

User Guide

APEX II CCD Diffractometer

December 9th, 2016

Matthias Zeller

Purdue University, West Lafayette, PA, 47907, USA

The following is guide for collecting data and solving structures using a Bruker APEX II single crystal diffractometer. It is intended as a “walk-through” user guide geared especially towards novice users, but tries to cover more advanced features of the software as well as far as they are important for the collection of normal “simple” small molecule structural data. A basic knowledge of the fundamentals of diffraction as it applies to crystallography is expected.

This manual is based on an instrument and software as manufactured around the year 2013. Most of the manual's content also applies to later instrument makes, such as a Bruker Prospector or Bruker Quest instruments and machines running Apex3 rather than Apex2 software. For the novice user, changes between different generation instruments are mostly limited to slightly different program layouts and colour schemes, and the general procedures described in this manual still apply.

For a more in depth description of the features of a CCD diffractometer the reader should refer to the manuals and technique guides on specific topics by the manufacturer of the type of

instrumentation you are using. The gold standard for a more in depth guide towards the use of Shelxtl for the refinement of single crystal structures Peter Müller's book Crystal Structure Refinement. Every crystallography lab should have at least one copy.

Among the many programs commonly used for crystal structure solution and refinement we recommend the Bruker *Shelxtl* package (including XPREP, XS and XM), George Sheldrick's refinement program *Shelxl2014*, and the graphical interface *Shelxle* by Hübschle, Dittrich and Sheldrick.

Instrument Overview

An ApexII instrument consists of:

- a) The actual diffractometer including the X-ray enclosure, detector, X-ray tube and optics, video microscope, goniometer, electronic controls, power supply and other miscellaneous pieces of equipment essential for the operation of the instrument.
- b) A variable temperature unit (pump, controllers and liquid nitrogen tanks located outside the enclosure)
- c) Haskris chillers that cool and circulate cooling water for the power supply and the X-ray tube.
- d) The Server computer: This computer runs the Bruker Instrument Service (BIS) and Bruker Configuration Program (BCP). These programs directly control the diffractometer (but not the variable temperature unit).
- e) The Client computer: This computer runs the Apex2 program and is the interface used to control data collections. Apex2 connects to BIS on the server computer to access the diffractometer.

Server and client computers are located to the left of the diffractometer enclosure, low temp unit and Haskris chillers to the right of the enclosure.

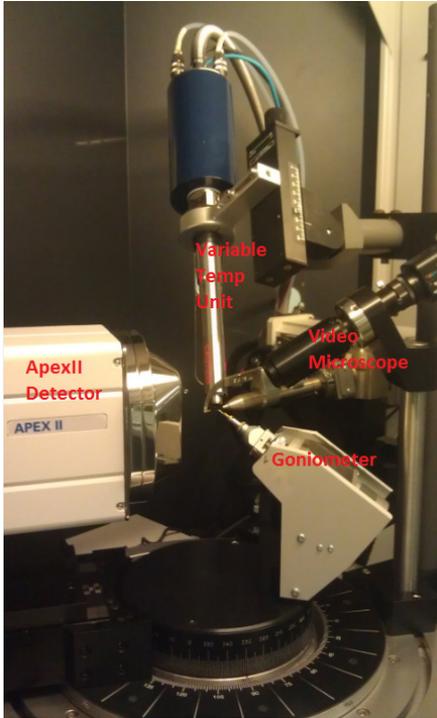


Figure 1, Inside of the X-ray enclosure with Detector, Video Microscope, Variable Temp Unit and Goniometer. Behind the Video Microscope are the X-ray tube (light grey), the Monochromator (not visible here) and the Collimator which also holds the beam stop. On the Goniometer is mounted a Goniometer Head holding the crystal specimen.

Having a promising sample of crystals it is best to start in the following order:

Adjusting the Temperature

All but very high melting samples (inorganics, ceramics, etc) should be measured at lower temperature to avoid extensive thermal motion of the atom cores, to obtain higher angle diffraction data and to minimize radiation damage of the sample.

The Oxford Cryosystems variable temperature unit is a stand-alone unit operated through its own controllers. It is not controlled through the computers running the Apex2 software or BIS. The sample is embedded in a stream of cold nitrogen gas supplied by a low pressure liquid nitrogen dewar. To avoid buildup of ice, the sample is insulated from ambient humidity by an outer layer of warm nitrogen supplied by a high pressure tank. This second tank is not electronically controlled; the nitrogen pressure and flow have to be adjusted manually.

Oxford Cryosystems variable temperature unit is able to achieve temperatures between 375 K (+102.5°C) down to ca. 85 K (-188.5°C). The recommended temperature for “low temperature” data collections is 100 K.

- Make sure, the tanks are properly connected and full enough for the planned experiment. See [Appendix A: changing the N₂ tanks](#) for changing tanks.

- The Oxford Cryostream N2 tank (white tank behind the diffractometer) is automatically refilled from the larger low pressure liquid nitrogen tank (to the right of the diffractometer). Check that the Liquid Level Controller (the smaller of the two controllers, Fig. 1) is set to **<AUTO>** for automatic refills. The fill level displayed on the controller should be between 40 and 60%. When it is below 40% (despite the controller set to AUTO) the low pressure N2 tank is empty and needs to be replaced.



Figure 1, The circulation pump and the two external controllers for the Oxford Cryosystems variable temperature unit.

- The pressure shield gas is provided from the high pressure liquid nitrogen tank. Check that the gage on the valve at the tank shows at least 20 psi. Check the fill level of the tank (move it to check its weight). When the tank runs low ice that builds up on its outside starts to melt and tanks should be changed.
- Check the display of the Cryostream Controller (larger of the two controllers) if the variable temperature unit might be already running. *If the unit is running* check if the temperature is appropriate for your experiment. If not, press the <ENTER> button, enter a <RATE> value (e.g. 240 K/hr) via the up and down keys, press the <ENTER> button, enter a target value for <TEMP>, press the <ENTER> button again, then press the blue <START> button. The controller will ramp the temperature to the desired value.
- *If the unit is switched off* it needs to be reset. Switch the unit off and on again using the Main Switch (in the back of the controller, right lower corner). Wait for the automatic checks to complete. When ready, press the <PROGRAM> button. Enter a <RATE> value (e.g. 240 K/hr) via the up and down keys, press the <ENTER> button, enter a target value for <TEMP>, press the <ENTER> button again, then press the blue <START> button. The controller will start the circulation pump (grey pump to the left of the controllers) and ramp the temperature to the desired value.

Starting the System

- Start BIS (Bruker Instrumentation System) if it is not already running (on the server computer, first computer to the left of the diffractometer housing). If you are connecting remotely to the instrument you can only connect to the client computer, not the server computer. Please ask a person on site to start BIS for you if it is not yet running. BIS must be running before starting Apex2.

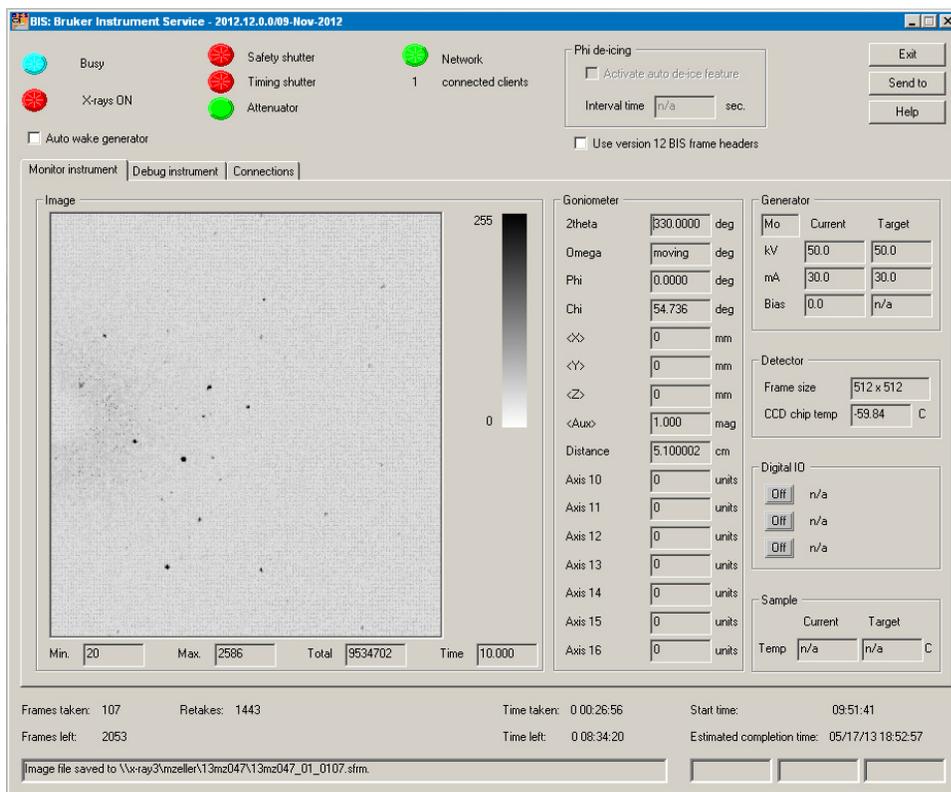


Figure 1, The BIS window

Unloading of the Previous Sample

- Check if the video is running. You can use the video on either the Client or the Server computer. If necessary start the program to watch the operations described below via the live video feed.
- Minimize any sessions of Apex2 that might be active
 - Start a new session of Apex2 and log on (<Sample>, then <Login>)
 - Connect to BIS (<Instrument>, then <Connect>).
- Either <Open> an existing sample (continuation of old data collection) or
- Start a <New Sample>
 - Fill in the project name. The name should be made up by the year (07 for 2007, 08 for 2008, etc), your initials, and a running number, e.g. 08trw001 or 08mz049
- Under <Setup>, go to <Center Crystal>
 - Click on <Mount> or <Right>, the angles will drive to a position convenient for mounting

- Carefully dismount the goniometerhead from the previous experiment (screw the ring at the base counter clockwise until completely loose, slowly lift out the whole goniometer head). Recover the crystal (especially if it is not yours!!) and use the goniometer head to mount the new crystal sample.

Selecting and Mounting of Sample

- We recommend to use Mitegen micromesh mounts for most of its samples and data collections. Crystals collected at low temperature are mounted on the micromesh with the help of a trace of mineral oil and flash frozen in the cold stream. Crystals collected at room or elevated temperature might need to be fixed to the mesh with a trace of glue to avoid crystal movement during measurement.
- For typical crystal selection and mounting, place a drop of mineral oil on a glass slide under a microscope. Place several representative crystals in the mineral oil. Inspect the crystals and select a suitable candidate. Crystals on the ApexII instrument should ideally be not larger than 0.5 mm in any direction (the size of the X-ray beam). If no single specimen with suitable dimensions can be found, use a sharp razor blade to cut off a single piece from a larger crystal or cluster. Remove all smaller pieces and dirt from the crystal as much as possible (the micromesh mount can be used to brush off loose pieces from the crystal). Scoop up the crystal with the micromesh mount (already mounted to the goniometer head) and place it on the inside of the concave face of the mount in the center of the mesh. Remove excess oil as much as possible. (If a crystal needs to be glued to the mount make sure that both crystal and mount are dry without any traces of oil left. Dip the mesh carefully in a small amount of glue and pick up the crystal without touching glass slide).

Figure 2, Crystal Centering Window and live video feed with mounted sample

Crystal Mounting and Centering

At this point you should have a session of Apex2 running, the <Center Crystal> window should be open, and the live video stream should be running (see above).



Figure 3, Goniometer head and adjustment screw

- Carefully mount the goniometer head with the mounted crystal. Pin and hole of mounting plate and the underside of the goniometer head have to line up.
 - Check the position of the mount by eye (is it aligned with the collimator and the beam stop?) and on the live video feed (is the crystal visible on the video screen?). If you cannot see the crystal the goniometer head is either not mounted properly, or its position is further off than usual and needs to be centered by eye before the live video feed can be used.
 - If the goniometer head is roughly aligned the tip of the mount is visible via the live video feed. There are three pins on the goniometer head: for up-down, right-left, and forward-backward. Using the bold end of the adjustment screw turn the pins to position the crystal in the center of the cross hair of the video feed. (if the pins are too tight or loose, adjust the small screws using the small hexagonal end of the adjustment screw. This is best done with the goniometer head not mounted on the instrument).
 - When the crystal is centered, click the large **<Spin Phi 90>** button (in the **<Center Crystal>** window of Apex2) to spin the mounting pin around its axis by 90°. Repeat the centering procedure using the adjustment screws as before. Repeat the process (**<Spin Phi 90>** followed by centering of the crystal in the cross hair of the video feed) until the crystal stays positioned in the center of the cross hair in all positions.
- For oddly shaped crystals, try to center the mass center of the crystal. For long needles, try to align them along of the axis of the mounting pin.

- Measure the dimensions of the crystal: Click on one side of the crystal, hold the left mouse key and drag to the other side. The value, in μm , is given in the bar below the video screen (second value from the right). Write down the value and repeat for the other two directions using the **<Spin Phi 90>** button (if no exact values can be obtained this way you will need to drive the angles manually using the **<Simple Scans>** option under **<Setup>**).

Crystal and compound description

- Under **<Setup>**, go to **<Describe>**
 - Fill in all values that apply, always give the full name of the person that provided the sample. Crystal dimensions need to be entered in mm (video screen values are in μm !).

The screenshot shows the 'Describe' tab in the APEX2 software. The fields are filled as follows:

Field	Value
Name	08an007
Compound	A.S.A.Hosseini Yazdi
Formula	C16O5H11
Crystal Color	n/a
Intensity	n/a
Primary Color	colourless
Secondary Color	n/a
Crystal Dimension	0.685 x 0.077 x 0.022 [mm]
Crystal Shape	needle

Figure 4, Crystal and compound description

Unit Cell Determination

- Under **<Evaluate>**, click on **<Determine Unit Cell>**.
 - Two procedures are available, **<Automatic Mode>** or **<Manual Mode>**.

