CrysAlis\textsuperscript{Pro}: Data reduction different image formats

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Area detector diffraction experiments

- Understand the experiment
- Gonirometer description
- Sense of rotation
- Area detector ‘perspective’
- Zero points

- Kappa vs. Euler geometry

Workflow on in-house instruments

- Describe instrument
- Run fully automatic calibration experiment describing fully the instrument at any distance
- Run test data collection on standard sample
- Run new experiment.
Workflow on inhouse instruments

Describe instrument
Workflow on in-house instruments
Run fully automatic calibration experiment describing fully the instrument at any distance

Full calibration experiment dialog (2.0.4)

Path
Name: HyPix6000_81016

Basic system parameters
- Dd zero(Cu) = -4.00000
- Dd zero(Mo) = 4.00000
- Overflow threshold = 1048900
- Beam step support orientation = gomni(nkll), HyPix 1x1 binning, Flood correction: off

Options
- Crystal: Cytidine
- Lattice type: P-lattice
- Lattice min: 2
- Lattice max: 30

Settings
- Generator at end of experiment: On
- Calibration mode: Optional

User mode parameters
- Parameters: No constraints, number of reflections to find = 5000 (2000 for quick calibration)

Cu
- Near
  - kV: 50.00
  - mA: 1.00
  - Exposure time the same for all theta positions: 10.0s/deg, Detector distance = 35.00, Scan range = 20.0, Scan width = 0.1,

Mo
- Near
  - kV: 50.00
  - mA: 1.00
  - Exposure time the same for all theta positions: 5.0s/deg, Detector distance = 30.00, Scan range = 10.0, Scan width = 0.1

User message
- Cytidine cell: a = 5.120, b = 14.000, c = 14.800, alpha = 90.000, beta = 90.000, gamma = 90.000
- You have changed cell type parameters, you must regenerate calibration run lists, please click 'Find experiments' button

End of calibration: Sat Oct 08 23:52:17 2016 (4h11m)
Workflow on in-house instruments

Run test data collection on standard sample
Workflow on in-house instruments
Run new experiment
Import: Worse situation

- Depending on the format the instrument model is only known so-so.
- Rather than on a 'Standard sample' the import is tried on something difficult.
- 'Easy': Import of known formats
- 'Complex': Esperanto import
First meeting with the data set

- Install CrysAlisPro: this will install a tool called 'CAPFrameView'. You can inspect all known image formats.
- Use of a file inspection program: 'Norton commander'
- A rename tool.
First meeting with the data set
First meeting with the data set
First meeting with the data set
First meeting with the data set

rename
Import tools

1. Easier access to import/export options (on power toolbar).
2. Organized export options.
3. Importing external images on one clique (instead of typing commands).
4. Esperanto importer for non standard image types.
Known formats and esperanto
External Frame & File Formats

Rigaku – dtrek format

Run list and aliases file generator for DTREK data collections

This dialog allows you to quickly generate a *.run file and aliases file for the data reduction of a DTREK data set.

1. Select an image, by clicking 'Browse' button in group box First dc DTREK dc file, e.g. name001.img
2. Choose whether instrument model will be loaded from selected image header or selected par file
3. Terminator format is automatically set, but you can change it if necessary
4. Also the last image will be automatically found, but you can change it if necessary (if it is not automatically found) Note: It is assumed that all frames between these two are available
5. Save the file

- You will be prompted for entering some critical parameters (usually default values are OK, as they are taken from image headers)
- Finally a new CrysAlisPro instance will be launched with the DTREK data set added to the experiment list

NOTE: Using CrysAlisPro you can process only DTREK images from selected Rigaku detectors! R-AXIS format is not supported!

