The following is a first short user guide for collecting data and solving structures. Having a promising sample of crystals it is best to start in the following order:

**Part 1: 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 and to minimize radiation damage of the sample.

With the Smart Apex machine, the cooling unit is controlled by the program CRYOFLEX. 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 controlled by the cryostat; the nitrogen pressure has to be adjusted manually.

- Make sure, the tanks are properly connected and full enough for the planned experiment. See [Appendix D: changing the N\textsubscript{2} tanks](#) for changing tanks.

- Open the `<CRYOFLEX>` program, wait some time until ready. Make sure, the valve of the high pressure nitrogen tank is open and nitrogen is flowing. The pressure should be app. 70 psi.
At this point best start a manual refill of the small white reservoir tank with low pressure nitrogen (select `<force refill to start>`). This may 10 - 15 minutes. When done, check `<automatic refill>`. Select the desired temperature (°C or K) and click on `<Start KRYO-FLEX>`.

Generally, the temperature will equilibrate at the desired value after about 0.5 h, but in some cases it may actually take several hours to finally cool down (e.g. when the cooling unit was off for a prolonged period).

For low temperatures around 100 K, Cryoflex is quite accurate (± 0.1 K). For higher temperatures (around 200 K) variations over time are more pronounced. The lowest possible temperature is 90 K.

The CRYOFLEX window can be minimized using the display in the lower taskbar of the computer screen.
• While cooling down, you can already go ahead with mounting and centering of the crystal, taking of the rotation frame and measuring of the dark current. Wait until the temperature has equilibrated before starting the unit cell measurement.

**Part 2: Data Collection**

**2a) Starting SMART**

• If the SMART program is not already open, then:

Open the SMART program: previous project: <Y>
The computer will ask several questions that can be answered <OK> or <YES> until you reach the type of measurement. Select <small molecule>.

• Then (or if SMART is already open)
Go to <CRYSTAL> on the toolbar and select <New Project> (If you want to modify an already open project, select <Edit Project>. Otherwise just proceed)

Fill in the following:
- Name: e.g. 04mz02a
For uniformity use the year, your initials and then a sample number (Use seven letters or less. SAINT, the integration program, will add one additional letter and will cut everything larger that eight letters off.)
- Chemical Formula (if known)
  e.g. C11H8Br2N2OZn
- Temp: type the temperature [°C]
  e.g. -173.5
- Working Directory: C:\frames\subfolder\filename\work
  e.g. C:\frames\zeller\04mz02a\work
- Data Directory: C:\frames\subfolder\filename
  e.g. C:\frames\zeller\04mz02a

Click on <OK>
Figure 2, New Project

The computer will ask several questions that can be answered <OK> or <YES> until you reach the type of measurement. Select <small molecule>.

2b Mounting and Centering

• Mounting
Mount a crystal, being not larger than 0.6 mm in any direction, on the tip of a glass fiber, a cryo loop, etc. Mount the glass fiber to the goniometer head. Lift the outlet of the cooling device using the lever at the side. Carefully screw the goniometer head at its place in the middle of the diffractometer and lower the lever again. Switch the lamp on and point it on the crystal. Using the small special wrench supplied adjust the metallic pins so that the crystal is centered roughly in the middle of the beam.

• Centering
Go to <CRYSTAL> on the toolbar and select <Evaluate>. Open the program Video, select <New Image> and click on the green arrow.
Take the external control pad, and press either <A> or <B> for a 180° or 90° rotation. The gonoimeter head will move from its former position (e.g. zero) to a position best for centering of
the crystal. (If the goniometer head does not start moving, hit 2THETA once and try again. If the goniometer head still does not move, get help).

Center the crystal in the crosshair. For this you have three metallic pins. That one being nearest to the top of the goniometer head controls **Up and Down**. That one pointing towards you controls **Right and Left**. That one at the side **Forward and Backward (Focus)**. Upon rotating by 90°, **Right and Left** and **Forward and Backward** are changing positions (**Right and Left always** points towards you). Use only **Up and Down** and **Right and Left** for centering the crystal.

When the crystal is in the crosshair, press again <A> or <B> on the external control pad. The crystal will move out of the crosshair. Adjust the position again using the **Up and Down** and **Right and Left** pins and repeat the rotation-centering sequence until the crystal stays in the crosshair when rotating. Check at the end once by pressing <C> on the external control pad.