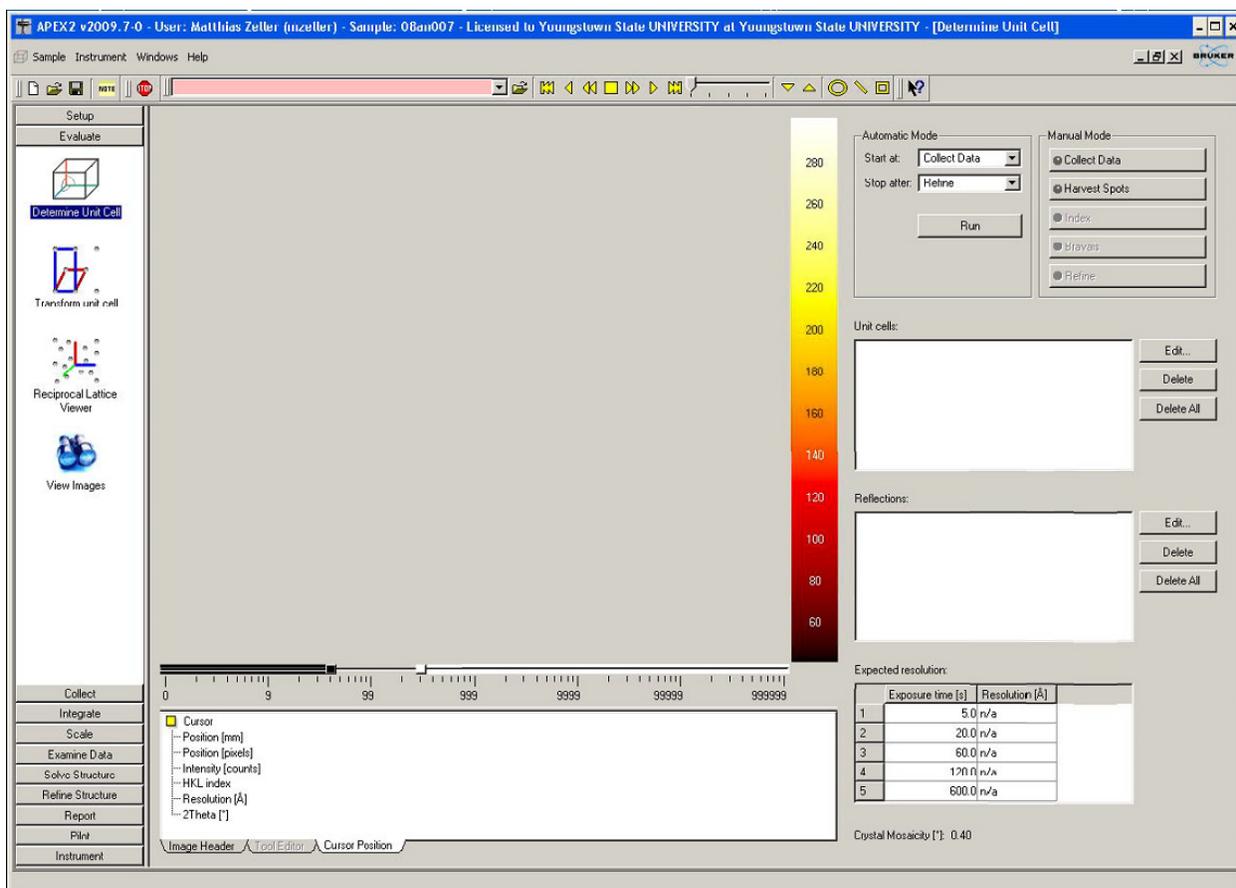


Figure 5, Starting Window of the Unit Cell determination

We encourage to use the manual procedure. To do so click [Collect Data](#), this will open the Unit Cell Data Collection Window.

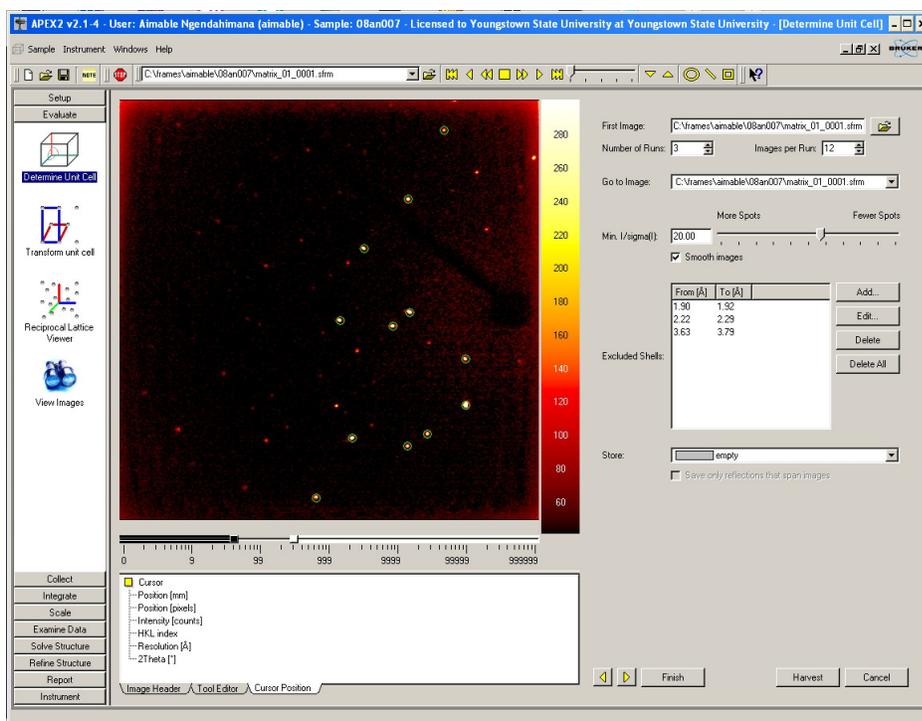


Figure 6, Unit Cell window with active data collection running

- Choose an exposure time (default is 10 seconds) and click **<Collect>**.
- 3 × 12 frames will be collected which will be displayed in the frame window.
- When finished proceed to **<Harvest Spots>**, select an I/sigma cutoff value for the diffraction spots to be used (see the frame to the left for the spots that will be used, a useful default value is 5.0) and click on **<Harvest>**. Reading in of the spots may take a few minutes.
- Proceed to **<Index>**, use all default values and click on **<Index>**.

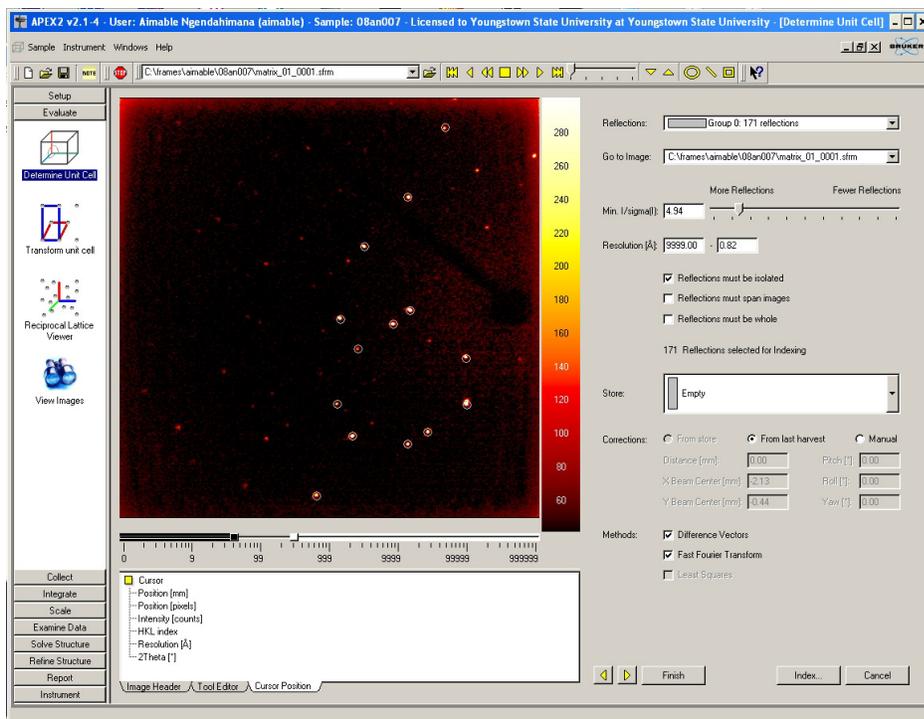


Figure 7, Unit Cell Indexing Window

Indexing might take a few minutes.

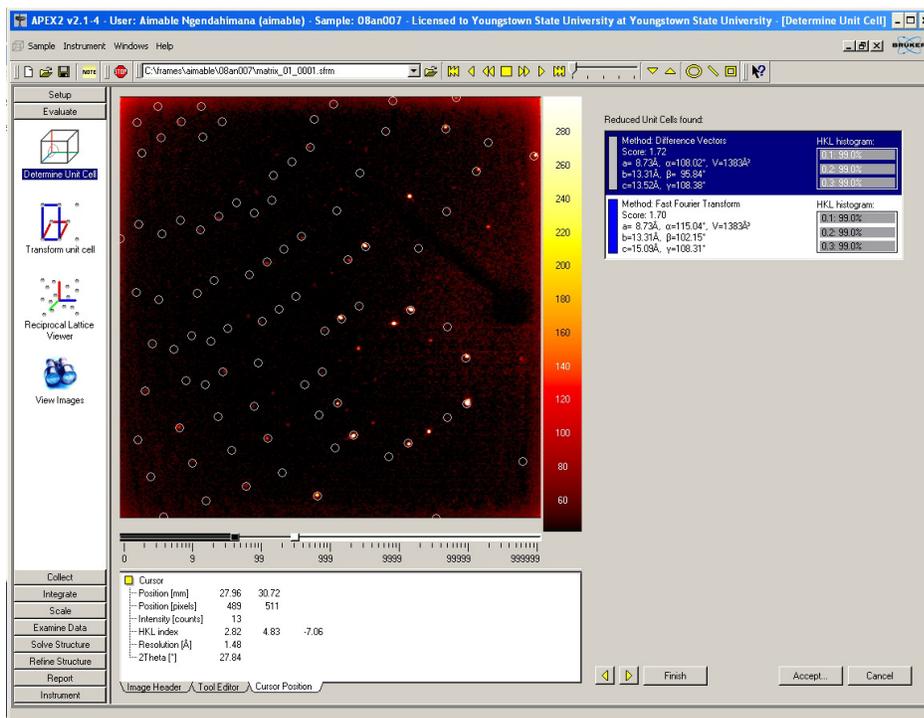


Figure 8, Unit Cell Indexing Result Window

Check the unit cell obtained. The positions of the diffraction spots should agree with the predicted positions (white or blue circles). The hkl histogram should have values around 90% or higher in the 0.1 line. If either of the two is not the case a non-merohedral twin might be present. If this case export the data as a p4p file using **<Sample>**, then **<Export>** and use the program Cell Now to determine the cell (see Appendix XXX, Twinning).

- If one of the two unit cells looks reasonable select it and proceed to **<Refine>**, take all defaults and click on **<Refine>**, then **<Accept>**.

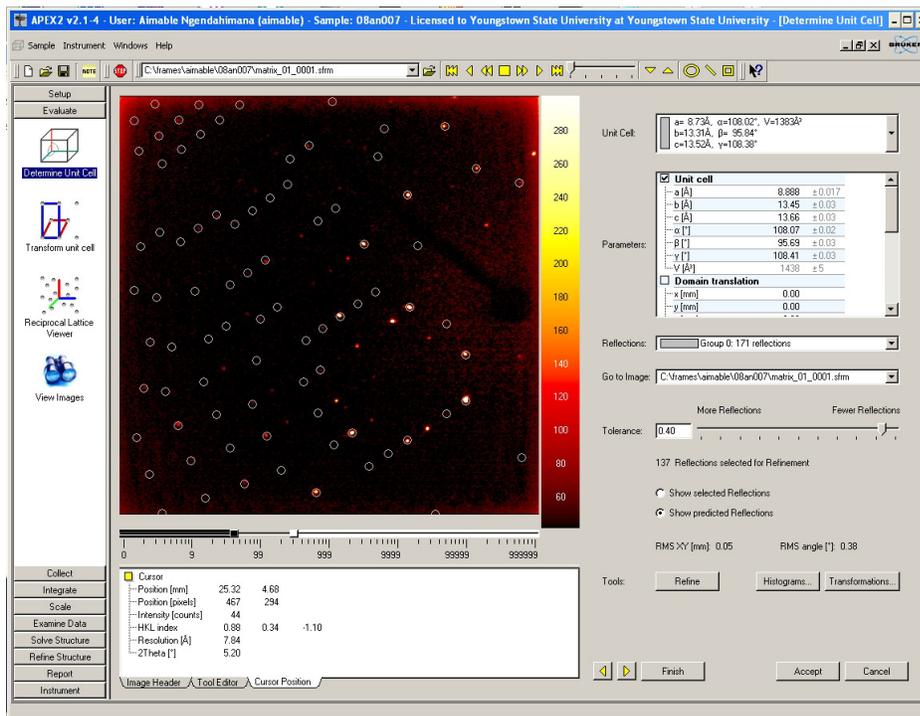


Figure 9, Unit Cell Refinement Window

- Proceed to **<Bravais>**. A list of possible Bravais lattices will be displayed with the software's choice highlighted.

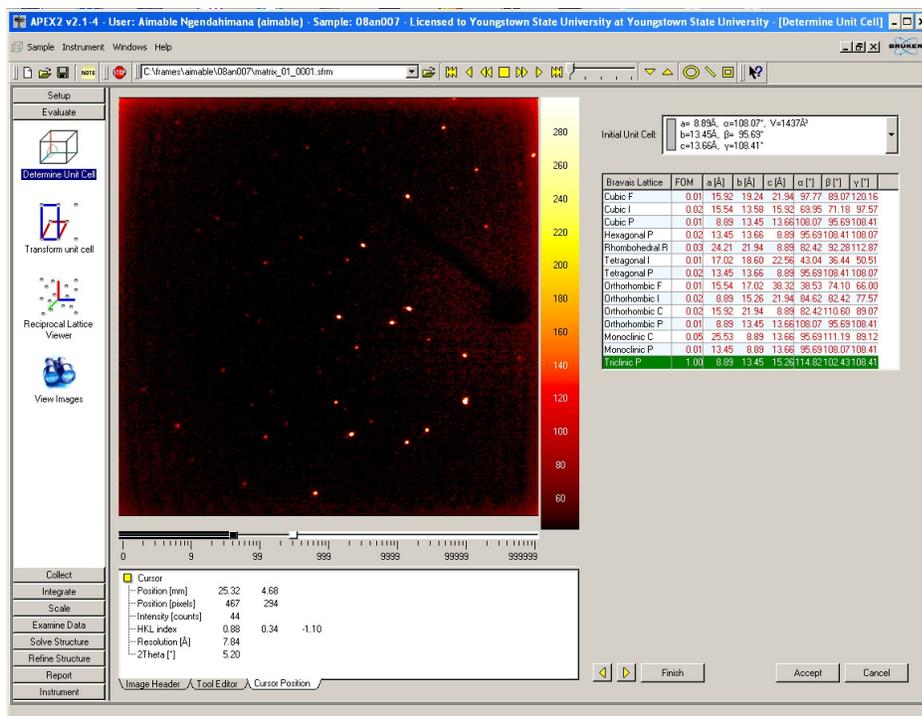


Figure 10, Bravais Result Window

The correct choice should have an FOM value significantly higher than the others. If several solutions have similarly high FOM values that with the highest symmetry is likely to be correct. If you are not sure (e.g. when a high symmetry solution has a high FOM value, but significantly lower than a lower symmetry solution) select the lower symmetry solution or your dataset might be incomplete when using **<Data Collection Strategy>** to set up a Strategy.

