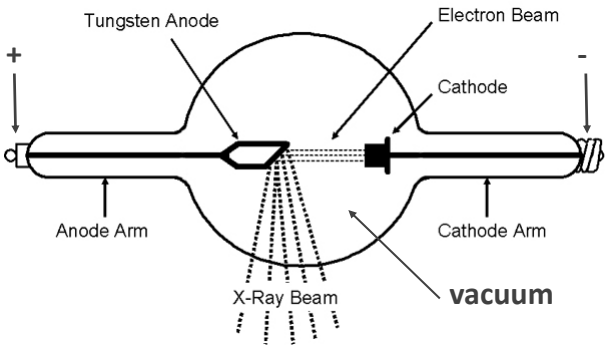
Data collection, reduction, and analysis (general)

Pedro M. Matias
Industry and Medicine Applied Crystallography

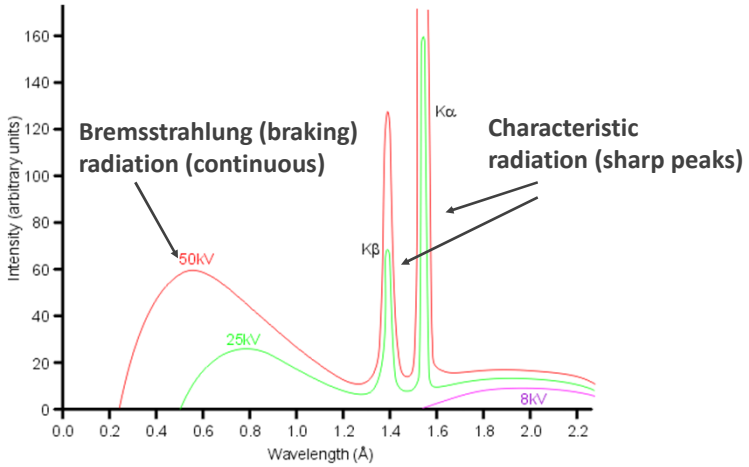
X-ray sources

Coolidge X-ray tube (William Coolidge, 1913)



- Cathode filament is **heated**, releasing **electrons**
- Electrons accelerated through ΔV
- Electrons hit anode, producing X-rays

X-ray sources



Typical X-ray emission spectrum of an X-ray tube

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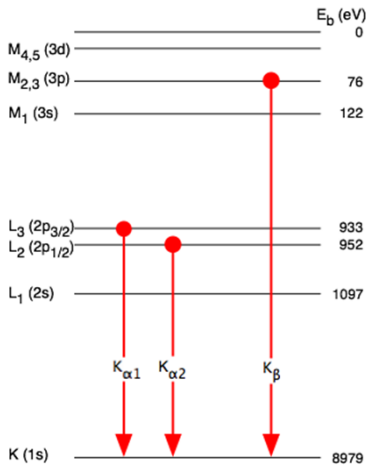
X-ray sources



Electronic transitions responsible for Cu X-ray emission lines

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X-ray sources

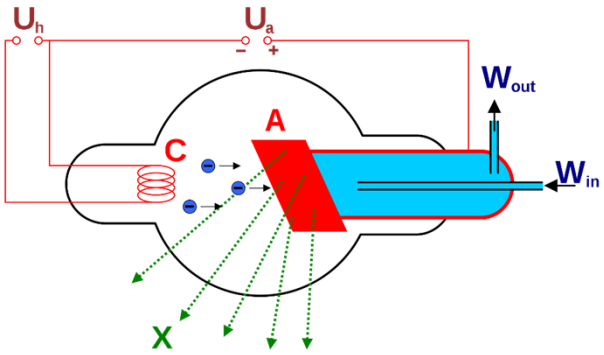

Diagram of an X-ray sealed tube (present day):
The anode water-cooling allows:

- higher loads
- stronger X-ray emission

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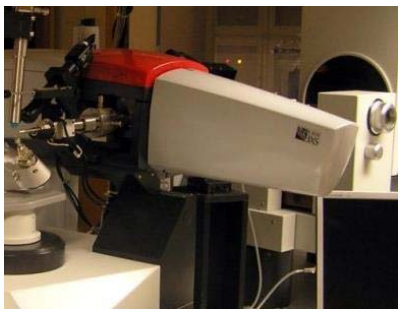
X-ray sources



A rotating anode (6000 rpm) X-ray tube (present day) allows:

- Even higher loads
- Even stronger X-ray emission

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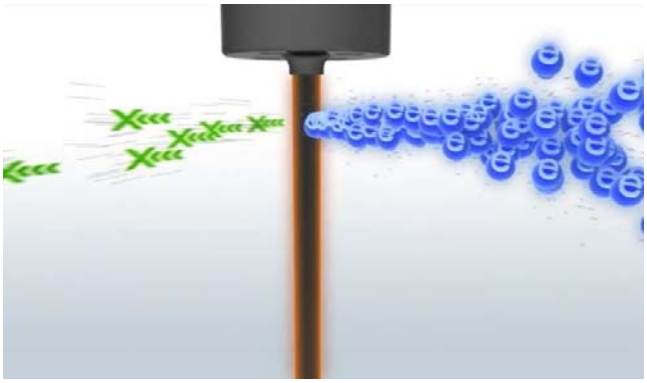
X-ray sources



Liquid anode (present day) allows:

- Even higher loads
- Even stronger X-ray emission

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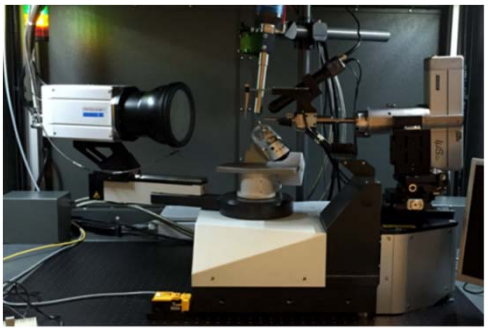
X-ray sources



A micro-source X-ray tube (present day) allows:

- Low power consumption (50 W) than RA (2.7 kW)
- Stronger X-ray emission than standard RA

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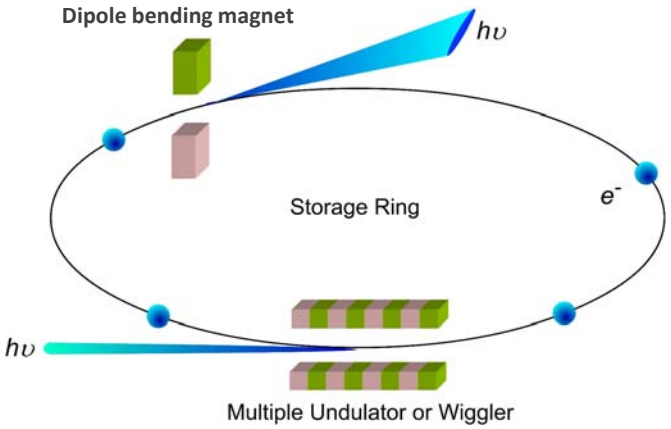
X-ray sources



Synchrotron X-ray sources:

- Very intense X-ray beams
- Tunable energy of X-ray beam

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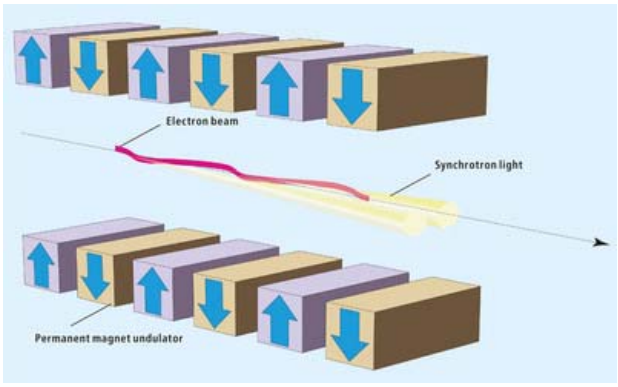
X-ray sources



