Crystallographic Information Files and Report Generation

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ver 1.0.0

Crystallographic Information Files

"The acronym CIF is used both for the *Crystallographic Information File*, the data exchange standard file format of Hall, Allen & Brown (1991), and for the *Crystallographic Information Framework*, a broader system of exchange protocols based on data dictionaries and relational rules expressible in different machine-readable manifestations, including, but not restricted to, Crystallographic Information File and XML." IUCR 2006

When you are ready to write-up your structure there are a few steps you should take.

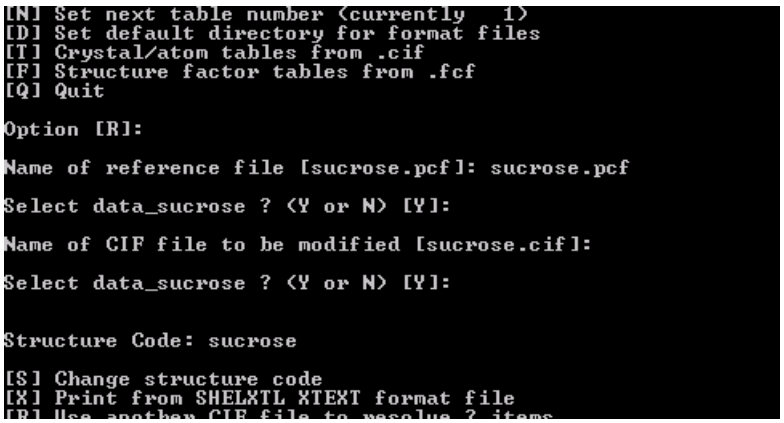
Add these lines to your final INS file

ACTA
SIZE .1 .1 .1
HTAB
CONF
TEMP -163
WPDB -1

make sure the SIZE is the correct size for your crystal. Don't guess!

Run a final refinement.

Point to XCIF. Select R and input the *.pcf file generated by XPREP. Select the default values for the next 3 questions.



Quit the program with Q.

If you do not have a *.pcf file you will need to input the values with the editor.

Point to Edit/Edit.Cif
Input crystal description and colour

_exptl_crystal_description cube
_exptl_crystal_colour colorless

Input correction_type :: "multi-scan" and process_details :: "sadabs"

_exptl_absorpt_correction_type multi-scan
_exptl_absorpt_correction_T_min 0.8861
_exptl_absorpt_correction_T_max 0.8861
_exptl_absorpt_process_details sadabs

Find _refine_ls_hydrogen_treatment mixed
change the mixed to riding
_refine_ls_hydrogen_treatment riding

Save the file and exit

For the GADDS data collection

Change _diffrn_measurement_device_type 'CCD area detector'
to
_diffrn_measurement_device_type 'MWPC area detector'

Change the SMART to FRAMBO

_computing_data_collection 'Bruker SMART'
_computing_cell_refinement 'Bruker SMART'

_computing_data_collection 'Bruker FRAMBO'
_computing_cell_refinement 'Bruker FRAMBO'

Next check the structure with CIFCHECK

http://journals.iucr.org/iucr-top/index.html

CIFCHECK on-line CIF checking routine

http://journals.iucr.org/services/cif/checkcif.html

Start a web browser and point to the service above. Find your CIF with the browse button and point to the Basic Structure Check.

The alert level A is most important. Here I have not reported a measured density so it gives me the ratio ... outside of range. This is ok. Alert 3 will always be present for Copper data. Our instrument will not read above two-theta of 120°.
O10 has a high Ueq but it is acceptable as is the short contact between calculated hydrogens.

checkCIF/PLATON report (basic structural check)

No syntax errors found. CIF dictionary
Please wait while processing .... Interpreting this report

Datablock: sucrose

Bond precision: C-C = 0.0073 A Wavelength=1.54178
Cell: a=7.7619(5) b=8.7112(8) c=10.8676(9)
alpha=90 beta=102.929(4) gamma=90