![Figure 3, Window of the Video Program](image-url)
Note the dimensions of the crystal (necessary for any publications and some types of absorption corrections): Click with the mouse at any corner of the crystal and drag it along one of the edges to another corner. The distance in micrometers is displayed at the bottom of the window (most right number). You will have to rotate the crystal to get accurate dimensions. Use the 2Theta, Omega, Phi and Fast, Slow and Up, Down buttons. Do not drive the goniometerhead into the detector!! Get a second person to assist you with this task. (If you do drive the goniometerhead to far, get help. It will take a while to fix this; you will most likely be doing your measurement not this day)

- Close the video program, switch the lights off and close the lead glass doors

- In SMART, hit <ESC> twice to leave the <Evaluate> mode, go to <CRYSTAL> on the toolbar and select <Edit Project>

Fill in the values for Crystal Color, Crystal Morphology (plate, needle, etc) and the three Dimensions in [mm].

![Options for Crystal > Edit Project](image)

Figure 4, Edit Project
- Go to <CRystal> on the toolbar and select <Evaluate>, type <U> and check that the following parameters are selected:
  - temp = -42 or -43
  - Service mode = off
  - Lead glass door = closed
  - X-Ray generator = Ready, 50 kV and 40 mA
  - everything else = Ready
Then hit <esc> three times (hitting only twice will start a 1 minute default rotation photo)

Figure 5, Evaluate/Status Mode

In case the X-Ray generator voltage and amperage are wrong, go to <Goniom>, then <Generator> and correct it. If the generator was off for longer than a few hours, the voltage has to be ramped up in steps (see Appendix A: Troubleshooting).
2c) Rotation Frame, Dark Current, etc

• Rotation Frame

Go to <ACQUIRE> on the toolbar and select <Rotation>. The value should be 60 seconds for average size crystals or up to 5 minutes for very small or weakly diffracting crystals.

Click on <OK>

![Figure 6, Rotation Frame Settings](image)

At this point check the photo. If the photo is good (i.e. no powder rings and sufficient intensity up to the outer parts of the frame) continue with the next steps. If necessary you can adjust the contrast using <ANALYZE> on the toolbar and selecting <Contrast>. Adjust the contrast using the sliding bar to the right hand side of the screen.
Figure 7. Rotation Frame

- Dark Current and Sample-Detector Distance
Depending on the size of the crystal and its diffraction power (rotation frame), you want to choose frame times between 5 and 30 seconds.
- Go to <DETECTOR> on the toolbar and select <Dark Current>. Set the time per frame between 5 and 30 seconds (default 10).
Name: xxxxxxxx._DK
e.g. 04mz02a10._DK (the ten signifies the dark current time setting)
You will be asked: Detector readout speed = 400 kHz, is this correct? Click <YES>
16 frames without X-ray exposure will be measured and added
- When done go to <EDIT> on the toolbar and select <Configuration>. Make sure that the
  sample-detector distance = 5.011 [cm] (this may change over time!)

**3d) Unit Cell Measurement**
- Go to <CRYSTAL> on the toolbar and select <Unit Cell> and check the settings:
  - 5.0, 251.6, 251.6 (5.0 is the rough sample detector distance, the two other numbers are the
    XBeam and YBeam Center of the 512 × 512 detector, which may vary slightly over time)
  If correct, click <YES>
Then check the frames and seconds/frame settings:
- frames = 20
Select a higher number, if you expect only few reflections (high symmetry, small unit cell, etc)
- seconds/frame = time for dark current (10 sec default)
Click on <OK>.
Figure 9, Unit Cell Measurement Settings

The instrument will now automatically run the unit cell measurement (3 directions × 20 frames). The approximate time needed will be displayed in the right lower corner of the SMART window.
When finished, SMART will automatically determine the unit cell (if possible). The program selects automatically all centered reflections, which are not bifurcated, too large/small etc. Then, it attempts to find a unit cell fitting for most of these reflections, sorts out non-fitting reflections, performs a least squares calculation and determines the most likely Bravais lattice. At this time, you should be present to ensure that the solution found by the program is actually a useful one.

