

Quick Start Users Guide for Bruker SMART APEX II Diffractometer

PRINTING THE RESULTS AND PREPARING THE FILES FOR SUBMISSION AND PUBLICATION

Software version APEX2 v2.1-0

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This guide is useful for completing a crystal structure and preparing the necessary files for submission and publication.

After data collection is complete, finish the data reduction according to **Steps 1.9-1.13** described in the *Routine Data Collection* section of this Quick Start Guide. Once the structure has been solved and preliminarily refined on the APEX2 computer, move to the IBM PC to finish the structure and print the results.

- 3.1** The rest of the process must be done in **SHELXTL**. Double click on the SHELXTL icon at the top of the desktop. Select **PROJECT-NEW**. Browse to the directory on the APEX2 computer that contains the structure file (*filename.hkl*):

/frames on Bruker-3b-server/guest/APEXUsers/.../Filename/work/...

Name the project, highlight *filename.hkl*, and click **OPEN**.

Note: *If you rename the structure file in XPREP after this point, you must create a new project using the newly created structure file.*

- 3.2** To finish the structure model, it is best to first refine the structure *anisotropically*, *label and sort* the atoms sensibly, then *add hydrogen atoms* and refine. There are also a number of things that need to be added to or edited in the instruction file (*filename.ins*) and the result file (*filename.res*). Access these files by using the **EDIT** menu in SHELXTL.

- i. To refine the structure anisotropically, add **ANIS** to the instruction file. Type this command on its own line, after the line containing **BOND**. Also enter the crystal size and collection temperature (in degrees Celsius) if these are not present in the instructions file. Use the **SIZE** and **TEMP** commands, each on their own line. Save and Refine.

So for JT373V, the instruction file would look similar to the following (*before refinement*):

```
TITL JT373V in P21
CELL 0.71073 9.5129 10.8084 14.3731 90 103.632 90
ZERR 4.00 0.0007 0.0007 0.0010 0.001 0.001 0.001
LATT 1
SFAC C CL N O V
UNIT 80 16 8 16 1
TEMP -148
SIZE 0.030 0.100 0.160
L.S. 4
BOND
ANIS
FMAP 2
PLAN 20
```

- ii. Label the atoms sensibly in XP and then sort them using the commands: **sort/n** (enter) **sort \$A1 \$A2 \$A3 ...** (enter), where A is an element in the structure. Type **info** to check that the atom list has been sorted and then save the information by typing **file** and then **quit**. Refine.

So for JT373V, the process for sorting would look similar to the following:

```
XP>> sort/n
XP>> sort $V $O $N $Cl $C
```

- iii. Once finished, open the result file and type **EXTI 0.00001** to refine the extinction coefficient. This command should be on its own line, after the line containing **BOND**. Save this file, select **EDIT-COPY .RES -> .INS**, and refine. If the extinction coefficient refines to zero, enter **REM** in front of **EXTI** and save.

- iv. For polar and non-centrosymmetric space groups, verify that the correct orientation of the polar axes has been chosen. This is done by typing **TWIN** and **BASF 0.5** into the instruction file, each on their own line. Type these commands after the line containing **BOND**. If the correct orientation has been chosen, the value of the BASF will refine to approximately zero. If the value refines to approximately one, type **MOVE 1 1 1 -1** into the instruction file, after the line containing the BASF value. Repeat the refinement. Certain space groups require special values; see p. 11-3 in the SHELX Manual.
- v. Hydrogen atoms can be added in XP by typing the command **hadd**. Verify that the hydrogen atoms are in the correct positions and that there are the correct number of hydrogen atoms at each position. Hydrogen atoms are labeled automatically based on the labeling scheme made in **Step ii**; there is no need to rename them. After adding hydrogens and saving, exit XP and refine. In the results file, replace all methyl hydrogen tags as **137** and save.

The weighting scheme should be repeatedly updated until it remains constant. Sometimes the suggested weighting scheme leads to a GooF value <1. The GooF value should be >1; if there is a discrepancy here, adjust the weighting scheme appropriately until the GooF is just above 1. When the refinement has converged, the max shift is ~0.00.

- vi. Check that all thermal parameters are reasonable; they should be approximately 0.04. Delete any disagreeable reflections (see JMT). Use SQUEEZE, as described in Appendix IV, for any horribly disordered solvent or counterions. Check suspicious space groups with Platon, as described in Appendix III.

