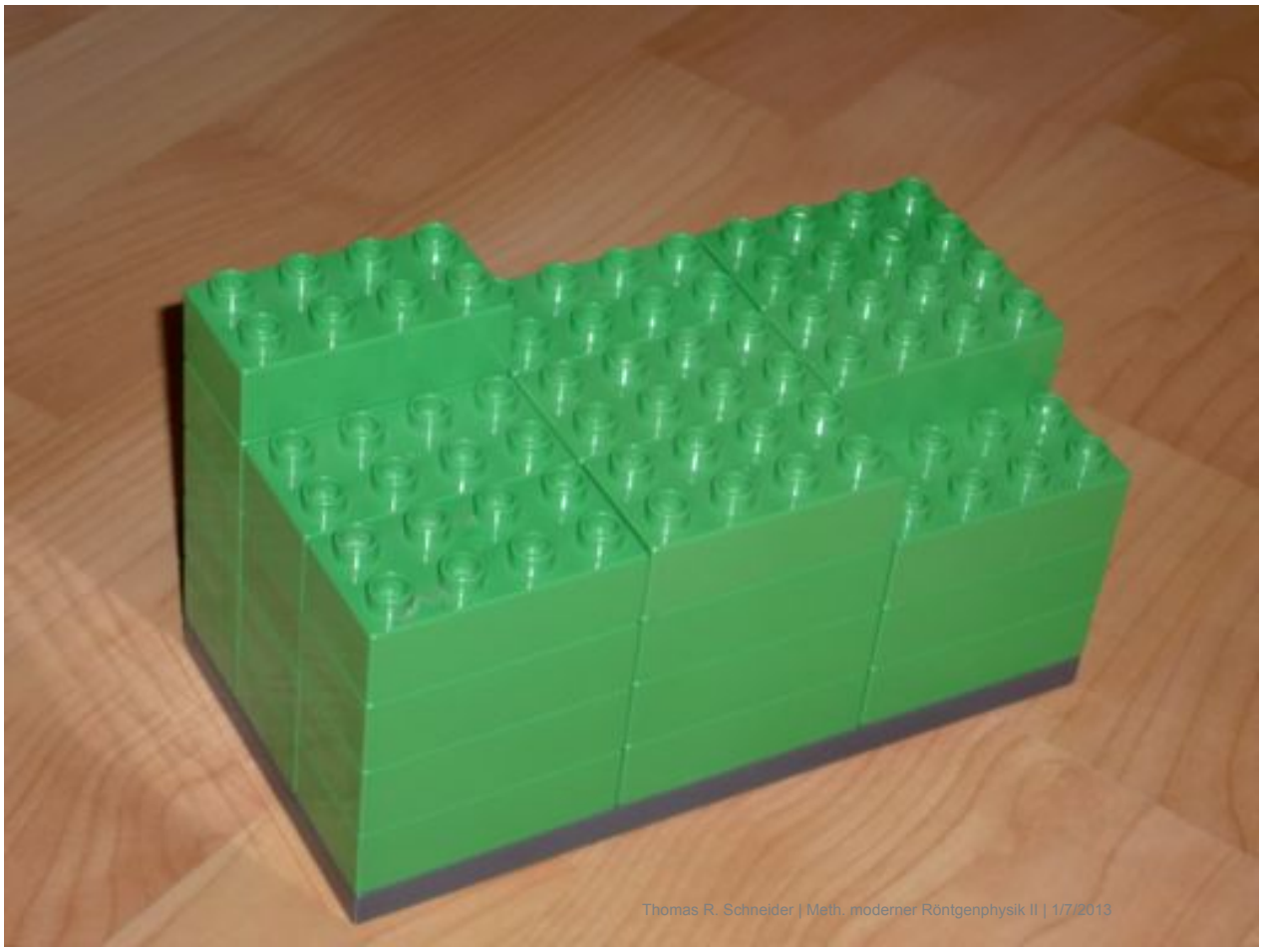
2 - rectangular

6 - hexagonal

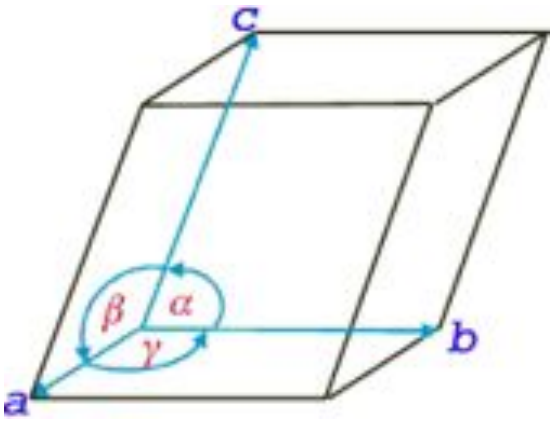
2 - rhombohedral

In 3D: Building crystals from unit cells

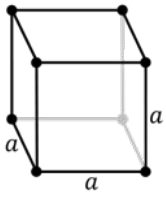
- A crystal is a homogeneous solid formed by a repeating three-dimensional pattern of atoms, ions, or molecules.
- In terms of translational symmetry, the repeating unit is the unit cell



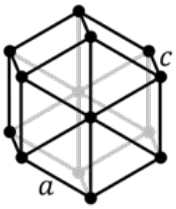
The general building block for a crystal is a parallelepiped



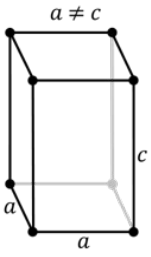
Lattice systems (primitive)