How an Undulator works

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X-ray sources

The European Synchrotron Radiation Facility
Grenoble (France)

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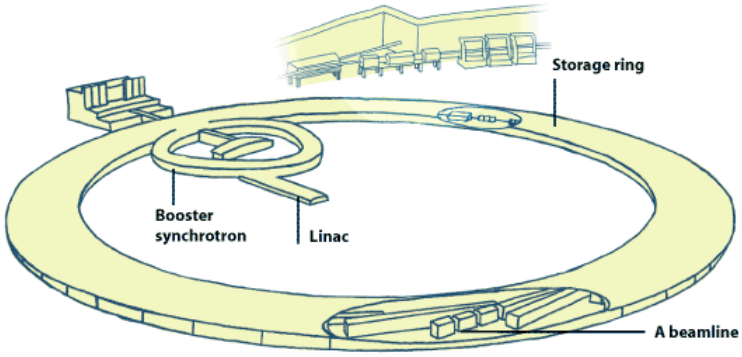
X-ray sources



The European Synchrotron Radiation Facility
Grenoble (France)

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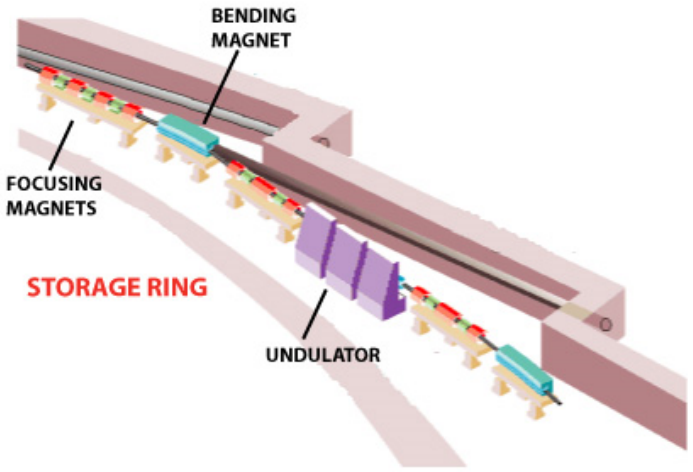
X-ray sources



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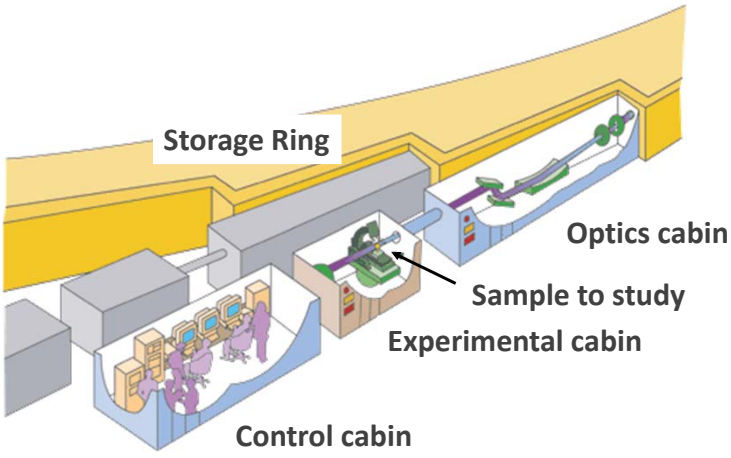
X-ray sources



http://www.esrf.fr/AboutUs/GuidedTour/Animation

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Interpreting the diffraction pattern
the reciprocal space

The diffraction pattern arising from a **Crystal Lattice** is made up of **discrete diffracted beams**. The **geometrical arrangement** of these beams can be described by the **Reciprocal Lattice** (related to the Crystal Lattice).

As for the Crystal Lattice, a **Unit Cell** can also be defined for the Reciprocal Lattice, with three edges denoted by ***a****, ***b****, ***c**** and three angles denoted by α^*, β^* and γ^*.

The coordinates of an atom in a Crystal Lattice are given by **three REAL numbers x,y,z**.

The coordinates of a Reciprocal Lattice point are given by **three INTEGER numbers h,k,l**.

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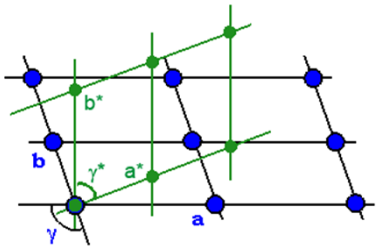
Interpreting the diffraction pattern
the reciprocal space

$a.a^*=1,\ a.b^*=0,\ a.c^*=0$
$b.a^*=0,\ b.b^*=1,\ b.c^*=0$
$c.a^*=0,\ c.b^*=0,\ c.c^*=1$



$$a^* = \frac{b \times c}{V}; \quad \cos\alpha^* = \frac{\cos\beta\cos\gamma - \cos\alpha}{\operatorname{sen}\beta\operatorname{sen}\gamma}$$

$$b^* = \frac{c \times a}{V}; \quad \cos\beta^* = \frac{\cos\alpha\cos\gamma - \cos\beta}{\operatorname{sen}\alpha\operatorname{sen}\gamma}$$

$$c^* = \frac{a \times b}{V}; \quad \cos\gamma^* = \frac{\cos\alpha\cos\beta - \cos\gamma}{\operatorname{sen}\alpha\operatorname{sen}\beta}$$

a^* perpendicular to b, c

b^* perpendicular to a, c

c^* perpendicular to a, b

$V = (a \times b) \,.\, c = a.\, b.\, c\,(1 - \cos^2\alpha - \cos^2\beta - \cos^2\gamma + 2\cos\alpha\cos\beta + 2\cos\alpha\cos\gamma + 2\cos\beta\cos\gamma)^{1/2}$

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Diffraction pattern and crystal symmetry

The **symmetry** of a Crystal Structure is reflected in the **symmetry** of its **Diffraction Pattern** in two ways:

1. The **symmetry** of the **Diffraction Pattern** results from that of the **Crystal System** to which the **Crystal Structure** belongs:

Triclinic: $\overline{1}$
Monoclinic: **2/m**
Orthorhombic: **mmm**
Tetragonal: **4/m** or **4/mmm**
Hexagonal: **6/m** or **6/mmm**
Trigonal: **$\overline{3}$m**
Cubic: **m3** or **m3m**

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Diffraction pattern and crystal symmetry

2. The presence of **face centering**, **screw axes** and **glide planes** leaves its **trace** in the Diffraction Pattern as **SYSTEMATIC EXTINCTIONS**, i.e., whole **groups of reflections** that are not present. In this way it is possible to determine the **SPACE GROUP** to which the Crystal Structure belongs.

Examples:

A-centered lattice: only reflections with **k+l even** are observed.

***c*-glide plane perpendicular to crystallographic *b*-axis:** of all the **h0l** reflections, only those with **l even** are observed.

2_1 screw axis parallel to crystallographic *b*-axis: of all the **0k0** reflections, only those with **k even** are observed.

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Reciprocal space and the Ewald sphere

How many diffracted beams can we measure from a crystal ?

The answer is given by a geometrical construction called the **Ewald sphere**.