	Calculated	Reported
Volume	716.19(10)	716.19(10)
Space group	P 21	P2(1)
Hall group	P 2yb	?
Moiety formula	C12 H22 O11	?
Sum formula	C12 H22 O11	C12 H22 O11
Mr	342.30	342.30
Dx,g cm-3	1.587	1.587
Z	2	2
Mu (mm-1)	1.239	1.239
F000	364.0	364.0
F000'	365.48	
h,k,lmax	8,9,12	8,9,12
Nref	1102[2045]	1979
Tmin,Tmax	0.883,0.883	0.886,0.886
Tmin'	0.883	

Correction method= 'MULTI-SCAN'
Data completeness= 1.80[0.97) Theta(max)= 58.85
R(reflections)= 0.0581(1848) wR2(reflections)= 0.1659(1979)
S = 1.096 Npar= 217

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A
DENSX01_ALERT_1_A The ratio of the calculated to measured crystal density lies outside the range 0.80 <> 1.20
Calculated density = 1.587
Measured density = 0.000
PLAT027_ALERT_3_A _diffrn_refins_theta_full (too) Low ..... 58.85 Deg.
PLAT035_ALERT_1_A No _chemical_absolute_configuration info given . ?
PLAT241_ALERT_2_A Check High Ueq as Compared to Neighbors for O10
PLAT415_ALERT_2_A Short Inter D-H..H-X H5 .. H12C .. 1.47 Ang.

And finally the No _chemical_absolute_configuration needs to be added to the cif.

For sugar the absolute configuration is based on relative configuration so the correct response is 'rm'

The responses are rm, ad, rmad, syn, unk see
http://www.iucr.org/iucr-top/cif/cifdic_html/1/cif_core.dic/Ichemical_absolute_configuration.html
for details.

Add the
_chemical_absolute_configuration 'rm'

line after
_chemical_formula_weight 342.30

in the cif.

Return to XCIF

```
Option [R]: T

Name of CIF file [sucrose.cif]:

Select data_sucrose ? (Y or N) [Y]:

Filename for tables (<CR> to print directly) [ ]: sucrose.rtf

The format definition file should be specified by means of the extensions
'rta' (Rich Text Format for input to MSWord / Angstroms), 'rtm' (RTF / pm),
'def' (plain text) or other extensions for local versions.

The three options 'ang' (Angstroms), 'met' (pm etc.) or 'ger' (pm / German)
generate .tex output files in the (obsolete) SHELXTL XTEXT format that may
be printed via the XCIF 'X' option on most HP printers. XTEXT format should
NOT be printed directly !

Filename extension for xcif.??? format definition file [ang]: rta

Crystal data table ? [Y]:
```

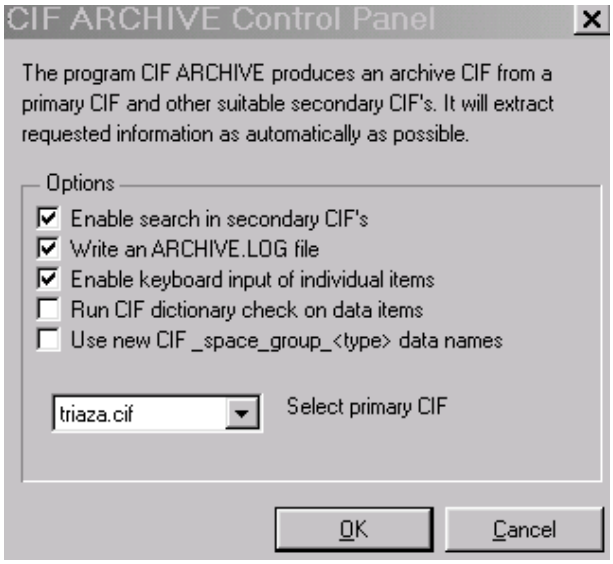
Choose T, for the file name enter *.rtf here I entered sucrose.rtf.
for the extension enter rta.
Choose Crystal Data Table, atomic coordinate table, bond/angle table full (not select), displacement table and hydrogen table. Answer N to the rest. Quit the program.