- 1. First dc DTREK dc file (*001.img)

   ![Browse Button]

   E:\data\2015\04\Alaska Images_2\MM007Cu\140304_screen0001.img

   Image information: detector distance=35.00, wavelength=1.541870, centerx=244.4, centery=206.2, Si thickness=1.000nm

- 2. Instrument model

   ![Browse Button]

   E:\data\2015\04\Alaska Images_2\MM007Cu\140304.par

   Par file information: detector distance=34.71, wavelength=1.540562, centerx=243.1, centery=204.4, beamstop type: DEF, b1=0.019; d1=0.052; d2=0.420; o0=90.205; t0=0.216; a1=90.000; be=0.000

- 3. Terminator format

   Run digits: 0, Separator: None, Frame digits: 4
   140304_screenFFFFF.img

- 4. Last dc DTREK dc file

   ![Browse Button]

   E:\data\2015\04\Alaska Images_2\MM007Cu\140304_screen0018.img

   [Help] [Run TC] [Cancel] [Save run file]
Example: Apex data from ECM-Basel

- Handling of twin data…
- Dr. Daniel Kratzert (Freiburg, Germany) reduced a Bruker Apex2 twin with CAP and Saint.
- This presentation gives the workflow for importing and reducing Bruker data and handling twinning
- For the evaluation the forum version 38.43 was used.
Data set import

- Open any existing experiment
- Import button
Data set import

- Open any existing experiment
- Import button
Data set facts

- Mo, Iμs, no Si filter showing 3λ effect
- Frame width: 0.4, correlated frames

CrysAlisPro run list data

Data collection mode: correlated
Total number of frames: 2889 (scan: 2889, reference: 0)
Disk space: 763.79Mb
Approximate data collection time (h:min): 10:55

<table>
<thead>
<tr>
<th>#</th>
<th>t</th>
<th>start</th>
<th>end</th>
<th>width</th>
<th>exposure</th>
<th>speed-rat</th>
<th>omega</th>
<th>theta</th>
<th>kappa</th>
<th>phi</th>
<th># to do</th>
<th># done</th>
</tr>
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<td>1</td>
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<td>1.386</td>
<td>184.186</td>
<td>0.400</td>
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<td>5.000</td>
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<td>30.000</td>
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<td>456</td>
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<tr>
<td>2</td>
<td>o</td>
<td>-11.114</td>
<td>171.686</td>
<td>0.400</td>
<td>3.000+</td>
<td>3.000</td>
<td>0.000</td>
<td>-</td>
<td>17.500</td>
<td>73.926</td>
<td>4.186</td>
<td>456</td>
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<td>o</td>
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<td>163.775</td>
<td>0.400</td>
<td>3.000+</td>
<td>3.000</td>
<td>0.000</td>
<td>-</td>
<td>10.000</td>
<td>73.926</td>
<td>86.686</td>
<td>356</td>
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<tr>
<td>4</td>
<td>o</td>
<td>21.786</td>
<td>174.186</td>
<td>0.400</td>
<td>3.000+</td>
<td>3.000</td>
<td>0.000</td>
<td>-</td>
<td>20.000</td>
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<td>5</td>
<td>o</td>
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<td>5.000+</td>
<td>5.000</td>
<td>0.000</td>
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<td>-27.500</td>
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<td>11.685</td>
<td>386</td>
</tr>
<tr>
<td>6</td>
<td>o</td>
<td>18.386</td>
<td>179.186</td>
<td>0.400</td>
<td>4.000+</td>
<td>4.000</td>
<td>0.000</td>
<td>-</td>
<td>25.000</td>
<td>73.926</td>
<td>9.184</td>
<td>401</td>
</tr>
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<td>7</td>
<td>o</td>
<td>-7.815</td>
<td>174.186</td>
<td>0.400</td>
<td>3.000+</td>
<td>3.000</td>
<td>0.000</td>
<td>-</td>
<td>20.000</td>
<td>73.926</td>
<td>-60.614</td>
<td>454</td>
</tr>
</tbody>
</table>
First opening of the data set

- Select SM/PX nature
Set the beam stop

- Apex system sometimes use a user angle for the beam stop
First peak hunting

• Use default
Automatic unit cell finding

- Use default, 66% indexed, in spite of slightly off model
First instrument model refinement

- The header info is not precise. Refine on full data.
- Use default
Use EwaldPro to find the twin

