The following is a guide for collecting data and solving structures using the Purdue Bruker Quest single crystal diffractometer with a sealed tube molybdenum source. It is intended as a “walk-through” user guide geared especially towards novice users, but also tries to cover more advanced features of the software where they are important for the collection of normal “simple” small molecule structural data. A basic knowledge of the fundamentals of diffraction and crystallography is expected.

This manual is based on an instrument and software produced around the year 2017. Most of the manual’s content also applies to newer as well as older instrument makes. For the novice user, changes between different generation instruments are mostly limited to slightly different program layouts and color schemes. The general procedures described in this manual still apply.

For a more in-depth description of the features of a CCD or CMOS diffractometer, the reader should refer to the manuals and technique guides on specific topics by the manufacturer of the type of instrumentation they are using. The gold standard for a more in-depth guide towards the use of Shelxtl for the refinement of single crystal structures is 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, XM and XT), George Sheldrick’s refinement program Shelx2018, the graphical interface Shelxle by Hübschle, Dittrich and Sheldrick, and Platon by Anthony Spek.
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**Instrument Overview**

The Bruker Quest instrument with sealed tube molybdenum source consists of:

- The diffractometer including the X-ray enclosure, detector, X-ray tube, monochromator and optics, video microscope, fixed Chi goniometer, electronic controls, power supply and other miscellaneous pieces of equipment essential for the operation of the instrument.
- An Oxford Cryosystems 800 plus variable temperature unit (pump, controllers and liquid nitrogen tanks located outside the enclosure) and an Oxford Cryosystems AD51 dry air unit.
- Haskris chillers that cool and circulate cooling water for the power supply and the X-ray tube.
- The instrument computer: This computer runs the Measurement Server, the Bruker Instrument Service (BIS), Diffrac.Maintenance and Apex3.

![Figure 1, Inside of the Quest X-ray enclosure with X-ray tube, Detector, Variable Temp Unit and Goniometer. Between X-ray tube and detector are the Optics (Monochromator, Collimator), the Video Microscope (in back, partially hidden), and the Beam Stop. On the Goniometer is mounted the Goniometer Head holding the crystal specimen.](image)
Having a promising sample of crystals, it is best to start in the following order:

**Checking the Status and Thermostat**

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 to the sample.

The Oxford Cryosystems variable temperature unit can be operated as a stand-alone unit through
its own controllers. More often, it is controlled via Apex3 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 dry air supplied by
an Oxford Cryosystems AD51 dry air unit (located outside the lab in a facilities closet).

The Oxford Cryosystems variable temperature unit is able to achieve temperatures between 475 K
(+202.5°C) down to ca. 85 K (-188.5°C). The recommended temperature for “low temperature”
data collections is 100 K in winter, and 150 K in summer (due to higher humidity levels that can
cause icing around the sample mount).

- Make sure, the tank is properly connected and full enough for the planned experiment.
- The Oxford Cryostream N₂ tank (silver dewar behind the diffractometer) is automatically refilled
  from the larger low pressure liquid nitrogen tank (to the left of the dewar). Check that the Liquid
  Level Controller (the smaller of the two controllers, Fig. 2) is set to <AUTOFILL>. The fill level
  displayed on the controller should be between 20 and 80%. When it is below 20% (despite the
  controller set to AUTOFILL) the low pressure N₂ tank is empty and needs to be refilled.
Figure 2, The external controller (bottom), the circulation pump (center), and the liquid level controller (top) of the Oxford Cryosystems variable temperature unit.

- **Check that no data collection is running** (e.g., look at the BIS interface, check for “Frames left” or “Time left”). If a running data collection can be stopped, abort the running data collection (see below), then make changes to the VT unit.
- Check that the dry air unit providing the shield gas flow is switched on.
- Tap the touchscreen of the Cryostream Controller (larger of the two controllers) to check the status of the VT unit. **If it is running** (“Cryostream running” beside the symbol on the screen), check if the temperature is appropriate for your experiment. If you need a different temperature, or if the system is on standby (“Cryostream ready” beside the symbol on the screen), use the <Run Experiment> interface of Apex3 to set the desired temperature (see below, Starting the System).
- **If the unit is completely switched off** it needs to be started. Press the main single button on the front of the controller. Follow the on screen prompts to switch on the unit. Use the <Run Experiment> interface of Apex3 to set the desired temperature (see below, Starting the System).
Starting the System

- Start BIS (Bruker Instrumentation System) if it is not already running. This will also start the measurement server if it is not active already.