The automatic unit cell determination can be repeated using `<CRYSTAL>` and then `<Redtn Cell>`. The automatic routine will look like the following:
Figure 11, Unit Cell Determination (using <Redt Cell>), Step 1: Settings

Figure 12, Unit Cell Determination, Step 2: Selection of Usable Reflections
Figure 13, Unit Cell Determination, Step 3: Autoindexing Output

Figure 14, Unit Cell Determination, Step 4: Re-indexing Output
Figure 15, Unit Cell Determination, Step 5: First Least Squares Output

Figure 16, Unit Cell Determination, Step 6: Bravais Lattice Determination

At this point SMART usually chooses the right Bravais lattice (indicated by >>>>). You can choose a different solution (#Sig should be small for a reasonable solution). Note the # of the solution you are going to use.
Figure 17, Unit Cell Determination, Step 7: Bravais Lattice Selection

At this point, type your choice for the solution # to use (if necessary)

Figure 18, Unit Cell Determination, Step 8: Final Least Squares Output

The final solution of the Unit Cell Determination will be stored as Matrix0.p4p. The unit cell information is automatically copied into all other (future) p4p files.

If the automatic unit cell determination fails, the unit cell determination can be performed step by step using <CRYSTAL> and then <Threshold>, <Index>, <Bravais>, <LS>. In case you think you might have an intergrown or twinned crystal, try the programs GEMINI (see Appendix B: Using Gemini) or RLATT. In case you don’t have enough reflections after the unit cell
measurement, you can perform the unit cell determination with more frames after the data collection.

3e) Data Collection

- Go to <ACQUIRE> on the toolbar and select <Edit-Hemi>. Go to the last column and check that the set time = dark current setting used before. You can change the settings of Hemisphere (See Appendix C: Changing the Hemisphere Settings).

![Figure 19, Hemisphere Settings](image)

Now select <ACQUIRE>, <Hemisphere> and start the collection of data.

![Figure 20, Hemisphere Run](image)
The approximate time needed will again be displayed in the right lower corner of the SMART window.

If the measurement is interrupted (power outage, shutter does not open, detector initialization failed, etc) you can resume the data collection by going to <ACQUIRE> on the toolbar and selecting <Resume>. (see Appendix A: Troubleshooting for details)

**Part 4: Preparing Data using the SaintPlus Program**

When the measurement is successfully finished, the machine will display the message <no more runs in edit runs array>. At this point, you can redetermine the unit cell using the frames of the actual measurement (e.g. if the original unit cell measurement was not very accurate or reliable)
4a) Integration

In order to determine the structure, not only the position of the peaks (known from the unit cell parameters), but also their intensity has to be determined. For the Smart Apex instrument, this is done by the program SAINT PLUS.

- Double click on the SaintPlus icon. Go to <PROJECT> on the toolbar and select <New>.

![Figure 22, Opening a New Saint Project](image)

Find the appropriate matrix file (in the data directory, it should end in .p4p)
e.g. 04mz02a.p4p
Select the file and type in the project name using up to 7 digits (when the files are merged later on an "m" will automatically be added giving the 8 digit maximum for the file names) Select <Open>

Go to the <SAINT> on the toolbar and select <Initialize>
If you are prompted for anything, use the values from the saint.ini files
Go to the <SAINT> on the toolbar and select <Execute>

- A window with integration parameters will pop up:

![Figure 23, Basic Saint Parameters](image)

- Check that you have the correct Laue class and lattice centering.
- The default for d-spacing should be 0.750000 (A d-spacing of 0.75 will give a maximum 2-Theta angle just above 28°. Lowering the value will include reflections at higher angles. With
the usual Hemisphere settings reflections down to d = 0.6 to 0.7 have been measured, 100 %
completeness ends at ca. d = 0.7)
- Max. wait for frame file = 0.000
- Get rid of all but the first line in matrix.p4p filename. If the cell parameters (a,b,c,α,β,γ) are not
correct, find the correct matrix.p4p filename using <…>, then click on <CELL>.

• Go to the top right of the screen (More Options) and select <Integrate>. A new window will
pop up:

![Integrate Window](image)

**Figure 24, Saint Integrate Window**

- If no values for reflection size are given, type in the default values 0.6(x); 0.6(y); 0.4(z).
- A check mark should be beside "narrow frame", "enable box size", and "decay" (if the sample
might have decomposed partly over the collection period)
- Under periodic o. m. updating a check should be beside "enable periodic updating", "constrain
Laue class", and "crystal translation" with frequency = 100
• Go to "advanced integrate" (bottom left button). Again, a new window will pop up:

![Advanced Integrate Window](image)

**Figure 25, Saint Advanced Integrate Window**

- Check the following parameters:
  
  Model Profiles: \(1/\sigma = 5.0000\)
  
  Fraction = 0.05000
  
  \(1/\sigma\) threshold = 4.0000
  
  Resolution lower limit = 9999.0000
  
  Active frame 1/2 width = 7
  
  Corrections to intensity esd's 0.00 and 1.00

• Click the "Integrate + Sort + Global" button and the integration will start. This will take several minutes.
The program first performs for each of the subsets of frames a box size and unit cell optimization using only the first frames. When finished with this step, all the other frames are processed and the reflections are integrated.