- Proceed to **<Refine>**, click **<Refine>**, then **<Accept>**.
- Please note the resolution prediction at the bottom of the window
- This finishes the Unit Cell Determination. If your dataset is high symmetry (orthorhombic or higher) and has no heavy absorbers proceed to **<Data Collection Strategy>** under **<Collect>** to use the program to develop the best data collection strategy. If your sample is low symmetry (or you don't yet know its symmetry, e.g. for twins), or if it is expected to be a heavily absorbing sample, set up a whole sphere data collection.

Data Collection

- **<Data Collection Strategy>**
 - Change the resolution to $d = 0.75$ (the ideal setting with our system).
 - Change **<Bijvoet Pairs>** to unmerged (in case the sample is non-centrosymmetric or chiral)
 - Change **<Exposure Time>** for the first three shells to a reasonable value. If the predicted resolution for 20 seconds was lower than 0.7 use 4 seconds, if it was higher than 0.7 use a longer time (up to 30-40 seconds is reasonable, in extreme cases up to several minutes)
 - Change **<Strategy>** to **<Best in x Hours>**. A well diffracting monoclinic sample can be finished in 2 or 4 hours. Higher symmetry samples need less time. Less well diffracting samples might need 8, 16 or even more hours.
 - In **<Execute>** select **<Refine Strategy>**.

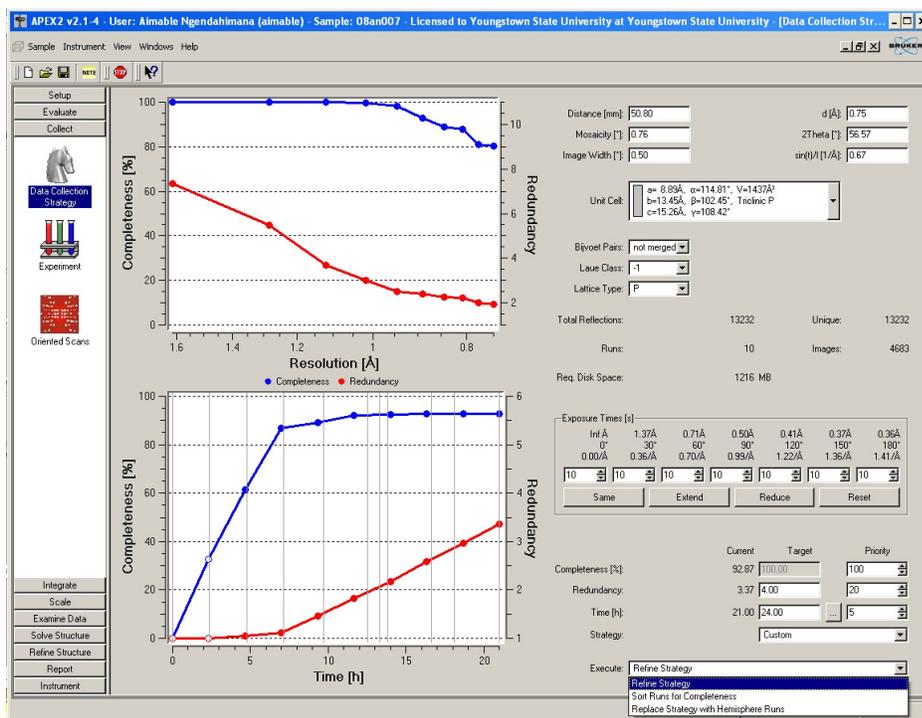


Figure 11, Strategy Starting Window

Watch **Completeness** and **Redundancy** as a function of Resolution and Time. Data should be >99 complete for $d = 0.75$, average redundancy should be greater than 3 to 4 (if possible). If the target time is too long or much more than objectively needed stop the process and select a different **<Best in x Hours>** option.

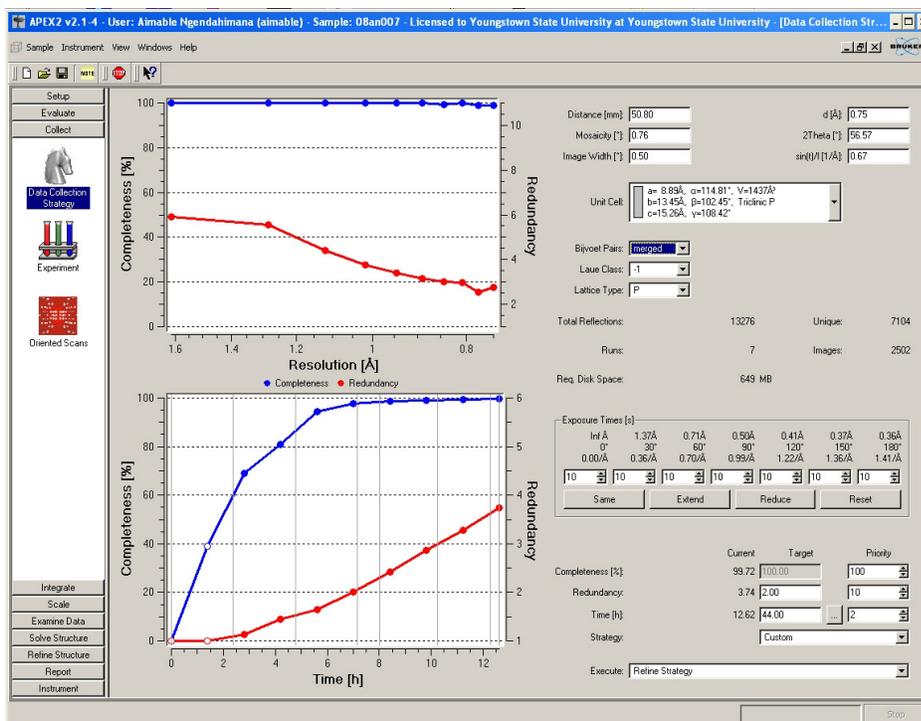


Figure 12, Strategy Window during optimization

- When the graphs are to your satisfaction (this might take a few minutes) Stop the process. The Strategy is now complete.
- Under <Collect> click on <Experiment>
 - If you did not use <Data Collection Strategy> collect a full sphere (e.g. for triclinic datasets). Click the <Load Table> button and select the <Sphere> file located in the root folder of every user. Good values for our instrument are:

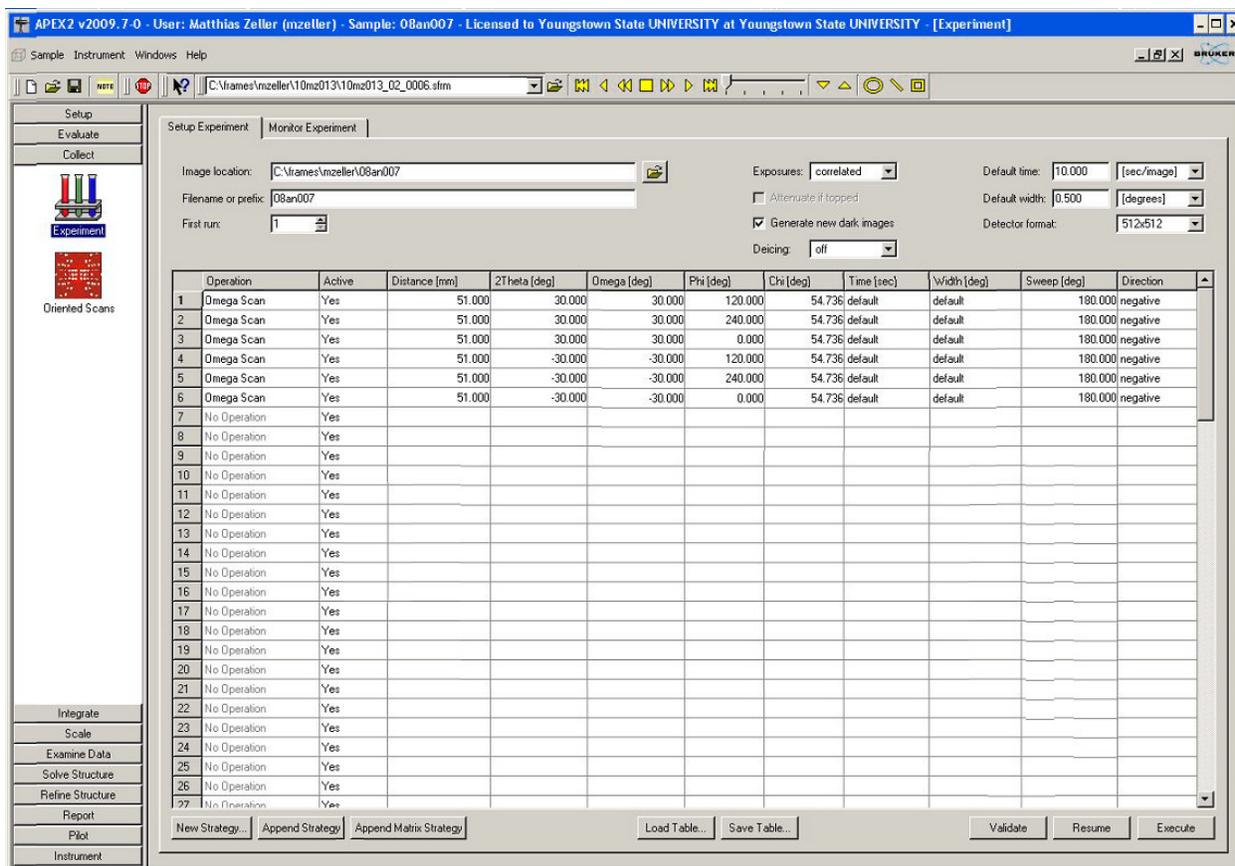


Figure 13, Experiment setup window with a standard strategy for collection of a full sphere up to a resolution of $d = 0.75$ (at 5 cm sample-detector distance)

- If you used **<Data Collection Strategy>** click on **<Append Strategy>**. Check the strategy by clicking on **<Validate>**.

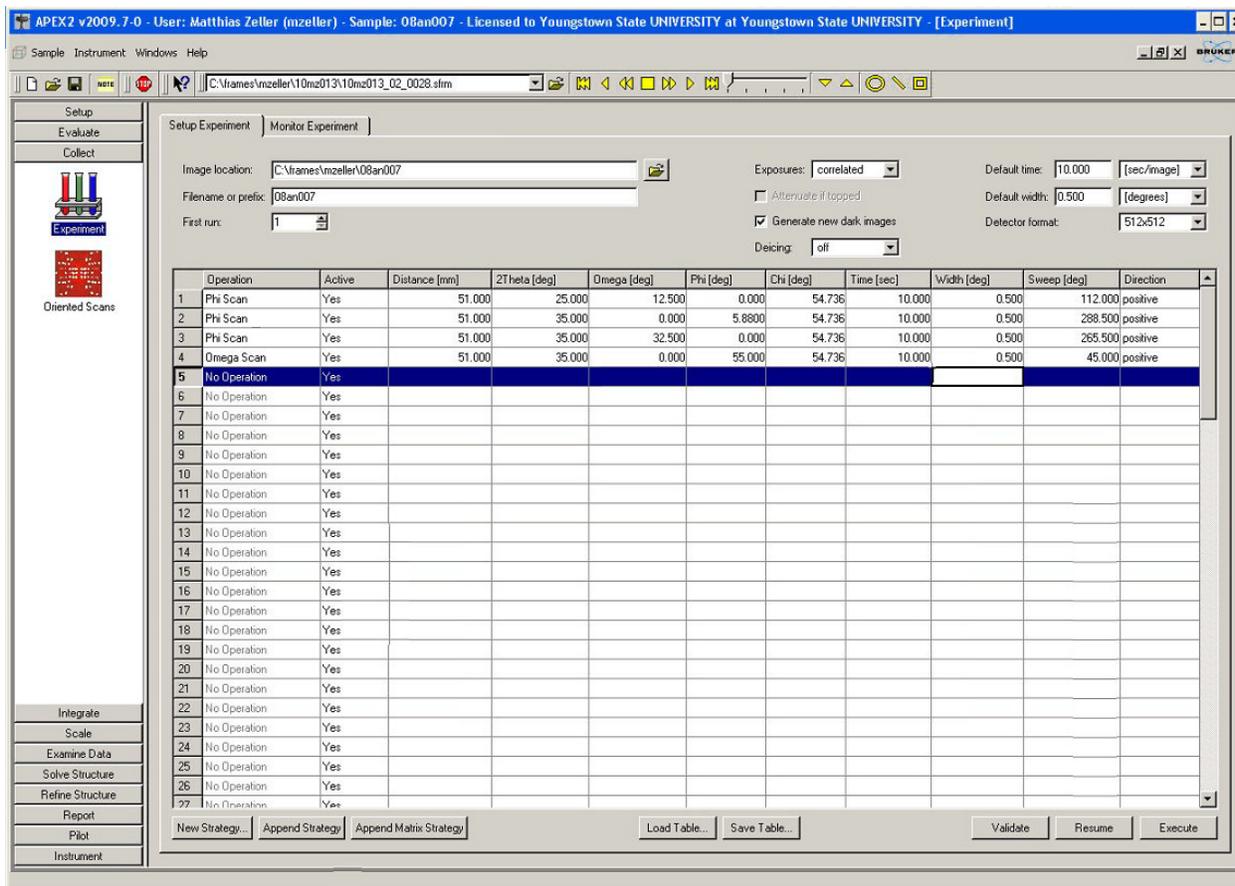


Figure 14, Experiment setup window with an typical strategy as determined by **<Data Collection Strategy>**

- Save the strategy by clicking **<Save Table>**. Then check that **<Filename or Prefix>** is correct (should be the same as the project name). If you used a **<Default Time>** in your Strategy change it to your value of choice.
- Proceed to **<Execute>**.
- The instrument will now collect your dataset. Open **<Check Status>** from the Instrument drop down menu to check variables and completion time. This will open the **<Instrument Status>** window.

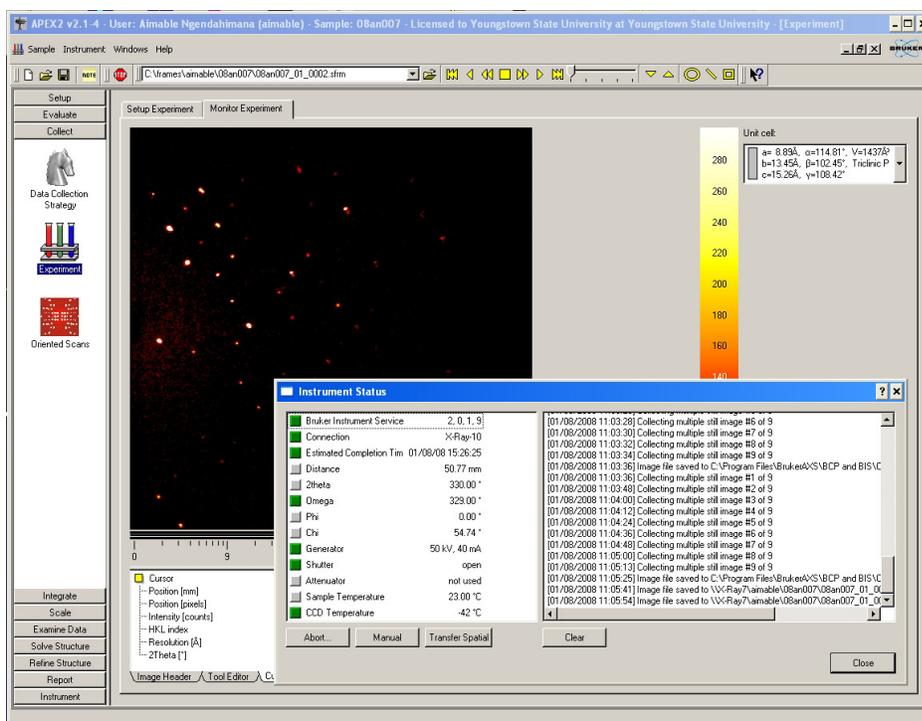


Figure 15, Data Collection Window with active data collection and Instrument Status Window open

- Users are encouraged to check their data during collection and to test-solve and refine the data while the data collection is still running to check that the data quality is good enough to proceed with the whole collection, that the unit cell is indeed correct, and if data might be already complete. It is highly advisable to not stop a data collection until you are sure the data are complete enough for a meaningful absorption correction and to pass checkcif (i.e. data should be solved, refined, the refinement quality checked, the completeness should be checked in the cif file, and preliminarily checked using checkcif prior to stopping the data collection!!). Once a crystal is taken off the instrument a data collection cannot be resumed!

