

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.

```
[N] Set next table number (currently 1)
[D] Set default directory for format files
[I] Crystal/atom tables from .cif
[F] Structure factor tables from .fcf
[Q] Quit

Option [R]:
Name of reference file [sucrose.pcf]: sucrose.pcf
Select data_sucrose ? <Y or N> [Y]:
Name of CIF file to be modified [sucrose.cif]:
Select data_sucrose ? <Y or N> [Y]:

Structure Code: sucrose

[S] Change structure code
[X] Print from SHELXTL XTEXT format file
[R] Use another CIF file to resolve ? items
```

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

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 Å | Wavelength=1.54178 |
|-------------------------------------|---------------|---------------------------------|--------------------|
| Cell: | a=7.7619(5) | b=0.7112(0) | c=10.0676(9) |
| | alpha=90 | beta=102.929(4) | gamma=90 |
| Volume | Calculated | 716.19(10) | Reported |
| Space group | P 21 | | P2(1) |
| Hall group | P 2yb | | ? |
| Moiety formula | C12 H22 O11 | | ? |
| Sum formula | C12 H22 O11 | | C12 H22 O11 |
| M _r | 342.30 | | 342.30 |
| D _x , g cm ⁻³ | 1.587 | | 1.587 |
| Z | 2 | | 2 |
| M _u (mm ⁻¹) | 1.239 | | 1.239 |
| F ₀₀₀ | 364.0 | | 364.0 |
| F ₀₀₀ ' | 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 = 1.507
 Measured density = 0.000
PLAT027 ALERT 3 A diffrs.refine.theta.full (no) Low 58.85 Deg.
PLAT025 ALERT 1 A No _chemical_absolute_configuration info given . 7
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.

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.