• Find next component in wrong peaks
Use EwaldPro to find the twin

- Easy: 180 deg rotation twin
Use EwaldPro to find the twin

- Back to lattice wizard…
Do twin data reduction

- Run the data reduction wizard. Twin reduction auto set…
Do twin data reduction

- Accept the run list
Do twin data reduction

- Special pars: All default; Limit the data to 0.75Ang (as done in the B set)
Do twin data reduction

- Background: Smart background

This is the most critical for the data set: It seems that the Apex detector has unstable background, thus introducing a bias on the average background method. Such behavior is also seen with other detectors where CAP uses by default Smart background.
Do twin data reduction

- Automatic outlier rejection: default
Do twin data reduction

- Chem. formula imported for AutoChem
Automatic result

- Auto: based on all hklf4 data.
Tuning your result

- Tuning means what kind of scaling is used and what reflection classes are in the final hklf4 and 5 files.
HKLF4 result
HKLF5 result
Generic image format ‘Esperanto’ generator

- Use of Esperanto format for unknown image formats with no compression or known formats with strange instrument configs.
- Esperanto fully supports 4 circle instruments.
- Command ‘dc rit’ rit = raw image transform.
- But it also supports the known formats to handle ‘unusual’, obstinate images.
- Pixel detectors have an automatic dead zone detection based on the special value -1.
- The Esperanto generator uses a proprietary Agilent bit field format. To get back the uncompressed Esperanto version, please use the export function.
Esperanto with bit field compression and pixel detector support

- For our Esperanto importer we now can use Agilent bitfield compression for Esperanto files. The format is not documented yet.

- There was also a pixel detector flag added to support the implemented pixel detector corrections ([dsithicknessmmforpixeldetector] - thickness of Si for pixel detectors; the presence of this number signals a pixel detector. Pixel detector gap zones are marked with -1).

- Publication describes the Esperanto format

Single-crystal diffraction at the Extreme Conditions beamline P02.2: procedure for collecting and analyzing high-pressure single-crystal data

André Rothkirch, G. Diego Gatta, Mathias Meyer, Sébastien Merkel, Marco Merlini and Hanns-Peter Liermann

Dc rit: ADSC 315

- Command dc rit
- Header bytes 1024, x 3072 y 3072 and other info from text header (f.ex with total commander)
- Then esperanto creatorunlist
- Slight play in EwaldPro to get the center right.
Dc rit: ADSC 210

- Command dc rit
- Header bytes 1024, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto creatorunlist
- Slight play in EwaldPro to get the center right.
Dc rit: MAR165ccd

- Command dc rit
- Known format MAR (mccd)
- Then esperanto create runlist
- Slight play in EwaldPro to get the center right.
Dc rit: A200 detector

- Command dc rit
- Header bytes 3584, x 2048 y 20482 and other info from text header (f.ex with total commander)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.
Dc rit: Diamond ID 19 Dectris turned

- Command dc rit
- Use of known format dectris. Header values are read.
- Camera turned 270deg. Non-square detector is padded by zeros.
- The header scan values are wrong by 3% (Scan scale err 0.97)
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.
- The several cycles to refine instrument model.
Dc rit: Diamond ID 19 Dectris turned II

- Command dc rit
- Use of known format dectris. Header values are read.
- Camera turned 270deg. Non-square detector is padded by zeros.
- This data had NO scan scale error!
- Then esperanto createrunlist
- Slight play in EwaldPro to get the center right.
- The several cycles to refine instrument model.
Dc rit: IPDS

- Command dc rit/Import button on power toolbar
- Read detector information from the sum file of IPDS (0.15mm pix, cen x=600, y=600)
- Stoe char as pixel type; .xi files (this is OD compression…)
- Make sure to use resolution limit due to round IP image (Mo typical 0.809Ang)
Dc rit: IPDS 2

- Command dc rit/Import button on power toolbar
- Read detector information from the sum file of IPDS (0.15mm pix, cen x=1133, y=1133)
- Stoe char as pixel type; .xi files (this is OD compression…)
- Make sure to use resolution limit due to round IP image (Mo typical 0.809Ang)
Dc rit: dtrek frame from Japanese synchrotron

- Command dc rit/Import button on power toolbar
- The issue was here that the dtrek image was turn 90deg relative to the inhouse image. The dtrek creatrunlist would not work on this.
- Thus the ‘dc rit’ command can be used to handle obstinate known images…
Dc rit: xpad detector