Integration

When the data collection is complete (or when enough data are collected for an initial

structure solution and refinement attempt) open **<Integrate>**, then **<Integrate Images>**

- Change the resolution limit to 0.75 or lower
- Click on **<Find Runs>**, check the runs you would like to integrate (leave out the Matrix runs). If some runs require mapping of the beam stop, integrate them separately.

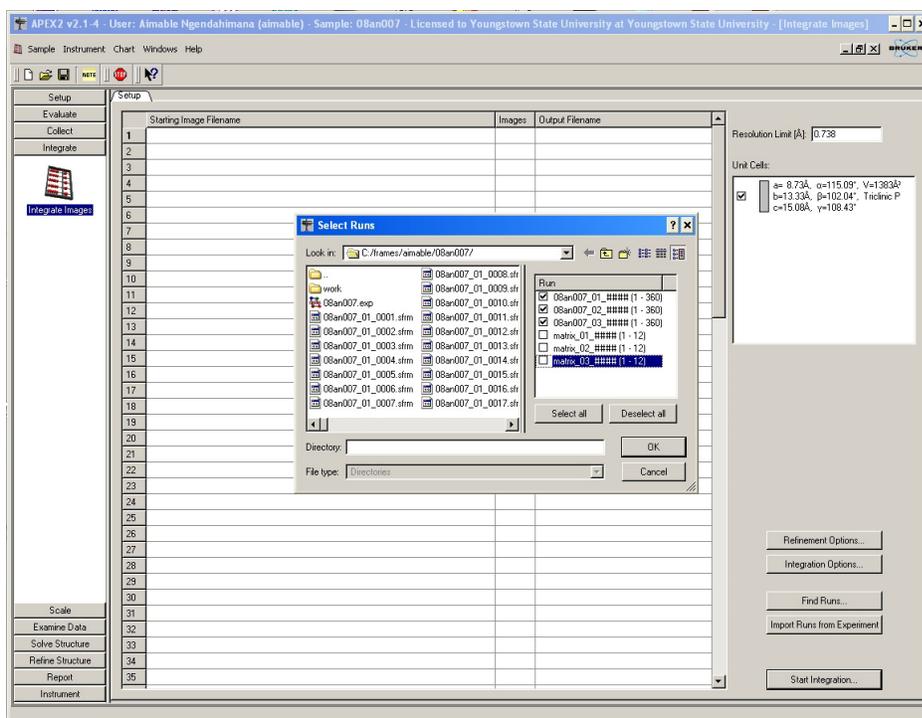


Figure 16, Integration Start Window with **<Find Runs>** Window open

- If the beam stop needs to be mapped, click on **<Integration Options>**, then **<More Options>**. Under **<Active Mask>** check **<Generate Mask>** and change the value from 0.00 to e.g. 0.70. For runs that do not require beam stop mapping, change it back to 0.00.
- Click on **<Start Integration>**. This will start the integration. Wait for it to finish, this might take several minutes.

Spot Shape Correlation should show values above 80% and be mostly even. Values of 40% or lower are statistically meaningless. Pixel Error should be on average between ± 0.2 . Average I/σ intensities should be above at least 3 (good datasets have values between 15 and 20). Shape profiles should be round to slightly ellipsoid. Double spots, large pixel errors and erratic uneven lines are possible signs for twinning. If you suspect a crystal to be split or twinned, consult Appendix XXX, Twinning.

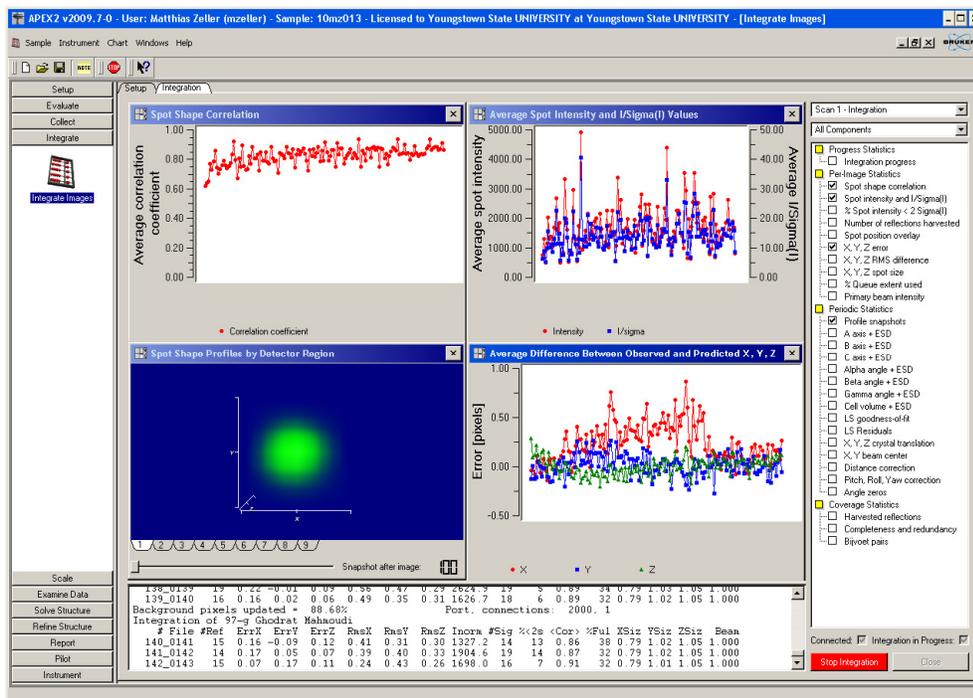


Figure 17, Integration Window with active Integration running

- Under <Scale> Click on <Scale>

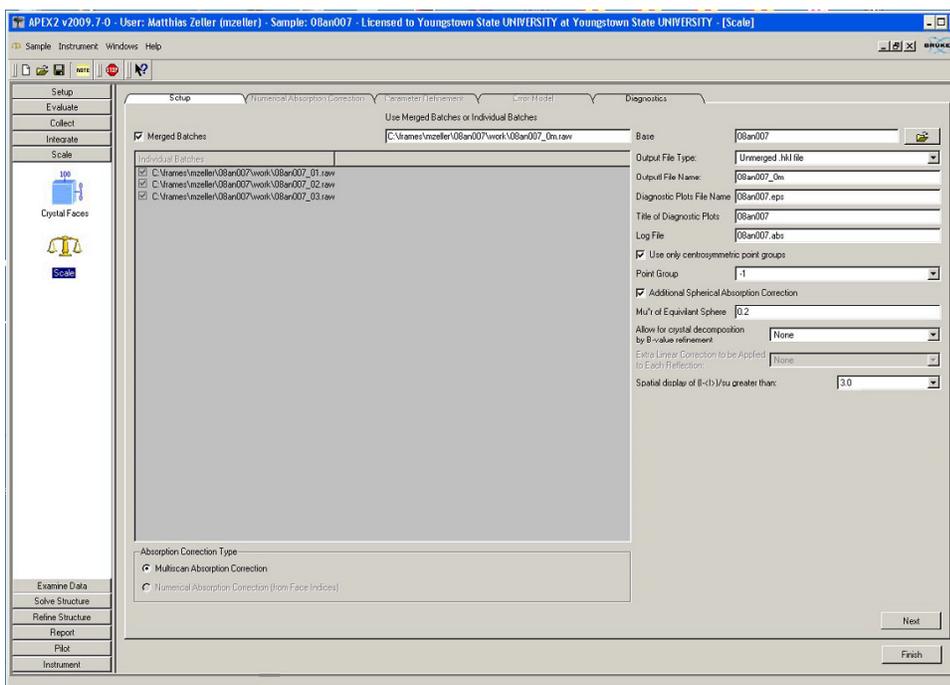
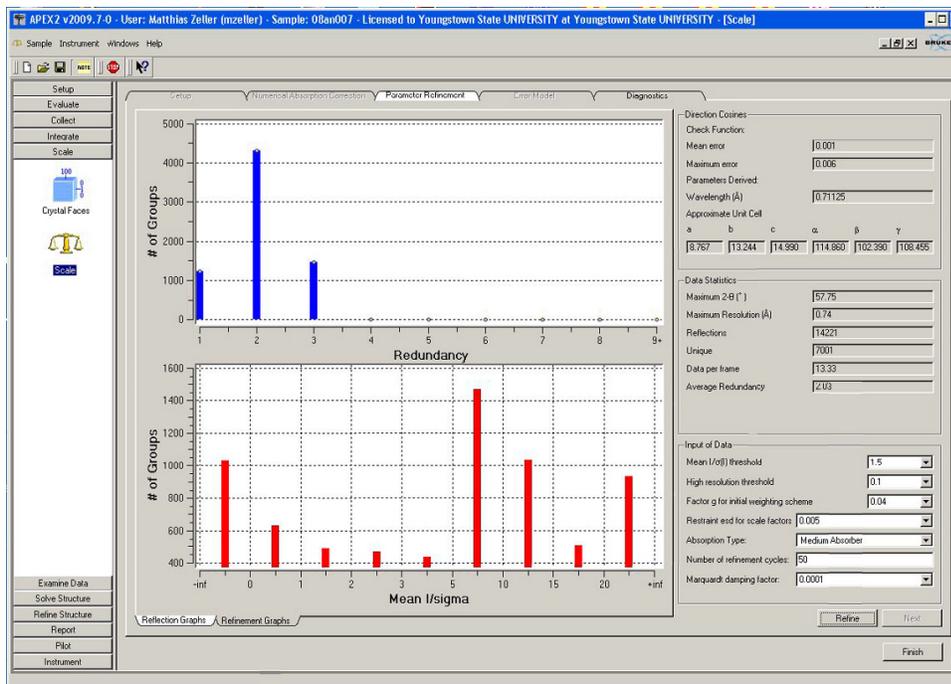


Figure 18, Absorption Correction and Error Model (<Scale>) Starting Window

- The merged batch raw file from the integration is loaded automatically. If you did integrate all runs at once you need to load individual raw files. Click the yellow browse symbol, click on the first raw file you want to load, check **<open all with base>** then click **<OK>** to load individual raw files and return to the main window. Uncheck **<use merged files>** box. Check the filenames and pointgroup and proceed by clicking **<Next>**.
- On the next page click **<Refine>**, then **<Next>**



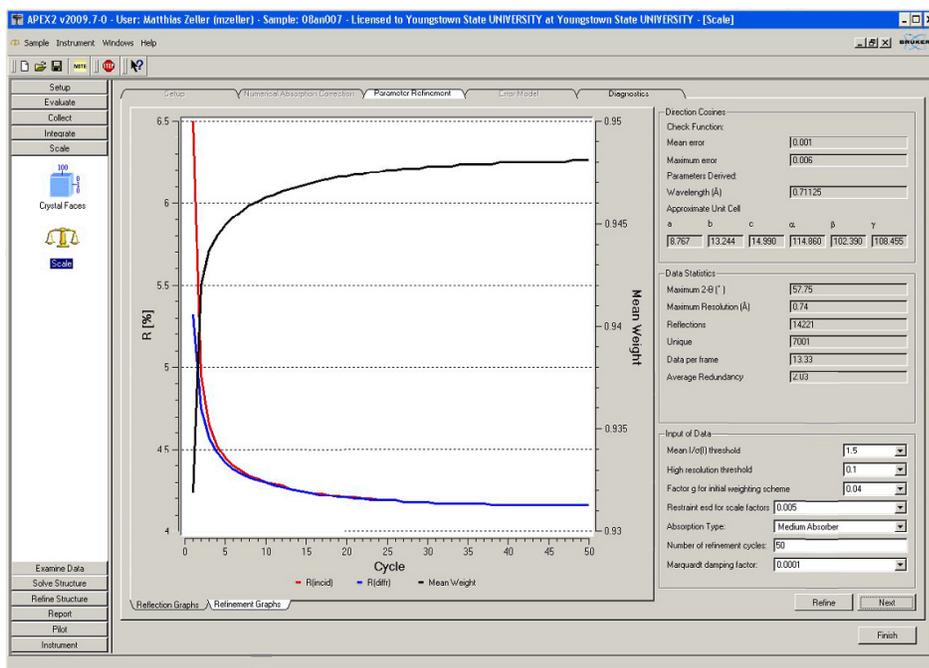


Figure 20, Absorption Correction window (second Scale Window) after refinement

- On the Error Model page click <Determine Error Model>, then <Finish>

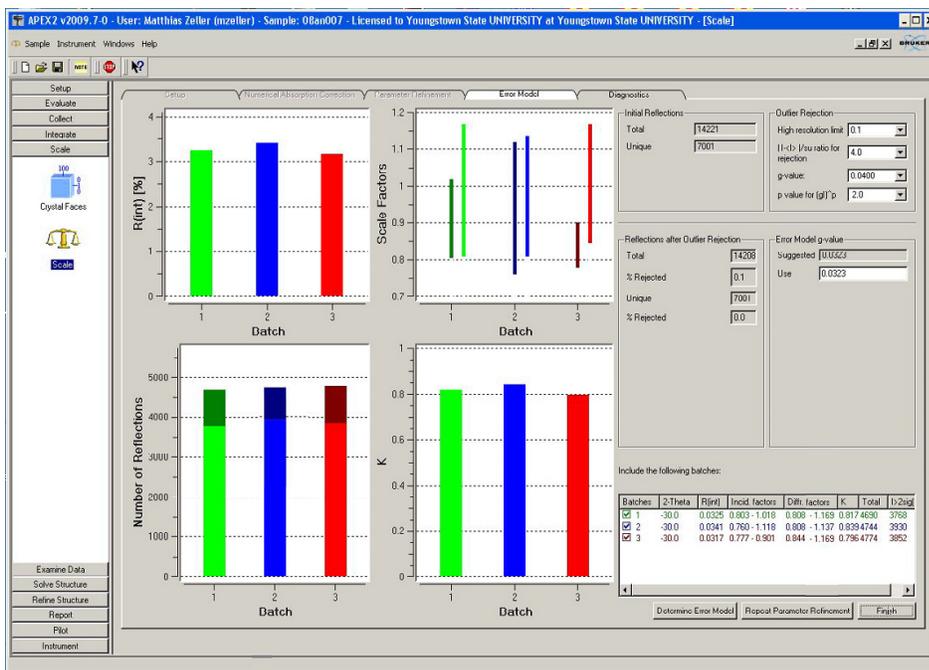


Figure 21, Error Model Window (third Scale Window) after determination of error model

- You have now finished the data collection and absorption correction. To allow other users to proceed with their data collection you should at this point move your data to another computer.