While running, have a look that the cross sections of the reflections displayed are round or ellipsoidal and that there is no intensity at the box edges (after box size optimization). $\#\text{sig}$ (number of reflections with intensities over sigma) should be high and $\%<2\text{sig}$ (percentage of reflections with intensities below 2 sigma) should be low (~ < 40%). Residual intensity should be around or over 90% for not twinned or multiple crystals.
The final GOF value should be below 5. If this is not the case, either some parameters are edited wrong, the selected unit cell is wrong, or the data quality may be poor.

- At the prompt hit <enter> to continue. Close all windows up to SaintPlus

**4b) SADABS**

SADABS will create the first hkl file and will automatically apply some absorption correction. SADABS uses a semi-empirical method based on multiple scanned equivalent reflections (“multi scans”). Absorption correction for thin plates should be applied separately.

- Select <SADABS> on the toolbar and hit <return> for the maximum # of reflections allowed being 100,000. (If you will have more than 100,000 reflections (you can check that in the final output listing of SAINT), give a high enough number)
Figure 28, SADABS

- Enter the name of the sad.abs filename: e.g. 04mz02am.abs, <Enter>
  (Saint has added an "m" to the filename to indicate, that all the data had been merged in one raw file)
- Enter the Laue group number: e.g. 3 (type this)
- For the Friedel Pairs, select [Y]. <enter>
  (type [N] only, if you already know, that your space group will be chiral (e.g. chiral non racemic molecule) and if you know, which space group that will be)
- Enter the filename for the raw file: e.g. 04mz02am.raw (must end in m.raw), <enter>, type < / >, <enter>

- Take the default values that are suggested until you are prompted for the number of refinement cycles. For a good crystal use approx. 20 cycles, for a poor crystal use approx. 100 cycles.

```
Enter filename (/ if no more) [ ]: 04mz02am.raw
Enter filename (/ if no more) [ ]: /

Mean and maximum errors in direction cosine check function = 0.000 0.002
The mean error should not exceed 0.005, and is usually caused by matrix changes during data processing.

Maximum 2-theta = 56.65 deg. Approximate wavelength = 0.71070 Angstroms

PART 1 - Refinement of parameters to model systematic errors

Thresholds should now be specified for excluding reflections from the parameter refinement; these reflections may still be corrected and included in the final output .hk1 file

13559 Reflections of which 3579 unique; 22.60 data per frame

Redundancy:  1  2  3  4  5  6  7  8  9+
Number of groups: 61  501  798  1249  714  256  0  0  0
Mean(I/sigma): -Inf  0  1  2  3  5  10  15  20  +Inf
Number of groups: 601  289  222  177  151  639  500  298  702

Enter mean(I/sigma) threshold (must be positive) [3]:
Highest resolution for parameter refinement [0.1]:
Factor g for initial weighting scheme w = 1/(sigma^2(I)+(g<I>)^2), where sigma(I) is estimated by SAINT and <I> is mean intensity [0.02]:
The following restraint esd could be increased for strong absorbers.
Restraint esd for equal consecutive scale factors [0.005]:
Suitable spherical harmonic orders are 4,1 for weak absorption and 8,5 for strong. Highest even order for spherical harmonics (0,2,4,6 or 8) [6]:
Highest odd order for spherical harmonics (0,1,3,5 or 7) [3]:
Number of refinement cycles [15]: 40.
```

Figure 29, SADABS

- After the iteration select [A] to accept the value
  - For the next three lines, take the default values
- After parameter refinement select [A] for accept
- Accept the suggested error model. If no error model is suggested, your data quality might be poor, but continue.

Figure 30, SADABS
- When prompted for “write diagnostics” type [Y] then <enter>, in general no name of the .eps file is needed
- For the next three lines, take the default values
- When prompted, type the name of the output file.hkl file: e.g. 04mz02am.hkl, <enter>
- If the crystal was not a thin plate no extra absorption correction is needed. Type <return> until you are back at the SaintPlus menu and close the SaintPlus program.
Part 5: Final Steps before Refinement

- Burn a CD or move the data online to your personal computer.