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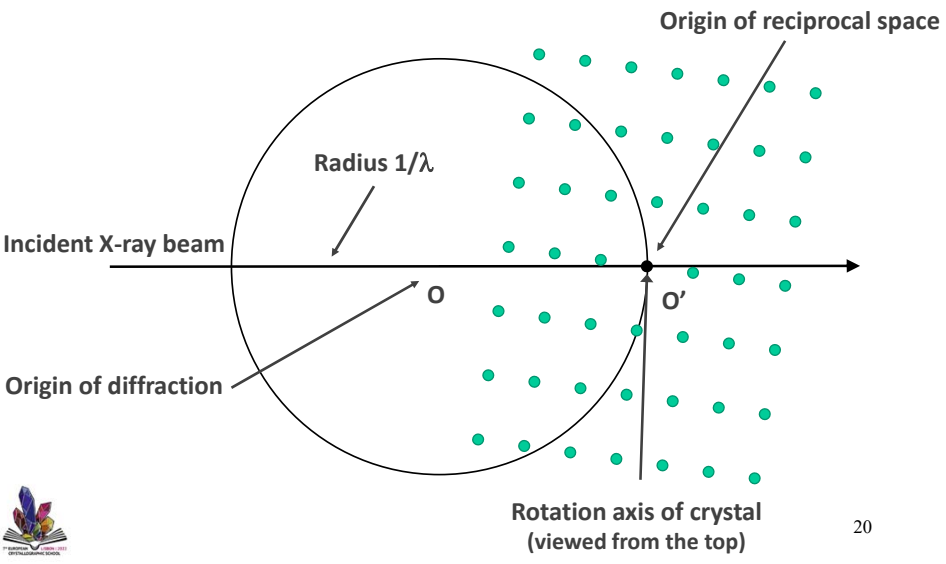
Reciprocal space and the Ewald sphere



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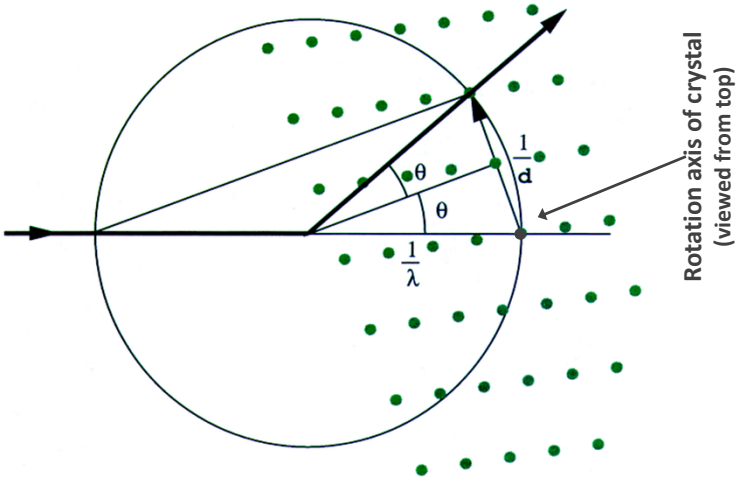
Reciprocal space and the Ewald sphere



Diffraction occurs whenever a **reciprocal lattice point** crosses the **Ewald sphere**.

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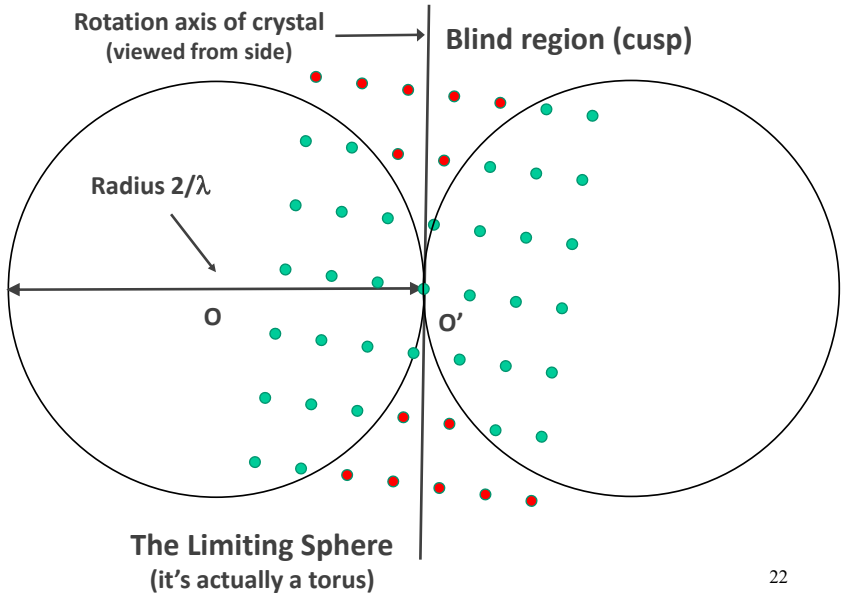
Reciprocal space and the Ewald sphere



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Reciprocal space and the Ewald sphere

The Limiting sphere determines the **theoretical extent of reciprocal space acessible for measurement**.

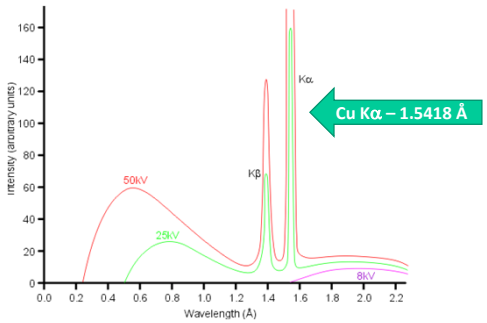
Depends on the **X-ray wavelength** used.

For Cu Kα, d = 1.5418 / 2 = 0.77 Å

From Bragg's Law this corresponds to **sin θ = 1**, i.e., **θ = 90°** and **2θ = 180°** but this Bragg angle is impossible in practice.

More importantly, if the crystal is rotated about a single axis, **many reciprocal lattice points never cross the Ewald sphere** – these points are located in a cusp named **blind region**.

Diffraction data collection – wavelength selection



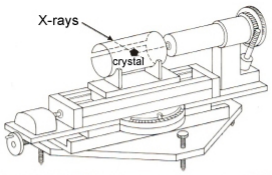
- Filter (*e.g.,* Ni-filter for CuKα)
- Monochromator (diffraction from graphite, Si, Ge crystals)
- Mirrors

Diffraction data collection – the Weissenberg camera

This camera was used in earlier days in X-ray crystallography.

The crystal **oscillates** about a certain angular range while a cylinder containing **photographic film** moves **back and forth** on a horizontal track.

A eucentric goniometer



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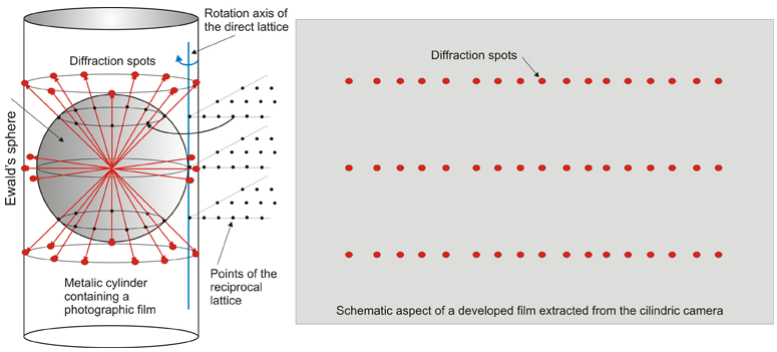
Diffraction data collection – the Weissenberg camera

When the crystal is correctly aligned **rows of diffraction spots** are obtained on film with a **stationary cylinder**.



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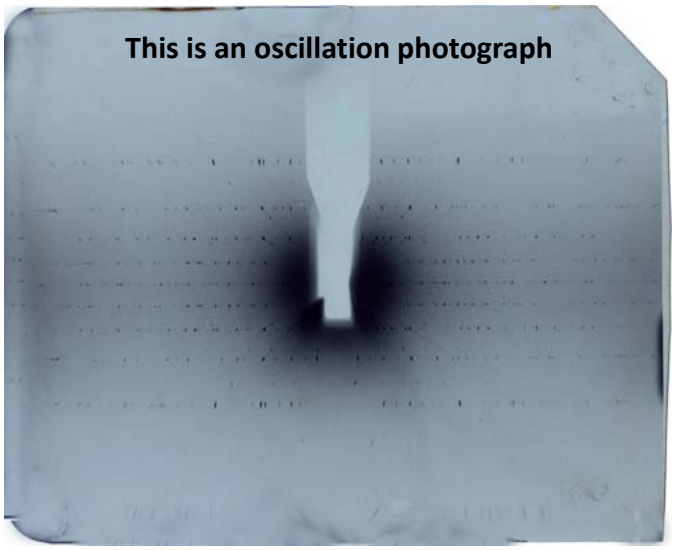
Diffraction data collection – the Weissenberg camera



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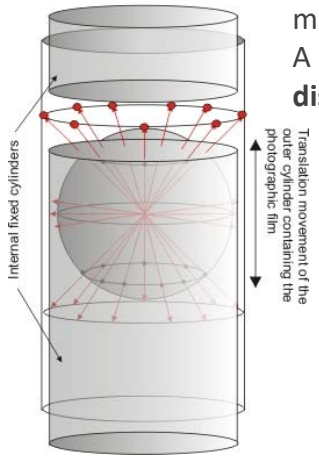
Diffraction data collection – the Weissenberg camera

A shield masks **all but one** of the rows and the crystal **oscillates** while the film cassette moves back and forth.