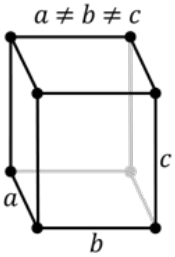
cubic



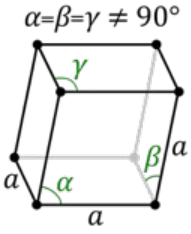
hexagonal



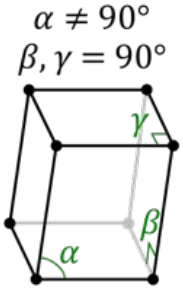
tetragonal



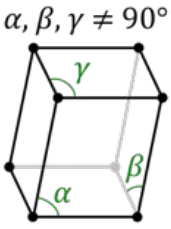
orthorhombic



rhombohedral



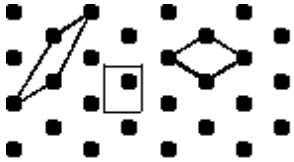
monoclinic



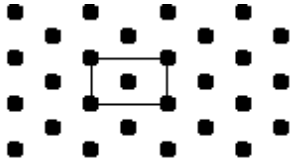
triclinic

Centered lattices

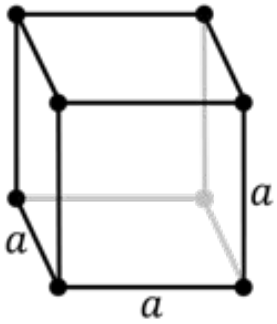
- Sometimes, it is more convenient to use a centered cell (lattice points on faces or inside the cell, to reflect the symmetry properly).
- This gives rise to a total of 14 Bravais lattices