Start word and read the *.rtf file as a rich text file.

WINGX report generator.

You can use wingx to generate the report. Start WINGX.

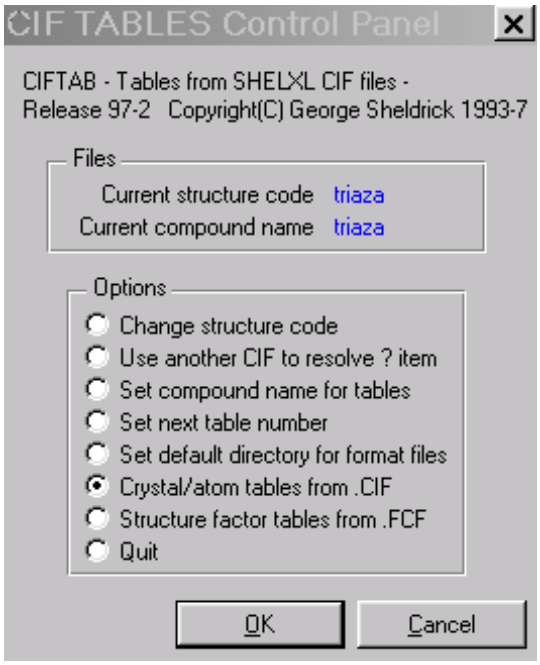
Point to Publish/CIFTABLES. Create the Archive.CIF file. Check the Enable keyboard input... and point to Ok. Do not ignore the Shelx97 values when asked.



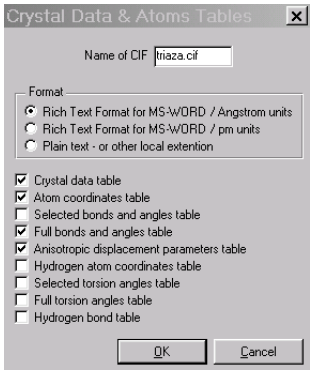
When prompted type auto.

Let the program run.

The next dialog box should look like this



Check the Crystal/Atom tables for .CIF



Check the Rich Text Format for MS-WORD/ Angstrom.

Point to OK.

Finish the program and select quit.

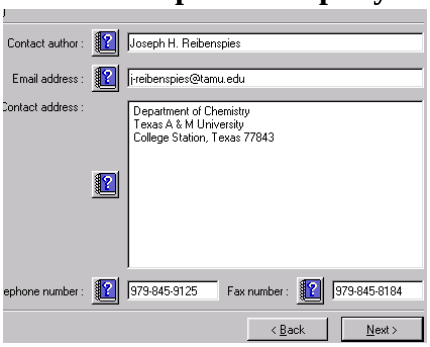
Rename the *.tex file that the program generated to *.rtf. Open WORD and read as a Rich Text Format document.

enCIFer - CIF checking, editing and visualisation software from the CCDC

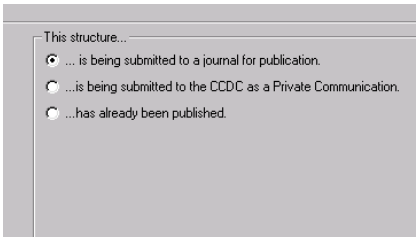
http://www.ccdc.cam.ac.uk/free_services/encifer/

How to use enCIFer to edit CIF files for publication.

Point to file/open and input your CIF file. (The file should have been ran through CIFCHECK first. Point to tools/publication wizard. Input contact information. Next input where you will publish the data. I will send this to ACTA CRYST



Next enter the authors etc.



When you finish the you will see the CIF will the authors and journal. This is fine if you do not send the file to ACTA CRYST. If you will need some more information.

