

Quick Start Users Guide for Bruker APEX II Diffractometer

TREATING NON-MEROHEDRALLY TWINNED CRYSTAL DATA

Software version APEX2 v2.5

Cell_Now 2008/1ⁱ

Twinabs 2008/2ⁱⁱ

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This guide is useful for integrating, scaling, and refining the data for nonmerohedrally twinned crystals.

- 4.1** CELL_NOW must be run in order to assign the reflections to different domains.
- i.** In **EVALUATE - DETERMINE UNIT CELL**, delete the cell and reflections. Harvest all images (364) from the first run, or first two runs as needed. The *mean I/σ* should be changed to a smaller value (about 3) for weak diffraction. Click **HARVEST**.
 - ii.** After harvesting, go to **SAMPLE – EXPORT – P4P FILES**. In the window that comes up, choose CELL_NOW in the *Export For* field.
 - iii.** Next, go to the **START** menu – **RUN**, and type *cmd* and hit **ENTER**. The command prompt window should show up. Return to the C drive directory by typing *cd ..* and hitting **ENTER** until you get to the C drive. Next, go to the directory in which your files are located. For example, if you want to go into the work directory of JT573Ti, you would type:

```
cd frames
cd guest
cd APEX users
cd Vassar users
cd Tanski
```

```
cd JT763Ti
cd work
```

Next, input *cell_now* and hit ENTER. Type *filename.p4p*, and accept the next three default options. If you know the bounds of the cell edge, change the min/max values in the next option; otherwise, accept the default [5 45]. CELL_NOW will run for about 8 minutes.

Once done, accept the default 0.25 value for the maximum deviation from integer index. When prompted for the name of the first domain, name it *dom1.p4p*.

You will be shown how many reflections fit to the first domain, and how many were not assigned. The [S] option will search for the new cell. Once done, name the second cell *dom2.p4p*. If the number of reflections not assigned to the second domain is small, you do not need to search for a third domain, so choose option [Q] to quit CELL_NOW.

4.2 Back in APEX2, go to **SAMPLE – IMPORT – P4P/SPIN FILE**, and choose the **dom2** file. Select all import options by clicking the check boxes, and click **OK**. Two unit cells will be generated. Perform a light refinement by refining all reflections with **REFINE**, choose the Bravais lattice, and refine all the reflections once again in **REFINE**. Repeat this procedure for the second unit cell, and close the window to save the data.

4.3 Integration

i. Go to **INTEGRATE – INTEGRATE IMAGES**, and go to **INTEGRATION OPTIONS**. For sharp diffraction spots, change the I/σ Lower Limit to 5.00 and the Upper Limit to 4.00. Also change the Fractional Lower Limit of Average Integration to 0.450, uncheck the Multiwire Reference Correction, and change the Separation Factor to 1.100. Click **OK**. Go to **REFINEMENT OPTIONS**, and in the Component Constraints boxes, under Twin Component 2, choose Refine Separately, in both boxes. Click **OK**.

In the Integrate Images window, make sure the Resolution Limit is reasonable. It must be at least 0.84 Å in order to be fit for publication (i.e. 0.84 or lower). Click **START INTEGRATION**.

- ii. Once the integration is done, check whether the distance refined to zero, and that the Completeness to Highest Resolution Limit is almost 100% (this is located just above the line “Local LS Refinement”). Click **CLOSE**, and recycle the integration by going to **SAMPLE – IMPORT – P4P/SPIN FILE**. Choose *filename_0m.p4p*, click all the import options, and **OK**. The unit cell values of the two cells should be closer together now.

Go to **REFINEMENT OPTIONS**, and in the Component Constraints boxes, under Twin Component 2, choose Unit Cell as Component 1 on both sides. Click **OK**, and **START INTEGRATION** (say **Yes** to overwriting).

- 4.4. Because *CELL_NOW* was used to integrate the data, the Scale menu cannot be used. Instead, *twinabs* will be run in command prompt.
 - i. In command prompt, type *twinabs*, accept the next default option, and when prompted for a file name, input *filename_0t.abs*.
 - ii. Enter the correct number corresponding to the Laue group of your crystal system.
 - iii. Choose the default [**Y**] for default parameter refinement and when prompted, enter *filename_01*, and accept the next three default options. Type the backslash (/) for the next option. Accept the matrix.
 - iv. Input the space group, using parentheses for subscripts. (e.g. *P2(1)/c*)
 - v. For the parameter refinement, it is best to experiment with the different options. 0 will fit the domains separately and 1 will fit only dom1. These options depend on the mean I/σ for each domain. Choose one of these options and hit **ENTER**.
 - vi. Accept the mean I/σ and accept the next six default options. Look at the mean weight generated for the chosen mean I/σ . It should be close to 1. If it is too low, type **R** to repeat, and try **3.0** as the new mean I/σ . Accept the next 6 defaults as before.
 - vii. Choose the next four default High Resolution options. The g value should be about 0.04.

