CCP4 School Chicago 2013

Data Processing with XDS

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X-ray Diffraction in a Nutshell
Model Building & Refinement — Ideal Crystal Data

- Goal of crystal structure determination: Model of some molecule

- Refinement of model against $h, k, l, F(hkl), \sigma_F$ or $h, k, l, I(hkl), \sigma_I$

- Ideal: independent of machine, independent of sample
Experimental Errors

Misfocused beam

Poorly diffracting crystal

Sources of Errors
- Beam/ beamline
- Crystal imperfections
- Overloads
- Detector Background Noise
- Settings inaccuracies
- ...

Courtesy N. Sanshvili, S. Corcoran; APS Chicago

Courtesy K. Pröpper, Uni Göttingen

P.S.: Nukri’s beam is NOT an experimental error, but simply the beam before it was focussed ;-)
Intensities and Amplitudes — Experiment vs. Theory

Intensities $I(hkl)$ are experimental quantities *measured* from the detector.

Amplitudes $|F(hkl)|$ are theoretical concepts necessary for refinement\(^*\)

$$I_{\text{exp}}(hkl) = \frac{e^4}{m e^2 c^4} \frac{\lambda^3 V_{\text{crystal}}}{V_{\text{u.c.}}} I_{0LPT E} |F_{\text{theor.}}(hkl)|^2$$ \hspace{1cm} (1)


Data reduction $\approx$ Determination of intensities $I_{\text{exp}}(hkl)$ from frames

Scaling $\approx$ Determination of amplitudes $|F_{\text{theor.}}(hkl)|$ from $I_{\text{exp}}(hkl)$ and experimental settings
Ewald Sphere Construction

Reciprocal Lattice:
\[
\vec{a}^* = \frac{\vec{b} \times \vec{c}}{(\vec{a} \times \vec{b}) \cdot \vec{c}}
\]
\[
\vec{b}^* = \frac{\vec{c} \times \vec{a}}{(\vec{a} \times \vec{b}) \cdot \vec{c}}
\]
\[
\vec{c}^* = \frac{\vec{a} \times \vec{b}}{(\vec{a} \times \vec{b}) \cdot \vec{c}}
\]

Lattice points at:
\[
h\vec{a}^* + k\vec{b}^* (l\vec{c}^*)
\]
(hollow circles)

The crystal rotates about the origin of the reciprocal lattice.
Ewald Sphere Construction

Draw a sphere with radius $1/\lambda$ that touches the lattice origin. The sphere centre lies aligned with the X-ray source.

This sphere is the **Ewald Sphere**.
Ewald Sphere Construction

- The scattering vector $\vec{S}$ points from the origin to the lattice point.

- Exactly those lattice points on the surface of the Ewald sphere fulfil the Laue conditions.

- They are the recordable reflections.
Ewald Sphere Construction

Some of these spots hit the detector.
Ewald Sphere Construction

X-ray source

Crystal rotation =
Lattice rotation =
New spots

(Rot. axis perpendicular to slide)
In this image, the inverse beam for anomalous phasing strategies is illustrated. The text states:

Inverse Beam for Anomalous Phasing Strategies

Wedges collected in the order
1. 0° – 5°
2. 180° – 185°
3. 5° – 10°
4. 185° – 190°
5. 10° – 15°
6. 190° – 195°
7. ...

Synchrotrons often offer ‘inverse beam’ collection strategies to accurately determine Bijvoet pairs for phasing based on anomalous dispersion.
Bijvoet Pairs and Inverse Beam

- Bijvoet are collected close together in space and time.
- Reduces random errors but may pronounce systematic errors.
XDS
The XDS Resources

originally written by W. Kabsch
co-authored by K. Diederichs

availability:

XDS http://xds.mpimf-heidelberg.mpg.de/ main program suite

Wiki and auxiliary programs http://strucbio.biologie.uni-konstanz.de/xdswiki/

GUIs

• xdsGUI http://strucbio.biologie.uni-konstanz.de/xdswiki/index.php/XDSgui

• XDSi http://cc.oulu.fi/~pkursula/xdsi.html

• Xdsapp http://www.helmholtz-berlin.de/forschung/funkma/soft-matter/forschung/bessy-mx/xdsapp
XDS Characteristics

Some of the special features of XDS:

- Arbitrary Geometry (axes orientation, etc.)
- 3-dimensional spot integration
- Correction for Radiation Damage available
- Optimised for new Pilatus Detector
- Command-line program (pro’s and con’s…)
- Parallelised: Fast!
- Clear documentation
xdsGUI

Load recent project:

/home/tg/uni/datasets/lehre/ggnb/xds
xdsGUI

***** INTEGRATE ***** (VERSION September 26, 2012)

PROFILE_FITTING= TRUE
NAME_TRUNCATE_OF_DATA_FRAMES=. /images/lysozyme_????_mar2
DATA_RANGE= 1 106
NX= 2300 NY= 2300 QX= 0.15000 QY= 0.15000
MINIMUM_VALID_PIXEL_VALUE= 0 OVERLOAD= 130000
BACKGROUND_PIXEL= 6.00 SIGNAL_PIXEL= 3.00
MAXIMUM_ERROR_OF_SPINDLE_POSITION= 5.0
MAXIMUM_ERROR_OF_SPINDLE_POSITION= 2.0
MINPK= 75.00000
STARTING_ANGLE= 0.000 STARTING_FRAME= 1
OSCILLATION_RANGE= 1.000000 DEGREES
RODATION_AXIS= 0.99999 0.00285 0.00187
X-RAY_WAVELENGTH= 1.54179 ANGSTROM
INCIDENT_BEAM_DIRECTION= -6.000291 -0.000699 0.648596
SPACE_GROUP_NUMBER= 89
UNIT_CELL_CONSTANTS= 76.82 76.82 38.84 90.000

RESOLUTION RANGE RECORDED BY DETECTOR (ANGSTROM) 20.
NUMBER OF TRUSTED DETECTOR PIXELS 3967122
MEAN CONTENTS OF TRUSTED PIXELS IN BACKGROUND TABLE 8
MEAN VALUE OF NON-XRAY BACKGROUND (OFFSET)= 0.00
NUMBER OF X-RAY COUNTS EQUIVALENT TO PIXEL CONTENTS IN
(Pixel_value - Offset) / GAIN, WITH GAIN= 1.20

**************************************************************************
*************** PROCESSING OF IMAGES 1 ***************
**************************************************************************

USING 5 PROCESSORS

*** DEFINITION OF SYMBOLS ***
ERR = ERROR CODE AFTER ACCESSING DATA IMAGE
0: NO ERROR
Documentation

XDS Program Package

for processing single-crystal monochromatic diffraction data recorded by the rotation method.

Version: December 6, 2010 Release Notes
Author: Wolfgang Kabach

- Very well organised html-documentation
- Every STEP documented
- Every keyword documented
- Coordinate systems are explained

© 2005-2010, MPI for Medical Research, Heidelberg imprint.
Wolfgang.Kabach@mpimf-heidelberg.mpg.de
page last updated: December 6, 2010
XDS Programs

**xds_par** Main program for data integration

**xscale** scaling program for multiple datasets. **N.B.:** Do not scale data that have already been scaled! Otherwise: corruption of standard deviations $\sigma_I$. Can hamper phasing

**xdscconv** Conversion to various other data formats

**cellparam** Weighted average unit cell parameters from several runs
Templates

- Templates of input scripts for all supported detector formats
- Only very few adjustments necessary to get started
- Beamlines often generate appropriate input scripts
- `generate_XDS.INP` from XDSwiki for MARCCD, ADSC, and the Pilatus 6M
- It is worth learning how to set it up from scratch!

http://strucbio.biologie.uni-konstanz.de/xdswiki/index.php/Generate_XDS.INP
XDS is controlled by one single input file: XDS.INP.

- Name cannot be changed
- **Each data set must be run in separate directory to avoid overwriting of files.**
- Contains about 100 Keywords \(^a\) of the form
  
  `KEYWORD=VALUE`

- Only about 10 Keywords must be modified for most data sets (*e.g.* image names, detector distance, number of images, etc.)

- Most important one:
  
  `JOB= XYCRR INIT COLSPOT IDXREF DEFPIX XPLAN INTEGRATE CORRECT`

  Each name stands for one of the steps XDS carries out during data integration.