Final Steps before Refinement

- Burn a CD or move the data via a USB flash drive or online to your personal computer.
- For the refinement, you will need the *.hkl, the *.p4p files (located in the work folder)
e.g. 04mz02a_0m.hkl
04mz02a_0m.p4p
- For publication purposes you will also need the *.abs and the *_ls files:
e.g. 04mz02am.abs or 04mz02a_0m.abs (A copy of what you did in SCALE, SADABS, or TWINABS, contains the ratio of Tmin/Tmax)
04mz02am_0m.ls (A copy of the last lines of the integration procedure, contains parameters of unit cell refinement (2THETA min, 2THETA max and the number of reflections used), crystal colour and shape, crystal dimensions)
- If you used Cell Now to obtain a unit cell you will also need to copy the *_cn file

Solving Structures

- **On your computer save the files from above** into a new file folder. Making a backup of your files is strongly recommended! (with older operating systems you need to uncheck the “read only” flag (by highlighting your files plus right mouse click, go into properties and uncheck.)
- Open the **SHELXTL** program. Select **<PROJECT>** and **<New>**. Find the appropriate file and open it. Give it a project name e.g. 04mz02a_0m, then **<open>**.

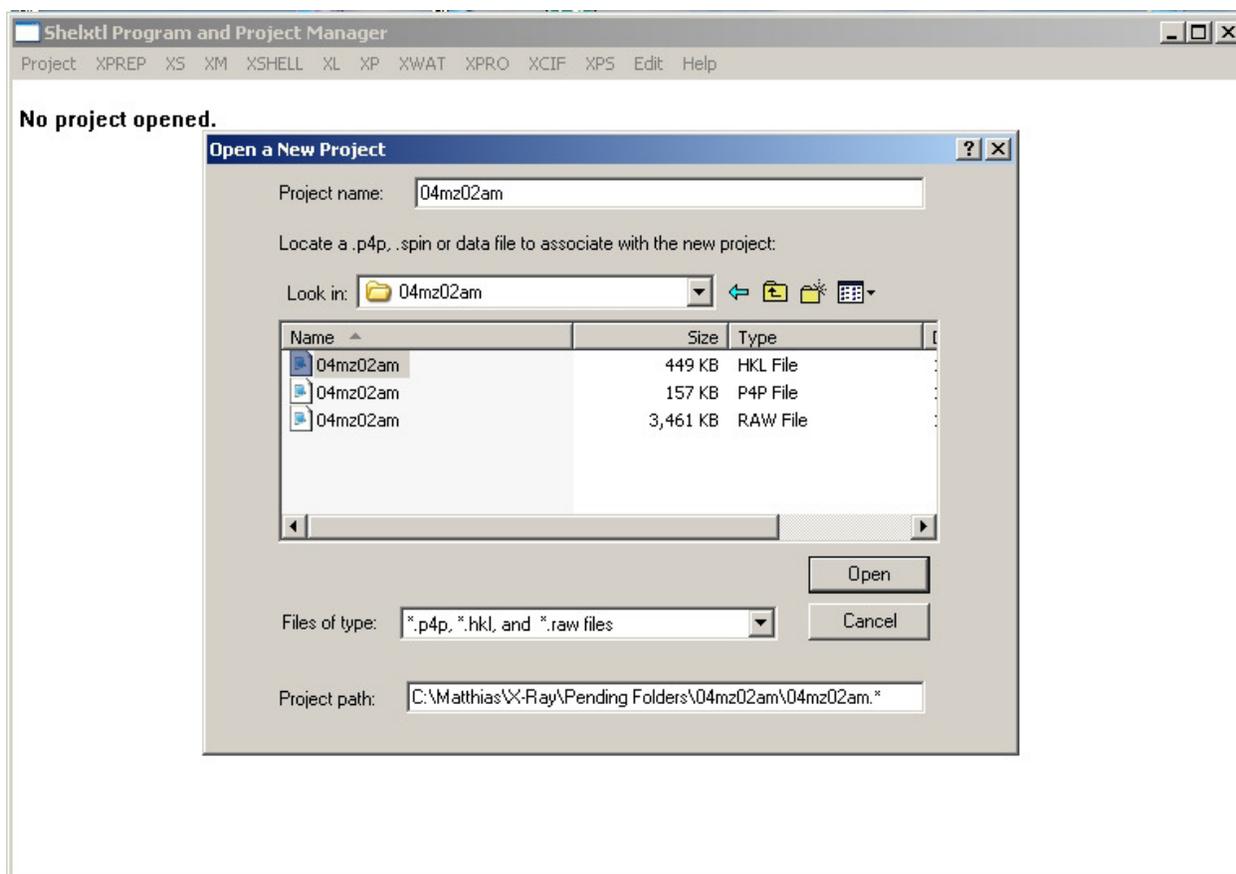
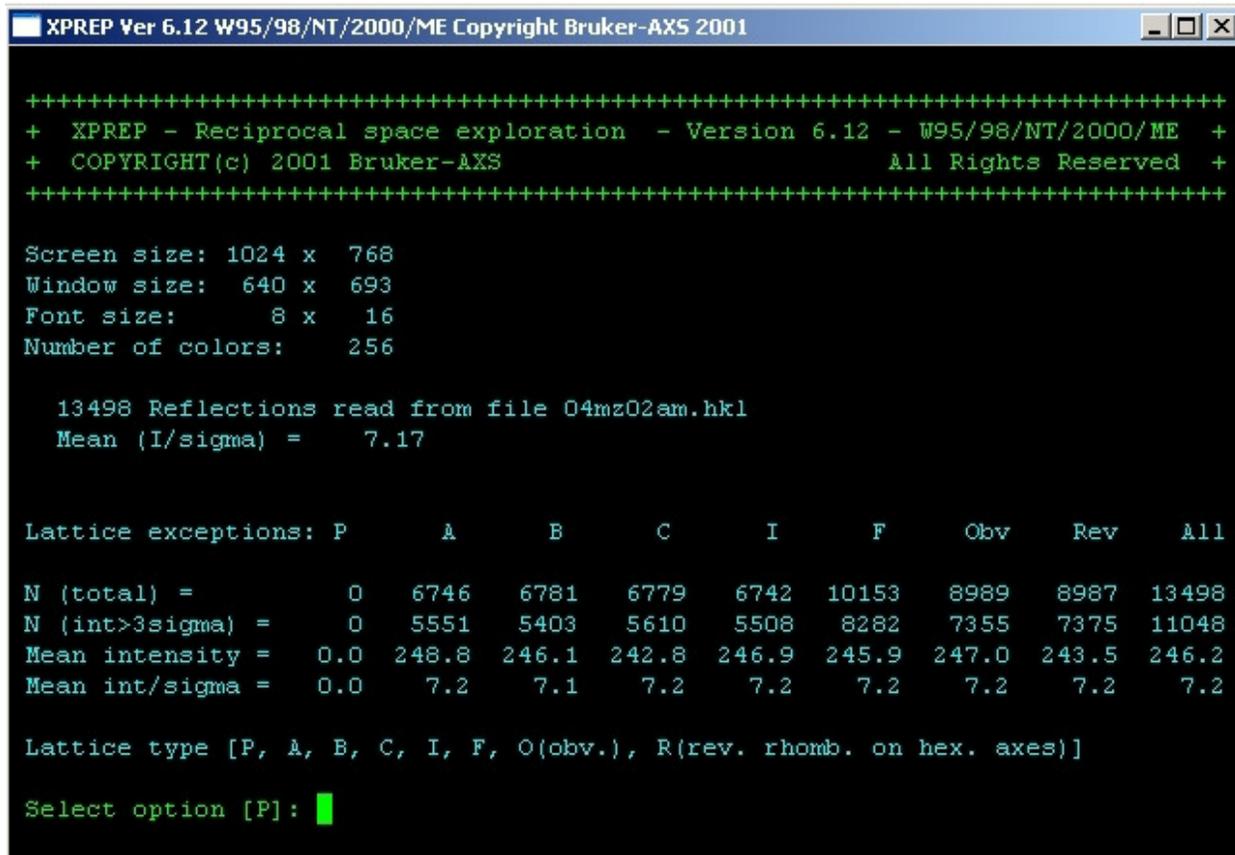


Figure 22, Shelxtl Program and Project Manager Window with New Project Window open

XPREP

- Select <XPREP> on the toolbar

In the next steps, the computer will make suggestions that can usually be accepted (i.e. for good quality data).



```
XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001

+++++
+ XPREP - Reciprocal space exploration - Version 6.12 - W95/98/NT/2000/ME +
+ COPYRIGHT(c) 2001 Bruker-AXS All Rights Reserved +
+++++

Screen size: 1024 x 768
Window size: 640 x 693
Font size: 8 x 16
Number of colors: 256

13498 Reflections read from file O4mzO2am.hkl
Mean (I/sigma) = 7.17

Lattice exceptions: P A B C I F Obv Rev All
N (total) = 0 6746 6781 6779 6742 10153 8989 8987 13498
N (int>3sigma) = 0 5551 5403 5610 5508 8282 7355 7375 11048
Mean intensity = 0.0 248.8 246.1 242.8 246.9 245.9 247.0 243.5 246.2
Mean int/sigma = 0.0 7.2 7.1 7.2 7.2 7.2 7.2 7.2 7.2

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]
Select option [P]: █
```

Figure 23, XPREP Window, initial lattice centering selection

- Select the suggested lattice type
- Choose [H] to search for higher metric symmetry
- Choose offered choice [A] for the Laue group (e.g. orthorhombic)

```

XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001
Current dataset: 04mz02am.hkl      Wavelength: 0.71073 Chiral: ?
-----
Original cell:  8.411  12.658  13.141  103.64  89.98  90.02  Vol  1359.6
                Esds:  0.001  0.001  0.001  0.00  0.00  0.00  Lattice: P
-----
Current cell:   8.411  12.658  13.141  103.64  89.98  90.02  Vol  1359.6
-----
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-----

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry      [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP         [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING          [O] Self-rotation function
[L] Reset LATTICE type of original cell    [Q] QUIT program

Select option [H]:

Determination of reduced (Niggli) cell

Transformation from original cell (HKLF-matrix):
-1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000 -1.0000

Unitcell:      8.411  12.658  13.141  76.36  89.98  89.98

Niggli form:   a.a =    70.75      b.b =   160.22      c.c =   172.68
                b.c =    39.24      a.c =    0.04      a.b =    0.03

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

-----
Option A: FOM = 0.026 deg.  MONOCLINIC  P-lattice  R(sym) = 0.059 [ 3093]
Cell:  12.658  8.411  13.141  90.02  103.64  89.98  Volume:  1359.61
Matrix: 0.0000  1.0000  0.0000 -1.0000  0.0000  0.0000  0.0000  0.0000  1.0000
-----
Option B retains original cell

Select option [A]: █
PgUp/PgDn scrolls text; only graphics window may be resized

```

Figure 24, , XPREP Window, initial lattice type selection

- Select [S] to determine or input space group
- Select [S] again to determine space group
- Select the suggested **Laue group** (e.g. [O] for orthorhombic)
- Select the suggested **lattice centering** (e.g. [C] for C-centered)

```

XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001
Current dataset: 04mz02am.hkl      Wavelength: 0.71073  Chiral: ?
-----
Original cell:  8.411  12.658  13.141  103.64  89.98  90.02  Vol  1359.6
                Esds:  0.001  0.001  0.001  0.00  0.00  0.00  Lattice: P
-----
Current cell:   12.658  8.411  13.141  90.02  103.64  89.98  Vol  1359.6
-----
Matrix: 0.0000  1.0000  0.0000 -1.0000  0.0000  0.0000  0.0000  0.0000  1.0000
-----
Crystal system: Monoclinic      Lattice: P
-----

[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu or [Q] QUIT program

Select option [S]:

[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal,
[H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT

Select option [M]:

Lattice exceptions: P      A      B      C      I      F      Obv      Rev      All
N (total) =             0   6781   6746   6779   6742  10153   8990   8985  13498
N (int>3sigma) =        0   5403   5551   5610   5508   8282   7367   7339  11048
Mean intensity =        0.0 246.1  248.8  242.8  246.9  245.9  247.3  247.4  246.2
Mean int/sigma =        0.0  7.1   7.2   7.2   7.2   7.2   7.2   7.2   7.2

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]: █

```

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

Figure 25, XPREP Window, new lattice centering selection

- Select the suggested for the **space group** e.g. C222(1) (If several solutions are offered, take that one with the lowest CFOM value. If that does not work out later on, try the next best in the list)

```

XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001
[E] EXIT to main menu or [Q] QUIT program

Select option [S]:

[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal,
[H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT

Select option [M]:

Lattice exceptions: P      A      B      C      I      F      Obv      Rev      All
N (total) =           0  6781  6746  6779  6742  10153  8990  8985  13498
N (int>3sigma) =       0  5403  5551  5610  5508  8282  7367  7339  11048
Mean intensity =     0.0 246.1 248.8 242.8 246.9 245.9 247.3 247.4 246.2
Mean int/sigma =     0.0  7.1  7.2  7.2  7.2  7.2  7.2  7.2  7.2

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:

Mean |E*E-1| = 0.903 [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

      -21-  -a-  -c-  -n-
N         12  456  485  465
N I>3s    0   202   5   203
<I>       1.9 262.0  2.5 257.0
<I/s>     0.9  4.5  0.8  4.5

Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym)  N(eq)  Syst. Abs.  CFOM
[A] P2(1)/c          # 14  centro  1 19410  0.059  3093  0.9 / 4.5  3.13

Select option [A]: █
PgUp/PgDn scrolls text; only graphics window may be resized

```

Figure 26, XPREP Window, space group selection

- Select [D] to read, modify or merge datasets
- Select [S] to display the **intensity statistics**

- Select [A] to not merge all equivalent reflections (including Friedel opposites). This does not actually merge the reflections (which would reduce your data to parameter ratio significantly), but only displays the data and statistics as if they would have been merged.

The intensity statistics will be displayed.