I have added this to the CIF.

```
------
;
Date of submission 7-19-2005

Please consider this CIF submission for publication as a
Regular Structural Paper in Acta Crystallographica E.
;


#------------------ TITLE AND AUTHOR LIST-----------------------------------#

_publ_section_title
;
The Crystal and Molecular Structure of 1,4,7-triazacyclononane hydrate
;

_publ_section_title_footnote
;
?


#------------------ TEXT ---------------------------------------------------#


_publ_section_abstract
;
The crystal and molecular structure of 1,4,7-triazacyclononane hydrate
has been determined at 110K.
;

_publ_section_comment
```

;
1,4,7-triazacyclononane ([9]aneN~3~) forms a variety of complexes with metallic and nonmetallic elements and has been extensively reviewed (Chaudhuri & Wieghardt, 1987). ....

.... (more text not important to this demo)

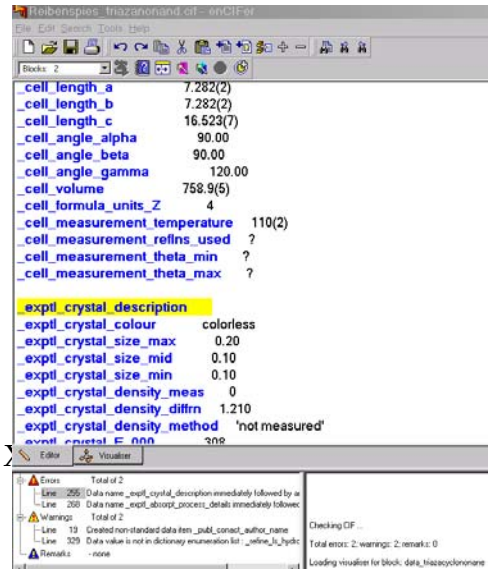
three symmetrical hydrogen atoms of the nitrogens may be directed inward [N3..N3 2.86(2) 3_665] (h10 conformation) as predicted by the molecular orbital calculations (Dahaoui-Gindrey, Lecomte & Guilard, 1998).
;
_publ_section_exptl_prep
;
1,4,7-triazacyclononane was purchased from Aldrich Chemical Company. The compound was transferred to a clean vial and gently heated past its melting point (317K) to 320K.
....
;

_publ_section_exptl_refinement
;
Systematic reflection conditions, for the data set, suggested the space group P-3c1. ...
....
;
_publ_section_references
;
Barbour, L.J.,(2001) J. Supramol. Chem. 1, 189-191.

Chaudhuri, P., Wieghardt, K. (1987) Prog. Inorg. Chem. 35, 329-436

;
_publ_section_figure_captions
;
Figure 1. View of 1,4,7-triazacyclononane (50% probability displacement ellipsoids)
;
_publ_section_acknowledgements
;
The X-ray diffractometers and crystallographic computing systems
.....
;
_publ_section_table_legends
;
Table 1. Fractional atomic coordinates and equivalent
isotropic displacement parameters (\%A^2^)

Table 2. Selected geometric parameters (\%A ,\%)
;



#---------- SECTION 2. COMPOUND(S) DETAILS ----------#

While typing I made a few mistakes that enCIFer has found. First at line 255 I left out the crystal description and at line 268 I left out the absorption correction details. On line 19 I entered conact instead of contact and at line 328 I input mixxed instead of mixed. I correct these mistakes and point to

check the CIF again. This time there are no errors and no warnings. Save the file and continue.