- For the refinement, you will need the *.hkl, the *.p4p and sometimes the *.raw files (located in the work folder)

  e.g. 04mz02am.hkl
       04mz02am.p4p
       04mz02am.raw

- For publication purposes you will need additionally the *.abs and the *._ls files:

  e.g. 04mz02am.abs (Copy of what you did in SADABS, contains the ratio of Tmin/Tmax)
       04mz02am._ls (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))
Part 6: Solving Structures

Note: you can revise the space group using XPREP in the next section if necessary. If you are actually changing the Laue group, you better re-integrate the data.

- On your computer save the files from above into a new file folder and uncheck “read only” (by highlighting your files, a right mouse click, go into properties and uncheck.) Making a backup of your files is strongly recommended!

- Open the SHELXTL program. Select <PROJECT> and <New>. Find the appropriate file and open it. Give it a project name e.g. 04mz02am (do not forget the "m") then <open>

![Image of a New Project dialog box in SHELXTL](image)

Figure 33, Shelxtl New Project

6a) XPREP

- Select <XPREP> on the toolbar
In the next steps, the computer will make suggestions, that can normally (that is for good quality data) be accepted.

Figure 34, XPREP

- Select the suggested lattice type
- Choose [H] to search for higher metric symmetry
- Choose offered choice [A] for the Laue group (e.g. orthorhombic)
Figure 35, XPREP

- 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)

Figure 36, XPREP
- 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 another one)
- Select [D] to read, modify or merge datasets

- Select [S] to display the intensity statistics

- Select [N] to not merge datasets. The default is [A], merge all equivalents (including Friedel opposites). This would reduce your data to parameter ratio significantly and should be only done when absolutely necessary.

After selecting [N] the intensity statistics will be displayed.

<table>
<thead>
<tr>
<th>Resolution</th>
<th>#Data</th>
<th>#Theory</th>
<th>%Complete</th>
<th>Redundancy</th>
<th>Mean I</th>
<th>Mean I/s</th>
<th>Rint</th>
<th>Rsigma</th>
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<tr>
<td>Inf - 2.20</td>
<td>663</td>
<td>482</td>
<td>137.6</td>
<td>1.38</td>
<td>944.6</td>
<td>16.43</td>
<td>0.0485</td>
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<td>482</td>
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<td>1.46</td>
<td>433.7</td>
<td>17.69</td>
<td>0.0492</td>
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<td>566</td>
<td>143.6</td>
<td>1.44</td>
<td>283.5</td>
<td>16.12</td>
<td>0.0500</td>
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<td>1.26</td>
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<td>14.25</td>
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<td>1.16</td>
<td>134.6</td>
<td>13.36</td>
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<td>87.3</td>
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<tr>
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<tr>
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<td>34.4</td>
<td>8.38</td>
<td>0.0852</td>
<td></td>
</tr>
</tbody>
</table>

Merged [N]; lowest resolution = 10.08 Angstroms; 0 outliers downweighted

Enter <CR> to continue

Have a look at the Completeness, Redundancy and Rsigma. The completeness should be ideally close or over 100 % down to a resolution of d = 0.75. Rsigma should be ideally below 10 %
down to \( d = 0.75 \). If \( R_{\text{sigma}} \) becomes very large at higher resolution (lower \( d \) values), it might be appropriate to cut the data at \( d = 0.8 \) and/or merge equivalent data in the next steps.

Hit the Enter key

- Select either [H] or [M] to apply a high resolution cutoff or to merge the data if necessary (best try without first and apply only if really necessary and
- Select [E] to return to the main menu

- Select [C] to define the unit cell contents.

**Figure 38, XPREP**

- If necessary, select [R] to change the radiation (Mo radn; \( \lambda = 0.71073 \); Cu radn; \( \lambda = 1.54178 \))
- If necessary, select [F] for new formula, and type the molecular formula
- 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 Laue group in XPREP, you must choose a new name for the hkl file (change it back to your old filename after closing XPREP)
- Select [Y] at the prompt to generate an .ins file
Figure 39, XPREP

- Select **[Q]** at the prompt
6b) Using XS

To perform your refinement, you will first need an starting point for your structure. Using the *.ins file provided by XPREP, the program XS will provide you with an initial guess.

- 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 actually the right ones. The software will select the solution with the smallest value for CFOM and the initial atom positions are written in the ins file.

Figure 40, 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), or, for compounds with lots of atoms heavier than sodium, use PATT instead of TREF. Select <file> and then <save> and run XS again.

