
Software fayre: raw data → hkl file

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Topics

□ **Software fayre: going from raw data to hkl file**

- Experimental and data processing challenges
- PD Vs. CCD
- Choice of the right software
- Quick overview of the software available

□ **Practical session**

- Area detectors (CCD)
- Point detectors (PD)

- Oxford diffraction (Diego Gatta)
- Bruker (Tonci Balic-Zunic)
- STOE (Andrzej Grzechnik, Karen Friese)
- XDS (Andrzej Grzechnik, Karen Friese)

- WinIntegrstp (Ross Angel)

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Practical sessions

SOFTWARE FOR AREA DETECTOR

- Oxford diffraction (Diego & Fabrizio)
- Bruker (Tonci)
- STOE (Karen & Andrzej)
- XDS (Karen & Andrzej)

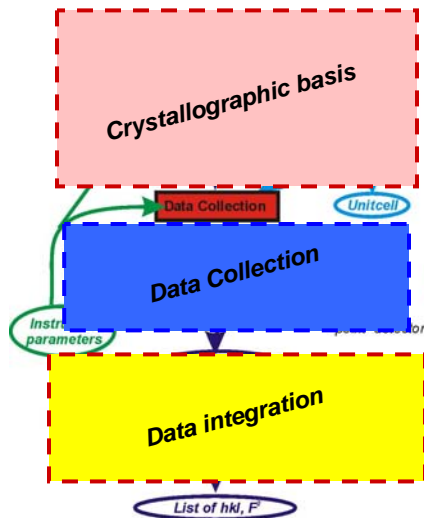
SOFTWARE FOR POINT DETECTOR

- WinIntegrstp (Ross)

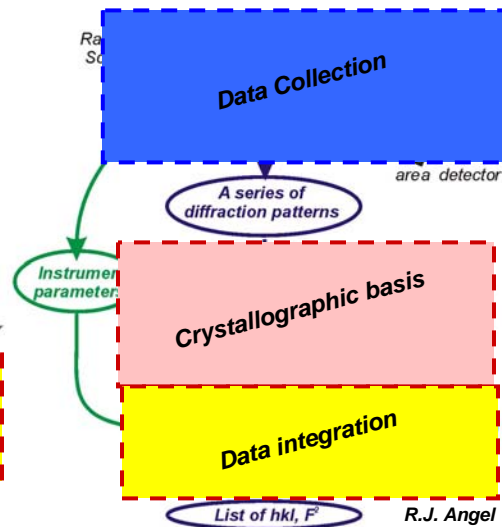
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Intensity data collection

Point detector.



Area detector.



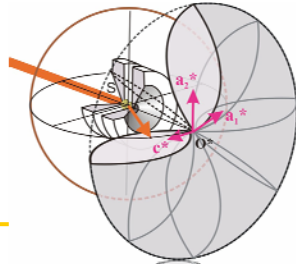
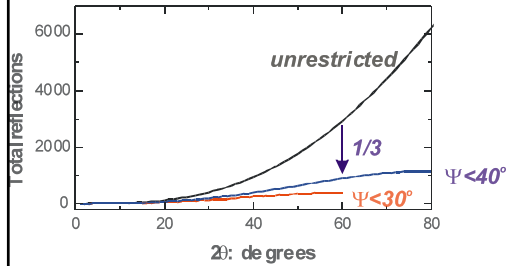
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CCD is "shoot first and ask questions later"

R.J. Angel

Intensities at high pressure: Challenges

Very precise data to measure very small changes in structures at high pressures.



Absorption by DAC's components

- Diffracted intensities reduced

Scattering by DAC's components

- Highly structured background
- Double diffraction

Limited access

- 1/3 or less than the entire dataset
- Limited equivalent reflections
- Rejection of aberrant intensities

Low signal

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Intensities at high pressure: Solutions

Point detector

□ Experimental

- Accurate peak positions (offset)
- Multiple scans
- Higher collimation
- Slits settings
- Detector distance

□ Data processing

- Optimized scan speed
- Optimized integration algorithms (Step scans)
- Profile fitting
- Recovery of weak data

Area detector

□ Experimental

- Poor peak positions (no offset)
- Beryllium cone (post integr. corr)
- Exposure time/generator power
- Detector distance