  *(XPLAN is optional and corresponds to the BEST [2] or STRATEGY [1] output to report optimal data collection range(s))*

\(^a\)also called “cards” for historical reasons
Steps of Data Processing

**Step**

- XYCORR
- INIT
- COLSPOT
- IDXREF
- DEFPIX
- INTEGRATE
- CORRECT

(Important) output files

- SPOT.XDS
- XPARM.XDS
- FRAME.cbf
- BKGPIX.cbf
- ABS.cbf
- INTEGRATE.HKL
- FRAME.cbf
- XDS_ASCII.HKL
- GXPARM.XDS

**red:** Text files with parameters or data

**cyan:** control images (use xds-viewer)

**black:** data files for further processing
The Steps

XYINIT writes files for positional corrections of the detector plane. Most modern detectors provide already corrected images so that these to files are normally flat.

INIT determines initial detector background

IDXREF indexing: unit cell dimensions and crystal orientation

DEFPIX set active detector area (exclude resolution cut-off, beam stop shadow, ...)

XPLAN (optional) generate “strategy” tables with data completeness

INTEGRATE extract reflection intensities from frames

CORRECT applies corrections (polarisation, Lorentz-correction, ...), scales reflections, reports data statistics
Program Flow

- Each step must be passed at least once - the subsequent steps depend on files produced by the previous steps.
- Each step creates a log-file (XYINIT.LP, INIT.LP,...).
- IDXREF is the main hurdle - once unit cell and crystal orientation are determined, integration usually runs smoothly.
- CORRECT summarises the quality of the data.
- Mostly IDXREF.LP and CORRECT.LP should be inspected.
### IDXREF

- **Indexing step:** Find cell parameters and cell orientation.

- **First refinement of experimental parameters** (Detector distance, ...)

- **Writes solution to XPARM.XDS**

<table>
<thead>
<tr>
<th>XPARM.XDS VERSION March 30, 2013</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 0.0000 0.1000 0.001263 -0.999998 -0.001591</td>
</tr>
<tr>
<td>0.976260 0.000701 -0.000942 1.024317</td>
</tr>
<tr>
<td>1 122.8045 140.1587 211.8464 109.309 99.583 90.012</td>
</tr>
<tr>
<td>-4.603119 87.195305 -86.352295</td>
</tr>
<tr>
<td>-8.289775 98.209831 99.652267</td>
</tr>
<tr>
<td>201.774994 -60.712154 -21.904343</td>
</tr>
<tr>
<td>201.774994 -60.712154 -21.904343</td>
</tr>
<tr>
<td>1 2463 2527 0.172000 0.172000</td>
</tr>
<tr>
<td>1231.734253 1265.753540 620.964600</td>
</tr>
<tr>
<td>1.000000 0.000000 0.000000</td>
</tr>
<tr>
<td>0.000000 1.000000 0.000000</td>
</tr>
<tr>
<td>0.000000 0.000000 1.000000</td>
</tr>
<tr>
<td>1 1 2463 1 2527</td>
</tr>
<tr>
<td>0.00 0.00 0.00 1.000000 0.000000 0.000000 0.000000 1.000000 0.000000</td>
</tr>
</tbody>
</table>
**SPOT.XDS**

- **COLSPOT**: Detector coordinates and intensity of strong spots to be used for indexing:

<table>
<thead>
<tr>
<th>X(pixel)</th>
<th>Y(pixel)</th>
<th>#image</th>
<th>counts</th>
</tr>
</thead>
<tbody>
<tr>
<td>1056.11</td>
<td>1529.51</td>
<td>15.35</td>
<td>2544.</td>
</tr>
<tr>
<td>1895.52</td>
<td>1525.49</td>
<td>9.19</td>
<td>2481.</td>
</tr>
<tr>
<td>1913.43</td>
<td>1547.90</td>
<td>2.63</td>
<td>1999.</td>
</tr>
</tbody>
</table>

- **IDXREF**: Miller-Indices according to XPARM.XDS

<table>
<thead>
<tr>
<th>X(pixel)</th>
<th>Y(pixel)</th>
<th>#image</th>
<th>counts</th>
<th>H</th>
<th>K</th>
<th>L</th>
</tr>
</thead>
<tbody>
<tr>
<td>1056.11</td>
<td>1529.51</td>
<td>15.35</td>
<td>2544.</td>
<td>14</td>
<td>13</td>
<td>18</td>
</tr>
<tr>
<td>1895.52</td>
<td>1525.49</td>
<td>9.19</td>
<td>2481.</td>
<td>-7</td>
<td>-13</td>
<td>-14</td>
</tr>
<tr>
<td>1913.43</td>
<td>1547.90</td>
<td>2.63</td>
<td>1999.</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

- 0 0 0: not indexed with current cell
"!!! ERROR !!! SOLUTION IS INACCURATE"

Correct indexing is crucial for data integration. If XDS cannot index more than 70 % of all reflections listed in SPOT.XDS, it stops with the above error message.

