**Cif File Entries for the Purdue Quest Diffractometer (December 2023)**

A CIF (Crystallographic Information File) is written by the refinement program, but it is usually incomplete, and some required items need to be added by hand before the CIF file will pass checking. Checking, done online via the web page <https://checkcif.iucr.org/>, is required if you want to publish crystallographic data in a peer reviewed journal.

This document contains general data collection items for all data sets collect on the two Purdue Bruker Quest diffractometer.

The CIF format uses text only (ASCII characters). This means, if it is not on the computer keyboard, don’t use it.

Each basic entry consists of two items: The identifier, and the value. The identifier usually starts with an underscore, and might consist of several words connected by underscores (e.g. \_diffrn\_radiation\_monochromator). It is followed by the value, which, depending on the item, is a number or text. It has to be either a single number or word, or it has to be marked as a single item with single parentheses (e.g. 'fine focus sealed tube'). Each line should not exceed 80 characters (that’s legacy from times when e-mails could not handle longer lines). If an entry is longer than 80 characters, you should use semicolons instead of the single parentheses. It may then stretch over several lines.

Values have to be in the cif “library”. Numbers have to be in an acceptable range, words have to be one of a list of allowed entries in the “CIF Dictionary” (e.g. at <http://www.iucr.org/resources/cif/dictionaries/cif_core>).

Tables in a cif file are following a more complicated procedure.

For a complete description of the CIF file format for reporting crystal structures, and on current developments regarding cif files, see <http://www.iucr.org/resources/cif>.

The CIF code allows to express some non-ASCII characters (such as Greek letters or Umlauts). H\"ubschle, for example, will be printed as Hübschle when converted to text using “printcif” or making tables.

Beware of typos!!

With a CIF file based on a dataset from one of the Purdue Bruker Quest instruments and refined with Shelxle and Shelxl2019, usually the items listed on the next pages have to be added or edited. They are listed in the order in which they will appear in the cif file and are given in blocks similar as in the cif file. Items in black can be copied directly. Items in red need a second look and a value looked up or a decision made.

Edits can be made using a simple text editor such as Notepad or programs like [Encifer](https://www.ccdc.cam.ac.uk/Community/csd-community/encifer/) or [FinalCif](https://dkratzert.de/finalcif.html). Do not use advanced text editors such as MS Word.

You may also try out [FinalCif](https://dkratzert.de/finalcif.html) to automatically extract missing items from the raw data files and to create tables and reports (read the [FinalCif software documentation](https://dkratzert.de/files/finalcif/docs/) first!).

1. Cell measurement details

\_cell\_measurement\_reflns\_used from p4p file, 1st value of SAINGL line

\_cell\_measurement\_theta\_min from p4p file, 2nd value of SAINGL line

\_cell\_measurement\_theta\_max from p4p file, 3rd value of SAINGL line

2. Crystal colour and shape

\_exptl\_crystal\_description from p4p file, MORPH entry

\_exptl\_crystal\_colour from p4p file, CCOLOR entry

\_exptl\_crystal\_size\_max from p4p file, CSIZE entry

\_exptl\_crystal\_size\_mid from p4p file, CSIZE entry

\_exptl\_crystal\_size\_min from p4p file, CSIZE entry

3. Absorption correction details

\_exptl\_absorpt\_correction\_type 'multi-scan'

\_exptl\_absorpt\_correction\_T\_min obtain value from “abs” file, 3rd last line

\_exptl\_absorpt\_correction\_T\_max obtain value from “abs” file, 3rd last line

\_exptl\_absorpt\_process\_details

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SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D. (2015). J. Appl. Cryst. 48, 3-10.

;

(These four entries might be already added if the original hkl file has been used rather than the one processed in XPREP).

4. Instrument details

Quest instrument with molybdenum source:

\_diffrn\_source 'fine focus sealed tube X-ray source'

\_diffrn\_source\_current '30 mA'

\_diffrn\_source\_voltage '50 kV'

\_diffrn\_radiation\_monochromator 'Triumph curved graphite crystal'

\_diffrn\_measurement\_device 'three circle diffractometer'

\_diffrn\_measurement\_device\_type 'Bruker AXS D8 Quest'

\_diffrn\_detector\_type 'PhotonII charge-integrating pixel array (CPAD)'

\_diffrn\_measurement\_method 'omega and phi scans'

\_diffrn\_detector\_area\_resol\_mean 7.4074

Quest instrument with copper source:

\_diffrn\_source 'I-mu-S 3.0 microsource X-ray tube'

\_diffrn\_source\_current '1.1 mA'