Figure 3, The BIS window

- Check if a data collection is still running (e.g., look at the BIS interface, check for “Frames left” or “Time left”). If a data collection is active but can be aborted, then stop this data collection first. Navigate to the active copy of Apex3 and click the STOP symbol in the Apex3 interface.

- Minimize any sessions of Apex3 that might be open.
  - Start a new session of Apex3. No login is required.
  - Connect to BIS (<Instrument>, then <Connect>).
- Via the <Sample> dropdown menu, either <Open> an existing sample (continuation of old data collection) or
- Start a <New Sample>
  - Fill in the project name. Avoid using special characters (including dashes and periods) or overly long project names.
- If necessary, ramp the temperature.
- Under <Collect> click on <Run Experiment>
- In the <Operation> column of the table, click on the top row and select <Thermostat>
Enter the target temperature (in K). Set the rate to 360 (K/hr). Click the <Execute> button. You can mount and center a crystal while the temperature is ramping. For unit cell measurements and data collections, wait for the temperature to have settled at the target value.

**Unloading the Previous Sample**

- Before using the video for crystal centering, make sure **only one copy of Apex3 is open** (trying to open the crystal centering tab from a second copy of Apex3 will crash the video feed).
- Under <Set Up>, go to <Center Crystal>
  - The video feed will automatically open showing you a life picture of the mounted crystal.
  - Click on <Center> in the lower right of the interface. The angles will drive to a position convenient for mounting a magnetic snap-on mount.
  - Press the <door open> button on the front face of the diffractometer enclosure (to the right of the front door, below the light button). Doors need to be closed to drive any mechanical parts other than the phi (spindle) axis of the goniometer.
  - Carefully dismount the magnetic snap-on mount from the previous experiment. Tilt it backwards until the magnet comes loose, then take it off.
Selecting and Mounting of Sample

- Crystals can be mounted on glass rods, inside glass capillaries, on Nylon loops, or using mesh mounts. The Purdue X-ray lab uses Mitegen micromesh mounts for most samples and data collections.
- Two types of micromesh mounts for small (<0.1 mm) and medium and large (0.1 – 0.5 mm) crystals are stocked in the lab (Fig 5). Crystals collected at low temperature are mounted on the micromesh with the help of a trace of mineral oil, Fomblin (fluorinated mineral oil) or polybutene oil (very viscous oil to protect extremely oxygen sensitive samples) 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 or Fomblin oil on a glass slide under a microscope. Place several representative crystals in the oil. Inspect the crystals and select a suitable candidate. Crystals on the Quest instrument with sealed tube molybdenum source should ideally be between 0.2 and 0.55 mm in any direction (size of the X-ray beam is 0.6 mm). The minimum size depends on the diffraction intensity of the crystal. For highly absorbing samples limit the size of the crystal (make sure the minimum transmission will be above 10-20%). 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 (the micromesh mount can be used to brush off loose pieces from the crystal).
• Scoop up a crystal with the micromesh mount and place it on the inside of the concave face of the mount in the center of the mesh. Remove as much excess oil 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. Dip the mesh carefully in a small amount of glue and pick up the crystal without touching the glass slide).

**Crystal Mounting and Centering**

• Make sure exactly one copy of Apex3 is running (trying to open the crystal centering tab from a second copy of Apex3 will crash the video feed).

• Carefully mount the magnetic snap on mount onto the goniometer.

• Check the position of the mount by eye (is it aligned with the collimator and the beam stop?)

• In Apex3, click *<Center Crystal>* in the *<Set Up>* submenue. The crystal centering live video feed will automatically open. Is the crystal visible on the video screen? If you cannot see the crystal, its position is further off than usual and needs to be adjusted by eye on the goniometer head before the live video feed can be used.
Figure 7, Crystal Centering Window and live video feed with mounted sample and green measuring line.

- There are three pins on the goniometer head: for up-down, right-left, and forward-backward. Use the bold end of the adjustment screw driver to turn the pins to position the crystal in the center of the cross hair of the video feed.

- When the crystal is centered in the cross hair, click the large `<Spin Phi 90>` button (in the `<Center Crystal>` window of Apex3) to spin the crystal around the phi 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. If the cross hair seems to be slightly off try to center the crystal so that it “rotates in place”.
  For oddly shaped crystals, try to center the mass center of the crystal. For long needles, try to align them along the axis of the mounting pin.

- Measure the dimensions of the crystal: Switch on the measuring tool (double sided arrow icon on top of `<Center Crystal>` window, Fig. 7). Click on one side of the crystal, hold the
left mouse key and drag to the other side. The length of the green line dragged is given in the bar below the video screen (“vector length”, value in µm). Write down the value and repeat for the other two directions using <Spin Phi 90>.