Most common reasons:

1. Wrong ORGX, ORGY
2. Poor data quality
• many programs take the direct beam position

• XDS takes the detector origin ORGX, ORGY: point normal to detector plane through crystal

\[
\begin{align*}
\text{ORGX} &= 1024, \text{ ORGY} = 1024 \\
\text{ORGX} &= -2048, \text{ ORGY} = 1024
\end{align*}
\]
Wrong ORGX, ORGY

The “detector origin” ORGX, ORGY in XDS.INP is provided in pixels. Typically it is in the centre of the detector, so a good start is ORGX=NX/2, ORGY=NY/2, i.e. half the detector dimensions.

Illustration of the sensitivity to ORGX, ORGY

Diffraction image from Thaumatin with a medium size cell 58 Å, 58 Å, 150 Å, 90°, 90°, 90°.

Coordinates of the four magnified spots:

<table>
<thead>
<tr>
<th>Coordinates</th>
<th>Difference</th>
<th>Difference(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1505 1800</td>
<td>9 7</td>
<td>0.66 0.51</td>
</tr>
<tr>
<td>1514 1793</td>
<td>8 6</td>
<td>0.59 0.44</td>
</tr>
<tr>
<td>1522 1788</td>
<td>8 7</td>
<td>0.59 0.51</td>
</tr>
</tbody>
</table>

An offset of the origin of about 0.5 mm means a shift of 1 in Miller-Indices

mis-indexing

Tim Grüne
Complaints about ORGX, ORGY

Indicators of a wrongly set origin:

1. refined detector distance far from real value

2. ORGX, ORGY drift far away from initial value

IDXREF.LP:

***** DIFFRACTION PARAMETERS USED AT START OF INTEGRATION *****

[...]

DETECTOR ORIGIN (PIXELS) AT 1580.99 1506.71 (XDS.INP: 1450 1450)
CRYSTAL TO DETECTOR DISTANCE (mm) 211.85 (XDS.INP: 200)

The detector distance reported during data collection (image header) should be correct within fractions of mm at a proper beamline and not drift by 11 mm.
Correcting ORGX, ORGY

1. Start with ORGX=NX/2, ORGY=NY/2 (use adxv [3] to read out header information). Often XDS refines these values reasonably well.
Correcting ORGX, ORGY

2. Check the image for the beam centre. At \( \theta = 0^\circ \) this corresponds well to ORGX, ORGY. Ice rings are particularly useful, they are centred at about the beam centre (use circle fitting option in mosflm).

In case you expect poorly diffracting crystals (3 Å and worse), consider recording one image with an empty loop filled with a drop of water to get an image with ice rings!!!
Carry on regardless

The XDS error message SOLUTION IS INACCURATE does not necessarily mean that something is seriously wrong. The step IDXREF still refines the experimental parameters and writes them into XPARM.XDS. This is all we need to continue.

Check IDXREF.LP for

- Number of indexed reflections

***** INDEXING OF OBSERVED SPOTS IN SPACE GROUP # 1 *****
  1909 OUT OF 2506 SPOTS INDEXED.
  0 REJECTED REFLECTIONS (REASON: OVERLAP)
  597 REJECTED REFLECTIONS (REASON: TOO FAR FROM IDEAL POSITION)

With $\approx 30\%$ of indexed reflections the parameters in XPARM.XDS are probably good enough to converged to the correct values during INTEGRATE and CORRECT.

- refined detector distance and detector origin do not shift too much

Set JOB = DEFPIX INTEGRATE CORRECT and integrate your data.