6b) Refinement Cycles

The refinement cycles can be performed using either <XP> and <XL> or <XSHELL>. The descriptions given here are based on using <XP> and <XL>. For a more detailed introduction into refinement methods use Allen D. Hunter’s manual (ask for one at adhunter@ysu.edu). For an in depth description of single crystal data refinement refer to the SHELXTL Software Reference Manual.

• Type "fmol" followed by <enter>, Hit the space bar until you reach the end of the Q peak listings
• Type "proj" followed by <enter>

A picture of the structure should appear that can be altered.
Have a close look at the “molecule” to identify Q-peaks that might be actual atoms. Rotate the molecule in an orientation you think is good to do this. (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)

- Exit "proj" again
- At this point you have to decide, which of the Q-peaks might be unwanted “ghost atoms” and which might be actual atoms. Not all atoms have to “pop out” in the first cycles. Then 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.

- The following commands are commonly used in the solution process but additional commands can be found in the manual.
"info" gives you some information about Q-peaks and atoms (after assignment). Often the actual atoms are showing a much higher intensity than “ghost-peaks”

"grow" - duplicates atoms in the structure in case of an inversion center, mirror plane or axis located in the molecule

"fuse" - opposite of grow command, has to be applied before assigning atom names

• When you have decided upon which peaks to assign or delete, use the following commands

"kill atomname(s)" - deletes specified atom(s)

"kill $q" - deletes all q peaks, "kill $C" deletes all carbon atoms etc

"pick" - assigns atoms

• Typing pick gives you a picture of your molecule (in the orientation when you have closed proj). The flashing atom can be deleted, assigned or skipped using the following commands:
  - typing a new atom name + <enter> = assigns new atom name
  - only <enter> = deletes selected atom
  - <backspace> = moves to previous (deleted) atom
  - <space> = skips the atom

Continue with the next atom. Other commands in pick are:
  - <tab> = enlarges the region of the selected atom
  - </> (backslash) this gets you out of the pick command and saves the structure “as is”

The result may be looking like this:
• When no Q peaks left unassigned or deleted, type "file" and then the filename e.g. 04mz02am followed by <enter> and <enter> again. Type <exit> to leave <XP>.

• Select <XL> for an optimization cycle and go on as described in Allen’s manual.
Figure 43, First XL Cycle
Appendix A: Troubleshooting

A) If the detector reinitialization fails:
Depending what you are doing at that time, the resume procedure is slightly different.

a) During Hemisphere etc measurements: These measurements can be resumed, but the detector has to be reinitialized first.
Step 1: Select <acquire> and <resume>. While this does not resume the measurement (the detector is still down), but a resume._tm file is “written”. Copy this file from the work folder into the data folder.
Step 2: Exit out of SMART, then press the red switch aside of the green “clear alarm” button at the left upper front side of the machine. Wait for the green light on the detector to come up and start SMART again. Select <acquire> and <resume> to restart the measurement.

b) During unit cell, rotation frame measurement etc: These measurements cannot be resumed, but has to be either started from scratch, or, if enough frames are already finished, just skip the remaining ones. In this case, go to <crystal>, <redtn cell> to start the cell determination.
To reinitialize the detector, follow the instructions of Step 2 in the above paragraph.

B) If the shutter doesn’t open / close

If you are lucky, it is hanging only once. In that case open and close the shutter several times (select <goniom>, <shutter> several times). The shutter should be ready now again. If your measurement can be resumed (not unit cell, rotation frames, but hemisphere etc) go to <acquire> and <resume> to restart the measurement.

If the shutter is hanging several times, 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 40 mA.

Power up procedure after prolonged downtimes:

<table>
<thead>
<tr>
<th>Downtime [days]</th>
<th>20 kV</th>
<th>25 kV</th>
<th>30 kV</th>
<th>35 kV</th>
<th>40 kV</th>
<th>45 kV</th>
<th>50 kV</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5 to 3</td>
<td>30 s</td>
<td>30 s</td>
<td>30 s</td>
<td>30 s</td>
<td>30 s</td>
<td>1 min</td>
<td>2 min</td>
<td>4 min</td>
</tr>
<tr>
<td>3 to 30</td>
<td>30 s</td>
<td>30 s</td>
<td>2 min</td>
<td>2 min</td>
<td>5 min</td>
<td>5 min</td>
<td>10 min</td>
<td>25 min</td>
</tr>
<tr>
<td>&gt; 30 or new generator</td>
<td>30 s</td>
<td>30 s</td>
<td>2 min</td>
<td>2 min</td>
<td>5 min</td>
<td>10 min</td>
<td>15 min</td>
<td>35 min</td>
</tr>
</tbody>
</table>
E) If the red alarm is lighted permanently and it is not the generator

If you are very familiar with the D8Tools program, 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 personnel computer. In really serious cases you may restart as well the computer of the SMART APEX machine.