Crystal and Compound Description

- Under <Setup>, go to <Describe>
  - Fill in all values that apply. In the “compound” line, it is best to 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!).

![Figure 8, Crystal and compound description](image-url)
**Unit Cell Determination**

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

![Figure 9, Starting Window of the Unit Cell determination](image)

We encourage to use the manual procedure. To do so, click **<Collect Data>**, this will open the Unit Cell Data Collection Window.
Choose an exposure time (default is 10 seconds) and click `<Collect>.

2 × 12 frames will be collected which will be displayed in the frame window.

Make sure that your specimen diffracts to sufficiently high angle. Use the resolution arc tool to check the resolution limit. Only samples that show at least some diffraction above ca. 0.9 Å (with ten second exposures) are likely to meet the IUCr requirements for publication. For crystals diffracting too weakly, consider using the copper microsource instrument.

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, spots that will be used are green circled). To obtain a reasonable suggestion for the exposure time use the default value of 20.0. Click on `<Harvest>`. 
Figure 11, Unit Cell Harvesting Window

- Proceed to <Index>, use all default values and click on <Index>.
Check the unit cells 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, or the crystal is not single. If this is the case, export the data as a p4p file using <Sample>, then <Export> and use the program Cell Now to determine the unit cells (see the Purdue Twinning Manual for details).

- If one of the two unit cells looks reasonable, select it and click <Accept>. This will open the Refine Window. Use all defaults and click on <Refine> to run a least squares optimization of the unit cell against the diffraction spot position. Select more or less reflections as desired and repeat, then click <Accept>.
Figure 13, Unit Cell Refinement Window

- Proceed to <Bravais>. A list of possible Bravais lattices will be displayed with the software's choice highlighted in green.
The correct choice should have an FOM value significantly higher than the others. If several solutions have similarly high FOM values, the one 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 to avoid collecting incomplete data.

- Make your choice of Bravais lattice, then click <Accept>. This will open the second Refine Window.
- Use all defaults and click on <Refine> to run a least squares optimization of the unit cell against the diffraction spot position (constrained to the chosen Bravais lattice). Select more or less reflections as desired and repeat, then click <Accept>.
- Please note the resolution prediction at the bottom of the window
- This finishes the Unit Cell Determination.
Data Collection

• Click `<Calculate Strategy>` under `<Collect>`.
  - Under Step 1., change the resolution to \( d = 0.75 \) or lower, as required. (The worst acceptable value, by IUCr standard, is 0.83 Å). Click `<Apply>` if you made any changes.

Figure 15, Strategy Starting Window

- Under Step 2., click `<Determine Strategy>`. In the pop-up window, it is usually save to use the default values. Click `<OK>`,
Under Step 3., click <Select Scan Parameters>. A popup window will emerge. Click the <reset> buttons for both “Frame angle” as well as “Frame time” to obtain (usually reasonable) suggested values.
The values can usually be accepted as they are (if you feel suggested exposure times are too long or too short, makes changes as you see fit). However, frame times have to be limited to under 90 seconds (the Photon100 detector of the instrument cannot handle longer exposure times). If the suggested exposure time is > 90 seconds, reduce the frame width and frame time values by equal factors until the frame time is equal or smaller than 90 seconds.

If the suggested exposure time is unusually long (> 3 minutes), consider collecting data using copper radiation on the other Quest instrument.

- If you feel the suggested values to be completely out of whack, make your own choice!!
- For most samples, “Theta Dependency” can be skipped when using Mo Kα radiation.
- Optional step to save data collection time: If you expect the structure to be centrosymmetric (most structures are), but would like the option to collect complete non-centrosymmetric data without needing to “starting over”, you can re-sort the frames so that complete centrosymmetric data are collected first, followed by frames that fill in Friedel pairs. Change <Symmetry> to the centrosymmetric case and click <Apply>, but do not click <Determine Strategy> again. Click the <Edit> button, then click the <SORT> button.

- Under <Collect> click on <Run Experiment>
  - In the <Operation> column of the table, click on the top row and select <Fast Scan> (Fig 18). The dynamic range of the Photon100 detector is limited. More intense diffraction spots can get saturated when using long exposure times suitable for good signal to noise for weak spots are used. The saturated spots are removed during data integration and have to be replaced with data from a fast scan.
Figure 18, Experiment setup window during addition of Fast Scan

- Click `<Append Strategy>` to add the runs from the Strategy calculation.
- Edit the exposure time for the fast scan. Use ten times less exposure time / degree for the Fast Scan than for the other runs.
Figure 19. Experiment setup window with Fast Scan and a typical strategy as determined by **Data Collection Strategy**

- Click **Execute** to start the data collection.