- Command dc rit/Import button on power toolbar
- The xpad detector is developed in France
- One of it’s raw format can be channelled through the Esperanto importer
- As the header info is unknown, it has to be given in the scan info section.
- Provide the raw data file contains the -1 marker for pixel detectors the esperanto creatorunlist command will automatically create a ccd file with dead zones.
- Such formats have to be transformed run by run as there is only one field for scan info.
Data finalization – optimal data

- Problems with...
  - Experiment
  - Unit cell
  - Data reduction
  - Finalization
  - Pseudo symmetry, twinning, incommensurate
Experiment

- Problems with...
  - Exposure time too low – diffraction limit
  - De-ice
  - Movie missing
  - Centering – sample mount
  - Choice of wavelength
Approach a data set...

- Inspect executive tab

- Warning signs:
  - Run list incomplete
  - High mosaicity
  - Scaling unusual
  - I/sig low; low redundancy
  - SG issues
Approach a data set...

- Run ‘Full auto analysis’ on all data
- Concurrent may get stuck

[Image of software interface]

- Load new experiment
- Full auto analysis (cell, red)
- Automatic data reduction
- Data reduction with options
Approach a data set…

- Inspect frames
- No diffraction at high angle $\rightarrow$ cut the data to that resolution

Diffraction limit!
Approach a data set…

- Inspect frames:
  - Low/high background
  - Diffuse scattering, split reflections, twin
  - Empty frames, strange frames

- Inspect the sample movie (if you have it…):
  - Sample mounting
Unit cell...

- Ewald Explorer
Unit cell finding – Automatic unit cell finding
Unit cell finding –
Influence of bad instrument model

Good instrument model

Bad instrument model:
Beam center deviation 40 pix
Unit cell finding –
Influence of bad instrument model

Bad instrument model:
Beam center deviation 40 pix

Bad instrument model:
Delta-peak table
Unit cell finding –
Influence of bad instrument model

![Graph showing the relationship between unit cell size and shift](image)

- **Peak table**: Normal peak table, Delta (differential) peak table
- **Find center**: On
- **Use search box**: On

Rigaku Oxford Diffraction
No! Just a “jumping” crystal
Unit cell (wobble).
Unit cell (ice ring)…
Unit cell (filters)…
Unit cell (filters)…
Unit cell (filters)…
Ewald Pro Collapse view

Collapse peak view – construction

1. Lattice vectors are multiplied by lattice overlay size

2. Subtract lattice vectors until given peak hits range
Ewald Pro Collapse view

Collapse peak view – twin example

Collapsed view:

Indexed twin

Unindexed twin

Orthogonal view
Ewald$^\text{Pro}$ Collapse view

Collapse peak view – other examples

- Moving crystal
- Multicrystal
- Incommensurate
Data reduction
During experiment - concurrent data processing

- In most cases provides good, close to optimal results

- Features improving data quality:
  - Robust prediction model refinement
  - Selection of background evaluation mode
Data reduction
Concurrent data processing - Robust cell / model refinement

• The key problem:
  • Inaccurate initial cell from pre-exp
  • Cell/Model can’t predict well low/high theta
  • Misaligned/jumping sample

• Solution:
  PROFFITPEAK module does standard peak-hunting and cell/orientation refinement before profile learning / analysis phase
Data reduction
Concurrent processing - Automatic selection of background

Average background level > threshold

No
Yes

• Average background (Re>10 frames)
  - Background image estimated once per specified frame range
  
Re = Fr (default, >10 frames)

• Smart background
  - Local background recomputed on every frame and fitted individually for every spot
  
Frame range: 1 (default), 3 or more available through the wizard

Rigaku
oxford diffraction
Data reduction
Use of lattice filters

Profile fitting data reduction

Step 1: Orientation matrix for data reduction

<table>
<thead>
<tr>
<th>qB matrix:</th>
</tr>
</thead>
<tbody>
<tr>
<td>-0.016289</td>
</tr>
<tr>
<td>-0.018899</td>
</tr>
<tr>
<td>0.027249</td>
</tr>
<tr>
<td>19.21267</td>
</tr>
<tr>
<td>0.922000</td>
</tr>
</tbody>
</table>