- To restart only the personnel computer, first switch the detector off (red switch at the upper left side of the control panel). Then restart the personnel 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 SMART program.

- To restart both computers, follow the above sequence. Additionally, after starting the restart of the personnel computer, switch the SMART APEX 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 SMART APEX computer will start up faster than the personnel computer and will be ready when the boot up sequence of the personnel computer is finished.
  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.
Appendix B: Using GEMINI

Sometimes SMART fails to provide a meaningful unit cell. This may be due to the presence of not only one, but several crystallites or due to twinning. Especially in the case of intergrown but not twinned crystals, GEMINI is an extremely easy method to extract the unit cell of the major crystallite, which very often can be used in SMART as in the case of a real single crystal.

Step 1: Use SMART to determine a unit cell as described before. Even without providing a (meaningful) unit cell, SMART writes a *.p4p file listing the reflections and their positions in reciprocal space (normally the MATRIX0.p4p file)

Step 2: Open GEMINI, kill the pop up advice. Click on <I> and load the p4p file using <browse>. Keep the default values and click on <Run>.
A new window with a listing of possible unit cells comes up. Choose one, which looks like a reasonable solution. You might have to try several possibilities to get the right one.

Some help is provided by the following (but don’t apply it strictly):
- The #Fits should be high. All values being higher than 1/3 of the number of reflections are reasonable.
- The cell volume should not be extremely large. Cell constants over 30 Å are rare. If you have several unit cells with similar #Fits, take the smallest one.
- Any of the offered unit cells looking like being monoclinic, orthorhombic or higher in symmetry is a good candidate.

Note the number of your favorite unit cell, together with its parameters and volume. Click on <U>.
Check <Option 1>, type in your selected solution number and hit <Run>. The frame from before comes up again (nothing has obviously changed.) Click <I> again, keep all parameters and click on <Run>.

A new list of possible unit cells comes up. In this case, only the unused reflections of the first run are used.
Generally you can assume that all components of your intergrown or twinned crystal have the same unit cell (This does of course not apply for mixtures of compounds). Look for a unit cell with approx. the same volume as before. (In rare cases it might happen that the length and angles are different to before. In that case the program might have picked a different setting for the same cell.) Pick the best solution (most #Fits etc) and note its number.
Check <Option 2>, type in your selected solution number and hit <Run>. The frame from before comes up again (nothing has obviously changed.) Click <I> again, keep all parameters and click on <Run>.
A new list of possible unit cells comes up. In this case, only the reflections of the first component are used, but all reflections which are fitting for the second component as well are excluded. Select a solution being similar to the two found before; note its number and click <U> again.
Check <Option 3a>, type in your selected solution number and hit <Run>. You will be asked, if you want to apply a transformation. Choose <Do Not Apply Transform>.
You are brought back to the main window. Click <U> again, check <Option 3b>, click on <Run>, again choose <Do Not Apply Transform>, which brings you back to the main window. Click <U> again, select <Option 4> and click <Run>. This brings you to the final output window.
This window lists the two final solutions, the number of reflections used for each, the orientation matrices, etc. For better visibility, best copy the whole output into Notepad (The columns are badly aligned).

Have a look at the last lines of the output.
In this example, you see, that 46 reflections have been used by the second component, but not by the first one (0-1+); 67 have been used by the first, but not the second (1+0-), but none have been used by both (1+1+). Some reflections have been used by none.

If a large part of the reflections are used by both components you can assume that the two components are twins, i.e. they are connected to each other by a mathematical twinning law. In this case special methods have to be used to solve the structure. (Not covered here)

If most of the reflections are used by only one component, you will not have a twinned sample, i.e. the components are not systematically related. The SMART and SAINT software can deal with that as if it would be a single crystal. Note the solution with the higher number of reflections. The corresponding files are MATRIX_a.p4p for the first solution and MATRIX_b.p4p for the second one.

Step 3:
Close GEMINI and go back to SMART. Go to <FILE> and <Read p4p>. Load the *.p4p file created by GEMINI (MATRIX_a.p4p or MATRIX_b.p4p) using the <…> symbol.