If something did seriously go wrong, the file XPARM.XDS would not been written.
DEFPIX: active detector mask

DEFPIX sets the area of the detector which is taken into account during integration. It takes into account:

1. INCLUDE_RESOLUTION_RANGE default: 20 Å to detector edge.
2. VALUE_RANGE_FOR_TRUSTED_DETECTOR_PIXELS exclude shadowed regions, e.g. beamstop, cryostream nozzle
3. UNTRUSTED_RECTANGLE exclude gaps of CCD chips e.g. Pilatus detector
4. EXCLUDE_RESOLUTION_RANGE exclude ice rings from data integration
VALUE_RANGE_FOR_TRUSTED_DETECTOR_PIXELS

ABS.cbf  BKGPIX.cbf, all included

VALUE_RANGE_FOR_TRUSTED_DETECTOR_PIXELS = 1000 30000
VALUE_RANGE_FOR_TRUSTED DETECTOR PIXELS

ABS.cbf

BKGPIX.cbf, shadows removed

VALUE_RANGE_FOR_TRUSTED_DETECTOR_PIXELS = 6100 30000
Pilatus and other gaps: UNTRUSTED_RECTANGLE

- Beamline provides rectangles for their detector
- UNTRUSTED_RECTANGLE = 981 989 0 2528
  \( X_{\text{from}} \rightarrow X_{\text{to}} \) and \( Y_{\text{from}} \rightarrow Y_{\text{to}} \): thin vertical strip
- Phasing: be generous to avoid shading!
INTEGRATE and CORRECT: Intensity determination and fine tuning

INTEGRATE determine spot intensities based on parameters (saved in XPARM.XDS)

CORRECT experimental corrections (e.g. Lorentz- and Polarisation-correction), refine parameters and determine space group (saved to GXPARM.XDS)

CORRECT write FRAME.cbf:
- predicted spot positions encircled
- check correctness of predictions
Recycling

- Parameters (in GXPARM.XDS) depend on measured intensities
- Intensities (including corrections) depend on Parameters

⇒ rename GXPARM.XDS to XPARM.XDS and rerun XDS (JOB = DEFPIX INTEGRATE CORRECT) to improve results.

This way one should also set the correct high- and low-resolution cut-offs
Resolution Cut-Off

The default resolution range in XDS is 20 Å to the detector edge

INCLUDE_RESOLUTION_RANGE=20.0 0.0

- Medium to low resolution data: increase 20 Å to 30 Å or even 50 Å (check BKGPIX.cbf)
- After second round of integration: determine high-resolution cut-off. My favourite: xprep - higher number of resolution shells than e.g. listed in CORRECT.LP

Why after second round?

- Correct space group rather than P1
  ⇒ more symmetry related reflections
  ⇒ more reliable data statistics, especially $I/\sigma_I$
## High Resolution Cut-Off

Subsetting of intensity data with signal/noise $\geq -3.0$ as function of resolution.