Y = 3653.09

Selected cell (from 0M 0M 0M ttt/OM 0M):
18 19.2129 19.4106 9.7602 89.9228 90.0620 90.0600 ti

Lattice extinctions (filter Bravais lattice extinctions)
- Don't use filter (C lattice)
- Use filter for: H lattice

Incommensurate structures
- Normal data reduction (HKL)
- Single q vector
- Other (reduction list)

Twining/Multi crystal (activated by UM TWIN entries)
- Use automatic twin/multi crystal data reduction with the following components:
  - Component 1
  - Component 2
  - Component 3
  - Component 4
  - Multi crystal
Data reduction
Data reduction wizard

Settings worth attention:

• Model refinement options

• Special parameters:
  - Integration mask size
  - Bad profile filter

• Background options
Data reduction
3D profile fitting

- Distorted rotation method data is mapped to ‘Kabsch-space’. Similar like XDS, but more complex

- Strong reflection data serve as reference profile. No shape assumption is made!

- All data is profile fitted to the ‘nearby’ reference profile. For strong data this means summation, for weak filtering

- You may choose to take less/more than 4sig of reference profile
Data reduction
Special pars- outliers

- Filter intruders by correlation coefficient.

- All strong reflections are self similar.
### Profile size analysis (per incidence angle)

<table>
<thead>
<tr>
<th>Incidence angle (deg)</th>
<th># of peaks</th>
<th>$\text{sig}_e_1$ (stddev)</th>
<th>$\text{sig}_e_2$ (stddev)</th>
<th>$\text{sig}_e_3$ (stddev)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0-12.6</td>
<td>769</td>
<td>1.639 (0.304)</td>
<td>1.430 (0.233)</td>
<td>1.757 (0.880)</td>
</tr>
<tr>
<td>12.7-18.1</td>
<td>769</td>
<td>1.624 (0.354)</td>
<td>1.430 (0.242)</td>
<td>1.649 (0.875)</td>
</tr>
<tr>
<td>18.1-22.5</td>
<td>769</td>
<td>1.624 (0.357)</td>
<td>1.431 (0.258)</td>
<td>1.572 (0.828)</td>
</tr>
<tr>
<td>22.5-26.2</td>
<td>769</td>
<td>1.594 (0.369)</td>
<td>1.414 (0.258)</td>
<td>1.542 (0.839)</td>
</tr>
<tr>
<td>26.2-29.9</td>
<td>769</td>
<td>1.627 (0.372)</td>
<td>1.410 (0.282)</td>
<td>1.440 (0.736)</td>
</tr>
<tr>
<td>29.9-32.8</td>
<td>769</td>
<td>1.630 (0.364)</td>
<td>1.382 (0.255)</td>
<td>1.391 (0.719)</td>
</tr>
<tr>
<td>32.8-35.8</td>
<td>769</td>
<td>1.594 (0.341)</td>
<td>1.341 (0.267)</td>
<td>1.392 (0.708)</td>
</tr>
<tr>
<td>35.8-38.9</td>
<td>769</td>
<td>1.632 (0.349)</td>
<td>1.337 (0.266)</td>
<td>1.315 (0.594)</td>
</tr>
<tr>
<td>38.9-41.9</td>
<td>769</td>
<td>1.638 (0.330)</td>
<td>1.294 (0.269)</td>
<td>1.303 (0.659)</td>
</tr>
<tr>
<td>41.9-51.6</td>
<td>769</td>
<td>1.618 (0.323)</td>
<td>1.221 (0.270)</td>
<td>1.188 (0.610)</td>
</tr>
<tr>
<td>0-51.6</td>
<td>7690</td>
<td>1.622 (0.347)</td>
<td>1.369 (0.269)</td>
<td>1.455 (0.769)</td>
</tr>
</tbody>
</table>