Go to <CRYSTAL> and <LS>, the Least Square options are coming up. Keep all the parameters and apply no constraints (triclinic). Choose an output p4p filename and click <OK>.

A warning <No spatial correction> comes up. Choose continue. SMART gets you to the Least Square output.
Click <OK>, choose <CRYSTAL> and <Bravais>.
Click <OK>.
Choose a reasonable Bravais lattice. The #Sig should be small for the right solution. In most cases the suggested one (indicated by >>>) is fine. Note the # of the solution you are going to use as well as its Bravais lattice. Click <OK> again.
Choose the Solution # to use. Click <OK>. Then choose <CRYSTAL> and <LS> again.
This time constrain the LS to the setting chosen before (e.g. monoclinic B, orthorhombic, etc). Click <OK>. A warning <No spatial correction> comes up. Choose continue. This gets you to the final Least Squares output.
Choose <OK>. When prompted to overwrite the existing p4p file, choose <Yes>. You can now go on with your data collection.

When starting the integration using SAINT, load the p4p file just generated as the MATRIX file and update the cell parameters clicking <CELL>. In Saint, don’t forget to check that the right Bravais and lattice setting are chosen.
Appendix C: Changing the Hemisphere Settings

You might want to change the settings of the hemisphere measurement. Each line of the a run list contains the following items. When changing any of the angles, check the collision limits first!
Go to `<ANALYZE>` and `<Limits>`.

A possible hemisphere setting with 2Theta = 30 deg may look like this:

<table>
<thead>
<tr>
<th>#</th>
<th>2Theta</th>
<th>Omega</th>
<th>Phi</th>
<th>Chi</th>
<th>Axis</th>
<th>Width</th>
<th>#</th>
<th>time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-30</td>
<td>-30</td>
<td>0</td>
<td>54.74</td>
<td>2</td>
<td>-0.3</td>
<td>620</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>-30</td>
<td>-30</td>
<td>120</td>
<td>54.74</td>
<td>2</td>
<td>-0.3</td>
<td>620</td>
<td>10</td>
</tr>
<tr>
<td>3</td>
<td>-30</td>
<td>-30</td>
<td>240</td>
<td>54.74</td>
<td>2</td>
<td>-0.3</td>
<td>620</td>
<td>10</td>
</tr>
<tr>
<td>4</td>
<td>-30</td>
<td>-30</td>
<td>0</td>
<td>54.74</td>
<td>2</td>
<td>-0.3</td>
<td>50</td>
<td>10</td>
</tr>
</tbody>
</table>

The meaning of the parameters:

#
Use 1, 2, 3, 4 etc

2-THETA (Swing)
The detector swing angle (goniometer 2-theta setting angle), in degrees, at which SMART positions the goniometer for the frame series scans. Valid values are -180° to 180°.

OMEGA
The omega angle, in degrees, at which SMART positions the goniometer at the start of the frame series scans. If you specify a scan axis (below) of 2, omega will be scanned as each frame is taken. Otherwise omega remains fixed throughout the frame series. Valid values are -360° to 360°.

PHI
The phi angle, in degrees, at which SMART positions the goniometer at the start of the frame series scans. If you specify a scan axis (below) of 3, phi will be scanned as each frame is taken. Otherwise phi remains fixed throughout the frame series. Valid values are -360° to 360°.
CHI
The chi angle, in degrees, at which SMART positions the goniometer for this frame series. Our goniometer is fixed at CHI=54.74°.

AXIS
The number of the axis to be scanned, either 2 for omega scans or 3 for phi scans.

WIDTH
The scan width, in degrees (either positive or negative), of each frame to be acquired. Thus, the starting scan angle for the Nth frame in the series (where the first frame is N=0) is OMEGA+N*WIDTH if the axis (above) is 2, or PHI+N*WIDTH if the axis (above) is 3. The width should be selected that way, that each reflection appears on several frames (> 4), so that it can be successfully centered and integrated. You can check this using <ANALYZE> and <Rocking> in the SMART menu. For high melting materials with less thermal motion you might have to lower width from its default value of 0.3° to a smaller number. Don’t forget to adjust the #FRAMES accordingly.

#FRAMES
The number of frames to be acquired in this series.

TIME
The accumulation time, in seconds, during which each frame is to be acquired. Use seconds.
Appendix D: Changing the N2 tanks

To set up the high pressure tank, connect the thin N$_2$ 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 N$_2$ line coming out of the white reservoir dewar to the liquid outlet of a liquid N$_2$ 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 Cryoflex program to adjust the temperature.