A Weissenberg photograph provides a **distorted image** of a **reciprocal lattice plane.**



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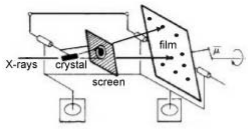
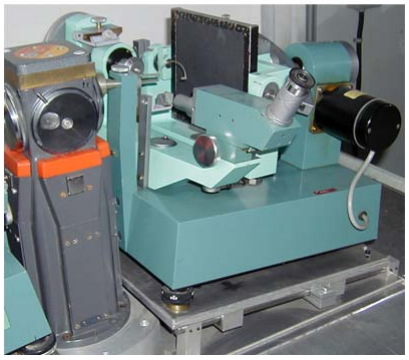
Diffraction data collection – the precession camera





This was an improvement over the Weissenberg camera.

The crystal and the film execute a **coupled precession motion** about an axis.

A **screen** isolates a reciprocal lattice plane for recording on film.

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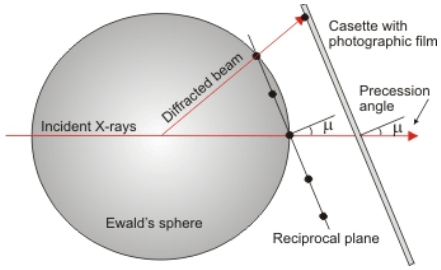
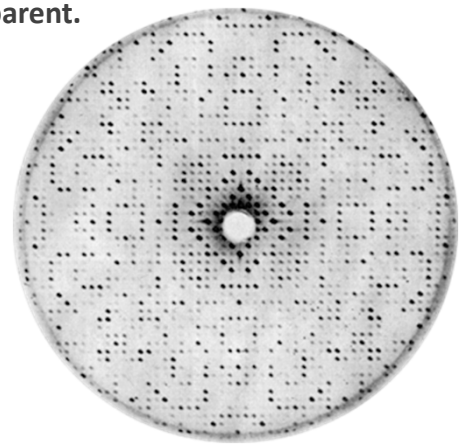
Diffraction data collection – the Weissenberg camera

A precession photograph provides an **undistorted image** of a **reciprocal lattice plane.**
The symmetry is readily apparent.





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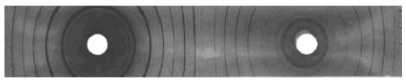
Diffraction data collection – the Guinier camera

The Guinier camera (or the Debye-Scherrer camera) were used to record diffraction from powder samples on photographic film.



The powder sample was loaded in a glass capillary and rotated during the experiment to avoid **preferred orientation** problems.

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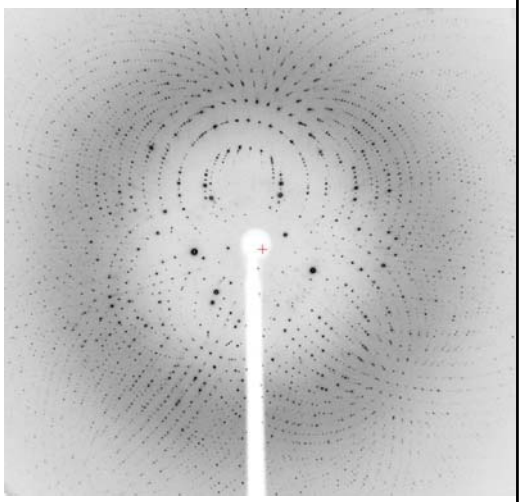
Diffraction data collection – the Laue method

White radiation is used to record images. The Bragg law is satisfied by the many different wavelengths in the ***primary beam***.



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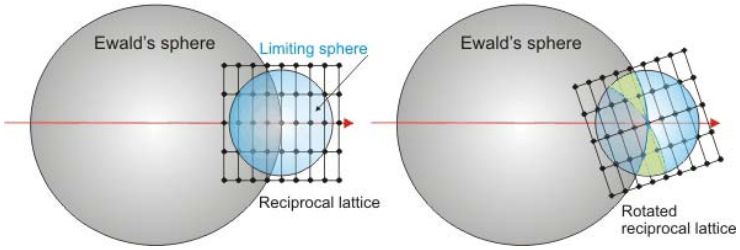
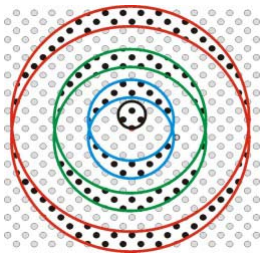
Diffraction data collection – the rotation method



Monochromatic radiation is used to record images.

The reciprocal lattice planes intersect the Ewald sphere and produce **lunes** on a detector (initially film, later **area detectors**).



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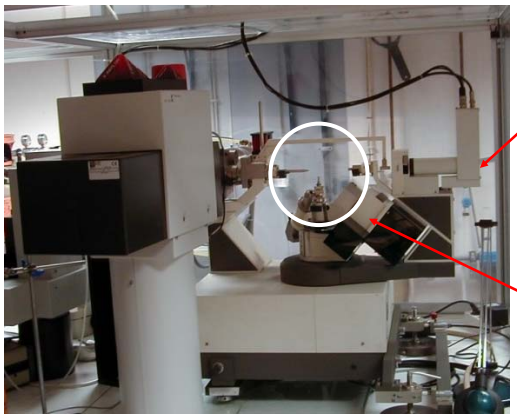
Diffraction data collection
the single-crystal diffractometer



Scintillation detector records diffraction intensities **individually.**

4-circle diffractometer allows crystal orientation about **an arbitrary direction**. **Eliminates the blind region**.

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Diffraction data collection
the single-crystal diffractometer

The **scintillation detector** records **diffraction intensities** (**Bragg peaks**) **individually.**

Each **Bragg peak** can be measured **individually** with a signal-to-noise ratio determined by the scan speed and limited by the patience of the experimenter.

This process becomes **extremely time-consuming** if more than a **few thousand** Bragg peaks need to be measured.