**Fitted profile normalization line parameters**

- $e_1$ dimension: $a=0.0022$ $b=0.99$
- $e_2$ dimension: $a=-0.0019$ $b=1.08$
- $e_3$ dimension: $a=-0.0136$ $b=1.46$
Data reduction
Prediction uncertainty – problem

- Problem lies in spot prediction uncertainty
- Higher inaccuracy at high theta

For profile size of 0.8 deg integration mask size is 8 pixels at incidence angle 0 deg (Atlas detector, 2x2 binning, at 55 mm)

<table>
<thead>
<tr>
<th>Detector theta (deg)</th>
<th>Std dev of misprediction (deg)</th>
<th>Std dev of misprediction (pix)</th>
<th>Max misprediction (pix)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11</td>
<td>0.014</td>
<td>0.13</td>
<td>0.78</td>
</tr>
<tr>
<td>24</td>
<td>0.02</td>
<td>0.2</td>
<td>1.2</td>
</tr>
<tr>
<td>40</td>
<td>0.025</td>
<td>0.25</td>
<td>1.5</td>
</tr>
<tr>
<td>111</td>
<td>0.04 – 0.08</td>
<td>0.4 – 0.8</td>
<td>2.4 – 4.8</td>
</tr>
</tbody>
</table>
Data reduction
Prediction uncertainty – solution

• Estimate prediction uncertainty at given theta

<table>
<thead>
<tr>
<th>Resolution</th>
<th># of peaks</th>
<th>delta_e1 (stdev)</th>
<th>delta_e2 (stdev)</th>
<th>delta_e3 (stdev)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.35-1.22</td>
<td>82</td>
<td>0.002</td>
<td>0.020</td>
<td>0.001 ( 0.013)</td>
</tr>
<tr>
<td>1.22-1.14</td>
<td>82</td>
<td>-0.002</td>
<td>0.025</td>
<td>0.009 ( 0.016)</td>
</tr>
<tr>
<td>1.14-1.06</td>
<td>82</td>
<td>0.003</td>
<td>0.024</td>
<td>0.004 ( 0.020)</td>
</tr>
<tr>
<td>1.08-1.04</td>
<td>82</td>
<td>-0.002</td>
<td>0.034</td>
<td>-0.002 ( 0.031)</td>
</tr>
<tr>
<td>1.04-0.99</td>
<td>82</td>
<td>0.001</td>
<td>0.026</td>
<td>-0.003 ( 0.021)</td>
</tr>
<tr>
<td>0.99-0.95</td>
<td>82</td>
<td>-0.007</td>
<td>0.033</td>
<td>-0.005 ( 0.030)</td>
</tr>
<tr>
<td>0.95-0.91</td>
<td>82</td>
<td>-0.005</td>
<td>0.036</td>
<td>-0.009 ( 0.039)</td>
</tr>
<tr>
<td>0.91-0.87</td>
<td>82</td>
<td>-0.008</td>
<td>0.036</td>
<td>-0.018 ( 0.041)</td>
</tr>
<tr>
<td>0.87-0.84</td>
<td>82</td>
<td>0.006</td>
<td>0.036</td>
<td>-0.012 ( 0.041)</td>
</tr>
<tr>
<td>0.84-0.80</td>
<td>73</td>
<td>0.005</td>
<td>0.035</td>
<td>0.017 ( 0.007)</td>
</tr>
</tbody>
</table>

• Enlarge integration mask according to prediction uncertainty

Standard mask → Enlarged mask
Data reduction
Model refinement options

No need to decide – program does it for you!
Data reduction
Model refinement options

- 2-cycle PROFFITPEAK
- Covers more difficult “wobbling” cases

PROFFITPEAK
For every run

- Profile analysis
- Per-run refinement
- Per-frame refinement
- Choose more accurate approach
- Profile analysis

PROFFITMAIN
For every run

- Cell/model refinement
- Per-run refinement
- Per-frame refinement
- Choose more accurate approach
- Integration
Data reduction
Model refinement options

- Model refinement with matrix orientation search
- For discontinuous sample jumps (LT, flexible holder, not fixed properly, etc.)