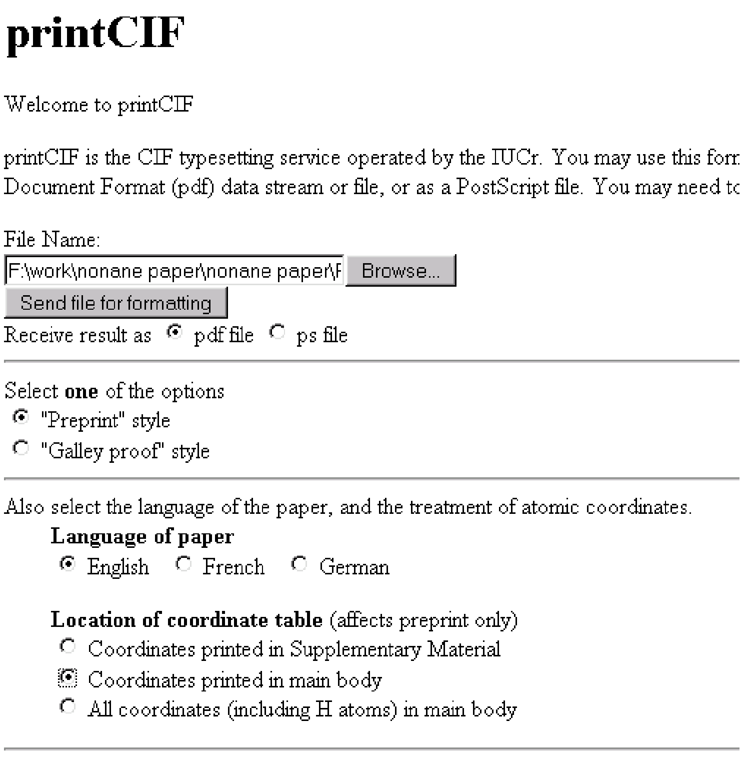
The visualize will display the structure for final approval.

Now go to CHECKCIF again and check for mistakes. Now I ask for publication check. The CIF passes the more rigorous test so its ready to go.

PRINTCIF : text layout program

http://journals.iucr.org/services/cif/printcif.html

Goto the on-line program PRINTCIF and input your CIF file. I choose to see a PDF file in the Preprint style, published in English with the coordinates in the text of the work. When you can view the results in adobe and save the file to your disk.



If all looks good then the CIF can be sent to the IUCR for publication.


PREVIEW 1

30 May 2006

Acta Cryst. (2004). C60, 000-000

The Crystal and Molecular Structure of 1,4,7-triazacyclononane hydrate

Abstract

The crystal and molecular structure of 1,4,7-triazacyclononane hydrate has been determined at 110K.