Insert any miscellaneous commands into the instruction file, such as **CONF** (torsional angles) or **BIND** (distance between specified atoms). Insert **ACTA** into the instruction file to create the *filename.cif* file. This command will also alert you to a discrepancy in the UNIT instruction. If there is a problem, see the *filename.lst* file. Modify the UNIT instruction in the instruction file, save and run the final XL refinement.

3.3 The parameter file (*filename.pcf*) must be modified so that the completed structure (contained in the .cif) is acceptable for submission to the Cambridge Structure Database and for publication.

Select **EDIT-EDIT** to open a notepad window. Select **FILE-OPEN** and under “Files of Type” select “All Files”. Browse to find *filename.pcf*.

Modify the .pcf file to resolve the important fields as shown below. Type the correct information into the fields; do not remove the quotation marks surrounding the field.

Note: Remember to put quotation marks around the ? in the *exptl_crystal_density_meas* field.

<pre> data_bisws _symmetry_cell_setting _symmetry_space_group_name_H-M _cell_measurement_temperature _cell_measurement_reflns_used _cell_measurement_theta_min _cell_measurement_theta_max _exptl_crystal_description _exptl_crystal_colour _exptl_crystal_size_max _exptl_crystal_size_mid _exptl_crystal_size_min _exptl_crystal_density_meas _exptl_crystal_density_method _exptl_absorpt_correction_type _exptl_absorpt_correction_T_min _exptl_absorpt_correction_T_max _diffn_ambient_temperature _diffn_radiation_type _diffn_radiation_source _diffn_radiation_monochromator _diffn_measurement_device_type _diffn_measurement_method _diffn_detector_area_resol_mean _diffn_standards_number _diffn_standards_interval_count _diffn_standards_decay_% _computing_data_collection _computing_cell_refinement _computing_data_reduction _computing_structure_solution _computing_structure_refinement _computing_molecular_graphics _computing_publication_material </pre>	<pre> 'Triclinic' 'P-1' 153(2) ? ? '? ' '? ' ? ? '? ' '? ' 'none' ? ? 153(2) 'MoK\alpha' 'sealed tube' 'graphite' 'CCD area detector' 'phi and omega scans' ? ? ? ? 'Bruker SMART' 'Bruker SMART' 'Bruker SAINT' 'Bruker SHELXTL' 'Bruker SHELXTL' 'Bruker SHELXTL' 'Bruker SHELXTL' </pre>	<div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Correct to 125</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">See JMT if not present</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Input description, e.g. block, plate...</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Input crystal color</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Input crystal size</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Correct to empirical</div> <div style="border: 1px solid black; padding: 2px; width: fit-content; margin-bottom: 5px;">Correct to 125</div>
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3.4 Generate a report by running **XCIF**. This should be done twice; the first time will be to print out tables that will go into the binder log book. The second time will be to generate the report such that it can be opened and modified in Microsoft Word. If **XL** is run after this step, **XCIF** must be run again. Select **XCIF**; hit **ENTER** to proceed through the options.

Choose **OPTION R** (“*Use another CIF file to resolve ? items*”) and hit **ENTER** four times to complete the option with the default settings.

OPTION C (“*Set compound name for tables*”) and enter a compound name after the “Input new name” prompt. Do not simply accept the sample filename for this.

OPTION N (“*Set next table number*”) and type **1** if starting from the beginning, or another number as appropriate (see below).

OPTION T (“*Crystal/atom table for .cif*”) and hit **ENTER** twice until the “Filename for tables” prompt appears. Enter *filename.tex*.

For the first run of **XCIF**, select the default [**ang**] definition file, and type **Y** for the first prompt only and **N** for the rest. For the second run of **XCIF**, type **def** and then **Y** or **N** as indicated below.

Crystal data table? – **Y** (Table 1)

Atom coordinates table? – **Y** (Table 2)

Selected bond lengths and angles table? – **N**

Full bond lengths and angles table? – **Y** (Table 3)

Anisotropic displacement params table? – **Y** (Table 4)

Hydrogen atom coordinates table? – **Y** (Table 5)

Selected torsion angles table? – **N** (Only **Y** if **CONF** is in **.ins** file)

Full torsion angles table? – **N**

Hydrogen bond table? – **N** (Only **Y** if **HTAB** completed)

OPTION F (“*Structure factor tables from .fcf*”) and hit **ENTER** twice until the “Filename for tables” prompt appears. Enter *filename.sft* and accept the next two defaults.

Select **OPTION X** and enter *filename.tex* to print. Make sure there is no missing data; select **OPTION Q** to quit.