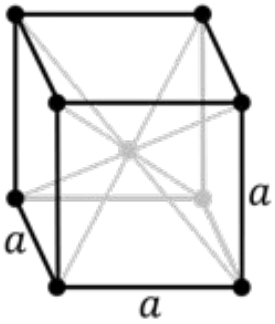
Primitive unit cell



Conventional unit cell

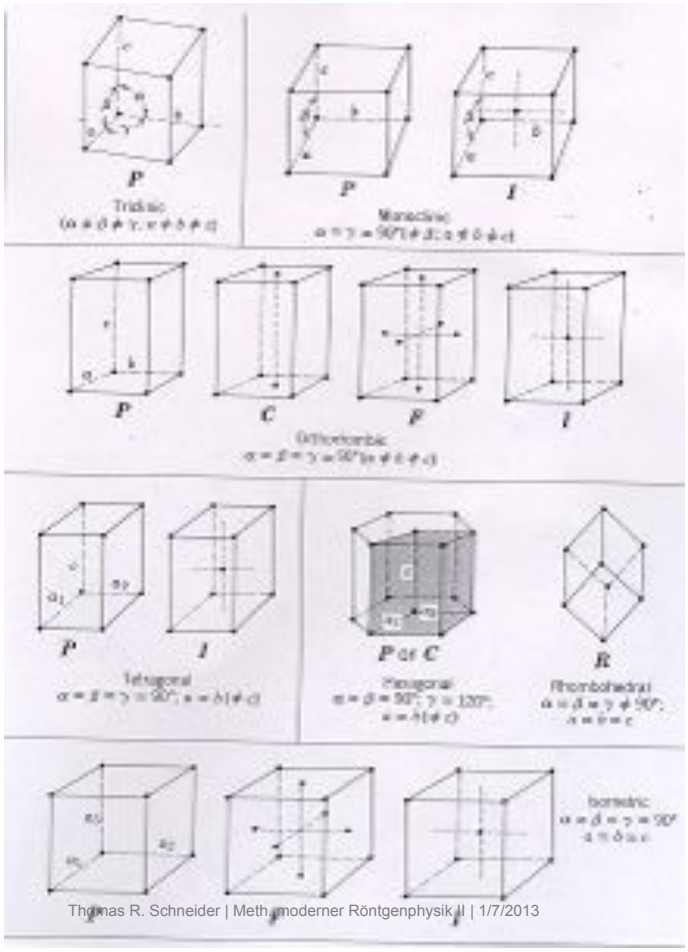


cubic (P432)



cubic body centered (I432)

14 Bravais lattices



Space groups

- Only certain point groups (local symmetry) are compatible with the translational symmetry in a crystal as expressed by the Bravais lattice.
- 32 point groups combined with the 14 Bravais lattices give rise to a total of **230 space groups**
  - Fyodorov (1891)
  - Schönflies (1891)
  - Barlow (1894)
- Only 1,2,3,4,6-fold axis are in fact allowed; 5, 7 and higher folds are not compatible with an infinite translational repetition

- For chiral objects (such as protein molecules) only 65 space groups are possible

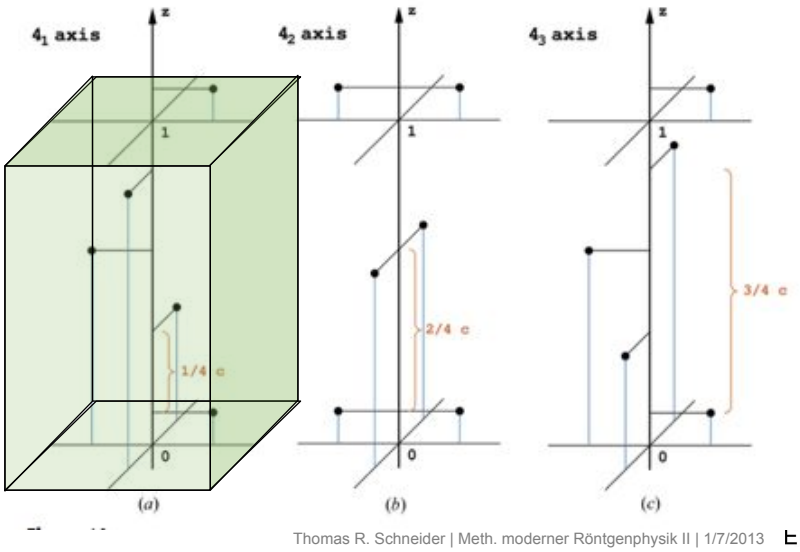
65 Space groups

That is all!