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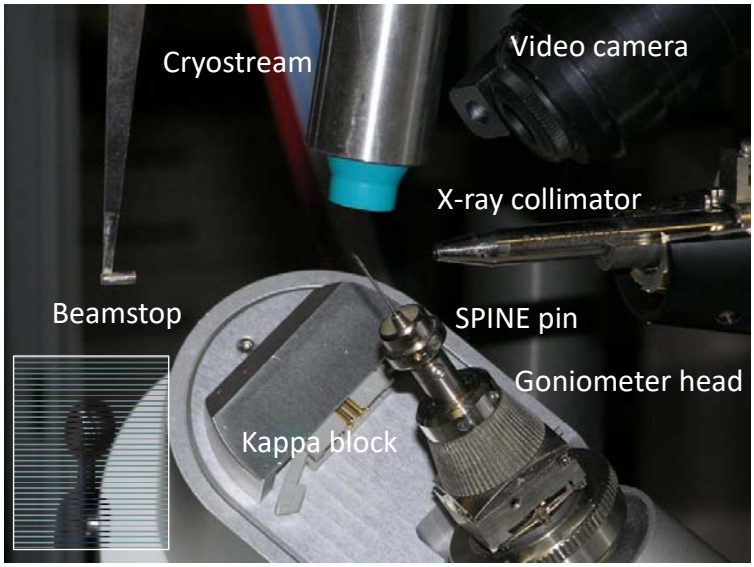
Diffraction data collection
the single-crystal diffractometer



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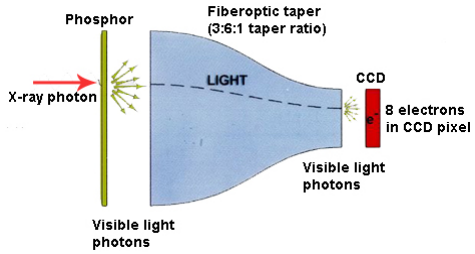
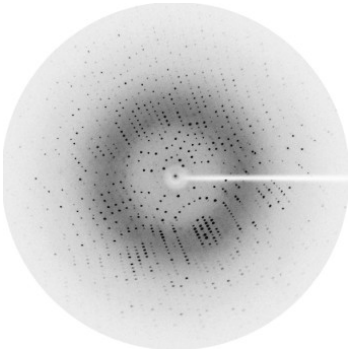
Diffraction data collection – the area detector

The area detector allows the **recording** and **readout** in **digital format** of a **2D diffraction image** containing **many** diffraction spots.





Film (minutes) → Imaging plate (2-5 minutes) → CCD (a few seconds) → Pixel (a few miliseconds)

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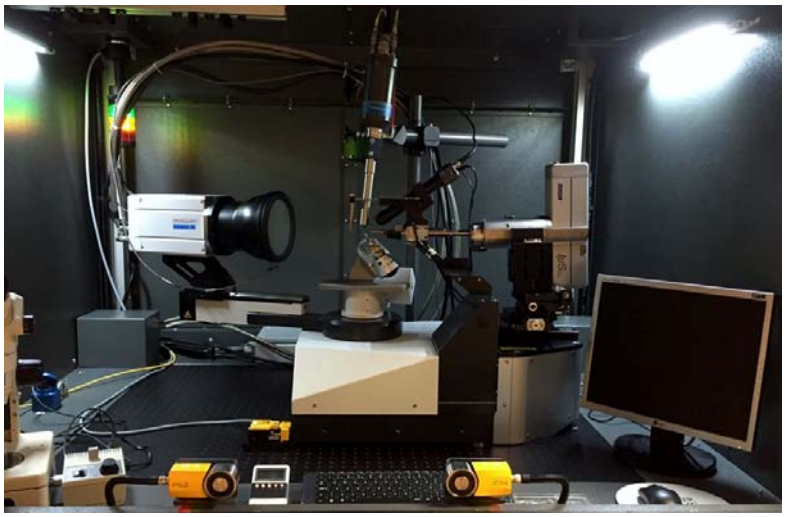
Diffraction data collection – the CCD detector



Bruker AXS Platinum 135 CCD at ITQB NOVA

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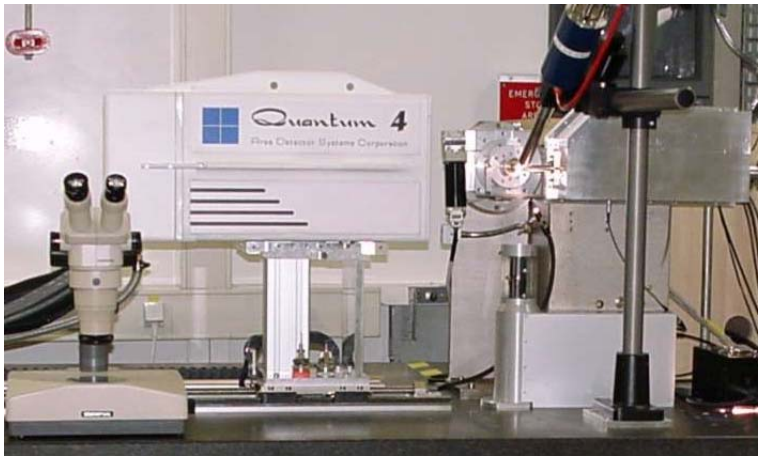
Diffraction data collection – the CCD detector



ADSC Quantum 4 CCD at an ESRF beamline

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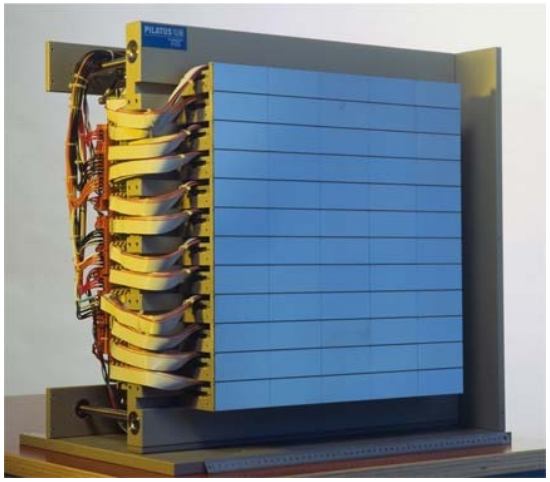
Diffraction data collection – the pixel detector



DECTRIS PILATUS 6M (under the hood)

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Diffraction data collection in practice

1. **M**ount and center crystal on the diffractometer.
2. **T**ake 2 or more **TEST** images 90° apart about an axis ⊥ to the X-ray beam.
3. **INDEX** the images, *i.e.,* determine the **reciprocal unit cell parameters** and the **direction of the reciprocal unit cell axes** in the **laboratory coordinate system** (orientation matrix).
4. **D**etermine the **BRAVAIS** lattice type – this hints at the possible Laue symmetries and space groups.
5. **C**alculate a data collection **STRATEGY** to obtain a data set as complete as possible.
6. **COLLECT** the diffraction images.
7. **INTEGRATE** the images to extract the diffraction intensities.
8. **SCALE** the data and determine the **TRUE** Laue Symmetry and possible space groups.

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Processing single-crystal diffraction data

Data Processing or Integration:

Determine the intensity of every **BRAGG PEAK** in the collected set of diffraction images.

This requires knowledge of the **reciprocal unit cell parameters** and the **direction of the reciprocal unit cell axes** in the **laboratory coordinate system** (orientation matrix) so that each Bragg peak can be identified by its **reciprocal lattice coordinates** – the three Miller indices h,k,l.

The raw intensities also need to be corrected for **Lorentz** and **polarization** effects.

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Processing single-crystal diffraction data

Lorentz correction:

The Lorentz correction is related to the time taken by each Bragg reflection to cross the Ewald sphere and depends on the geometry of the diffraction experiment.

For example, for the rotation method, when the incident X-ray beam, the monochromatized beam and the scattered beam are all coplanar, $L = 1/\sin 2\theta$.

https://dictionary.iucr.org/Lorentz%E2%80%93polarization_correction
http://epgp.inflibnet.ac.in/epgpdata/uploads/epgp_content/S001174BS/P001202/M010937/ET/1526545878Module_5_ET.pdf

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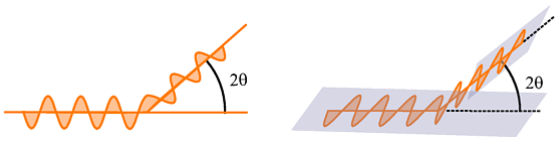
Processing single-crystal diffraction data

Polarization correction:

The radiation from an X-ray tube is unpolarized, but it becomes partially polarized when a monochromator is used. The component with the electric vector normal to the plane of scattering is attenuated by $\cos^2\theta$ while that with the electric vector in the plane of scattering is not attenuated.