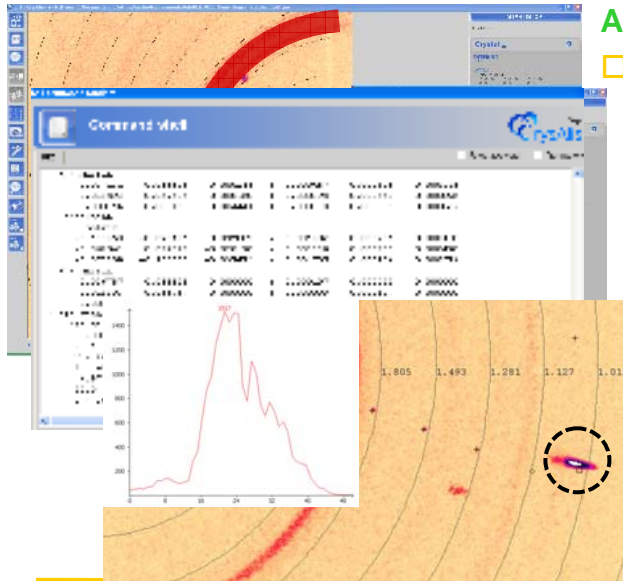
□ Data processing

- Masking solutions (global, local)
- Multiple indexing (diamonds, Qz)
- Overlap checking

Every reflection profile in a data set **must be visually examined** by the experimentalist (open to human misjudgement and errors!!)

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Integration: Area detectors

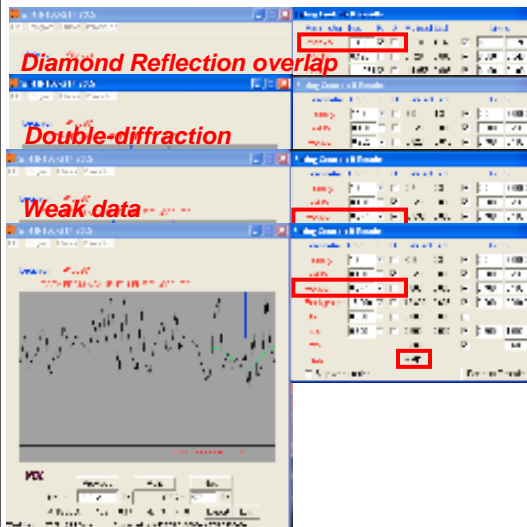


Area detector

- Data processing
 - Masking solutions
 - Global, local
 - Static, dynamic
 - Multiple indexing
 - Diamonds, Qz
 - Overlap checking
 - 3D indexing algorithm

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Integration: Point detector



Point detector

- Data processing
 - Diamond reflections
 - Double-diffraction
 - Optimized scan speed
 - Optimized integration algorithms (Step scans)
 - Profile fitting
 - Recovery of weak data
 - 2D indexing algorithm

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What to know: CCD and point detectors

Point detector

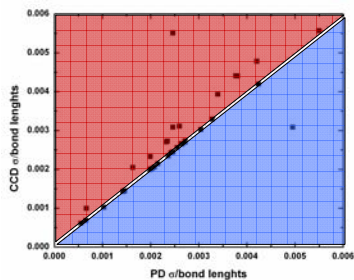
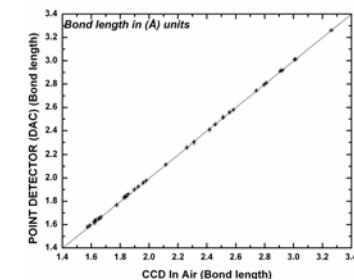
- **Pros**
 - Better/lower background
 - Better handling of the refls
 - Recovery weak data
 - Avoid bad reflections
 - Reflections Offsets
 - Optimised scan speed
 - Optimized integration algorithms (Step scans)
 - Higher collimation (small cells)
- **Cons**
 - Long data collections
 - UB matrix determination
 - Long
 - Not allowed by all the software
 - Possible misindexing

Area detector

- **Pros**
 - Quick Peak hunting
 - Fast intensity data collection
 - Large number of reflections
 - High Redundancy
- **Cons**
 - High background
 - Diffuse scattering
 - Approx equal intensities
 - Equal scan speed
 - Static images
 - No handling of the refls
 - No refls offsets
 - Black box

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CCD and point detectors: Practical example



Structure refinements data

- **Bond distance values**
 - Good agreement between DAC-PD and CCD in air
 - Almost in agreement DAC-PD DAC-CCD
- **Bond distance esd's**
 - Always higher value on CCD
 - Agreement between CCD in air and PD in DAC

Data obtained on the same instrument with two different detectors

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Commercial software available

XDS

http://xds.mpimf-heidelberg.mpg.de/html_doc/downloading.html



http://www.stoe.com/pages/products/software_single.html



<http://www.oxford-diffraction.com/products/software-crysalis-pro/>



<http://www.bruker-axs.de/dataintegration.html>

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CCD – XDS – software

Exchange capabilities

- Support many detector format
 - Pilatus, Marr555, Marr345, marCCD, R-AXIS II, R-AXIS IV, R-AXIS IV++, RAXIS-V, SATURN 92, CRYNALIS, STOE, SIMENS
- HKL conversion to different format

Indexing optimization

- Learned reflection profile
- Spatial correction at each pixel of a detector
- Centering the diffraction peaks
- Refine orientation matrix (UB)
- Defines the obscured regions of the detector

Post integration feature



Wolfgang Kabsch, author of the 100 000th publication on Crystallography Journals Online.