TRICLINIC					
P1					
MONOCLINIC					
P2	**$P2_1$**	**C2**			
ORTHORHOMBIC					
P222	**$P222_1$**	**$P2_12_12$**	**$P2_12_12_1$**		
$C222_1$	**C222**	**F222**	**I222**	**$I2_12_12_1$**	
TETRAGONAL					
P4	**$P4_1$**	**$P4_2$**	**$P4_3$**		
I4	**$I4_1$**				
P422	**$P4_212$**	**$P4_122$**	**$P4_12_12$**	**$P4_222$**	
$P4_22_12$	**$P4_322$**	**$P4_32_12$**			
I422	**$I4_122$**				
TRIGONAL					
P3	**$P3_1$**	**$P3_2$**	**R3**		
P312	**P321**	**$P3_112$**	**$P3_121$**		
$P3_212$	**$P3_221$**	**R32**			
HEXAGONAL					
P6	**$P6_1$**	**$P6_5$**	**$P6_2$**	**$P6_4$**	**$P6_3$**
P622	**$P6_122$**	**$P6_522$**	**$P6_222$**	**$P6_422$**	**$P6_322$**
CUBIC (minus sign in front of triade optional)					
P23	**F23**	**I23**	**$P2_13$**	**$I2_13$**	
P432	**P_4232**	**F432**			
$F4_132$	**I432**	**P4332**	**P4132**	**$I4_132$**	

Understanding space group symbols

- **P222**: 'P-two-two-two', a primitive lattice with three 2-fold axis perpendicular to each other. Orthorhombic.
- **P4**: a primitive lattice with a 4 fold along the z-axis. tetragonal.
- **P4$_1$**: a primitive lattice with a 4-fold screw axis along the z-axis. tetragonal.



65 Space groups

That is all!

TRICLINIC					
P1					
MONOCLINIC					
P2	**P2$_1$**	**C2**			
ORTHORHOMBIC					
P222	**P222$_1$**	**P2$_1$2$_1$2**	**P2$_1$2$_1$2$_1$**		
C222$_1$	**C222**	**F222**	**I222**	**I2$_1$2$_1$2$_1$**	
TETRAGONAL					
P4	**P4$_1$**	**P4$_2$**	**P4$_3$**		
I4	**I4$_1$**				
P422	**P4$_2$12**	**P4$_1$22**	**P4$_1$2$_1$2**	**P4$_2$22**	
P4$_2$2$_1$2	**P4$_3$22**	**P4$_3$2$_1$2**			
I422	**I4$_1$22**				
TRIGONAL					
P3	**P3$_1$**	**P3$_2$**	**R3**		
P312	**P321**	**P3$_1$12**	**P3$_1$21**		
P3$_2$12	**P3$_2$21**	**R32**			
HEXAGONAL					
P6	**P6$_1$**	**P6$_5$**	**P6$_2$**	**P6$_4$**	**P6$_3$**
P622	**P6$_1$22**	**P6$_5$22**	**P6$_2$22**	**P6$_4$22**	**P6$_3$22**
CUBIC (minus sign in front of triade optional)					
P23	**F23**	**I23**	**P2$_1$3**	**I2$_1$3**	
P432	**P$_4$232**	**F432**			
F4$_1$32	**I432**	**P4332**	**P4132**	**I4$_1$32**	

International Tables Volume A

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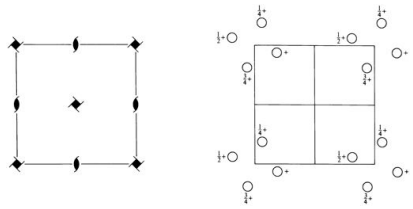
Tetragonal 4 C_4^2 $P4_1$