The diffracted X-ray beam is again partly polarized, so the polarization factor becomes:

$(1+ A \cos^2 2\theta)/(1 + A)$, where $A = \cos^2 2\theta_M$ and θ_M is the Bragg angle of the monochromator crystal.

https://dictionary.iucr.org/Lorentz%E2%80%93polarization_correction
http://epgp.inflibnet.ac.in/epgpdata/uploads/epgp_content/S001174BS/P001202/M010937/ET/1526545878Module_5_ET.pdf

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Processing single-crystal diffraction data

Absorption correction:

When X-rays encounter any form of matter, they are partly transmitted and partly absorbed. For a crystal, the diffracted beam intensity can be written as $I_x=I_o e^{-\mu x}$ where μ is the linear absorption coefficient of the material and **x** the path length traversed by the X-ray beam.

If the crystal shape and orientation is precisely known, the absorption correction can be calculated analytically for each Bragg peak.

In the days of scintillation detectors, scans of several strong Bragg peaks around the diffraction vector (**ψ-scans**) were used to derive absorption corrections for the whole set of diffraction intensities.

Nowadays, an **empirical correction** is applied during the **scaling** process.

https://dictionary.iucr.org/Lorentz%E2%80%93polarization_correction
http://epgp.inflibnet.ac.in/epgpdata/uploads/epgp_content/S001174BS/P001202/M010937/ET/1526545878Module_5_ET.pdf

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Processing single-crystal diffraction data

In addition to proprietary software for integrating diffraction data recorded in the home laboratory, there are several software packages that can process many different diffraction image formats:

- iMOSFLM (Andrew Leslie, University of Cambridge, UK)
- XDS (Wofgang Kabsch and Kay Diederichs, Max Planck Heidelberg, DE)
- autoPROC (Gerard Bricogne, GlobalPhasing, Cambridge, UK)
- HKL3000 (HKL Research, Inc., Charlottesville, VA USA)

They all use a few images to INDEX the diffraction pattern and then use that information to go through the whole dataset and extract the individual diffraction intensities.

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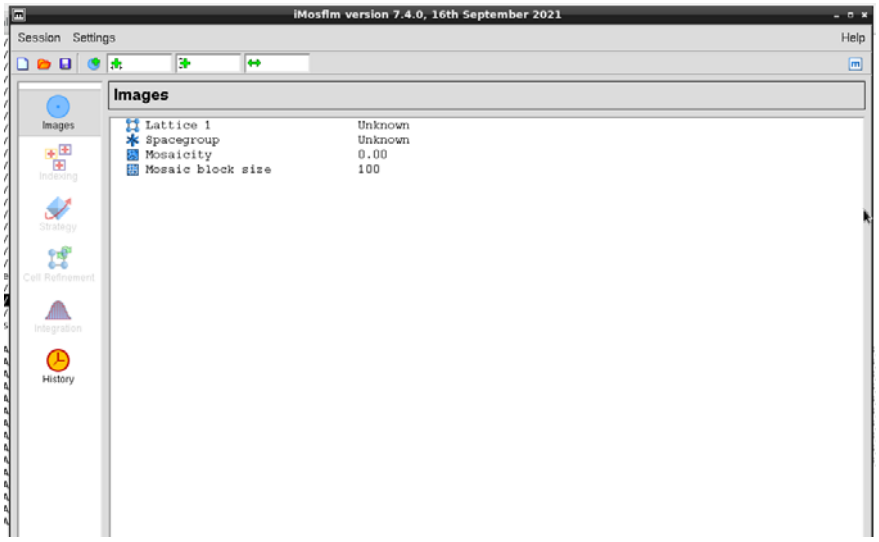
Processing single-crystal diffraction data - iMOSFLM



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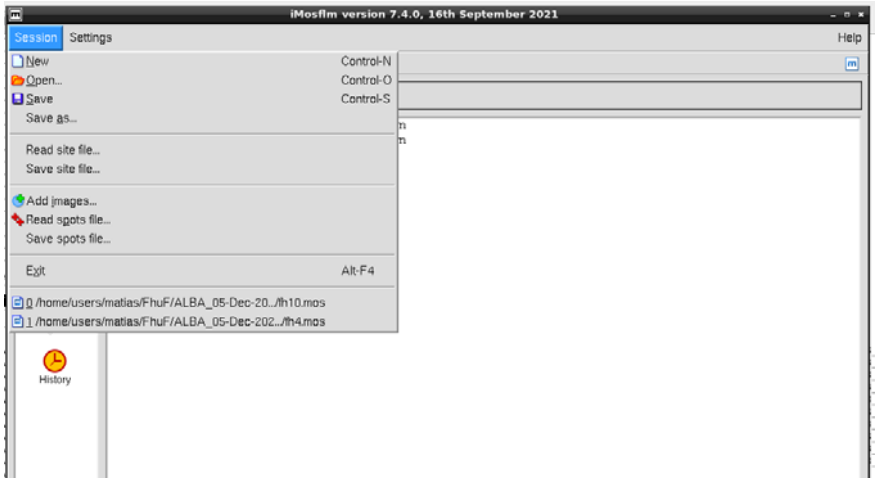
Processing single-crystal diffraction data - iMOSFLM



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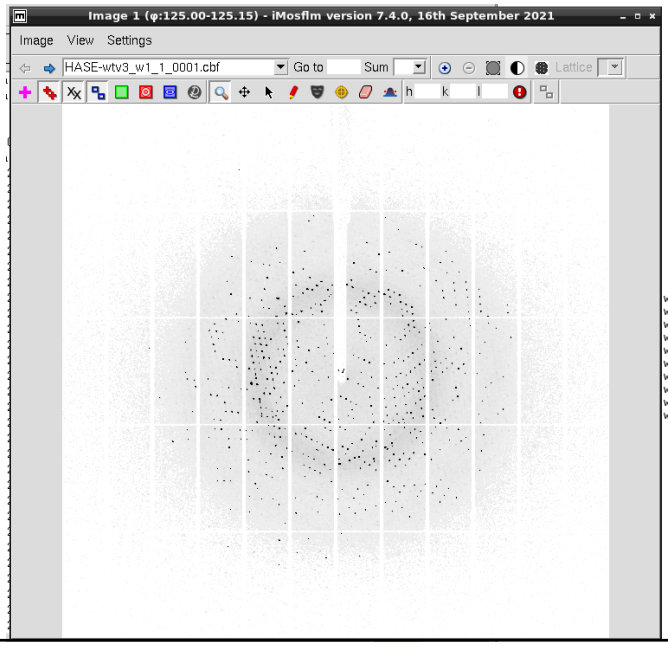
Processing single-crystal diffraction data - iMOSFLM



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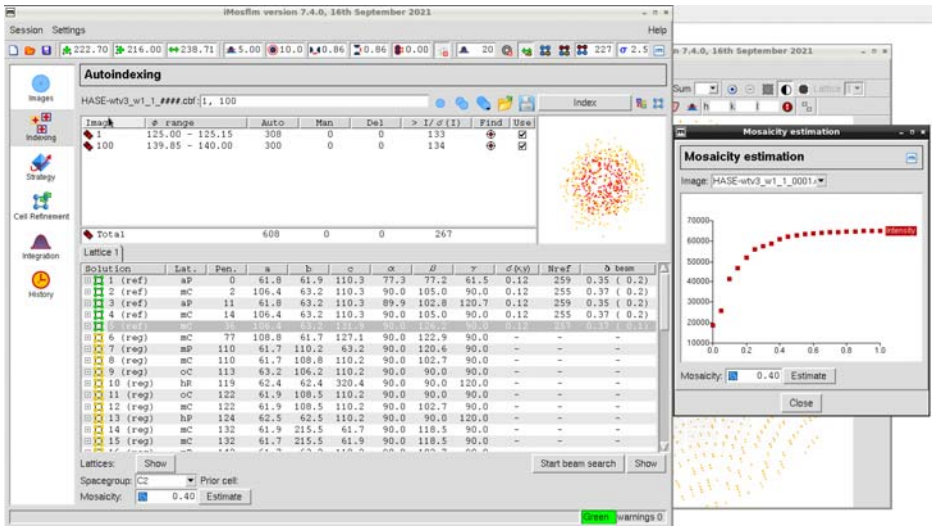
Processing single-crystal diffraction data - iMOSFLM



More details in the MX tutorials

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Processing single-crystal diffraction data - XDS

X-ray **D**etector **S**oftware for processing single-crystal monochromatic diffraction data recorded by the rotation method.