<table>
<thead>
<tr>
<th>Resolution Limit</th>
<th>Observed</th>
<th>Unique</th>
<th>Possible</th>
<th>Completeness</th>
<th>R-factor observed</th>
<th>R-factor expected</th>
<th>I/\sigma</th>
<th>R-meas</th>
<th>CC(1/2)</th>
<th>Anomalous</th>
<th>SigAno</th>
<th>Nano</th>
</tr>
</thead>
<tbody>
<tr>
<td>13.63</td>
<td>9181</td>
<td>1459</td>
<td>1511</td>
<td>96.6%</td>
<td>2.5%</td>
<td>2.9%</td>
<td>9180</td>
<td>55.25</td>
<td>2.7%</td>
<td>100.0*</td>
<td>10</td>
<td>0.901</td>
</tr>
<tr>
<td>9.64</td>
<td>17093</td>
<td>2592</td>
<td>2672</td>
<td>97.0%</td>
<td>3.1%</td>
<td>3.5%</td>
<td>17091</td>
<td>53.02</td>
<td>3.0%</td>
<td>99.9*</td>
<td>4</td>
<td>0.858</td>
</tr>
<tr>
<td>7.87</td>
<td>23196</td>
<td>3381</td>
<td>3433</td>
<td>98.5%</td>
<td>3.2%</td>
<td>3.4%</td>
<td>23195</td>
<td>46.27</td>
<td>3.5%</td>
<td>99.9*</td>
<td>-1</td>
<td>0.848</td>
</tr>
<tr>
<td>6.81</td>
<td>27489</td>
<td>3965</td>
<td>4049</td>
<td>97.9%</td>
<td>4.4%</td>
<td>4.8%</td>
<td>27483</td>
<td>35.88</td>
<td>4.8%</td>
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<td>-2</td>
<td>0.851</td>
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<td>6.09</td>
<td>31672</td>
<td>4498</td>
<td>4565</td>
<td>98.5%</td>
<td>5.7%</td>
<td>6.3%</td>
<td>31671</td>
<td>28.72</td>
<td>6.3%</td>
<td>99.8*</td>
<td>-3</td>
<td>0.834</td>
</tr>
<tr>
<td>5.56</td>
<td>35456</td>
<td>4969</td>
<td>5040</td>
<td>98.6%</td>
<td>6.6%</td>
<td>7.3%</td>
<td>35451</td>
<td>25.07</td>
<td>7.3%</td>
<td>99.7*</td>
<td>-5</td>
<td>0.816</td>
</tr>
<tr>
<td>5.15</td>
<td>37852</td>
<td>5387</td>
<td>5465</td>
<td>98.6%</td>
<td>7.1%</td>
<td>7.8%</td>
<td>37847</td>
<td>23.06</td>
<td>7.8%</td>
<td>99.7*</td>
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<td>0.818</td>
</tr>
<tr>
<td>4.82</td>
<td>40555</td>
<td>5829</td>
<td>5894</td>
<td>98.9%</td>
<td>7.1%</td>
<td>7.8%</td>
<td>40546</td>
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<td>7.8%</td>
<td>99.7*</td>
<td>-5</td>
<td>0.795</td>
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<td>4.54</td>
<td>41126</td>
<td>6125</td>
<td>6213</td>
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<td>7.7%</td>
<td>8.4%</td>
<td>41105</td>
<td>20.88</td>
<td>8.4%</td>
<td>99.7*</td>
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<td>0.807</td>
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<tr>
<td>4.31</td>
<td>46588</td>
<td>6568</td>
<td>6647</td>
<td>98.5%</td>
<td>9.5%</td>
<td>10.3%</td>
<td>45578</td>
<td>17.93</td>
<td>10.3%</td>
<td>99.5*</td>
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<td>0.804</td>
</tr>
<tr>
<td>4.11</td>
<td>47633</td>
<td>6842</td>
<td>6900</td>
<td>99.2%</td>
<td>12.4%</td>
<td>13.4%</td>
<td>47622</td>
<td>14.06</td>
<td>13.4%</td>
<td>99.2*</td>
<td>-1</td>
<td>0.805</td>
</tr>
<tr>
<td>3.93</td>
<td>50103</td>
<td>7192</td>
<td>7284</td>
<td>98.7%</td>
<td>16.8%</td>
<td>18.1%</td>
<td>50091</td>
<td>10.89</td>
<td>18.1%</td>
<td>98.7*</td>
<td>-1</td>
<td>0.800</td>
</tr>
<tr>
<td>3.78</td>
<td>51899</td>
<td>7414</td>
<td>7519</td>
<td>98.6%</td>
<td>23.0%</td>
<td>24.9%</td>
<td>51889</td>
<td>8.35</td>
<td>24.9%</td>
<td>97.7*</td>
<td>-1</td>
<td>0.796</td>
</tr>
<tr>
<td>3.64</td>
<td>54983</td>
<td>7766</td>
<td>7842</td>
<td>99.0%</td>
<td>31.6%</td>
<td>34.1%</td>
<td>54975</td>
<td>6.30</td>
<td>34.1%</td>
<td>96.0*</td>
<td>-1</td>
<td>0.781</td>
</tr>
<tr>
<td>3.52</td>
<td>56772</td>
<td>8033</td>
<td>8114</td>
<td>99.0%</td>
<td>43.3%</td>
<td>46.7%</td>
<td>56759</td>
<td>4.72</td>
<td>46.7%</td>
<td>93.2*</td>
<td>0</td>
<td>0.774</td>
</tr>
<tr>
<td>3.41</td>
<td>58909</td>
<td>8313</td>
<td>8412</td>
<td>98.8%</td>
<td>59.1%</td>
<td>63.8%</td>
<td>58897</td>
<td>3.45</td>
<td>63.8%</td>
<td>87.7*</td>
<td>-1</td>
<td>0.748</td>
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<tr>
<td>3.31</td>
<td>60300</td>
<td>8607</td>
<td>8702</td>
<td>98.9%</td>
<td>83.0%</td>
<td>89.7%</td>
<td>60285</td>
<td>2.42</td>
<td>89.7%</td>
<td>79.6*</td>
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<td>0.728</td>
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<tr>
<td>3.21</td>
<td>62086</td>
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<td>8904</td>
<td>98.9%</td>
<td>116.7%</td>
<td>126.0%</td>
<td>62073</td>
<td>1.72</td>
<td>126.0%</td>
<td>64.9*</td>
<td>1</td>
<td>0.705</td>
</tr>
<tr>
<td>3.13</td>
<td>64144</td>
<td>9057</td>
<td>9159</td>
<td>98.9%</td>
<td>156.4%</td>
<td>168.9%</td>
<td>64134</td>
<td>1.28</td>
<td>168.9%</td>
<td>49.9*</td>
<td>-2</td>
<td>0.666</td>
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<tr>
<td>3.05</td>
<td>61751</td>
<td>9009</td>
<td>9395</td>
<td>95.9%</td>
<td>206.2%</td>
<td>223.0%</td>
<td>61650</td>
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<td>223.0%</td>
<td>34.5*</td>
<td>-2</td>
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<tr>
<td>total</td>
<td>877788</td>
<td>125810</td>
<td>127720</td>
<td>98.5%</td>
<td>10.5%</td>
<td>11.4%</td>
<td>877522</td>
<td>13.37</td>
<td>11.4%</td>
<td>99.9*</td>
<td>-1</td>
<td>0.769</td>
</tr>
</tbody>
</table>