- The instrument will now collect your dataset. Open **Show Status** from the Instrument drop down menu. This will open the **Instrument Status** window where you can check variables and completion time. You can also check many of these items via the BIS interface.
Figure 20, Data Collection Window with active data collection and Instrument Status Window

- Users are encouraged to check the progress of their data 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, that the structure is indeed of interest, that the unit cell is 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 an efficient 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!
Data Integration

When the data collection is complete (or when enough data are collected for an initial structure solution and refinement attempt) open, <Reduce Data> 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).

![Integration Start Window with Select Runs Window open](image)

Figure 21, Integration Start Window with <Select Runs> Window open

- 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 around or above 80% and be mostly even. Values of 40% or lower are statistically meaningless. Pixel Errors should be on average between $\pm 0.2$. Average $I/\sigma$ intensities should be above at least 3 (good datasets have values larger than 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 the Purdue Twinning Manual.

- Open the “work” folder of your experiment and open the file ending in _0m._ls. Inspect the table at the beginning of this file. Look for the values for “Spots exceeding frame queue size” and “Spots left in Write-Behind Cache”. If these are unusually large (a substantial fraction the total data), then repeat the integration with different settings.

Click <Integration Options> and change the values for “Profile XYX Half-Widths” from 4 x 4 x 4 to 9 x 9 x 9. Repeat the integration. If you used very narrow frame widths you also need to increase the number for “Active Image Queue Half-Widths” (default is 7, which will be too low when using frame width smaller than ca. 0.5). Check the file ending in _0m._ls again to ensure that the number of reflections rejected due to “Spots
Scaling and Absorption Correction

- Under <Reduce Data> Click on <Scale>

![Figure 23, Absorption Correction and Error Model (<Scale>) Starting Window](image)

- Unless only one raw file exists the merged batch raw file from the integration is loaded automatically.
  
  *If you did initially integrate a single run you will have to manually load the merged raw file. Click the blue browse symbol, click on the first raw file (ending in _0m.raw), then click <OK>.*
  
  *If you did not integrate all runs at once you need to load individual raw files. Click the blue browse symbol, click on the first raw file you want to load, then click <OK>. Uncheck raw files you would like to not use.*

- Check the Laue Group and Point Group.
- Keep Multi-scan as the absorption correction method. It is sufficient for > 99% of all samples. For heavy absorbers, adjust the number of Mu*r (r is the average radius of the crystal in mm, mu is the absorption coefficient in mm\(^{-1}\); its value can be looked up in the cif after assignment of all atoms. If the value of mu is higher than initially expected, repeat scaling and absorption correction with a more suitable Mu*r value prior to writing the final cif).

- Proceed by clicking <Start>.

![Figure 24, Absorption Correction window (second Scale Window) before refinement](image)

- The refinement window will open (Fig. 24). An average redundancy of around 3.0 is usually sufficient for weak to medium absorbing samples. For oddly shaped and heavily absorbing samples a higher redundancy is recommended.

- Click <Refine>, wait for the refinement to finish and inspect the results. The parameter refinement curve should show a smooth and steady decline (as in Fig. 25) and the final R value be reasonably low. Large up and down fluctuations combined with higher than expected R values could be a sign that scaling and absorption correction effects are highly correlated. This will seldom be a problem with a large beam and penetrating little absorbed MoK\(\alpha\) radiation. In
the rare case when the refinement results appear less than ideal, try dampening scaling by selecting a higher value for “Restraint ESD for Scale Factors” and repeat the parameter refinement (click <Refine> once more). Default is 0.005, the maximum allowed value is 0.02.

Figure 2, Absorption Correction window (second Scale Window) after refinement

- Once satisfied, click <Next>. The Error Model page will open.
- Check the R value of the Fast Scan. If it is substantially larger than the R values of the other scans, check the “Fast Scan” checkbox and click <Repeat Parameter Refinement>.
- Check the number of rejected reflections. If this number is extremely high, in conjunction with higher than expected data R values, then there is a possibility that the selected Laue group is too high in symmetry (“metric pseudosymmetry”). Revise your unit cell assignment and reintegrate the data.
- The number of rejected reflections can be reduced (assuming that the Laue group is correct!) by increasing the value for “<|I-I>|/su ratio for rejection” (default is 4.0). If you do so click <Error Model> to update the outlier rejection.
Figure 3, Error Model Window (third Scale Window)

- Click <Finish>, then <Exit> to finalize scaling and absorption correction.
- You have now finished the data collection and absorption correction. To allow other users to use the instrument you should move your data to a different computer at this point.