**PROFFITMAIN**

For every run

- Cell orientation search
- Profile analysis
- Per-run refinement
- Per-frame refinement

Choose more accurate approach

**PROFFITPEAK**

For every run

- Cell orientation search
- Profile analysis
- Per-run refinement
- Per-frame refinement

Choose more accurate approach

Integration
Manual Data Reduction
Sample slippage example

- Superficially gives impression of twin
  - Twin indexing matches 96.8% of reflections

TWIN ANALYSIS

<table>
<thead>
<tr>
<th>Component</th>
<th>Ratio</th>
<th>Isolated</th>
<th>Overlapped</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.37</td>
<td>1676</td>
<td>24</td>
</tr>
<tr>
<td>2</td>
<td>0.63</td>
<td>1690</td>
<td>24</td>
</tr>
</tbody>
</table>

DECOMPOSED TWIN DATA STATISTICS (<0.80 overlap)

<table>
<thead>
<tr>
<th>Component</th>
<th>Redundancy</th>
<th>F2/sig(F2)</th>
<th>Rint</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.5</td>
<td>9.7</td>
<td>0.245</td>
</tr>
<tr>
<td>2</td>
<td>1.5</td>
<td>7.0</td>
<td>0.658</td>
</tr>
</tbody>
</table>

Overlap limit for HKLF4 export: 0.80

TWIN HKLF5 STATISTICS FOR OVERLAPPED OBS

<table>
<thead>
<tr>
<th>Components</th>
<th>Redundancy</th>
<th>F2/sig(F2)</th>
<th>Rint</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2</td>
<td>1.3</td>
<td>24.8</td>
<td>0.223</td>
</tr>
</tbody>
</table>

... but twin integration doesn’t help
Manual Data Reduction
Sample slippage example

Lattice wizard

LATTICE
Current cell (CSD: 6 hits)
3.977(2) 15.622(7) 9.424(12) 90.08(7) 98.83(8) 89.98(4) 578.5(8)
Constrained current cell
No constrained unit cell present!
Lattice reduction
selected cell
3.9790 9.4181 15.6360 89.9940 89.9391 81.0464 578.8
reduced cell
3.9790 9.4181 15.6360 89.9940 89.9391 81.0464 578.8

PEAK TABLE
Peak hunting table
UB fit with 477 obs out of 893 (total:893,skipped:0) (53.42%) After per-run orientation adjustment:
UB fit with 860 obs out of 893 (96.30%)

INSTRUMENT MODEL
Goniometer
beam: 0.06483 alpha: 50.06949 beta: -0.06608
om zero: 0.08435 th zero: -1.35848 ka zero: -0.05339
Detector
x-rot: 0.45333 y-rot: -0.14434
x-cen: 1026.96119 y-cen: 1038.70213 distance: 50.00000
Wavelength Cu (Ang): A1 1.54056 A2 1.54439 B1 1.39222
Manual Data Reduction

Sample slippage example

RESULTS (466 frames) - SYM: P2/m (b-unique)
Resolution(A) Redundancy F2/sig(F2) Rint
inf - 0.80 1.5 22.4 0.016
inf - 0.84 1.5 23.2 0.016
Completeness: 98.4% (0.84 ANG)
# Manual Data Reduction

Sample slippage example

<table>
<thead>
<tr>
<th>No slip</th>
<th>Intentional sample slip</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Reference data</td>
</tr>
<tr>
<td>$R_{\text{int}}$ (%)</td>
<td>1.6</td>
</tr>
<tr>
<td>$I/\sigma$</td>
<td>22.0</td>
</tr>
<tr>
<td>$R_1$ (%)</td>
<td>3.32</td>
</tr>
<tr>
<td>Comp (%)</td>
<td>98.5</td>
</tr>
</tbody>
</table>
Manual Data Reduction
Background correction

Choice of background correction:

- Average background
  - Define range and frequency
- Smart background
  - Combination of local and average background
  - Improves statistics \((I/\sigma)\) for samples with varying background features
Manual Data Reduction
Background correction

Well-mounted sample

Standard peak-hunting produces many non-lattice peaks.