3.5 After running XCIF, some information may still be missing from the generated .cif file. These must be edited manually. Select **EDIT-EDIT .CIF** to open the file.

```

.
.
.
_cell_length_a          9.8190(3)
_cell_length_b          14.0298(4)
_cell_length_c          11.5874(3)
_cell_angle_alpha       90.00
_cell_angle_beta        110.244(2)
_cell_angle_gamma       90.00
_cell_volume            1497.66(7)
_cell_formula_units_Z   2
_cell_measurement_temperature 388(2) ← Correct to 125
_cell_measurement_reflns_used 2026
_cell_measurement_theta_min 2.37
_cell_measurement_theta_max 23.07
.
.
.
_exptl_absorpt_coefficient_mu 3.051
_exptl_absorpt_correction_type 'empirical'
_exptl_absorpt_correction_T_min 0.5806
_exptl_absorpt_correction_T_max 0.7501
_exptl_absorpt_process_details '?' ← Type sadabs
.
.
.
_diffrn_ambient_temperature 388(2) ← Correct to 125
_diffrn_radiation_wavelength 0.71073
_diffrn_radiation_type MoK\alpha

```

In most structures some of the hydrogen atoms are refined (Q peaks are assigned as H's and then refined when running XL), and the rest of them are located in calculated positions through Add Hydrogen in XShell (or the command HADD in XP). *If all the hydrogen atoms in the structure are in calculated positions (none of them was located and refined), scroll down to the section after the `_refine_special_details` paragraph and change the corresponding line:*

```

_atom_sites_solution_hydrogens geom
_refine_ls_hydrogen_treatment mixed ← Change to constr
_refine_ls_extinction_method SHELXL

```

Note: If XL was run after running XCIF, XCIF must be redone and the .cif file manually edited.

3.6 It is easiest to print ORTEP diagrams from XP.

- i. Open the structure in XP. Type “**fmol/n**” and then “**proj**” to see and rotate the structure. After exiting, type “**kill \$q**” to remove the Q peaks. If desired, type “**kill \$h**” to remove the hydrogens. If you want to save certain hydrogens, type “**pick \$h**” to delete (enter), skip (space bar), and assign atoms (type label, return). Type “/” to get out of pick. If hydrogens will be present in the plot, enter the command “**ATYP \$H 1**” (correct shading) and “**LABL 4 500**” (correct labeling).
- ii. Type “**PROJ**” and orient the structure in a reasonable manner. The command “**MPLN atom1 atom2 atom3**” will orient the molecule such that the named atoms are in the plane of the screen.
- iii. π -bonded ligands (i.e: Cp rings, centroids) should be created using the “**CENT/X**” command. The centroid should be joined to the metal (“**JOIN X1A M**”, for example) and the bonds from the ring to the metal deleted (“**UNDO C1 M**”, for example). To save it for later, type “**SAVE name**”. This creates a file called *name.sav*, which can be read in again with the “**NEXT name**” command.
- iv. To create the ORTEP diagram, type “**TELP 0 -20 0.04**”. Hit **ENTER** until prompted for the plot file. Type a name.
- v. The ORTEP drawing should appear on the screen. A small rectangle will appear next to the first atom in the list; move it to a good location and click the left mouse button to label that atom. Hit **ENTER** if you want to skip a label. Type “**b**” to get out of the labeling screen.
- vi. Type “**DRAW filename**” to print the diagram. Use this command to create .pdf files of the diagram also.

- 3.7** Submit the .cif file to the **CHECKCIF/PATON** service. Upload the file at <http://journals.iucr.org/services/cif/checkcif.html>.

checkCIF/PLATON

Welcome to the *checkCIF/PLATON* service, which is operated by the [IUCr](#).

Please upload your CIF using the form below.

File name:

Browse...

Send CIF for checking

Select report type

Publication check Full structural check Basic structural check

After selecting **SEND CIF FOR CHECKING**, a series of alerts will be given. Special attention must be given to alerts category A and B.

- 3.8** *In both log books, record that the structure was completed. From the APEX2 computer, transfer the entire sample folder to the external hard drive. Place the print out of Table 1 and an ORTEP diagram of the structure in the binder log book.*
- 3.9** When the structure is submitted for publication, the .cif file must be submitted to the Cambridge Structural Database. See the instructions at <http://www.ccdc.cam.ac.uk/products/csd/deposit/>.

Any available information on the journal, authors and title of the publication must be added at the top of the .cif file. Copy the contents of the file *CifPubTemplate.pcf* (IBM PC) into the beginning of the .cif file, and enter the journal, author, and title information.