

# Phenix and CCP4

## Data input



### Phenix

- Reads both SHELX HKLF4 (intensity,  $F^2$ ), Scalepack and mtz files.

### CCP4

- Use Import merged data to read in a Scalepack file.
- Use Convert to/modify/extend (f2mtz) MTZ to read in a SHELX HKLF4 file. However will the resulting mtz file will be merged.
- Use pointless to create an unmerged mtz file which can be imported into Aimless.
- Use f2mtz to inport a SHELX phs file from phasing runs.

# Determine Space Group

## Writing out Scalepack File



- Click **Determine space group**
- Click the output sca file button and run it as you normally work
- The sca file will be written to the work directory when you finish

PROTEUM2 v2015.9-0 - User: (guest) - Sample: lyso\_1216 - Licensed to Matt Benning at Bruker

Sample Instrument Windows Help

Determine Space Group

Setup Lattice Exceptions Space Group Determination Statistics Cell Information Diagnostics

Input Files

hkl file: lyso\_1216\_0m.hkl

p4p file: lyso\_1216\_0m.p4p

Output Files

ppp file: lyso\_1216\_0m.ppp

output\_hkl file: lyso\_1216\_0m.hkl

output\_sca file: lyso\_1216\_0m.sca

Unit Cell

	a	b	c	alpha	beta	gamma
cell	76.9861	76.9861	38.6451	90	90	90
cell esds	0.0016	0.0000	0.0008	0	0	0

Experimental Parameters

Must be chiral

Radiation Type: Ga

Solve Structure Report

Finish Start Over Exit

# XPREP

## Writing out Scalepack File



- Select **Analyze Data** from the **Examine Data** menu
- Browse for the correct p4p and HKL file if not already shown the files box and click Ok
- The XPREP window will open
- Hit the CR once, this will take you to the main menu

The screenshot shows the Proteum2 v2015.0-0 software interface. The main window has a menu bar (Sample, Instrument, Windows, Help) and a toolbar. A vertical sidebar on the left contains several icons, with the 'Analyze Data' icon circled in red. Below the sidebar is a 'Select Files For XPREP' dialog box with two text input fields: 'P4P file: C:\frames\guest\lyso\_1216\work\lyso\_1216\_0m.p4p' and 'HKL file: C:\frames\guest\lyso\_1216\work\lyso\_1216\_0m.hkl'. The 'XPREP Version 2014/2 for Windows' window is open, displaying a text-based menu and data. The text includes copyright information, screen size (1280 x 1024), window size (640 x 323), font size (8 x 16), and number of colors (256). It also provides instructions on how to use XPREP, including file formats and command-line options. A table of lattice exceptions is shown at the bottom of the XPREP window.

Lattice exceptions:	P	A	B	C	I	F	Obv	Rev	All
N (total) =	0	63798	63759	63739	63786	95648	85004	85067	127538
N (int>3sigma) =	0	49623	49562	49779	49754	74482	66056	66132	99120
Mean intensity =	0.0	23.0	23.2	22.7	22.8	23.0	22.7	22.7	22.8
Mean int/sigma =	0.0	14.6	14.7	14.7	14.7	14.7	14.5	14.6	14.6

Select option [P]: █

PgUp/PgDn scrolls text; only graphics window may be resized

# XPREP

## Writing out Scalepack File



- Follow the normal path to select the metric symmetry and space group
- At the main menu, select **option D** for Read, Modify and merge datasets
- Select **option W** to write out a HKL file
- Select **option H** for exporting a Denzo/scalepack formatted file and input a filename

```
[4] SHELX HKLF 4 format (F-squared)
[3] SHELX HKLF 3 format (F)
[C] CNS format (F, with headers)
[H] HKL2000 or Denzo/Scalepack format
[X] X-PLOR format (F)
[E] EXIT to main menu
[Q] QUIT program

Select option [4]: H

Output filename: test.sca
```

The screenshot shows the XPREP software interface. At the top, the title bar reads "XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014". Below the title bar is a table with columns "Index", "# Data", and "Filename or Source of Data". The table contains one entry: "1 127538 lyso\_1216\_0m.hkl <- current dataset". Below the table is a main menu with two columns of options. The first column includes options like "[M] Sort-MERGE current data (no scaling)", "[L] LEAST-SQUARES scale and merge datasets", "[I] INCLUDE Rfree flags from another file", "[S] Display intensity STATISTICS", "[F] FACE-indexed absorption corrections", "[T] Copy file, TRANSFORM hkl and cosines", "[H] Apply HIGH/low resolution cutoffs", "[G] Generate simulated powder diagrams", "[Y] Self or cross correlation coefficients", "[Z] Expand data to triclinic", "[X] Parsons Q values and Flack x parameter", and "[K] Check for consistent indexing". The second column includes options like "[C] Change CURRENT dataset", "[W] WRITE dataset to file", "[R] READ in another dataset", "[D] DELETE stored dataset", "[A] MAD, SAD, SIR or SIRAS", "[N] NORMALIZE/scale sigmas", "[U] Anisotropic scaling", "[J] Change dataset label", "[E] EXIT to main menu", and "[Q] QUIT program". At the bottom of the menu, it says "Select option [S]:".

```
XPREP Version 2014/2 for Windows Copyright(C) Bruker-AXS 2014
Index # Data Filename or Source of Data
1 127538 lyso_1216_0m.hkl <- current dataset

[M] Sort-MERGE current data (no scaling) [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file [R] READ in another dataset
[S] Display intensity STATISTICS [D] DELETE stored dataset
[F] FACE-indexed absorption corrections [A] MAD, SAD, SIR or SIRAS
[T] Copy file, TRANSFORM hkl and cosines [N] NORMALIZE/scale sigmas
[H] Apply HIGH/low resolution cutoffs [U] Anisotropic scaling
[G] Generate simulated powder diagrams [J] Change dataset label
[Y] Self or cross correlation coefficients [E] EXIT to main menu
[Z] Expand data to triclinic [Q] QUIT program
[X] Parsons Q values and Flack x parameter
[K] Check for consistent indexing

Select option [S]:
```