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma
Inf - 2.20	663	482	137.6	1.38	944.8	18.43		0.0485
2.20 - 1.75	704	482	146.1	1.46	433.7	17.69		0.0492
1.75 - 1.50	813	566	143.6	1.44	283.5	16.12		0.0500
1.50 - 1.35	816	576	141.7	1.42	225.5	15.46		0.0509
1.35 - 1.25	673	512	131.4	1.31	189.3	15.47		0.0519
1.25 - 1.15	953	758	125.7	1.26	149.3	14.25		0.0534
1.15 - 1.05	1246	1072	116.2	1.16	134.6	13.36		0.0548
1.05 - 1.00	771	712	108.3	1.08	101.3	12.97		0.0579
1.00 - 0.95	870	842	103.3	1.03	87.3	12.23		0.0601
0.95 - 0.90	1025	1050	97.6	0.98	66.6	11.46		0.0649
0.90 - 0.85	1219	1322	92.2	0.92	52.8	10.32		0.0702
0.85 - 0.80	1400	1648	85.0	0.85	42.9	9.28		0.0766
0.80 - 0.75	1682	2160	77.9	0.78	34.4	8.38		0.0852
0.75 - 0.70	3082	3808	80.9	0.81	38.3	8.79		0.0808
Inf - 0.75	12835	12182	105.4	1.05	170.5	12.70		0.0536

Merged [N]; lowest resolution = 10.38 Angstroms; 0 outliers downweighted

Enter <CR> to continue

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

Figure 27, XPREP Window, intensity statistics

Have a look at the **Completeness**, **Redundancy** and **Rint** and **Rsigma**. The completeness should be ideally close or over 100 % down to a resolution of $d = 0.75$. Rsigma and Rint should be ideally below 10 % down to $d = 0.75$. If they become very large at higher resolution (lower d values), it might be appropriate to cut the data at $d = 0$.

Hit the Enter key

- Select [H] to apply a high resolution cutoff.
 - Type 0.75 for the high resolution cutoff for data collected on our Smart Apex Instrument with the usual strategy.
- Use the default value (inf) for the low resolution cutoff.
- Select [E] to return to the main menu
 - Select [C] to define the **unit cell contents**.
 - If no unit cell contents are given type the most likely molecular formula (element symbols and numbers without any spaces)
 - If the given formula seems wrong, select [F] for new formula, and type the most likely formula

XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001

```

Current formula is:
C11H8O1N2I2Zn1

Tentative Z (number of formula units/cell) = 4.0 giving rho = 2.459,
non-H atomic volume = 20.0 and following cell contents and analysis:

C      44.00    26.25 %      H      32.00    1.60 %
N       8.00     5.57 %      O       4.00    3.18 %
Zn      4.00    12.99 %      I       8.00   50.42 %

[Z] change Z, [F] new FORMULA, [R] change RADIATION,
[E] EXIT to main menu or [Q] QUIT program

Select option [E]: █

```

Figure 28, XPREP Window, formula definition

- If necessary, select [R] to change the radiation (Mo radn; $\lambda = 0.71073$; Cu radn; $\lambda = 1.54178$)
- If necessary, select [Z] to change the number of (symmetrically independent) molecules **Z** per unit cell, (note: not all values of Z are possible with all space groups, 5 and 7, 9, 11 etc are impossible for all space groups)
- Select [E] to exit to the main menu

Note: If you had been using SADABS for the generation of your *.hkl file, no absorption correction has to be applied. If you still need to apply absorption correction, this can be done here by choosing [A] (not covered here)

- Select [F] to setup the new hkl file. If you have changed the unit cell or its orientation in XPREP, the program will force you to choose a new name for the hkl file to avoid losing the original data (change it back to your old filename after closing XPREP after making a backup of the original hkl file)

- Select [Y] at the prompt to generate an .ins file

```

XPREP Ver 6.12 W95/98/NT/2000/ME Copyright Bruker-AXS 2001
-----
Original cell:  8.411  12.658  13.141  103.64  89.98  90.02  Vol  1359.6
                Esds:  0.001  0.001  0.001  0.00  0.00  0.00  Lattice: P
-----
Current cell:  12.658  8.411  13.141  90.02  103.64  89.98  Vol  1359.6
-----
Matrix: 0.0000  1.0000  0.0000 -1.0000  0.0000  0.0000  0.0000  0.0000  1.0000
-----
Crystal system: Monoclinic  Space group: P2(1)/c  # 14 [cen]  Laue: 2
-----
Formula: C11H8O1N2I2Zn1  Formula wt: 503.36
Z: 4.00  Density: 2.459  At.vol: 20.0  F(000): 928.00  Mu[mm-1]: 6.33
-----

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry      [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP         [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING          [O] Self-rotation function
[L] Reset LATTICE type of original cell    [Q] QUIT program

Select option [D]: f

Output file name (without extension) []: O4mzO2am
** Filename MUST be different if matrix is changed **
Output file name (without extension) []: O4mzO2am1

File O4mzO2am1.ins set up as follows:

TITL O4mzO2am1 in P2(1)/c
CELL 0.71073  12.6580  8.4112  13.1408  90.000  103.644  90.000
ZERR  4.00  0.0013  0.0008  0.0013  0.000  0.002  0.000
LATT 1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N O ZN I
UNIT 44 32 8 4 4 8
TEMP -173.5
TREF
HKLF 4
END

Do you wish to (over)write the intensity data file O4mzO2am1.hkl ? [N]: y
PgUp/PgDn scrolls text; only graphics window may be resized

```

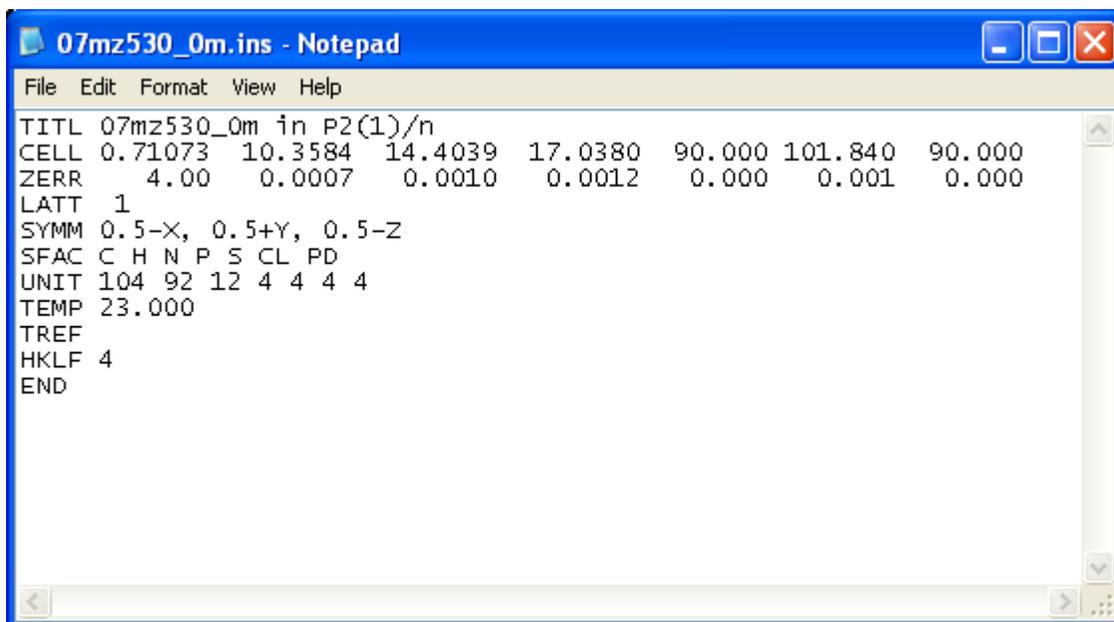
Figure 29, XPREP Window, ins and hkl file setup

- Select [Q] at the prompt

The Initial INS File

The initial ins file written in XPREP, together with the hkl file, contains all the information to get started solving and refining your crystal structure. The ins file can be opened and edited in any text editor. Open it via **<Edit>** and **<Edit .ins>** in the SHELXTL menu.

The initial ins file may look like this:

A screenshot of a Notepad window titled "07mz530_0m.ins - Notepad". The window contains the following text:

```
File Edit Format View Help
TITL 07mz530_0m in P2(1)/n
CELL 0.71073 10.3584 14.4039 17.0380 90.000 101.840 90.000
ZERR 4.00 0.0007 0.0010 0.0012 0.000 0.001 0.000
LATT 1
SYMM 0.5-X, 0.5+Y, 0.5-Z
SFAC C H N P S CL PD
UNIT 104 92 12 4 4 4 4
TEMP 23.000
TREF
HKL 4
END
```

Figure 30, typical initial INS file

The **TITL** line gives the initial name of the ins file and the space group from XPREP.

The cell line gives the wavelength used, then the unit cell parameters a, b, c, α , β , γ .

The **ZERR** line gives the Z value (number of molecules in the unit cell as given in XPREP) and the estimated standard deviations for a, b, c, α , β , γ .

LATT gives the lattice type (see SHELXTL manual).

The **SYMM** line(s) give the symmetry operators of the space group that create symmetry dependent atoms (these lines define the actual space group, not the TITL line).

The **SFAC** line gives the atom types and defines the atom structure factors to be used by the program. New elements have to be added in this line before they can be used during structure refinement.

The **UNIT** line gives the number of atoms per unit cell as listed in the SFAC line.

The **TEMP** line gives the temperature in degrees Celsius. **Change the value to the actual temperature used in your experiment.**

The **TREF** line defines the type of structure solution method to be used (see below)

The **HKLF 4** line defines the type of refinement to be used (HKLF 4 is the standard single crystal refinement for a not non-merohedrally twinned dataset).

The **END** lines defines the end of the file.

Select **<file>** and then **<save>**.

Using XS

To perform the refinement of your structure, you will first need an initial guess (a starting point). Using the *.ins file provided by XPREP, the program XS will provide you with this initial guess. In the Shelxtl software there are two methods to choose from: Direct methods (indicated by the line TREF in the INS file), and the Patterson method (indicated by the line PATT in the ins file). Direct methods can be used for all datasets with better than atomic resolution. Patterson methods can only be used for structures with at least one heavy atom (i.e. sodium or heavier). Direct methods usually provide a more complete initial guess (more atoms already found), but they tend to fail more often than Patterson methods and they have occasionally problems to place inversion centers correctly.

- Select **<XS>** on the toolbar and the computer should begin to process data. The software tries in this step to find an “initial guess” for the atom positions. This can only be successful, if the atoms listed in the formula are correct. The software will select the solution with the smallest value for CFOM and the initial atom positions are written in the ins file.

```

C:\WINNT\System32\cmd.exe
+-----+
+ XS - CRYSTAL STRUCTURE SOLUTION - SHELXTL Ver. 6.12 W95/98/NT/2000/ME +
+ Copyright(c) 2001 Bruker AXS All Rights Reserved +
+ 04mz02am started at 15:40:24 on 20-Jan-2004 +
+-----+

Read instructions and process reflection data
Data: 3402 unique, 3292 observed R(int) = 0.0678 R(sigma) = 0.0599
Systematic absence violations: 4 Bad equivalents: 1
ESEL Emin 1.200 Emax 5.000 DelU 0.005 renorm 0.700 axis 0
OMIT s 4.00 2theta(lim) 180.0
INIT nn 11 nf 16 s+ 0.800 s- 0.200 wr 0.200
PHAN steps 10 cool 0.900 Boltz 0.400 ns 153 mtp 40 mnqr 10
TREF np 256. nE 246 kapscale 0.900 ntan 2 wn -0.950
FMAP code 8
PLAN npeaks -28 del1 0.500 del2 1.500
MORE verbosity 1
TIME t 9999999.

153 Reflections and 2010. unique TPR for phase annealing
246 Phases refined using 5742. unique TPR
394 Reflections and 11123. unique TPR for R(alpha)
1791 Unique negative quartets found, 1584 used for phase refinement
186 Unique NQR employed in phase annealing
128 Parallel refinements, highest memory = 7328 / 73612

Try Ralpha Nqual Sigma-1 M(abs) CFOM Seminvariants
738873. 0.042 -1.000 0.963 1.251 0.042* +--+ -+++ +---- -+--+ -+--+
Freq: 0 0 112 0 0 0 0 0 0 0 0 0 0 0 1 7 4 0 0 0 1 0 1 0 0 0 1 1 0 / 128
1029041. 0.042 -1.000 0.963 1.251 0.042 +--+ -+++ +---- -+--+ -+--+
Freq: 0 0 224 0 0 0 0 0 0 0 0 0 0 0 2 13 8 1 0 1 2 0 2 0 0 1 1 1 0 / 256

256. Phase sets refined - best is code 738873. with CFOM = 0.0417

Fourier and peaksearch
RE = 0.171 for 16 atoms and 1031 E-values
Fourier and peaksearch
RE = 0.156 for 23 atoms and 1031 E-values
Fourier and peaksearch

+-----+
+ 04mz02am finished at 15:40:25 Total elapsed time: 2.0 secs +
+-----+
Press any key to continue . . .

```

Figure 31, the XS window

- For good quality data the right solution is normally “falling out” when continuing with XP or XSHELL.

If this is not the case, you can change the settings for XS in the ins file. Go to **<Edit>** on the toolbar and select **<edit .ins>**

Either, change **TREF** to **TREF 2000** (or up to 10000) to run more iterations, or, for compounds with atoms heavier than sodium, use **PATT** instead of **TREF**. Select **<file>** and then **<save>** and run XS again.

ADD XM

Structure Refinement

The refinement cycles can be performed using either **<XP>** and **<XL>** or the graphical user interface **<XSHELL>**.

The descriptions given here are based on **<XSHELL>**. For a detailed introduction into refinement methods using **<XP>** and **<XL>** please refer to the book Crystal Structure Refinement by Peter Müller or the Shelxl Manual.

The SHELX GUI consists of a graphical user interface displaying the atoms, and a range of sub-windows to change settings, define properties and perform operations. The typical windows layout is mostly self explaining, but the user will need to get familiar with all the features to use the program to its full extent.

- Select **<XSHELL>** on the toolbar to open the graphical interface.