- viii. For Output, accept the four default options. Generate the HKLF 4 format by accepting the default [4] option. Name the file *filename_0m.hkl*, and hit **ENTER**.
- ix. When prompted to make a file using domain N, choose option **0** or **1**.
- x. Always average the Friedel Opposites (hit **Y**), and accept the next three default options.
- xi. An R_{int} value will be generated for all observations – write this down! It will be needed later. It may also be found in the *filename_0t.abs*.
- xii. Generate the HKLF 5 format by accepting the default option, hit **ENTER** to average equivalent reflections, and input *filename_0t.hkl* as the output name.
- xiii. When prompted to make a file using dom N only, input either **0** or **1**.
- xiv. Average Friedel Opposites: only do so if the space group is centrosymmetric; if it is not, input **N** for no.
- xv. Accept the point group and the next 4 default options, and hit enter to quit [Q].

NOTE: *filename_0m.hkl* contains roughly detwinned HKLF 4 the data that is used for the structure solution. It generates the *filename_0m.p4p/hkl/ins...* files.
filename_0t.hkl contains all data in HKLF 5 format (depending on the options selected in Twinabs) and generates *filename_0t.p4p/hkl/ins...* files.

- 4.5** Go to **EXAMINE DATA – SPACE GROUP**. The filenames should already be generated: *filename_0m.p4p*, which came from the integration, and *filename_0m.hkl*, which came from HKLF 4. Continue with routine XPREP procedures. When prompted for a filename, leave it as *filename_0m*, and overwrite the *.hkl*.
- 4.6** Go to **SOLVE STRUCTURE – STRUCTURE SOLUTION**. Change the base to *filename_0m.ins* if it is not already there. Click **SOLVE STRUCTURE**, and exit once it is finished.

4.7 Go to **REFINE – STRUCTURE REFINEMENT**.

- i. Choose the *filename_0m.res* file and refine as normally.
- ii. Once finished refining, go back to command prompt, and type *copy filename_0m.res filename_0t.ins*, and hit **ENTER**.
- iii. Go back to the refinement, and change the base to *filename_0t.ins*. In the instructions file, change the title to *filename_0t*, and at the bottom, change HKLF from 4 to 5. Add BASF 0.5. Make note of the R factor and Weight. Refine.

4.8. It is advisable to go back to twinabs and experiment with fitting the domains separately (the 0 option) and fitting domain 1 only (the 1 option). For example, you may want to do 1/1/1, 1/0/1/, 0/1/1, etc, and see which combination gives you the best R_{int} . Always make note of the R_{int} , R factor, and weighting scheme.

4.9 **Important:** Do not enter the **BASF 0.5** and **TWIN** commands together. The **BASF** will refine to somewhere between 0 and 1, showing the percentage of each twin present. If the space group is chiral, the Flack x parameter can be found in the .lst file.

4.10 Refer to steps 3.3 - 3.5 for editing the parameter file (*filename.pcf*), and generating and the .cif file. In addition to the steps outlined in 3.5, the following lines in *filename.cif* must be edited manually before submitting to the **CHECKCIF/PLATON** service.

<code>_exptl_absorpt_process_details</code>	<code>`twinabs 2008/2'</code>
<code>_diffrn_reflns_av_R_equivalents</code>	(value can be found in the .abs file, it is the R_{int} value "for all observed...")
<code>_diffrn_reflns_av_sigmaI/netI</code>	(R_{int} for Highest Resolution limit (e.g. Inf-0.84), found in .prp file; generated in XPREP.)
<code>_diffrn_reflns_number</code>	(Value found in .abs file,

"HKLF 5 dataset constructed
from all observations
involving domain 1")

_diffn_reflns_number_total

(Value found in .lst file,
"R1 = for __ unique
reflections")

_exp_special_details

'details of twinning from .abs file'
'BASF w/esd from .lst file'

ⁱ Sheldrick, G. M. (2008). CELL_NOW, University of Göttingen.

ⁱⁱ Sheldrick, G. M. (2008). TWINABS, University of Göttingen.