Logfile: XSCALE.LP

Tim Grüne

XDS

41/47
Low Resolution Cut-Off

Default resolution range (20 Å - edge)
- includes noise at edge
- loose low resolution reflections

Adjusted resolution range (40 Å - 2.85 Å)
- \( I/\sigma_I \approx 2 \) in outer shell
- low resolution reflection important for “shape” of molecule (MR, refinement)
Format Conversion

The final integrated data are written to the file XDS_ASCII.HKL.

Data are best scaled with XSCALE.

1. for phasing with shelx c/d/e

2. for refinement (mtz-file)
HKL → hkl

How to:

1. Phasing with shelx c/d/e requires no conversion

2. shelxc and xprep both read XDS and XSCALE output (HKL-) files

3. format detected automatically
HKL → mtz

How to (suggested by Clemens Vonrhein, Globalphasing):

1. Conversion to mtz, hklref: indexing correction if necessary (e.g. space group $C2$)

   ```
   pointless -copy xdsin xscale.HKL hklref ${freeRefMtz} hklout pointless.mtz
   ```

2. Merging of equivalent indices:

   ```
   aimless hklin pointless.mtz hklout aimless.mtz << eoaimless
   ONLYMERGE
   eoaimless
   ```

3. Intensities to amplitudes ($I \rightarrow |F|$):

   ```
   truncate hklin aimless.mtz hklout truncate.mtz << eotruncate | tee truncate.log
   eotruncate
   ```
HKL $\rightarrow$ mtz (continued)

4. Copying $R_{\text{free}}$ flags if necessary

```shell
cad hklin1 truncate.mtz hklin2 ${\text{freeRefMtz}}$ hklout cad.mtz << eocad | tee cad.log
LABIN FILE 1 ALL
LABIN FILE 2 E1=FreeR_flag
eocad
```

5. Correction for reported resolution, e.g. by mtzdmp

```shell
sftools << eosftools | tee sftools.log
read cad.mtz
SELECT ONLY COLUMN IMEAN PRESENT
write sftools.mtz
eosftools
```

6. Extension of $R_{\text{free}}$ reflections to full resolution

```shell
freerflag hklin sftools.mtz hklout freerflag.mtz << eofreerflag | tee freerflag.log
COMPLETE FREE=FreeR_flag
eofreerflag
```
References

3. Andrew Arvai, [http://www.scripps.edu/~arvai/adxv.html](http://www.scripps.edu/~arvai/adxv.html)