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/chemical_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
'rtf' (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), 'net' (pm etc.) or 'ger' (pm / German)
generate .tex output files in the (obsolete) SHELXTL XTEFI 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]: rtf
Crystal data table ? [Y]:
```

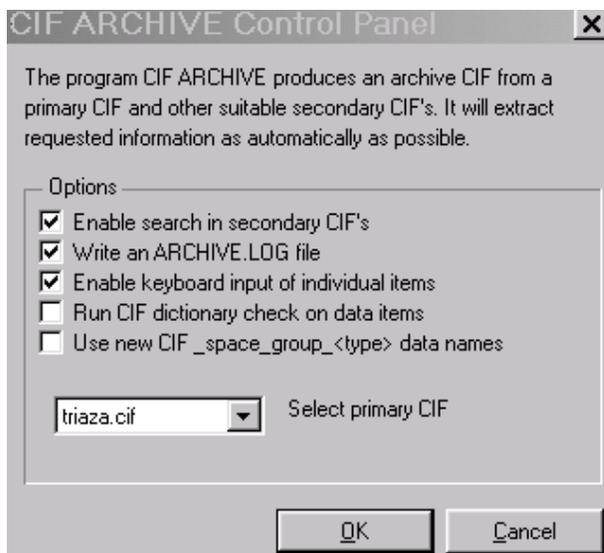
Choose T, for the file name enter *.rtf here I entered sucrose.rtf. for the extension enter rta. Choose Crystal Data Table, atomic coordinate table, bond/angle table full (not select), displacement table and hydrogen table. Answer N to the rest. Quit the program.

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.

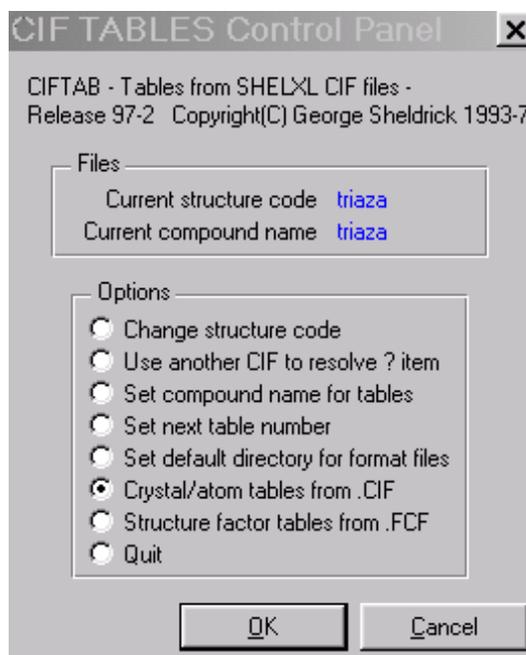
Point to Publish/CIFTABLES. Create the Archive.CIF file. Check the Enable keyboard input... and point to Ok. Do not ignore the Shelx97 values when asked.



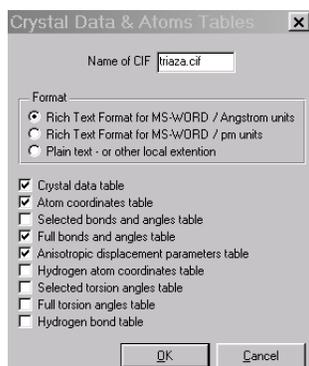
When prompted type auto.

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.

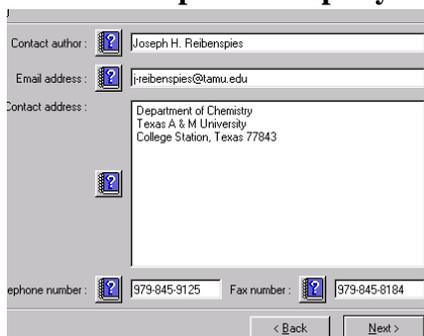
Rename the *.tex file that the program generated to *.rtf. Open WORD and read as a Rich Text Format document.

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

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

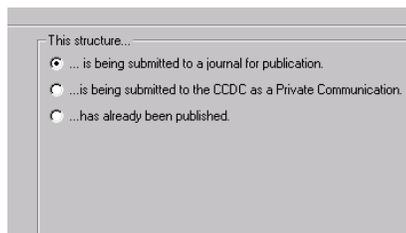
How to use enCIFer to edit CIF files for publication.

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



The screenshot shows a web form with the following fields: 'Contact author:' with the value 'Joseph H. Reibenspies'; 'Email address:' with the value 'jreibenspies@tamu.edu'; 'Contact address:' with the value 'Department of Chemistry, Texas A & M University, College Station, Texas 77843'; 'Telephone number:' with the value '979-845-9125'; and 'Fax number:' with the value '979-845-8184'. There are 'Back' and 'Next >' buttons at the bottom.

Next enter the authors etc.



The dialog box titled 'This structure...' contains three radio button options: the first is selected and reads '... is being submitted to a journal for publication.'; the second is '... is being submitted to the CCDC as a Private Communication.'; and the third is '... has already been published.'

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 & Guillard, 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

;

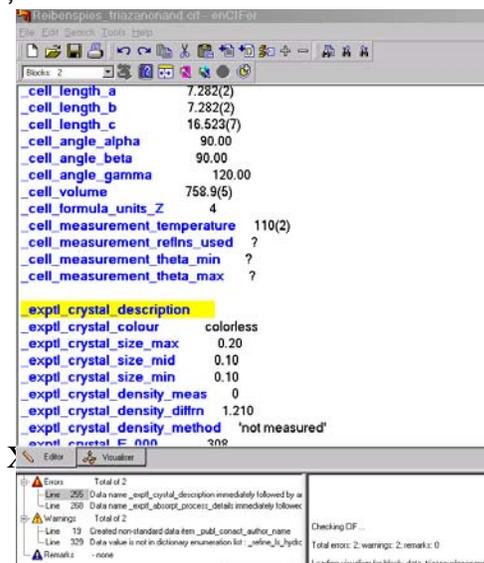
Table 1. Fractional atomic coordinates and equivalent

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

printCIF

Welcome to printCIF

printCIF is the CIF typesetting service operated by the IUCr. You may use this for: Document Format (pdf) data stream or file, or as a PostScript file. You may need to

File Name:

F:\work\anonane paper\anonane paper\ Browse...

Send file for formatting

Receive result as pdf file ps file

Select one of the options

- "Preprint" style
 "Galley proof" style

Also select the language of the paper, and the treatment of atomic coordinates.