PgUp/PgDn scrolls text; only graphics window may be resized



# Inputting data to Phenix

## SHELX or Scalepack HKL file

Phenix will read either a SHELX or Scalepack formatted file. However with the SHELX format, you have to define whether it is a HKLF4 (intensities) or HKLF3 (amplitudes). Typically you will be reading in intensities.

- If the name of the file is `data.hkl`, make a copy of the file in the same directory and call it `data.hkl=hklf4`
- Load the `data.hkl=hklf4` into Xtrriage
- Input the cell constants and space group
- Outputs a mtz file



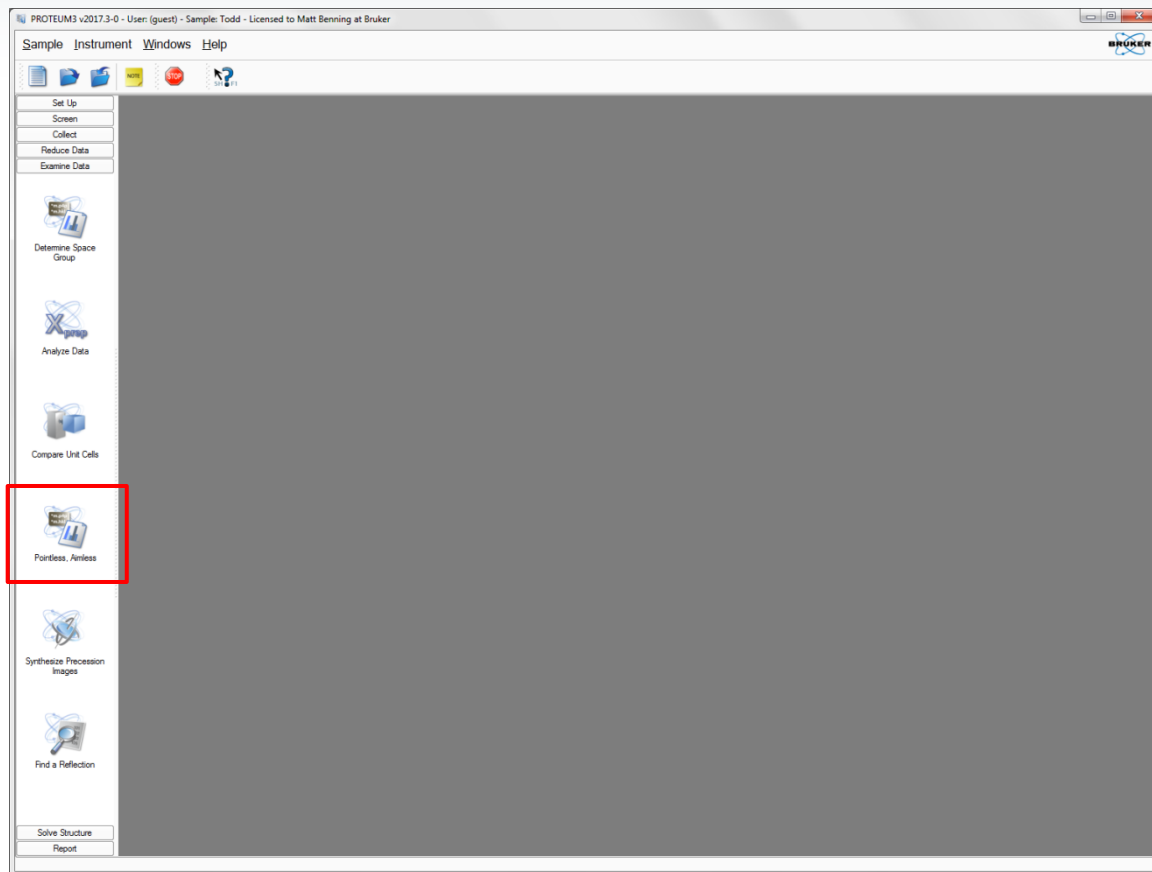
# Pointless, Aimless

If you have CCP4 installed, add the following 3 lines to the end of the bn-config.py file