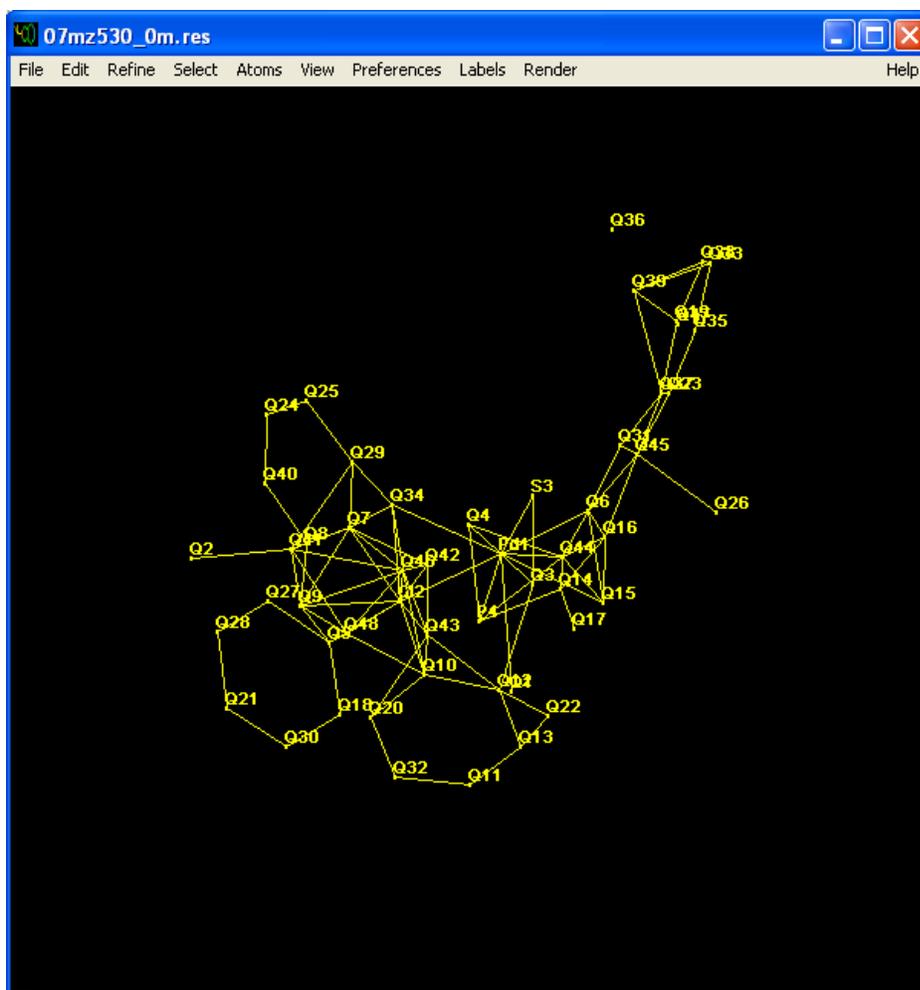


Figure 32, XSHELL window with initial result from XS

- The initial result from the structure solution will usually not display atoms, but mostly “Q-peaks” (some atoms might be displayed, but they are likely to be incomplete and wrongly assigned). Q-peaks are positions within the unit cell that might represent a possible atom, and you will have to decide which Q-peaks might be actual atoms. Q-peaks with a lower number have a higher electron density count and are more likely to be real atoms. Move the cursor atop of the atom, this will display the actual electron count in the upper left corner of the graphical interface. (If you are not able to identify any fragments of the expected molecule, the initial guess might be wrong. Go back and try XS again with higher TREF numbers or using PATT).

- At this point you have to decide, which of the Q-peaks might be unwanted “ghost atoms” and which might be actual atoms (in the above screenshot the Q-peaks of the easily visible six membered rings can be obviously assigned initially as carbon atoms). Not all atoms have to “pop up” in the first cycles. You will have to delete the “ghost peaks” and assign atom symbols and names to the remaining peaks. If you are not sure, rather delete some actual atoms than assigning questionable ones.

To delete a Q-peak move the cursor atop of it (into the crosshair) and hit K on the keyboard.

To assign a single Q-peak with an atom symbol and number move the cursor atop of it and make a right mouse click. Select [<edit>](#). A new window will pop up:

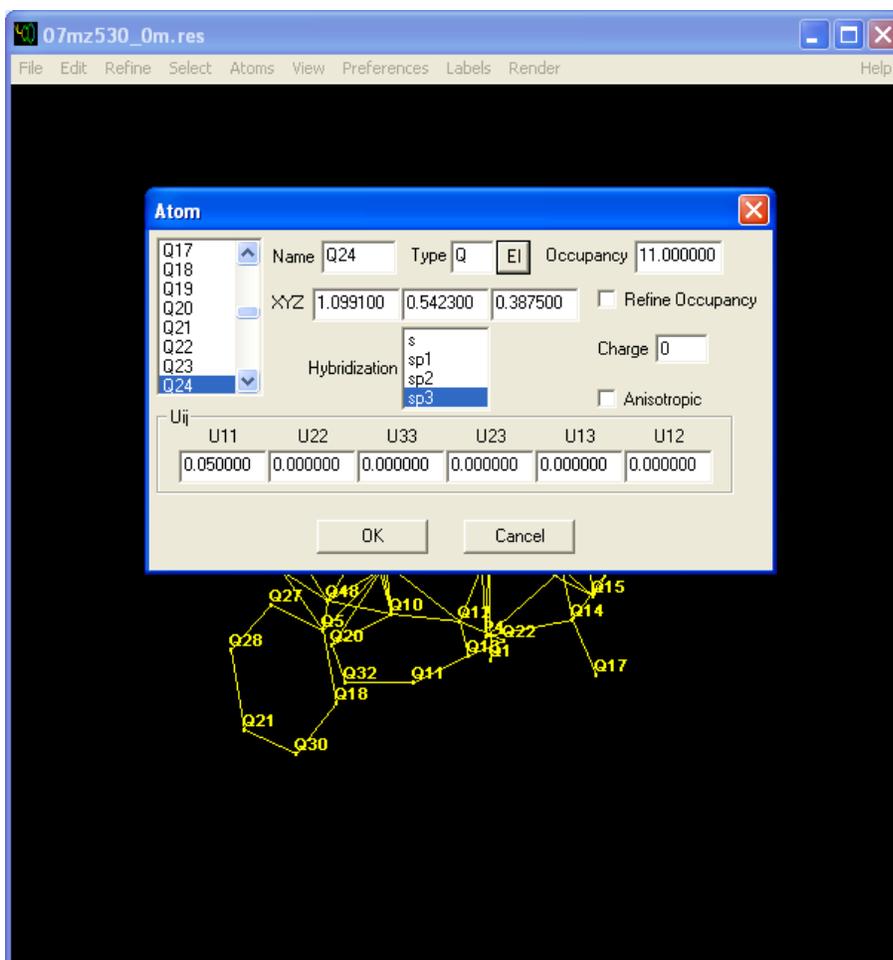


Figure 33, XSELE window with atom assignment window for individual atom open

Select a new atom type (use the [EI](#) symbol for a periodic table) and change the Name as needed.

- To assign a group of atoms of the same type select them by moving the cursor atop of each atom (into the crosshair) and hitting S on the keyboard in the order you want to name them. Then go to [Labels](#) and [Group Label](#).

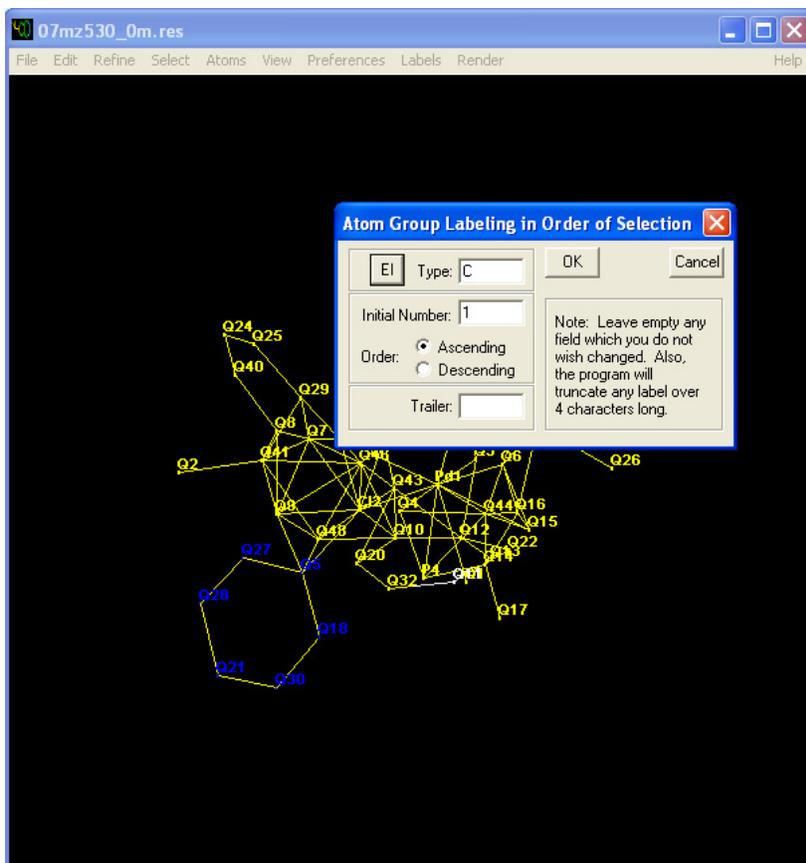


Figure 34, XSHL window with atom assignment window for multiple atoms open

Again select the atom type using the **EI** button, and the number for the first atom. Upon clicking **<OK>** all selected atoms will be named consecutively in the order they had been selected.

- Continue until all Q-peaks are either assigned and named or deleted. Use the electron count of the Q-peaks to guess which ones might be heavy atoms and use geometric features to identify fragments such as phenyl or cyclopentadienyl rings or functional groups.
- You can also open the modified ins file via **<Edit>**, then **<Edit current file>** to make manual changes directly in there.
- The structure may now look like this:

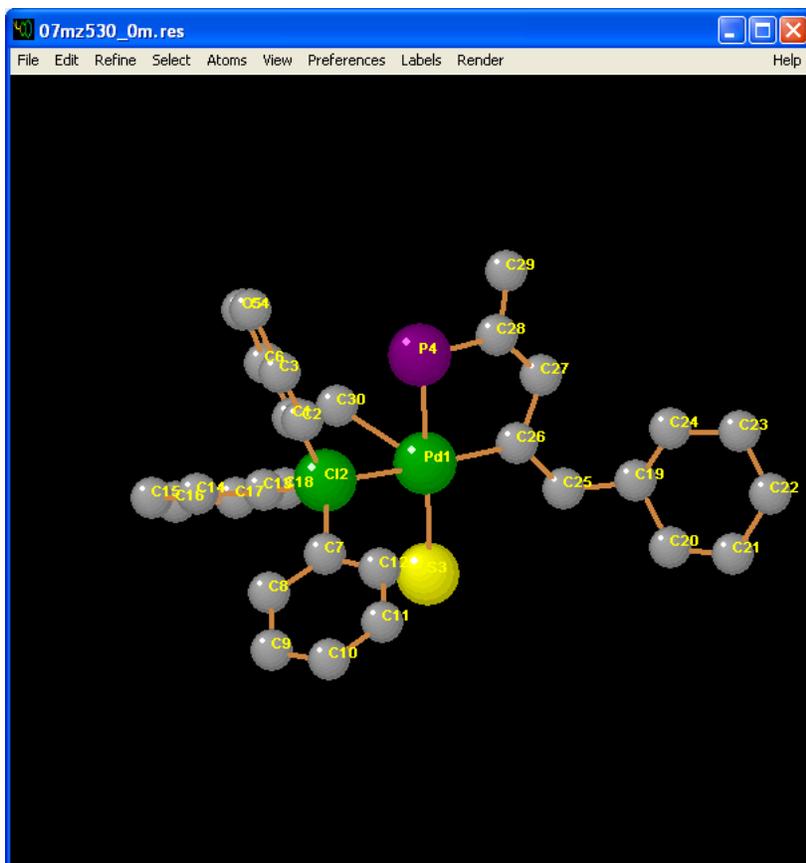


Figure 35, XSELE window with all atoms deleted or assigned (some possibly wrong)

If you cannot identify all fragments or the molecule seems to be incomplete it might be located on a center of symmetry, a mirror plane or a crystallographic axis. Make a right mouse click on the graphical interface and select <Grow> or <Pack> to see the whole molecule or the whole unit cell.

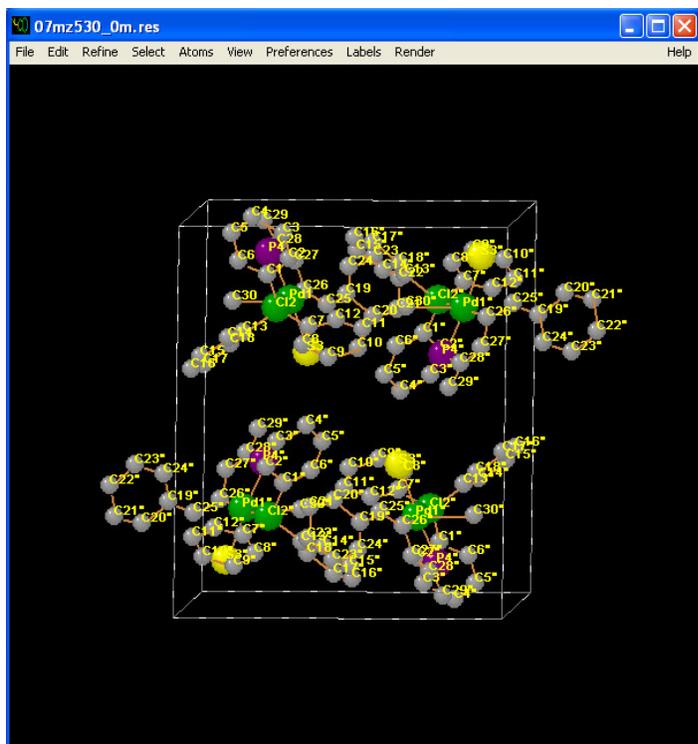


Figure 36, XSHL window in packing mode showing the whole unit cell content

- When all Q-peaks are assigned or deleted go to **<Refine>**. The refinement window provides a hook to the refinement program **<XL>**. Leave all parameters unchanged and start a refinement cycle by clicking **<OK>**.

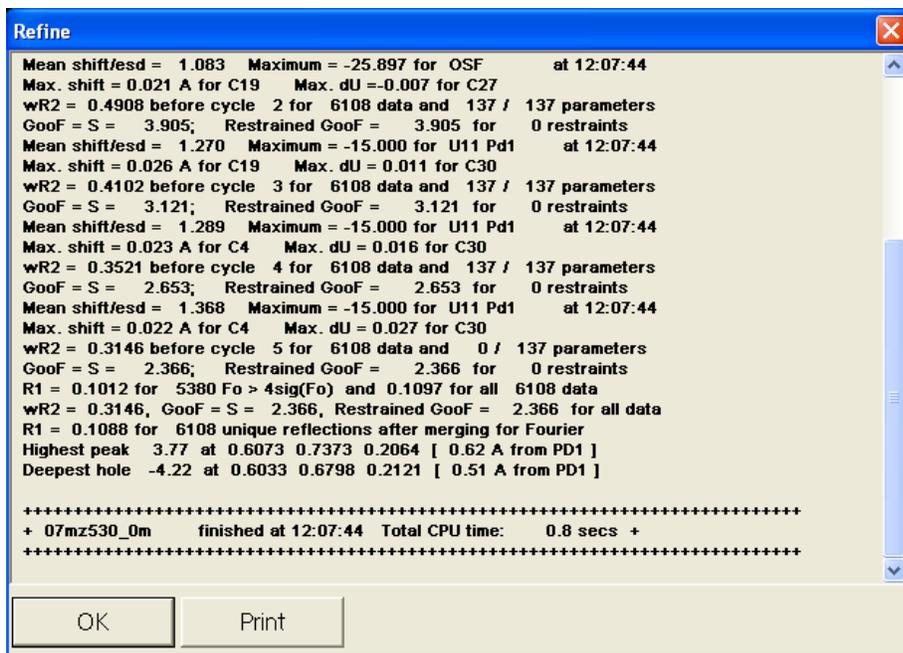


Figure 37, XL window as spawned from the XSHEL interface

The refinement program <XL> will run four consecutive cycles. Before clicking <OK> closely inspect the parameters obtained during all cycles. R1, Goof, Max. dU and Maximum should all decrease.