Transferring the Data

- Move the data via e-mail, a USB flash drive or online to your personal computer.

- For the refinement, you will need the *.hkl and the *.p4p files (located in the work folder of your project)
  
e.g.  04mz02a_0m.hkl
       04mz02a_0m.p4p

- For publication purposes you will also need the *.abs and the *._ls files:
e.g. 04mz02a.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

Setting up Data for XPREP

• 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 4, 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 high quality data).

Figure 5, 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. monoclinic).

![Figure 6, XPREP Window, initial lattice type selection](image)

- Select [S] to determine or input space group.
- Select [S] again to determine space group.
- Select the suggested Laue group (e.g. [M] for monoclinic)
- Select the suggested lattice centering (e.g. [P] for primitive, [C] for C-centered, etc)

Figure 30, XPREP Window, new lattice centering selection

- Select the suggested option for the space group e.g. P2(1)/c or C222(1) (If several solutions are offered, take the one with the lowest CFOM value. If that does not work out later on, try the next best in the list).
**Figure 31, 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 is not recommended to do), but only displays the data and statistics as if they would have been merged.
The intensity statistics will be displayed.

Have a look at the Completeness, Redundancy, Rmerge and Rsigma (Rmerge is the same as Rint in Shelxl and Sadabs). The completeness should be ideally close to 100 % down to a resolution of \( d = 0.75 \) Å. Rsigma and Rmerge should be ideally below 10 % down to \( d = 0.75 \) Å. If they become very large at higher resolution (lower \( d \) values), it might be appropriate to cut the data at an appropriate resolution during refinement.

Hit the Enter key

- Select [E] to exit to the main menu, then [C] to define the unit cell contents.
- If no unit cell contents are given type the most likely molecular formula (element symbols and numbers only).
- If the given formula seems wrong, select [F] for new formula, and type the most likely formula.

- 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 for 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 overwriting and losing the original data (change it back to your old filename after closing XPREP, or start a new project with the new hkl filename).
- Select [Y] at the prompt to generate an .ins file.

Figure 34, 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:

![Typical initial INS file](image)

Figure 7, 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. 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. If needed, 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>.

**Solving Structures using XS and XM**

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 programs XS or XM will provide you with this initial guess (other programs and methods can be used, such as DirDiff, charge flipping, etc). In the program XS 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. XM is utilizing direct methods originally written for macromolecular data, but can also be used for small molecule data. It is more likely to work where XS/TREF fails, but requires more computing resources.

- To use XS, select <XS> on the toolbar and the computer begins 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 (combined figure of merit) and the initial atom positions are written to the *.res file. The res file cannot be opened while XS is running.
- For good quality data the correct solution is normally “falling out” when continuing with Shelxle, Olex or Crystals graphical refinement interfaces.

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 even higher) to run more iterations, or, for compounds with at least one atom heavier than sodium, use PATT instead of TREF. Select <file> and then <save> and run XS again.

If XS fails using both PATT and TREF methods, try using <XM>. In the *.ins file, replace the TREF line by the following four lines:

FIND 58
PLOP 77
MIND 1.0 -0.1
NTRY 1000
Save the ins file and run Click <XM> in the in the SHELXTL menu. The software will run 1000 tries (or the number specified in the NTRY line) and selects the solution with the highest correlation coefficient, CC. The initial atom positions are written to the res file. This will take substantially longer than XS (a fast computer may be required), but solutions can be analyzed while XM is still running. If a valid solution is found, XM can be aborted.

XM does not write all required lines to the *.res file. Before running the first refinement cycle, add the following lines:

FMAP 2
L.S. 4
PLAN 20

Solving Structures using ShelXT

In the rare case that XS (TREF or PATT) and XM both fail, try using XT (ShelXT). ShelXT uses a combination of Patterson and Direct methods and will try a number of space groups compatible with the metric symmetry. The output name of the res file will be appended by a, b, c etc (depending on the number of solutions created) and a new hkl file will be written. You can use XT from within Apex3, or via command line. ShelXT as an extensive array of options, but these are rarely needed and simple execution via “XT.exe filename” does usually work (filename = name of the .ins file, type without file extension). The output res and hkl files can be used as they are (but check if the input unit cell has been changed in case you are still processing data and plan to update the hkl file).

ShelXT can also be added as an external program to Shelxle via its “Extra” dropdown menu.

Structure Refinement

Open the result of the structure solution in a graphical interface such as Shexle, Olex or Crystals. See separate manuals for details of the refinement procedure, and for writing and validating the crystallographic information file.