- `ccp4 = "C:/CCP4-7/7.0"`
- `ccp4_range = [22.0,1.85]`
- `ccp4_autoprocess = True`

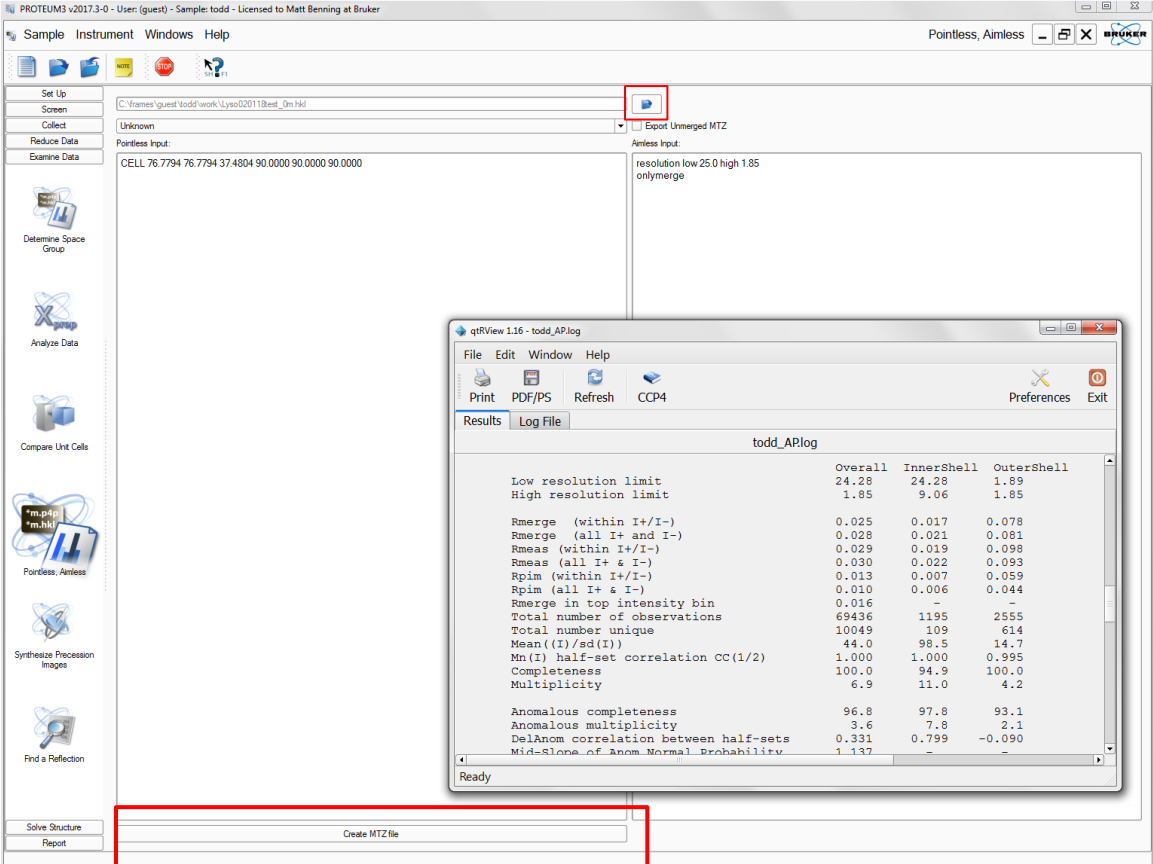
# Pointless, Aimless

- Open the “Examine Data” menu
- Select the “Pointless, Aimless” icon



# Pointless, Aimless

- If there is no MTZ file in the work folder, PROTEUM will automatically run Pointless and Aimless based on default values and display the aimless output.
- Default resolution 25 – 1.85 Å
- The pointless and aimless fields are editable so you can add keywords, change the defaults and click “create MTZ file” at the bottom left to rerun the programs. The new Aimless log will appear when both programs are finished.
- If the space group is not assigned (default), PROTEUM lets pointless perform a space group search.
- The plugin will search for the [HKL filename\\_0m.hkl](#) in the work directory but you can also search for a HKL file using the browser button.