Patterson symmetry $P4/m$ $P4_1$ No. 76



Origin on 4_1

Asymmetric unit $0 \le x \le \frac{1}{2}$; $0 \le y \le \frac{1}{2}$; $0 \le z \le 1$

Symmetry operations

(1) 1 (2) $2(0,0,\frac{1}{2})$ $0,0,z$ (3) $4^+(0,0,\frac{1}{2})$ $0,0,z$ (4) $4^-(0,0,\frac{1}{2})$ $0,0,z$

Generators selected (1); $t(1,0,0)$; $t(0,1,0)$; $t(0,0,1)$; (2); (3)

Positions

Multiplicity, Wyckoff letter, Site symmetry	Coordinates				Reflection conditions
	(1) x,y,z	(2) $\bar{x},\bar{y},z+\frac{1}{2}$	(3) $\bar{y},x,z+\frac{1}{4}$	(4) $y,\bar{x},z+\frac{3}{4}$	
4 a 1					General: $00l:\ l=4n$

Symmetry of special projections

Along [001] $p4$ — $\mathbf{a}' = \mathbf{a}$ $\mathbf{b}' = \mathbf{b}$ — Origin at $0,0,z$

Along [100] $p1g1$ — $\mathbf{a}' = \mathbf{b}$ $\mathbf{b}' = \mathbf{c}$ — Origin at $x,0,0$

Along [110] $p1g1$ — $\mathbf{a}' = \frac{1}{2}(-\mathbf{a}+\mathbf{b})$ $\mathbf{b}' = \mathbf{c}$ — Origin at $x,x,0$

Maximal non-isomorphic subgroups

I [2] $P2_1$ (4) 1; 2

IIa none

IIb none

Maximal isomorphic subgroups of lowest index

IIc [2] $C4_1$ ($\mathbf{a}' = 2\mathbf{a}, \mathbf{b}' = 2\mathbf{b}$) ($P4_1$, 76); [3] $P4_1$ ($\mathbf{c}' = 3\mathbf{c}$) (78); [5] $P4_1$ ($\mathbf{c}' = 5\mathbf{c}$) (76)

Minimal non-isomorphic supergroups

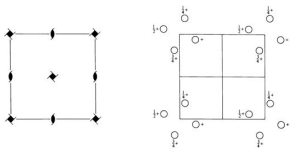
I [2] $P4_122$ (91); [2] $P4_12,2$ (92)

II [2] $I4_1$ (80); [2] $P4_1$ ($\mathbf{c}' = \frac{1}{2}\mathbf{c}$) (77)

Mores space groups ...

Tetragonal 4 C_4^2 $P4_1$

Patterson symmetry $P4/m$ $P4_1$ No. 76



Origin on 4_1

Asymmetric unit $0 \le x \le \frac{1}{2}$; $0 \le y \le \frac{1}{2}$; $0 \le z \le 1$

Symmetry operations

(1) 1 (2) $2(0,0,\frac{1}{2})$ $0,0,z$ (3) $4^+(0,0,\frac{1}{2})$ $0,0,z$ (4) $4^-(0,0,\frac{1}{2})$ $0,0,z$

Generators selected (1); $t(1,0,0)$; $t(0,1,0)$; $t(0,0,1)$; (2); (3)

Positions

Multiplicity, Wyckoff letter, Site symmetry	Coordinates				Reflection conditions
	(1) x,y,z	(2) $\bar{x},\bar{y},z+\frac{1}{2}$	(3) $\bar{y},x,z+\frac{1}{4}$	(4) $y,\bar{x},z+\frac{3}{4}$	
4 a 1					General: $00l:\ l=4n$

Symmetry of special projections

Along [001] $p4$ — $\mathbf{a}' = \mathbf{a}$ $\mathbf{b}' = \mathbf{b}$ — Origin at $0,0,z$