XDS is a command-line based data processing program.

Input files always have extension **.INP**

Text output files have extension **.LP**

File names are always in **UPPER CASE** and take the name of the XDS program that will read them as input.

Processed data file is named **XDS_ASCII.HKL** by default

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Processing single-crystal diffraction data - XDS

Main programs in the XDS Program Package

xds	Processes rotation images using a single CPU.	**XDS.INP**
xds_par	Multi-processor version of **xds** for up to 32 CPUs.	**XDS.INP**
xscale	Scaling program for XDS-produced data sets (single-processor version).	**XSCALE.INP**
xscale_par	Multi-processor version of **xscale** for up to 32 CPUs.	**XSCALE.INP**
xdsconv	Converts reflection intensities determined by XDS to amplitudes in a format suitable for various other program packages.	**XDSCONV.INP**

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XDS.INP Example

The file **XDS.INP** contains several lines, which define:

1. Job control (which sub-programs are run)
2. Parameters defining detector and data collection geometry
3. Selection of diffraction data images
4. Parameters controlling indexing of diffraction images
5. Criteria for accepting reflections
6. Parameters controlling integration and peak profile
7. Background and peak pixels

Many of these parameters have **sensible default values** and need not be changed by the user.

N.B. XDS can read **compressed** image files (tested with .gz). Compress images to save disk space but use uncompressed image name in XDS.INP!

At synchrotron beamlines, an XDS.INP file is automatically created by the data collection system. At home sources, template files exist which have to be modified by hand prior to data processing.

Job control

JOB= ALL !XYCORR INIT COLSPOT IDXREF DEFPIX XPLAN INTEGRATE CORRECT

IDXREF – index diffraction pattern
XPLAN – data collection strategy (usually not needed)
INTEGRATE – integrate images
CORRECT – scale reflection intensities

Main output files:
IDXREF.LP
CORRECT.LP
XDS_ASCII.HKL

More details in the MX tutorials

Processing single-crystal diffraction data - Scaling

Scaling is the process whereby the differences between the **different observations** of the same **independent Bragg peak** (normally due to Laue symmetry) are minimized by applying scale factors to the different images where these observations are recorded.

These differences may arise from several factors, e.g.,

- ✓ Differences in **absorption path** through the crystal (thin plate vs. thick prism)
- ✓ Differences in the **crystal volume** being irradiated (at the synchrotron, the beam size is often much smaller than the crystal size)

If **anomalous dispersion** is present, then the Friedel or Bijvoet mates are treated separately (i.e., h,k,l and –h,-k,-l are no longer considered equivalent).

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Processing single-crystal diffraction data - Scaling

In addition, crystals of macromolecules can experience **radiation damage** when exposed to the very intense X-ray beams at synchrotron sources.

This effect will gradually decrease the effective diffraction power of the whole crystal and is modeled in the scaling process by a **negative exponential function** (the decay B-factor).

Examples of scaling software:

- SCALA or AIMLESS in the CCP4 suite
- CORRECT in the XDS suite
- SCALEPACK in the HKL3000 suite

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Processing single-crystal diffraction data - Scaling

The scaling software will print out different statistics related to the diffraction data set:

The **merging R-factor** is a measure of the **internal consistency** of a diffraction data set

$$R_{merge} = \frac{\sum_{hkl}\sum_{i=1}^{n} |I_i(hkl) - \bar{I}(hkl)|}{\sum_{hkl}\sum_{i=1}^{n} I_i(hkl)}$$

$I_i(hkl)$ is the i-th observation of the intensity with indices hkl

$\bar{I}(hkl)$ is the mean value of that intensity

The **redundancy-independent R-factor** removes the effect of data multiplicity

$$R_{meas} = \frac{\sum_{hkl}\sqrt{\frac{n}{n-1}}\sum_{i=1}^{n} |I_i(hkl) - \bar{I}(hkl)|}{\sum_{hkl}\sum_{i=1}^{n} I_i(hkl)}$$

and is usually higher than R_{merge}

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Processing single-crystal diffraction data - Scaling

The **precision-indicating R-factor** considers the effect of the data multiplicity and estimates the precision of merged data

$$R_{pim} = \frac{\sum_{hkl}\sqrt{1/n-1}\sum_{i=1}^{n} |I_i(hkl) - \bar{I}(hkl)|}{\sum_{hkl}\sum_{i=1}^{n} I_i(hkl)}$$

$I_i(hkl)$ is the i-th observation of the intensity with indices hkl

$\bar{I}(hkl)$ is the mean value of that intensity

Data R-values: $R_{pim} < R_{merge} < R_{meas}$

These and other statistics are usually calculated as a function of resolution: the dataset is divided in resolution shells containing approximately the same number of independent intensities and values are calculated (and output) for each resolution shell.

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Processing single-crystal diffraction data - Scaling

	Overall	InnerShell	OuterShell
Low resolution limit	106.634	106.634	1.425
High resolution limit	1.401	3.801	1.401
Rmerge (all I+ & I-)	0.122	0.081	1.246
Rmerge (within I+/I-)	0.102	0.067	1.055
Rmeas (all I+ & I-)	0.149	0.099	1.518
Rmeas (within I+/I-)	0.144	0.095	1.469
Rpim (all I+ & I-)	0.084	0.056	0.856
Rpim (within I+/I-)	0.101	0.067	1.018
Total number of observations	388915	19536	19264
Total number unique	137073	6952	6855
Mean(I)/sd(I)	3.2	8.5	0.5
Completeness	98.8	97.5	99.5
Multiplicity	2.8	2.8	2.8
CC(1/2)	0.987	0.980	0.335
Anomalous completeness	75.4	81.4	71.3
Anomalous multiplicity	1.6	1.6	1.6
CC(ano)	0.000	-0.030	0.043
|DANO|/sd(DANO)	0.594	0.617	0.530

Processing single-crystal diffraction data - Scaling

Completeness measures how many of the theoretically accessible intensities were measured.

Multiplicity (or Redundancy in some programs) measures how many observations were made on average of each independent intensity.

The **mean I/sig(I)**, represented by <I/sig(I)> is a measure of the diffraction intensity (or signal-to-noise ratio). The value of <I/sig(I)> can be used to determine the high-resolution cut-off for the dataset (e.g., **2.0**)

$CC_{1/2}$ is a correlation coefficient between randomly generated half-datasets. Presently, a $CC_{1/2}$ of **0.3** (30%) is an acceptable high-resolution cut-off for use in phasing and refinement

Processing single-crystal diffraction data - Scaling

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Processing single-crystal diffraction data - Twinning

What is twinning?

Twinning is a phenomenon where multiple domains with differing orientations form within a single crystal - think of it as having several "sub-crystals" contained within a single sample, and the resulting diffraction images from such a twinned crystal will be made up of contributions from each of the sub-crystals.

Twinning can occur in crystals of both small and macromolecules. The likelihood of its occurrence is determined to a large extent by the unit cell parameters and the crystal symmetry.

http://legacy.ccp4.ac.uk/peter/documents/twinning/twinning.html

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Processing single-crystal diffraction data - Twinning

There are two different types of twinning:

Epitaxial (2D overlap): two distinct interpenetrating reciprocal lattices can be seen in the diffraction pattern (two diffraction patterns overlayed in a single image). This can be solved at least in principle, by indexing the two patterns separately, especially if the diffraction pattern from one of them is much stronger than the other.

Merohedral (3D overlap): in this case, the lattices are superimposed. The most common type of merohedral twinning for macromolecules is hemihedral twinning, where there are two distinct domains within the twinned crystal.