qtRView 1.16 - todd\_AP.log

Results Log File

todd\_AP.log

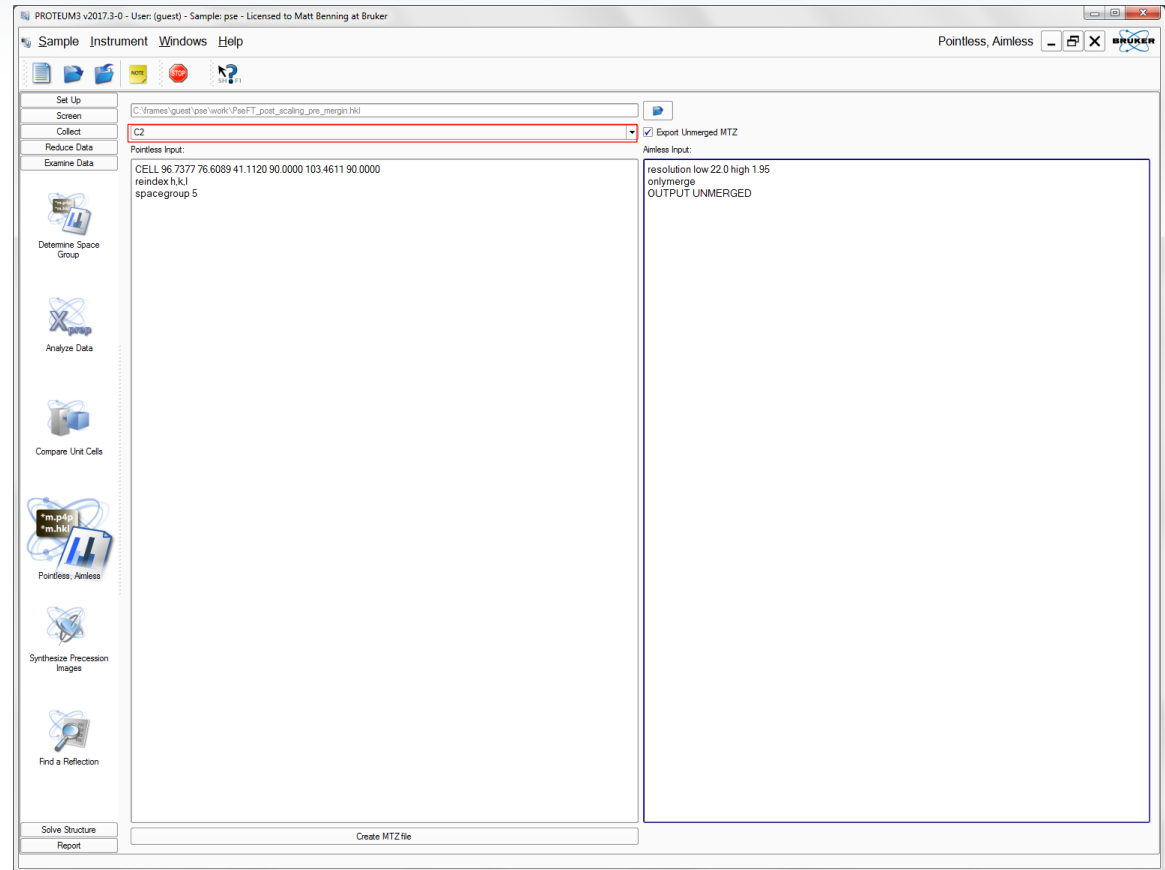
	Overall	InnerShell	OuterShell
Low resolution limit	24.28	24.28	1.89
High resolution limit	1.85	9.06	1.85
Rmerge (within I+/I-)	0.025	0.017	0.078
Rmerge (all I+ and I-)	0.028	0.021	0.081
Rmeas (within I+/I-)	0.029	0.019	0.098
Rmeas (all I+ & I-)	0.030	0.022	0.093
Rpim (within I+/I-)	0.013	0.007	0.059
Rpim (all I+ & I-)	0.010	0.006	0.044
Rmerge in top intensity bin	0.016	-	-
Total number of observations	69436	1195	2555
Total number unique	10049	109	614
Mean (I)/sd (I)	44.0	98.5	14.7
Mn (I) half-set correlation CC(1/2)	1.000	1.000	0.995
Completeness	100.0	94.9	100.0
Multiplicity	6.9	11.0	4.2
Anomalous completeness	96.8	97.8	93.1
Anomalous multiplicity	3.6	7.8	2.1
DelAnom correlation between half-sets	0.331	0.799	-0.090
Mid-Slope of Anom Normal Probability	1.137	-	-