Comment

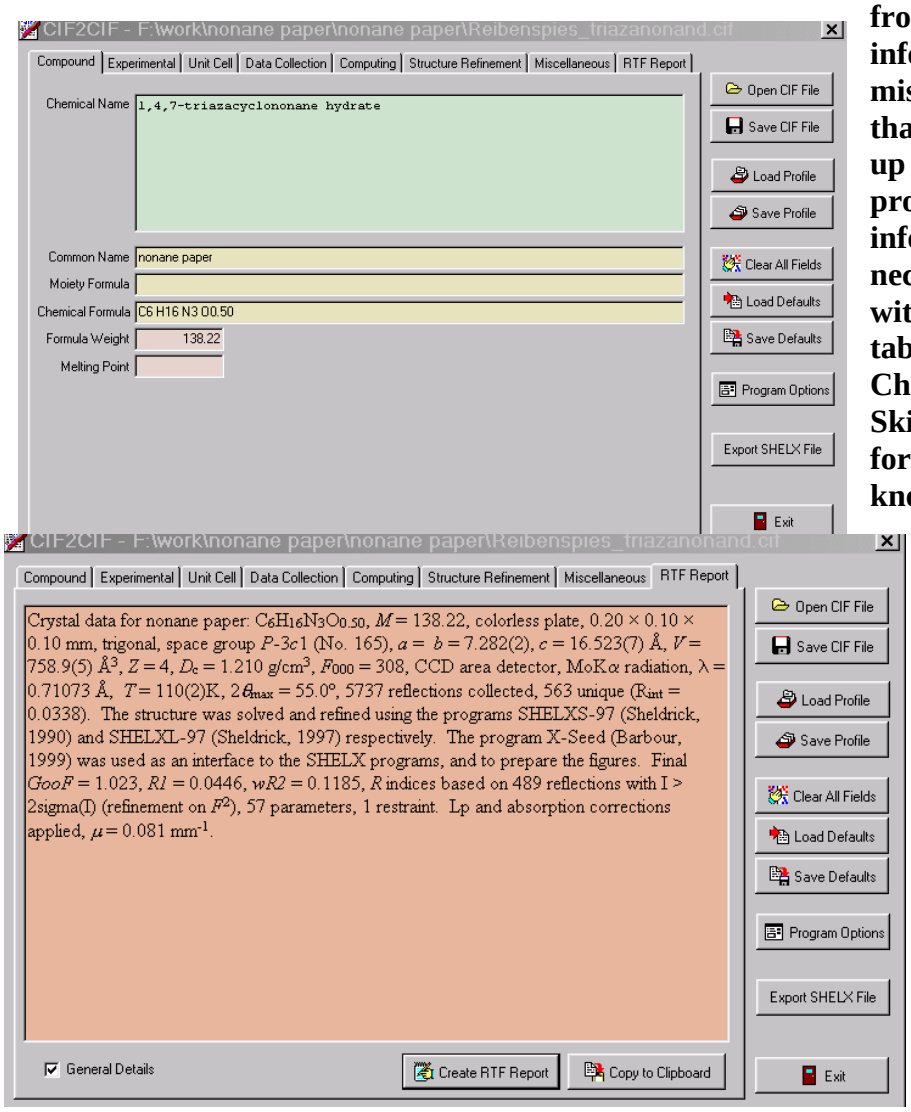
[illegible]

Experimental

1,4,7-triazacyclononane was purchased from Aldrich Chemical Company. The compound was transferred to a clean vial and gently heated past its melting point (317K) to 320K. The neat solution was then slowly cooled to 290K. Small needles formed overnight and a suitable crystal was chosen and quickly mounted and moved to a BRUKER SMART APEX diffractometer, equipped with a Nitrogen cold stream maintained at 110K.

Another handy program is the CIF2CIF found in the XSEED package of programs.

Start CIF2CIF and point to Open CIF file. The program will read the information from the CIF. If information is missing it will leave that line blank. It is up to you to provided the correct information where necessary. Starting with the Compound tab input the Chemical Name. Skip the Moiety formula (unless it is known) and goto to the Experimental tab. Add any information you deem necessary and continue to the Unit Cell Tab. Repeat for each tab. Finally point to the RTF report and create a RTF report. Copy this to Clipboard and then to your file.



RTF report .

Crystal data for nonane paper: $C_6H_{16}N_3O_{0.50}$, M = 138.22, colorless plate, 0.20 × 0.10 × 0.10 mm, trigonal, space group P-3c1 (No. 165), a = b = 7.282(2), c = 16.523(7) Å, V = 758.9(5) Å^3, Z = 4, D_c = 1.210 g/cm^3, F_{000} = 308, CCD area detector, MoKα radiation, λ = 0.71073 Å, T = 110(2)K, 2θ_{max} = 55.0°, 5737 reflections collected, 563 unique (R_{int} = 0.0338). The structure was solved and refined using the programs SHELXS-97 (Sheldrick, 1990) and SHELXL-97 (Sheldrick, 1997) respectively. The program X-Seed (Barbour, 1999) was used as an interface to the SHELX programs, and to prepare the figures. Final *GooF* = 1.023, *R1* = 0.0446, *wR2* = 0.1185, *R* indices based on 489 reflections with I >2sigma(I) (refinement on F^2), 57 parameters, 1 restraint. Lp and absorption corrections applied, μ = 0.081 mm^{-1}

Use this in the experimental or footnote section and be sure to add the correct references.