- After the refinement cycle the atom positions and thermal parameters of already defined atoms will be optimized and positions of still missing atoms will be suggested (new Q peaks). Correct atom assignments and assign new atoms as necessary. Probe the thermal parameters by opening the ins file via <Edit>, then <Edit current file>. The isotropic thermal parameter is the last value listed for each atom (see the Shelxtl manual or the Müller for a description of the values listed for each atom). Atoms with very large thermal parameters (when compared to the other atoms) may be wrongly assigned and are much lighter atoms or no atoms at all. Atoms with very small thermal parameters may be heavier atoms.

Correct changes should result in lower values for R1, wR2 and goof in the next set of refinement cycles.

- Continue with corrections, additions and refinement cycles until no changes are observed for R1, Goof, Max. dU and Maximum. Then check the <Anis> checkbox in the refinement window prior to the next refinement cycles. After the cycles make a right mouse click and select <Thermal Ellipsoid> to display the anisotropic displacement parameters for all non-hydrogen atoms.

The display will now (after deleting of all Q-peaks) look like this:

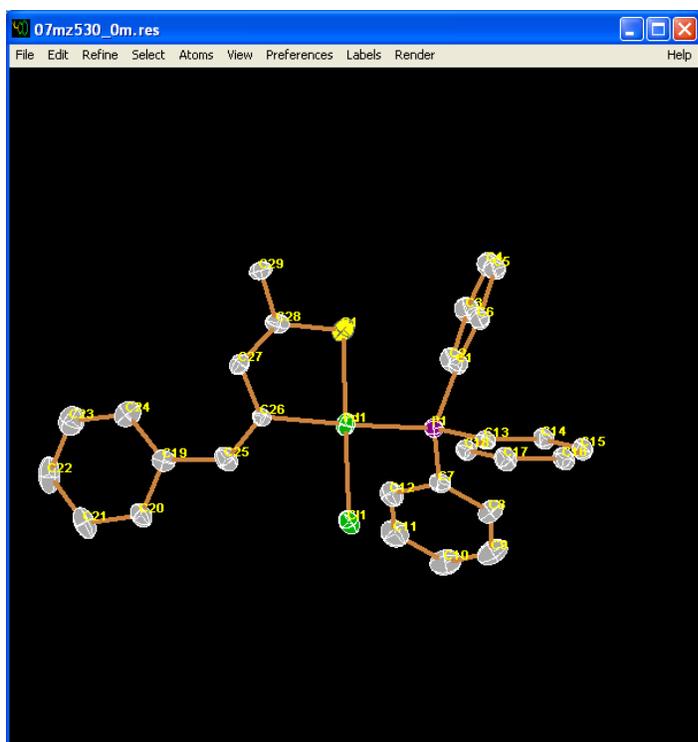


Figure 38, XSELE window with all anisotropic atoms (some atoms still wrongly assigned)

Make last corrections to atom types and give all atoms their final names:

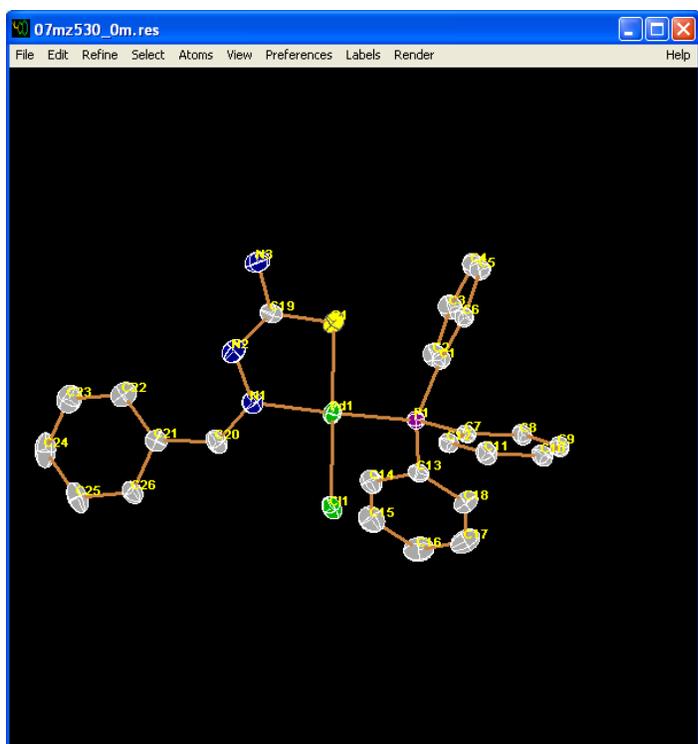


Figure 39, XSELE window with all anisotropic atoms (all heavy atoms correctly assigned)

- Sort all atoms using the `<Atoms>` and `<Sort>` sub-window.
- Add hydrogen atoms in calculated positions using `<Atoms>` and `<Hybridize All>`, then the `<Atoms>` and `<Calculate Hydrogens>`. Use manual commands in the ins file to add hydrogen atoms that are added wrongly using the automatic routine (see the Shelxtl manual or the Müller for details).

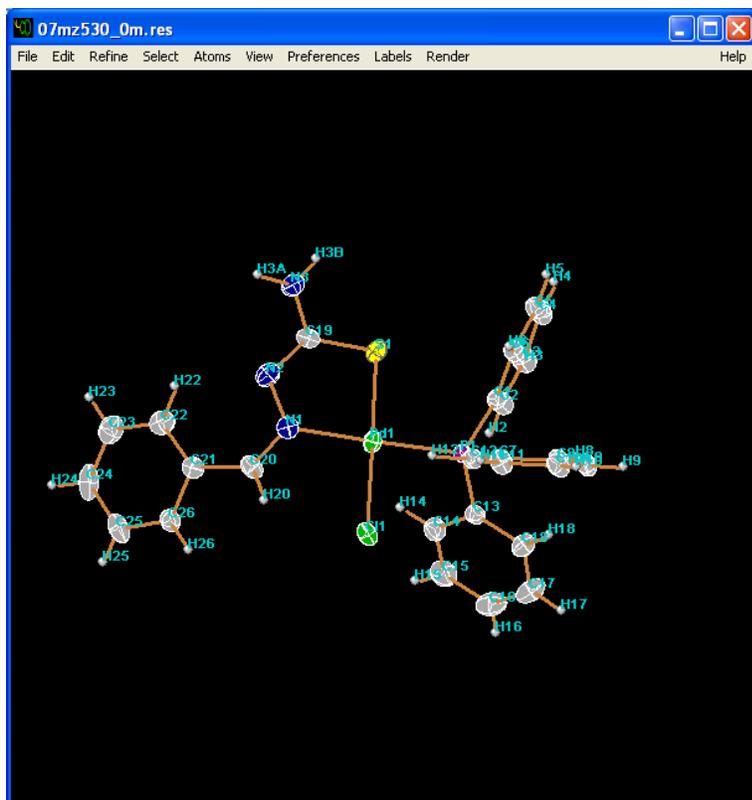


Figure 40, XSELE window after addition of hydrogen atoms

- Continue with corrections, additions and refinement cycles until no changes are observed for **R1** and **Goof**, and until **Max. dU** and **Maximum** are basically zero.
- Open the ins file via **<Edit>**, then **<Edit current file>** and make a range of final changes:
 Add a line **ACTA** (a refinement cycle will now create a cif file)
 Change **BOND** to **BOND \$H** (a refinement cycle will now add X-H bonds and H-X-H angles to the cif file)
 Add a line **CONF** (a refinement cycle will now add dihedral angles to the cif file)
 Add a line **HTAB** (possible hydrogen bonds will be listed in the listing file)
- Save the ins file and run another set of refinement cycles. Check for alarm messages in the XL window.
- If the message “**Merging of Friedel pairs is highly recommended**” appears add the command **<MERG 3>** to the ins file.

- If the message “Cell content from Unit instruction and atom list do not agree” appears open the listing file (via **<Edit>** and **<Edit .lst>** in the **<SHELXTL Program and Project Manager>**. Search for “**Unit-cell contents**” in the file and use the values in the second column to correct the **<UNIT>** instruction in the ins file.

- Also in the listing file search for “Hydrogen bonds”. If possible hydrogen bonds are listed add a command as follows to the ins file:

HTAB <Donor-Atom> <Acceptor Atom>_\${<Symmetry Operator Number>} and
EQIV \${<Symmetry Operator Number>} <Symmetry operator>

e.g.

HTAB N3 C11_ \$1

EQIV \$1 x-1/2, -y+3/2, z-1/2

A refinement cycle will now add hydrogen bonds to the cif file (see the Shelxtl manual or the Müller for details).

- Run refinement cycles until no changes are observed for **R1** and **Goof**, and until **Max. dU** and **Maximum** are basically zero.

Creating and Validating the CIF File (Crystallographic Information File)

- Open **<XCIF>** in the **<SHELXTL Program and Project Manager>**.

Click the enter key using the default values until you reach **<option N:>**. Type **Q** to quit the program.

- Open the Cif file via **<Edit>** and **<Edit .cif>**. You will have to make some manual changes:

_exptl_crystal_density_meas ?

_exptl_absorpt_correction_type 'multi-scan'

_exptl_absorpt_correction_T_min Add the Tmin value from the *.abs file here

_exptl_absorpt_correction_T_max Add the Tmax value from the *.abs file here

_exptl_absorpt_process_details 'Apex2 v2009.7-0 (Bruker, 2009)'

_diffrn_measurement_device_type 'Bruker AXS SMART APEX CCD diffractometer'

_diffrn_measurement_method '\w scans'

```

_diffrn_detector_area_resol_mean ?
_diffrn_standards_number      0
_diffrn_standards_interval_count .
_diffrn_standards_interval_time .
_diffrn_standards_decay_%     ?

_reflns_threshold_expression   I>2\s(I)

_computing_data_collection     'Apex2 v2009.7-0 (Bruker, 2009)'
_computing_cell_refinement     'Apex2 v2009.7-0'
_computing_data_reduction      'Apex2 v2009.7-0'
_computing_structure_solution
'SHELXTL 6.14 (Bruker, 2000-2003; Sheldrick, 2008)'
_computing_structure_refinement 'SHELXTL 6.14'
_computing_molecular_graphics  'SHELXTL 6.14'
_computing_publication_material 'SHELXTL 6.14'

```

If all H atoms were added in calculated positions:

```

_refine_ls_hydrogen_treatment  constr

```

(use "refall" if positions and thermal parameters were refined, "refxyz" if positions were refined but thermal parameters constrained, and "mixed" if combinations were used).

If a chiral space group without heavy atoms was used (Merg 3) add the following line:

```

_chemical_absolute_configuration syn

```

- Check your cif file using the checkcif web page provided by the International Union of Crystallography at <http://checkcif.iucr.org/> and make any corrections to either the ins or the cif file as necessary. All Level A Alerts should be corrected. If this is not possible, or if the Alert reflects an real property of the compound, a Validation Report has to be appended to the cif file (Copy the lines starting with "# start Validation Reply Form" at the end of the Checkcif report

that pertain to the Alerts you need to address to the end of the cif file; add explanations for the Alerts in the fields provided).

Creating Word Tables and Graphics from the CIF File

After finalizing the cif file you can create a table in word format from the cif file with the most important results using the web page <http://publCIF.iucr.org/services/tools/>

Graphics can be created with a multitude of different programs that can read cif format starting with Chem3D (of the Chemdraw suite of programs) to more specialized Crystallography oriented programs. A very versatile program is Mercury. A free version (for installation on your own computer) can be obtained from the Cambridge Crystallographic Data Centre at <http://www.ccdc.cam.ac.uk/products/mercury/>.

Appendix A: Changing the N₂ tanks

To set up the high pressure tank, connect the thin N₂ line (coming out the back of the SMART APEX machine, coated in black plastic) via a reducing valve to the **gas outlet** of a 200 psi nitrogen tank. Set the outgoing pressure at the reducing valve to zero, open the valve and adjust the outgoing pressure to about **70 psi**.

To set up the low pressure tank, connect the insulated N₂ line coming out of the white reservoir dewar to the **liquid outlet** of a liquid N₂ ambient pressure tank. Near the connector there is a small metal tube branching off the line containing a flow sensor. Make sure, this tube is **pointing upwards**. Open all valves and use the Kryoflex program to adjust the temperature.

Appendix B: Troubleshooting

B) If the shutter doesn't open / close

If the shutter is hanging, get help.

C) If the red alarm light is flashing

The lead glass doors are not closed. Check the doors. You might have to fasten the small screws at the door handles.

D) If the red alarm is lighted permanently

Most likely, the **X-ray generator power** is off. Check at the display located at the left side of the instrument panel of the machine. If the power is zero, or if an **error code 30** is displayed, clear the error and power the generator up again. If the power was at zero for more than **several hours**, the voltage has to be **raised in steps (see below)**. If you are not familiar with this, get help.

Procedure for clearing the error and powering up the generator:

Aside of the **X-ray generator power display** there are three buttons: **<Off>** (left), **<Heat>** (middle, small circle in big circle)) and **<On>** (right). For restarting the X-ray generator, press the **<Heat>** button and wait about 1 minute for the generator to heat up. Then press the green **<clear alarm>** button (located at the left upper side of the panel, well hidden), then press the **<On>** button to power the generator up again. (The alarm light should be off by now. If not, try the whole sequence again. If this does not help, get help.) When the alarm is off, go in the SAMRT program to **<goniom>** and **<generator>** and change the values to 50 kV and 30 mA.

Power up procedure after prolonged downtimes:

Downtime [days]	voltage and duration							Total
	20 kV	25 kV	30 kV	35 kV	40 kV	45 kV	50 kV	
0.5 to 3	30 s	30 s	30 s	30 s	30 s	1 min	2 min	4 min
3 to 30	30 s	30 s	2 min	2 min	5 min	5 min	10 min	25 min
> 30 or new generator	30 s	30 s	2 min	2 min	5 min	10 min	15 min	35 min

E) If the red alarm is lighted permanently and it is not the generator

If you are familiar with the **D8Tools program** (located on the server computer), look up the details of your current problem. If not, get help.

F) Restarting the personnel computer and the SMART APEX machine

If you are explicitly allowed to do so, and, if really nothing else helps, you may restart the client or the server computer. In really serious cases you may restart these machines as well the computer of the SMART APEX machine.

- To restart only client computer simply shut it down and restart it. You will need a password to log on again.
- To restart only the server computer, **first switch the detector off** (red switch at the upper left side of the control panel). Then restart the server computer. You will need a password to log on

again. After the boot up sequence is finished, switch first the detector on again. Wait for the green light at the detector. Restart the BIS.

- To restart the server and the X-ray diffractometer computer, follow the above sequence. Additionally, switch the diffractometer computer off (red button at the most upper right side of the control panel, below the emergency switch off). Then turn it on again (yellow button just below the off button). The generator will show an alarm when the boot up sequence is finished. You will have to clear the alarm and power up the generator again.