Ready

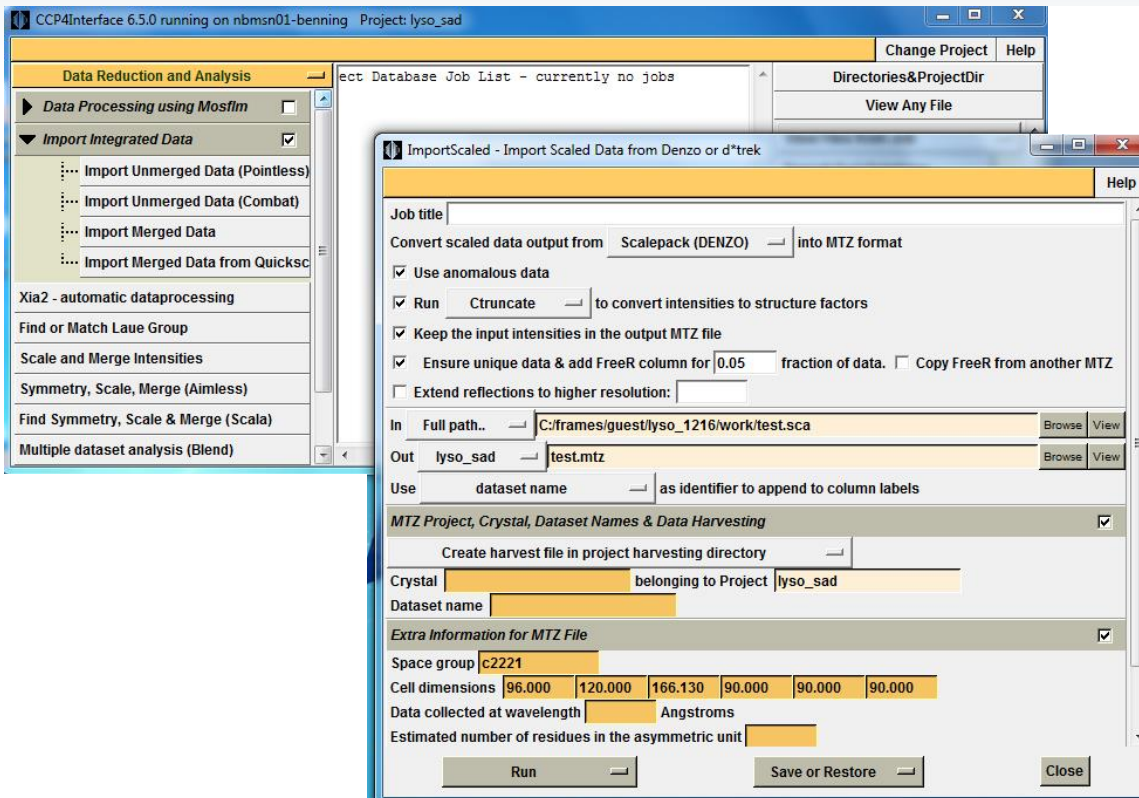


# Pointless, Aimless

- If you want to assign a space group, select the desired group in the box below the input HKL filename. This will fix the space group to the that group assigned.
- A merged MTZ file is written out by Aimless, if you want to write out a unmerged MTZ file as well, check the "Export Unmerged MTZ"
- Output files are written to the work folder.
  - *Entry prefix\_AP.log* is the output logfile from Aimless
  - *HKL filename\_merged.mtz* is the merged MTZ file output by Aimless
  - *HKL filename\_unmerged.mtz* is the corresponding unmerged MTZ

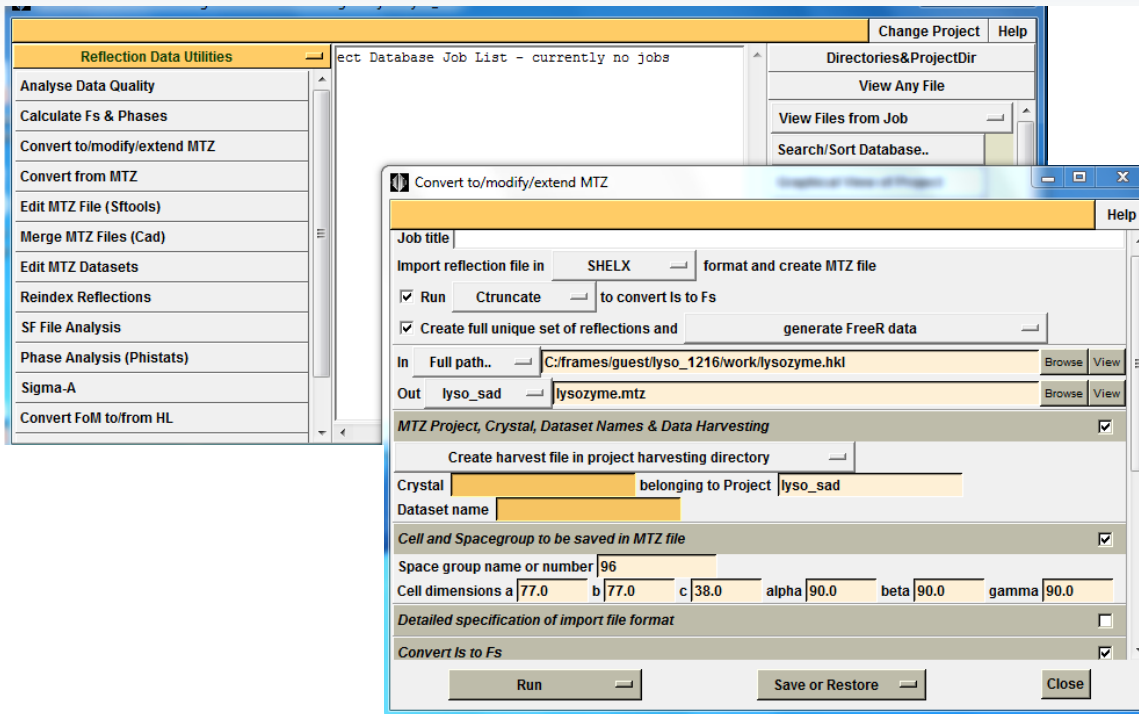


# Inputting data to CCP4 Scalepack file



- From the **Data Reduction and Analysis** menu, select **Import integrated Data>Import Merged Data**
- Browse for the scalepack file
- Fill in any missing information