Intentionally badly-mounted sample
# Manual Data Reduction

## Background correction

<table>
<thead>
<tr>
<th></th>
<th>Well-mounted</th>
<th>Badly-mounted</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Reference Data</td>
<td>Auto analysis</td>
</tr>
<tr>
<td><strong>Time</strong></td>
<td>3h 8m</td>
<td>16h 18m</td>
</tr>
<tr>
<td><strong>Diff. Limit</strong></td>
<td>0.92</td>
<td>1.41</td>
</tr>
<tr>
<td><strong>R(_{\text{int}}) (%)</strong></td>
<td>3.1</td>
<td>14.4</td>
</tr>
<tr>
<td><strong>I/(\sigma)</strong></td>
<td>13.5</td>
<td>3.0</td>
</tr>
<tr>
<td><strong>R(_1) (%)</strong></td>
<td>5.28</td>
<td>-</td>
</tr>
<tr>
<td><strong>Comp (%)</strong></td>
<td>99.5</td>
<td>99.5</td>
</tr>
</tbody>
</table>
Manual Data Reduction

Bad profile rejection

Simulation of cryo-failure (shield flow turned off)
## Manual Data Reduction

### Bad profile rejection

<table>
<thead>
<tr>
<th></th>
<th>No ice</th>
<th>Large ice build-up</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Reference Data</td>
<td>Auto analysis</td>
</tr>
<tr>
<td>$R_{\text{int}}$ (%)</td>
<td>2.6</td>
<td>10.5</td>
</tr>
<tr>
<td>$I/\sigma$</td>
<td>17.7</td>
<td>8.4</td>
</tr>
<tr>
<td>$R_1$ (%)</td>
<td>3.83</td>
<td>6.11</td>
</tr>
<tr>
<td>Comp (%)</td>
<td>99.9</td>
<td>99.9</td>
</tr>
</tbody>
</table>
Manual Data Reduction
XX PROFFITLOOP - ultimate tool

- Not satisfied with the results, consider trying different option combinations using PROFFITLOOP tool

Caution! This tool may run for a long time and generate a lot of output files with gibberish names
Manual Data Reduction

XX PROFFITLOOP - ultimate tool

• Afterwards browse the results in the finalizer window
Post-integration empirical corrections

- Scaling
- Empirical absorption based on spherical harmonics
- Decay
- Detector sensitivity

Try manual settings!
Remark on scaling of non-centro data

Unit cell finding
Profile learning
Integration
Post correction

Integration
Average unit cell
Absorption
Beam illumination
Empirical corrections
Error model
Outlier rejection
Point/space group
Optional merging
Remark on scaling of non-centro data

• Organic samples – Friedel pairs equivalent
• Samples with heavier atoms – scaling with Friedel pairs merged may destroy anomalous signal info
• Version 38 makes automatic decision based on provided formula (“heavy” means Si for Cu, Sc for Mo)
Optimal data – hints at experiment time

- Centering/Sample choice/holder/amount of oil
- Low T: de-ice runs
- Absorption: make movie 1-6 deg
- Concurrent data red re-start
- Cu – Mo choice
- Collect redundant data
- Rather reduce scan width than increase dd
- Check your diffraction limit
Optimal data – hints at unit cell finding time

- Check for non-indexing reflections
- Garbage (ice rings, powder), twin, sample jump
- Re-run refine model
- Ewald
  - Use filters (intensity, lattice type) and groups
  - Use intensity view
- Check chemical formula unit cell consistency
Optimal data – hints at data reduction time

- Check for experiment artifacts (empty frames, trips)
- Apply Bravais lattice where obvious
- Special pars
  - Use bad reflection filter
  - Use reduced profile size if overlapping
  - Incidence correction, prediction accuracy
- Smart background on high background data
- xx proffitloop
Optimal data – hints at data finalization time

- Hand set empirical parameters
- Use shape based absorption correction
- Apply filters carefully (e.g. R int)
- Interactively decide space group
- Remove unnecessary data via d-value filter
Software Updates

- CrysAlisPro is frequently updated with fixes for known problems
- New features are introduced in annual major updates
- All updates are Free and available from our user forum, http://www.rigakuxrayforum.com
- Free multi-user, multi-site license
Thank you for listening!

Find out more at

www.rigaku.com