As a result of hemihedral twinning, each observed diffraction intensity is a weighted sum of two crystallographically distinct, twin-related, reflections.

http://legacy.ccp4.ac.uk/peter/documents/twinning/twinning.html

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Processing single-crystal diffraction data - Twinning

Hemihedral twinning can be characterized by two parameters:

- The **Twin Fraction** α represents the fractional volume of the crystal that the smaller of the two domains occupies. It takes values between 0.0 and 0.5.
- The **Twin Operator** is the symmetry operation that relates the orientations of the two domains. It is expressed as a mapping of reflection indices from hkl to h'k'l'. Possible twin operators can be deduced from the space group.

http://legacy.ccp4.ac.uk/peter/documents/twinning/twinning.html

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Processing single-crystal diffraction data - Twinning

The **observed intensities** from the twinned crystal are related to the intensities from each of the two domains:

$I_{hkl}(twin) = \alpha\ I_{hkl}(crystal1) + (1-\alpha)\ I_{h'k'l'}(crystal2)$

Depending on the value of α there are two possible situations:

- Perfect twin, with a twin fraction α = 0.5
- Partial twin, with a twin fraction α < 0.5

For a **perfect twin** it is not possible to deconvolute the intensity data.

For a **partial twin** where α is not too close to 0.5 it is possible to deconvolute ("detwin") the data and perform a normal structure determination and refinement.

http://legacy.ccp4.ac.uk/peter/documents/twinning/twinning.html

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Processing single-crystal diffraction data - Twinning

Twinning Tests - there are two tests that can be used to indicate twinning:

Perfect Twinning Test: $\langle I^2 \rangle / \langle I \rangle^2$ should take a value of 2 for untwinned data, and 1.5 for a perfect twin.

Partial Twinning Test: $\langle H \rangle = \langle |I_h - I_{h'}| / (I_h - I_{h'}) \rangle$ can be calculated and gives an indication of the twin fraction from $\alpha = (0.5 - \langle H \rangle)$. However, this requires that the twin operator is already known.

One consequence of perfect or nearly perfect twinning is that the **apparent symmetry of the diffraction pattern will be higher than the true one**, which will make the structure determination very difficult or impossible, and the refinement R-factors (more on this on Wednesday) will be much higher than expected.

http://legacy.ccp4.ac.uk/peter/documents/twinning/twinning.html

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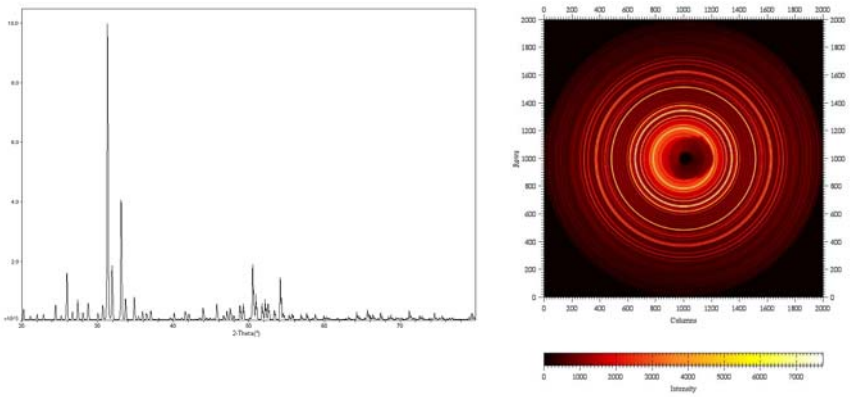
What about powder diffraction data?



In a powder diffraction experiment, a **3-dimensional pattern** is collapsed into **1-dimension** (the Bragg angle 2θ).
This makes it extremely difficult to extract individual intensities due to problems with peak overlap at higher 2θ values and peak shape definition.

https://chem.libretexts.org/Courses/Lafayette_College/CHEM_212_213%3A_Inorganic_Chemistry_%28Nataro%29/03%3A_Solid_state/3.11%3A_Powder_X-ray_Diffraction
https://www.esrf.fr/computing/Forum/imgCIF/PAPER/olivine.html

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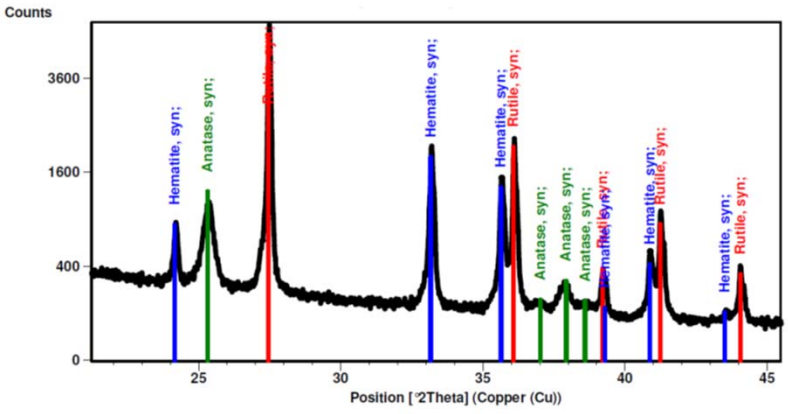
What about powder diffraction data?

The powder diffraction pattern of pure substances is like a fingerprint and can be used to identify which substances are present in a chemical mixture.



http://prism.mit.edu/xray/introduction%20to%20xrpd%20data%20analysis.pdf

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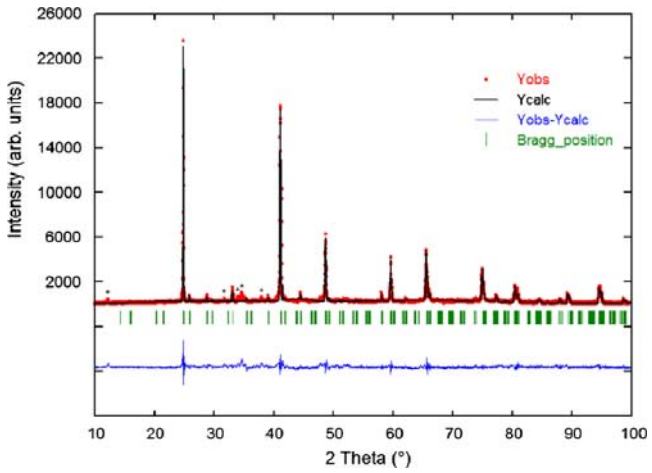
What about powder diffraction data?

If structural information is available, the whole diffraction pattern can be fitted using the Rietveld method and the crystal structure can be refined.



https://www.researchgate.net/figure/Final-plot-of-the-Rietveld-refinement-showing-the-observed-calculated-and-difference_fig2_243230896

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Web resources to explore

https://www.youtube.com/watch?v=QHMzFUo0NL8 - **What is X-ray diffraction?**

https://www.youtube.com/watch?v=H3NeiImTJOA - **Explains the Ewald sphere (in French)**

http://www.doitpoms.ac.uk/tlplib/reciprocal_lattice/reciprocal_lattice.php - **Explains the relationship between direct and reciprocal lattices**

http://www.doitpoms.ac.uk/tlplib/reciprocal_lattice/absences.php - **Explains systematic extinctions (absences) – bottom diagram**

http://www.doitpoms.ac.uk/tlplib/xray-diffraction/bragg.php - **Explains Bragg's Law**

http://www.doitpoms.ac.uk/tlplib/miller_indices/index.php - **Describes Miller indices and Lattice planes**

http://pd.chem.ucl.ac.uk/pdnn/pdindex.htm - **Advanced Certificate in Powder Diffraction on the Web at the Birkbeck College School of Crystallography**

http://www.xtal.iqfr.csic.es/Cristalografia/index-en.html - **General resource for X-ray crystallographic topics**

https://onlinelibrary.wiley.com/iucr/doi/10.1107/S0907444903007947 - **Padilla & Yates paper on the L-test for twinning**

Microsoft PowerPoint - Introduction to XRPD Data Analysis.pptx - introduction to xrpd data analysis.pdf

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