# Inputting data to CCP4 SHELX HKLF4 file



- From the Reflection data Utilities, select Convert to/modify/extend MTZ
- In the input window, change the Import reflection filetype to SHELX
- Browse for the SHELX HKL file
- Input the Space group and Cell information and any other missing information

# Using Pointless to Create MTZ

## File prep



- With a HKL file written by SADABS, you have to remove the lines at the end of the file after the last reflection line (highlighted in blue below). If you write out a HKLF4 file from XPREP, you can skip this step.

```
9  1 -23 2.02095 11.0166  1  
  0  0  0  0.00  0.00  0
```

```
_exptl_absorpt_process_details
```

```
;
```

```
SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,  
J. Appl. Cryst. 48 (2015) 3-10
```

```
;
```

```
_exptl_absorpt_correction_type multi-scan
```

```
_exptl_absorpt_correction_T_max 1.0000
```

```
_exptl_absorpt_correction_T_min 0.8067
```

```
_exptl_special_details
```

```
;
```

```
The following wavelength and cell were deduced by SADABS from the  
direction cosines etc. They are given here for emergency use only:
```

```
CELL 1.54369  42.505  45.457  47.886 101.541 114.565  89.042
```

```
;
```



## Using Pointless to Create MTZ

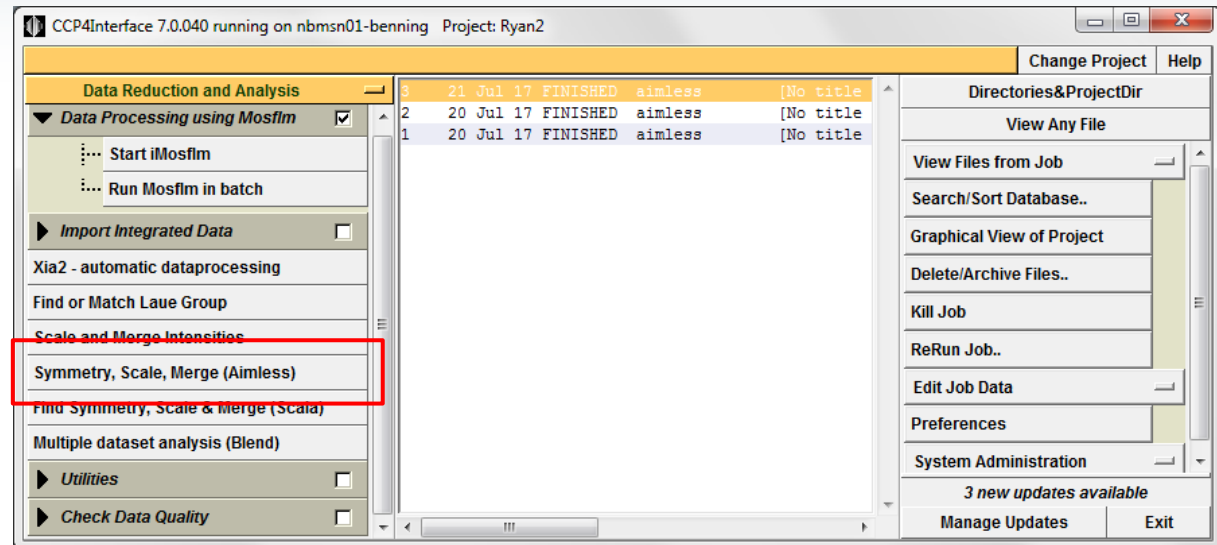
```
Pointless -c hklin filename.hkl hklout filename.mtz <pointless.inp
```

To create the input file you only need to add the three lines below. Just update the cell and space group information (space group is optional). The cell constants just have to be separated by a space.

- Pointless.inp  
cell 119.3466 45.1348 74.2698 90.0000 120.9434 90.0000  
reindex h,k,l  
spacegroup 19