Along [100] $p1g1$ — $\mathbf{a}' = \mathbf{b}$ $\mathbf{b}' = \mathbf{c}$ — Origin at $x,0,0$

Along [110] $p1g1$ — $\mathbf{a}' = \frac{1}{2}(-\mathbf{a}+\mathbf{b})$ $\mathbf{b}' = \mathbf{c}$ — Origin at $x,x,0$

Maximal non-isomorphic subgroups

I [2] $P2_1$ (4) 1; 2

IIa none

IIb none

Maximal isomorphic subgroups of lowest index

IIc [2] $C4_1$ ($\mathbf{a}' = 2\mathbf{a}, \mathbf{b}' = 2\mathbf{b}$) ($P4_1$, 76); [3] $P4_1$ ($\mathbf{c}' = 3\mathbf{c}$) (78); [5] $P4_1$ ($\mathbf{c}' = 5\mathbf{c}$) (76)

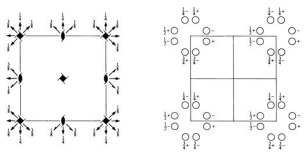
Minimal non-isomorphic supergroups

I [2] $P4_122$ (91); [2] $P4_12,2$ (92)

II [2] $I4_1$ (80); [2] $P4_1$ ($\mathbf{c}' = \frac{1}{2}\mathbf{c}$) (77)

$P4_122$ D_4^3 422 Tetragonal

No. 91 $P4_122$ Patterson symmetry $P4/mmm$



Origin on $2\,[010]$ at 4_1 $(1,2)\,1$

Asymmetric unit $0 \le x \le 1$; $0 \le y \le 1$; $0 \le z \le \frac{1}{8}$

Symmetry operations

(1) 1 (2) $2(0,0,\frac{1}{4})$ $0,0,z$ (3) $4^-(0,0,\frac{1}{2})$ $0,0,z$ (4) $4^-(0,0,\frac{3}{4})$ $0,0,z$
(5) 2 $0,y,0$ (6) 2 $x,0,\frac{1}{8}$ (7) 2 $x,x,\frac{1}{8}$ (8) 2 $x,\bar{x},\frac{1}{8}$

Diffraction and Symmetry

- If a crystal has e.g. a four-fold symmetry axis along the rotation axis, the diffraction pattern will repeat itself every 90 deg. of rotation.
- That means, one needs to collect only 90 deg. of data to catch every plane at least once.
- In fact, due to Friedel's law, I(hkl) = I(-h,-k,-l), only half of this rotation is needed.

- In reality, crystals end up in random orientations and it is not trivial to 'guess' how many degrees of data are needed to complete a data set -> strategy programs such as BEST.
- Radiation damage needs to be taken into account!

- If the crystal permits (in terms of radiation damage), symmetry equivalent reflections can be collected and compared.

Rejections

- When compared to symmetry equivalents, some reflections do not fit into the picture; these reflections are marked as outliers and will be rejected in subsequent rounds of scaling

From scale.log

Reflections printed out due to failing chi**2 test during scaling

H	K	L	Total chi**2,	<I>	resol
9	1	46	40.2	4736.3	2.94

original hkl			film#,	dev./sigma	I,	%PROB,	sigma
-9	-1	46 a+	147	0.7	4982.1	0	213.6
-1	9	46 f+	160	-1.4	4406.7	0	305.0
1	9	-46 a+	77	1.9	5124.8	0	166.4
1	9	-46 a+	178	0.2	4861.6	0	188.9
-1	-9	-46 a+	95	-1.3	4487.7	0	281.2
-9	**1**	**-46 f+**	**190**	**-5.4**	**3180.9**	**100**	**295.7**
-9	-1	-46 a-	178	-0.3	4742.5	0	365.1
-1	9	-46 a-	27	-1.7	4531.7	0	188.7
1	-9	-46 a-	81	0.4	5012.1	0	407.1
1	9	46 f-	172	-0.3	4748.7	0	316.3
-9	1	46 a-	127	1.0	5060.7	0	244.0