Language of paper

- English French German

Location of coordinate table (affects preprint only)

- Coordinates printed in Supplementary Material
 Coordinates printed in main body
 All coordinates (including H atoms) in main body

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

20 May 2006

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

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

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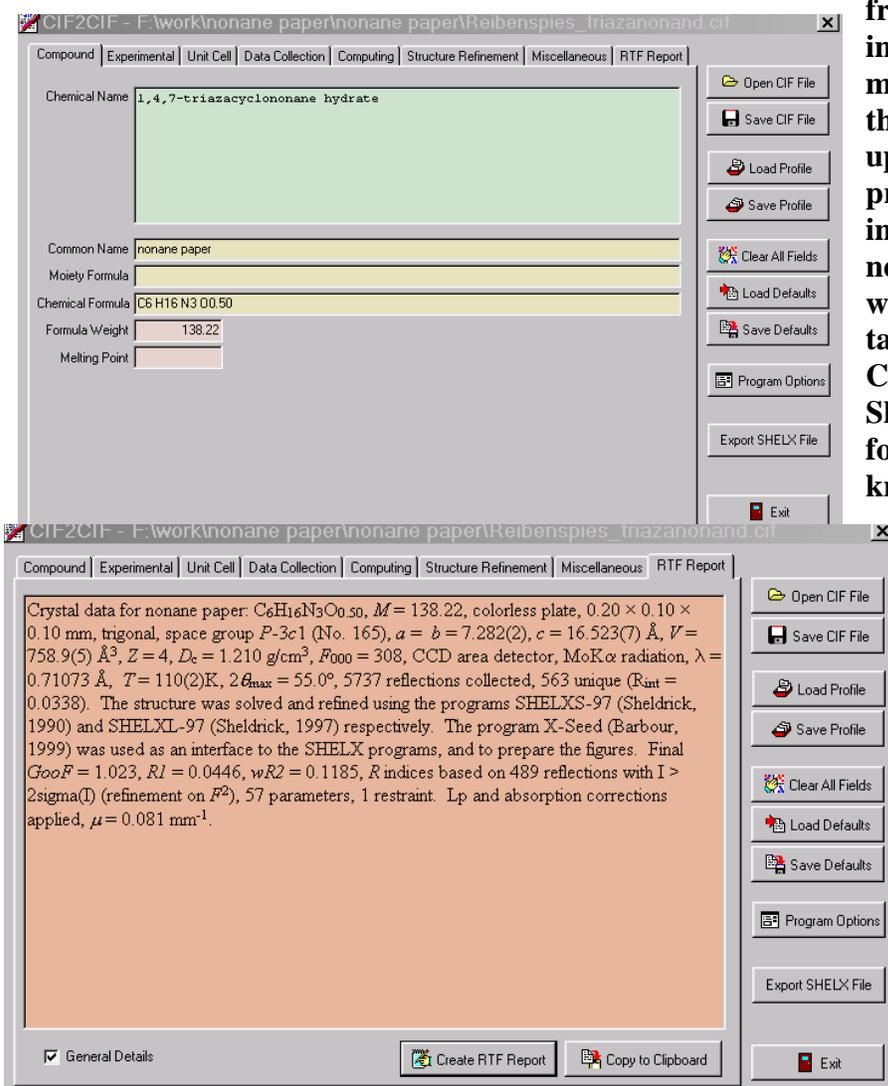
Abstract
The crystal and molecular structure of 1,4,7-triazacyclononane hydrate has been determined at 110K.

Comment
1,4,7-triazacyclononane ($[H_{10}N_3]$) forms a variety of complexes with metallic and nonmetallic elements and has been extensively reviewed (Stamandri & Weylandt, 1977). Only 12 of the $[H_{10}N_3]$ molecule is unique with the remaining atoms generated by inherent symmetry. The structure of $[H_{10}N_3]$, reported in this manuscript, displays the trigonal $[33-3]$ (R0) ring conformation (Dale, 1972; Ribeiro-Claes, Assaki, Marques & Teixeira-Dias, 1996; Meyer) and is similar to the ring conformation seen in the protonated 1,4,7-triazacyclononane $[H_{11}N_3]^+$ ($[H_4][H_{10}N_3]$) $[B(OH)_3]_2[NO_3]$ (Fleming, 5 January & Slav, 1993). The results reported herein confirm the molecular orbital ab initio calculations that predicts the $[33-3]$ conformation for $[H_{10}N_3]$ (Dabasi-Gladys, Lescotte & Galarud, 1996). An included water molecule (1/2 mole per mole of $[H_{10}N_3]$) is observed and forms a hydrogen bond with the N atoms of the $[H_{10}N_3]$ ($N \cdots O = 2.99(2)$ Å); the H atoms on the nitrogen was located in a difference Fourier map and is directed away from the center of the ring system and toward the included water (R0 conformation). However because of the high symmetry of the molecule and the site symmetry, a doublet in the hydrogen position with one of the three symmetrical H atoms of the N atoms may be directed toward $N \cdots O = 2.98(2)$ Å (R0 conformation) as predicted by the molecular orbital calculations (Dabasi-Gladys, Lescotte & Galarud, 1996).

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; liquid over the heated container and a suitable crystal was chosen and quickly mounted and sealed 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, $0.20 \times 0.10 \times 0.10$ mm, trigonal, space group $P-3c1$ (No. 165), $a = b = 7.282(2)$, $c = 16.523(7)$ Å, $V = 758.9(5)$ Å³, $Z = 4$, $D_c = 1.210$ g/cm³, $F_{000} = 308$, CCD area detector, MoK α radiation, $\lambda = 0.71073$ Å, $T = 110(2)$ K, $2\theta_{max} = 55.0^\circ$, 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 $Goof = 1.023$, $RI = 0.0446$, $wR2 = 0.1185$, R indices based on 489 reflections with $I > 2\sigma(I)$ (refinement on F^2), 57 parameters, 1 restraint. Lp and absorption corrections applied, $\mu = 0.081$ mm⁻¹

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