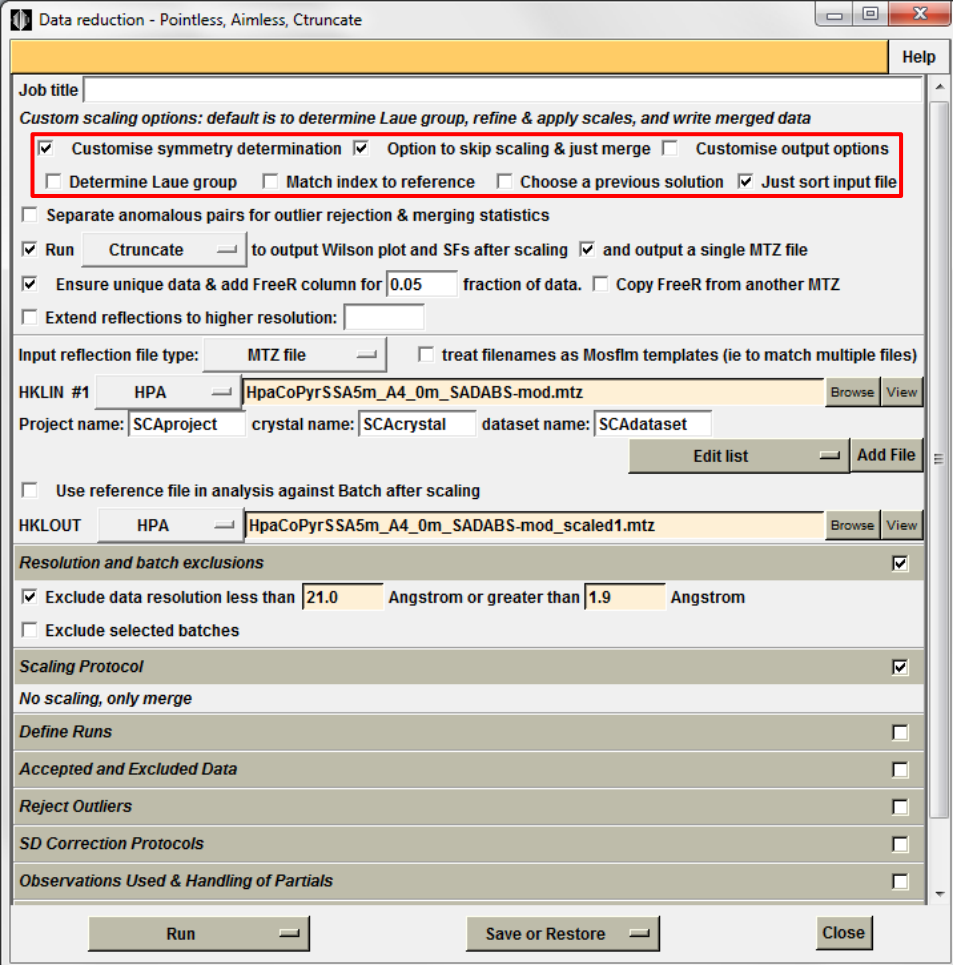
# Importing SHELX mtz file into Aimless

- Click on “Symmetry, Scale & Merge” as you normally would to run Aimless



# Running Aimless

- Select “Option to skip scaling & just merge”
- To disable Aimless from changing the space group click “Customise symmetry determination” and uncheck “Determine Laue group”
- Click to add a Rfree flag
- Browse for the mtz file created with pointless
- Set the resolution range if desired



Data reduction - Pointless, Aimless, Ctruncate

Job title

Custom scaling options: default is to determine Laue group, refine & apply scales, and write merged data

Customise symmetry determination  Option to skip scaling & just merge  Customise output options

Determine Laue group  Match index to reference  Choose a previous solution  Just sort input file

Separate anomalous pairs for outlier rejection & merging statistics

Run  to output Wilson plot and SFs after scaling  and output a single MTZ file

Ensure unique data & add FreeR column for  fraction of data.  Copy FreeR from another MTZ

Extend reflections to higher resolution:

Input reflection file type:   treat filenames as Mosfilm templates (ie to match multiple files)

HKLIN #1

Project name:  crystal name:  dataset name:

Use reference file in analysis against Batch after scaling

HKLOUT

Resolution and batch exclusions

Exclude data resolution less than  Angstrom or greater than  Angstrom

Exclude selected batches

Scaling Protocol

No scaling, only merge

Define Runs

Accepted and Excluded Data

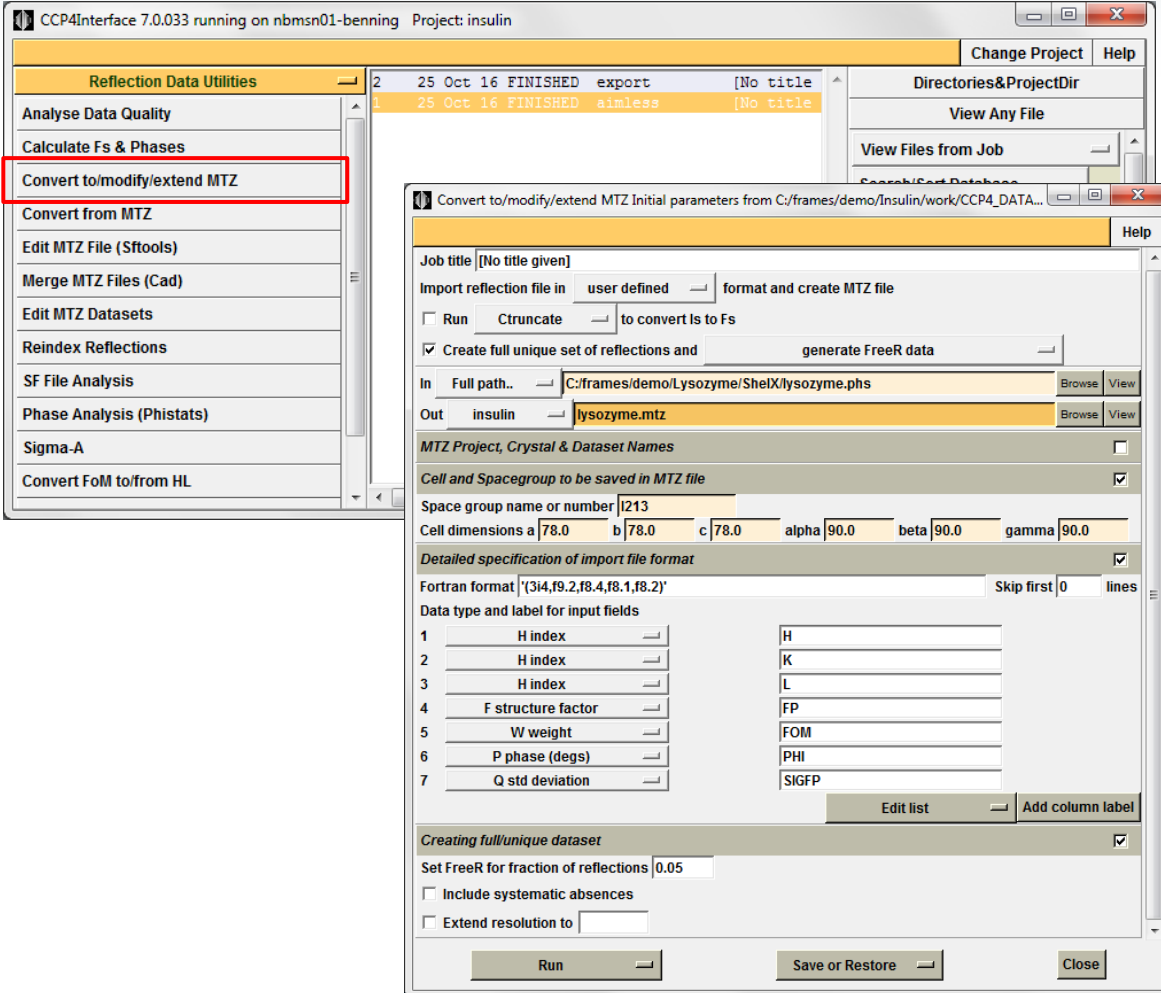
Reject Outliers

SD Correction Protocols

Observations Used & Handling of Partial

# Converting a PHS file to MTZ format

- Click on “convert to/modify/extend MTZ” under the Reflections data Utilities menu
- Set import file to “user defined”
- Click the box to generate a FreeR data
- Browse for the phs file and assign a output mtz filename
- Input the space group and cell dimensions
- Input the file format
  - 3i4,f9.2,f8.4,f8.1,f8.2
- Set the data type and labels as shown
- Click run



CCP4Interface 7.0.033 running on nbmsn01-benning Project: insulin

Reflection Data Utilities

- Analyse Data Quality
- Calculate Fs & Phases
- Convert to/modify/extend MTZ**
- Convert from MTZ
- Edit MTZ File (Sftools)
- Merge MTZ Files (Cad)
- Edit MTZ Datasets
- Reindex Reflections
- SF File Analysis
- Phase Analysis (Phistats)
- Sigma-A
- Convert FoM to/from HL

Convert to/modify/extend MTZ Initial parameters from C:/frames/demo/Insulin/work/CCP4\_DATA...

Job title [No title given]

Import reflection file in **user defined** format and create MTZ file

Run  Truncate to convert to Fs

Create full unique set of reflections and generate FreeR data

In Full path.. C:/frames/demo/Lysozyme/SheIX/lysozyme.phs

Out insulin lysozyme.mtz

MTZ Project, Crystal & Dataset Names

Cell and Spacegroup to be saved in MTZ file

Space group name or number |213

Cell dimensions a |78.0 b |78.0 c |78.0 alpha |90.0 beta |90.0 gamma |90.0

Detailed specification of import file format

Fortran format '(3i4,f9.2,f8.4,f8.1,f8.2)' Skip first 0 lines

Data type and label for input fields

1	H index	H
2	H index	K
3	H index	L
4	F structure factor	FP
5	W weight	FOM
6	P phase (degs)	PHI
7	Q std deviation	SIGFP

Creating full/unique dataset

Set FreeR for fraction of reflections 0.05

Include systematic absences

Extend resolution to

Run Save or Restore Close





# Converting a PHS file to MTZ format

```
f2mtz -c hklin filename.hkl hklout filename.mtz <phs_to_mtz.inp
```

To create the input file you only need to add the three lines below. Just update the cell and space group information (space group is optional). The cell constants just have to be separated by a space.

- Phs\_to\_mtz.inp  
symmetry H3  
cell 78.0 78.0 78.0 90.0 90.0 90.0  
format '(3i4,f9.2,f8.4,f8.1,f8.2)'  
skipline 0  
labout H K L FP FOM PHI SIGFP  
ctypout H H H F W P Q  
PNAME S100  
DNAME  
XNAME