Data Quality – R-factors

From output.sca (10 symmetry equivalent reflections for (9,1,46)

```
 -9  -1  46   9   1  46   147 1 0  2  4973.3   215.4
 -1   9  46   9   1  46   160 1 0  4  4402.4   306.1
  1   9 -46   9   1  46    77 1 0  5  5121.4   168.9
  1   9 -46   9   1  46   178 1 1  5  4855.8   191.1
 -1  -9 -46   9   1  46    95 1 1  6  4490.0   282.9
 -9  -1 -46   9   1  46   178 2 1  1  4736.8   365.9
 -1   9 -46   9   1  46    27 2 0  3  4545.1   191.2
  1  -9 -46   9   1  46    81 2 1  4  5125.4   416.2
  1   9  46   9   1  46   172 2 0  6  4732.4   316.8
 -9   1  46   9   1  46   127 2 0  8  5044.3   245.2

              9   1  46              4836.2    75.7
```

- The standard R-factor:

$$R_{lin} = \frac{\sum_{hkl} \left| I_{hkl} - \langle I \rangle_{hkl} \right|}{\sum_{hkl} I_{hkl}}$$

describes how well the measurements of symmetry equivalent reflections agree with their mean.

Scaling and merging

From output.sca (10 symmetry equivalent reflections for (9,1,46)

```
 -9  -1  46   9   1  46   147 1 0  2  4973.3   215.4
 -1   9  46   9   1  46   160 1 0  4  4402.4   306.1
  1   9 -46   9   1  46    77 1 0  5  5121.4   168.9
  1   9 -46   9   1  46   178 1 1  5  4855.8   191.1
 -1  -9 -46   9   1  46    95 1 1  6  4490.0   282.9
 -9  -1 -46   9   1  46   178 2 1  1  4736.8   365.9
 -1   9 -46   9   1  46    27 2 0  3  4545.1   191.2
  1  -9 -46   9   1  46    81 2 1  4  5125.4   416.2
  1   9  46   9   1  46   172 2 0  6  4732.4   316.8
 -9   1  46   9   1  46   127 2 0  8  5044.3   245.2

              9   1  46              4836.2    75.7
```

Scaled and merged intensity for (9,1,46)

Error estimate corrected for multiple measurements (sqrt(N))

- For this unique reflection, the R-factor is 4.6%

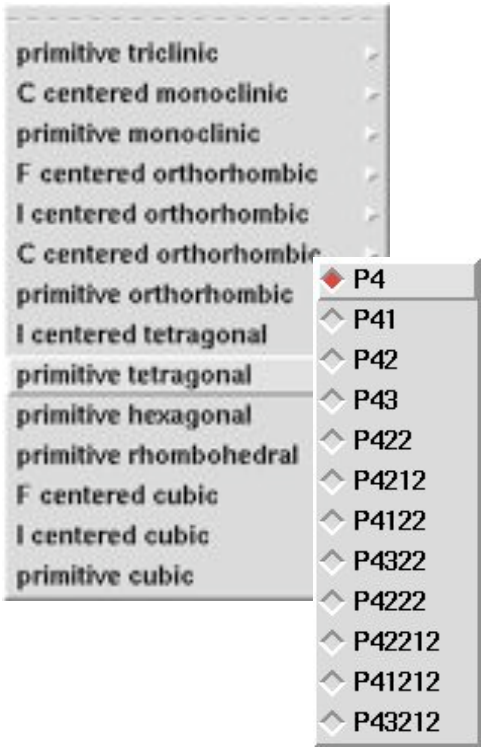
R-factors and Multiplicity

- The standard R-factor does not reflect the improvement in data quality when more measurements are made:

4973.3										4973.3	0.0000
4973.3	4402.4									4687.9	0.0609
4973.3	4402.4	5121.4								4832.4	0.0593
4973.3	4402.4	5121.4	4855.8							4838.2	0.0450
4973.3	4402.4	5121.4	4855.8	4490.0						4768.6	0.0541
4973.3	4402.4	5121.4	4855.8	4490.0	4736.8					4763.3	0.0462
4973.3	4402.4	5121.4	4855.8	4490.0	4736.8	4545.1				4732.1	0.0458
4973.3	4402.4	5121.4	4855.8	4490.0	4736.8	4545.1	5125.4			4781.3	0.0497
4973.3	4402.4	5121.4	4855.8	4490.0	4736.8	4545.1	5125.4	4732.4		4775.8	0.0453
4973.3	4402.4	5121.4	4855.8	4490.0	4736.8	4545.1	5125.4	4732.4	5044.3	4802.7	0.0461

Space group determination

- Combination of symmetry elements with the 14 Bravais lattices results in 230 space groups.
- Space groups can be identified by checking:
  - How well the data fit the symmetry (R_{merge})
  - The systematic absences
- Some space groups (e.g. enantiomorphic space groups) can only be distinguished by solving/ refining the structure.



Space groups – Checking for symmetry

XPREP output

Identical indices and Friedel opposites combined before calculating R(sym)

Option	Space Group	No.	Type	Axes	CSD	R(sym)	N(eq)	Syst. Abs.	CFOM
[A]	P4	# 75	chiral	1	4	0.051	57875	0.0 / 11.0	23.80
[B]	P4(2)	# 77	chiral	1	7	0.051	57875	0.0 / 11.0	16.30
[C]	P4(1)	# 76	chiral	1	114	0.051	57875	0.0 / 11.0	4.67
[D]	P4(3)	# 78	chiral	1	114	0.051	57875	0.0 / 11.0	4.67
[E]	P422	# 89	chiral	1	4	0.056	72030	0.0 / 11.0	20.94
[F]	P42(1)2	# 90	chiral	1	4	0.056	72030	0.0 / 11.0	20.94
[G]	P4(2)22	# 93	chiral	1	4	0.056	72030	0.0 / 11.0	20.94
[H]	P4(2)2(1)2	# 94	chiral	1	20	0.056	72030	0.0 / 11.0	5.70
[I]	P4(1)22	# 91	chiral	1	8	0.056	72030	0.0 / 11.0	12.05
[J]	P4(3)22	# 95	chiral	1	8	0.056	72030	0.0 / 11.0	12.05
[K]	P4(1)2(1)2	# 92	chiral	1	245	0.056	72030	0.0 / 11.0	1.34
[L]	P4(3)2(1)2	# 96	chiral	1	245	0.056	72030	0.0 / 11.0	1.34

Summary - Symmetry

- All chiral objects can only crystallize in one of the 65 chiral space groups
- These space groups arise from the combination of the condition of rotational and translational invariance of the crystal with the fact that the repeating object is chiral.
- The symmetry of the crystal can be used to short-cut data collection.
- Collection of symmetry-related reflections allows to do statistics about the accuracy and precision of the measured diffraction intensities.

- WARNING: The packing introduced by the crystal symmetry does not always correspond to the biologically relevant oligomer.

Sources

- **Dauter1999**. Zbigniew Dauter (1999) 'Data-Collection Strategies' Acta Cryst. D55:1703-1717
- **Dauter2010**. Zbigniew Dauter & Mariusz Jaskolski (2010) 'How to read (and understand) Volume A of International Tables for Crystallography: an introduction for nonspecialists' Acta Cryst D43:1150-1171
- **Blow2007.** David Blow (2007): 'Outline of Crystallography for Biologists' Oxford University Press
- **Glusker1994**. Jenny Glusker, Mitchell Lewis, Miriam Rossi (1994): 'Crystal Structure Analysis for Chemists and Biologists' VCH Publishers