\_diffrn\_source\_voltage '50 kV'

\_diffrn\_radiation\_monochromator 'HELIOS multilayer Montel optics'

\_diffrn\_measurement\_device 'four circle diffractometer'

\_diffrn\_measurement\_device\_type 'Bruker AXS D8 Quest'

\_diffrn\_detector\_type 'PhotonIII\_C14 charge-integrating and photon counting pixel array'

\_diffrn\_measurement\_method 'omega and phi scans'

\_diffrn\_detector\_area\_resol\_mean 7.4074

The entries \_diffrn\_radiation\_monochromator, \_diffrn\_measurement\_device, \_diffrn\_detector\_type, \_diffrn\_source\_current and \_diffrn\_source\_voltage are not automatically added to the cif file. Add them anyway (it also helps to populate all entries in reports you might create using FinalCIF).

5. Software details:

\_computing\_data\_collection ' Apex4 v2022.10-1 (Bruker, 2022) '

\_computing\_cell\_refinement ' SAINT V8.40B (Bruker, 2020)'

\_computing\_data\_reduction ' SAINT V8.40B (Bruker, 2020)'

\_computing\_structure\_solution 'SHELXS-97 (Sheldrick, 2008)' or 'SHELXM (Sheldrick, 2008)' or 'SHELXT (Sheldrick, 2015b)'

\_computing\_structure\_refinement

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SHELXL-2019/2 (Sheldrick, 2015a, 2019),

SHELXLE Rev1573 (H\"ubschle et al., 2011)

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6. Structure solution methods

\_atom\_sites\_solution\_primary 'direct', 'patt', 'dual space' or 'isomor'

\_atom\_sites\_solution\_secondary 'difmap' or 'none'

Use 'direct' if you used TREF in XS or XM to solve the structure (direct methods). Use 'patt' if you used PATT in XS to solve the structure. Use 'dual space' if you used ShelXT to solve the structure. Use 'isomor' if you solved the structure by isomorphous replacement.

Use difmap if you assigned Q-peaks as atoms. Use none if no atoms needed to be added during refinement.

7. Absolute Configuration:

\_chemical\_absolute\_configuration 'ad' or 'syn' or 'unk'

This entry only applies for chiral and non-centrosymmetric structures.

Use 'ad' (anomalous dispersion) if you refined a Flack parameter and got a reliable value close to zero with a small esd.

Use 'syn' (synthesis) if you assigned the absolute structure based on a known chiral center, e.g. from the starting material that did not change during synthesis.

Use 'unk' (unkown) if you don’t know.

8. Special Details:

If you used any non-standard techniques to process the data or refine the structure, add these to the \_refine\_special\_details section of the cif. Add here details on twinning, disorder, any constraints or restraints you used, pseudosymmetry, use of Squeeze and other details you would like to add that fit nowhere else.

\_refine\_special\_details

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Typical example text:

The two disordered moieties were restrained to have similar geometries.

Uij components of ADPs for disordered atoms closer to each other than

2.0 Angstrom were restrained to be similar. Subject to these conditions

the occupancy ratio refined to ??? to ???.

Water H atom positions were refined and O-H and H...H distances were

restrained to 0.84(2) and 1.36(2) Angstrom, respectively. Some water

H atom positions were further restrained based on hydrogen bonding

considerations.

Water H atom positions were initially refined and O-H and H...H

distances were restrained to 0.84(2) and 1.36(2) Angstrom,

respectively, while a damping factor was applied. Some water

H atom positions were further restrained based on hydrogen bonding

considerations. In the final refinement cycles the H atoms were

constrained to ride on their carrying oxygen atom (AFIX 3) and

the damping factor was removed.

(see the cif template files for other example text)

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9. Authors, software cited above and acknowledgements. Add below the data\_ line at the top and above the remainder of the cif text. Repeat the author loop (below \_publ\_author\_address) as often as needed to include all authors (you, advisor, etc). Delete any software you did not use.

\_publ\_contact\_author\_name 'Last Name, First Name(s)'

\_publ\_contact\_author\_address

; Your Department

 Purdue University

 Your Street Address

 West Lafayette

 IN Your ZIP-code

 USA

;

\_publ\_contact\_author\_email 'your e-mail'

\_publ\_contact\_author\_fax 'your department fax number'

\_publ\_contact\_author\_phone 'your department phone number'

loop\_

\_publ\_author\_name

\_publ\_author\_address

 'Last Name, First Name(s)'

; Department

 University or Company

 Street Address

 City

 State, ZIP-code

 Country

;

\_publ\_section\_references

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\_publ\_section\_acknowledgements

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