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CCD – XDS – software

Pro

- Free of charge
- High compatible with several instruments (home labs and large scale facilities)
- High level useful high pressure features

Cons

- No reciprocal space reconstruction
- No incommensurate phases
- Not so much interactive (black box)

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CCD – STOE – software



Exchange capabilities

- Support many detector format
 - Stoe IPDS II, Stoe IPDS 2T, IPDS 2T dual beam, Pilatus,
 - HKL conversion to different format

Indexing optimization

- Overlap Check
- Masking (beam stop, Be ring and diamond peaks)
- Multi orientation matrix feature, up to 6 matrices (twins, diamond overlap etc..)
- Refine orientation matrix (UB)
- Defines the obscured regions of the detector (each frames has it own mask)
- Incommensurate structures
- Reciprocal space reconstruction

Post integration feature

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CCD – STOE – software



Pro

- Free of charge
- High level useful high pressure features
- Reciprocal space reconstruction
- Quite much interactive (allowed a lot of user intervention)

Cons

- Not Highly compatible with several instruments
- Hand made masking of the shadowed areas of the detector
- Use of the mask to optimize the integration profile

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CCD – Oxford Diffraction – software



Exchange capabilities

- Support many detector format
 - Marr555, Marr345, marCCD,
- HKL conversion to different format

Indexing optimization

- Centering the diffraction peaks
- Refine orientation matrix (UB)
- Defines the obscured regions of the detector
- Masking (beam stop and Be ring)
- Multiple cells handling (twins, diamonds etc...)

Post integration feature

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CCD – Oxford Diffraction – software



□ Pro

- Free of charge
- Compatible with different instruments (home labs and large scale facilities)
- High level useful high pressure features

□ Cons

- No feature for the masking of a single reflection (e.g. Diamond peaks)
- Not extremely interactive

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CCD – Bruker – software (SaintPlus)



□ Exchange capabilities

- Support many detector format
 - Marr555, Marr345, marCCD, Pilatus coming soon (other detector formats via conversion software)
- HKL conversion to different format

□ Indexing optimization

- Hand picking of the reflections
- Centering the diffraction peaks
- Refine orientation matrix (UB)
- Multiple cells handling
- Masking areas of the detector

□ Post integration feature

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CCD – Bruker – software (SaintPlus)



□ Pro

- Comes with the instrument (\$\$\$)
- Compatible with many instrument (Thanks to the conversion software)
- Hand picking of the reflections

□ Cons

- Tricky handling of the unit cell parameters and multiple cells
- Difficult handling of the overlapping (diamond peaks)
- Not so much interactive (black box)

Very recommended OLD versions of the software!!!

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PD – WinIntegrSTP – software



□ Exchange capabilities

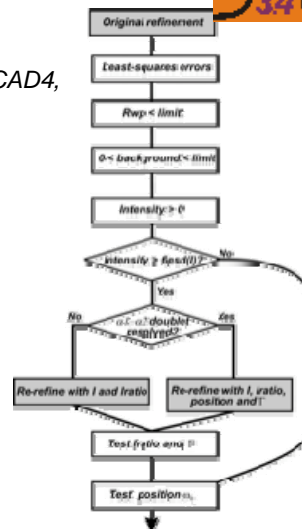
- Support many point detector format
 - Xcalibur, Xscans, STOE, Philips Febo, CAD4, old version Single
- HKL (shelx or Rfine formats)

□ Indexing optimization

- Step scans integration
- Visual examination of the refls
- Centering the diffraction peaks
- Refine orientation matrix (UB)
- Different background refinements choice

□ Post integration feature

- No post integration features
- Need other softwares



Angel R.J. (2003) J. Appl. Cryst. 36, 295–300

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PD – WinIntegrSTP – software



□ Pro

- *Free of charge*
- *High compatible with several instruments (home labs and large scale facilities)*
- *Mainly designed for High pressure data integration*
- *Continuously developed*
- *Flexible!!*

□ Cons

- *.....Please ask to Ross.....*