Crystallographic Information Files and Report Generation

J. Reibenspies,
N. Bhuvanesh

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

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

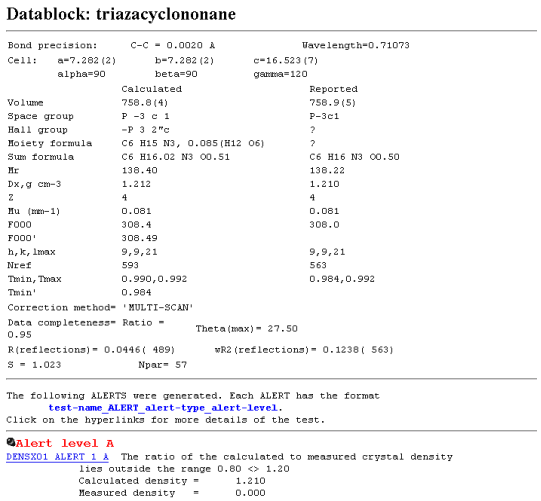
CIF TOOLS

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 to it gives me the ratio ... outside of range. This is ok.

My CIF checks out so I can use it in the next program



Datablock: triazacyclononane

Bond precision: C-C = 0.0020 A Wavelength=0.71073
Cell: a=7.282(2) b=7.282(2) c=16.523(7)
alpha=90 beta=90 gamma=120

	Calculated	Reported
Volume	758.8(4)	758.9(5)
Space group	P -3 c 1	P-3c1
Hall group	-P 3 2"c	?
Moiety formula	C6 H15 N3, 0.085(H12 O6)	?
Sum formula	C6 H16.02 N3 O0.51	C6 H16 N3 O0.50
Mr	138.40	138.22
Dx,g cm-3	1.212	1.210
Z	4	4
Mu (mm-1)	0.081	0.081
F000	308.4	308.0
F000'	308.49	
h,k,lmax	9,9,21	9,9,21
Nref	593	563
Tmin,Tmax	0.990,0.992	0.984,0.992
Tmin'	0.984	

Correction method= 'MULTI-SCAN'
Data completeness= Ratio = 0.95 Theta(max)= 27.50
R(reflections)= 0.0446(489) wR2(reflections)= 0.1238(563)
S = 1.023 Npar= 57

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.

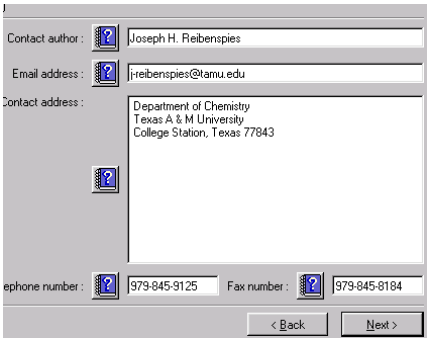
Alert level A
DENSZ01_ALERT_1_A The ratio of the calculated to measured crystal density lies outside the range 0.80 <> 1.20
Calculated density = 1.210
Measured density = 0.000

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

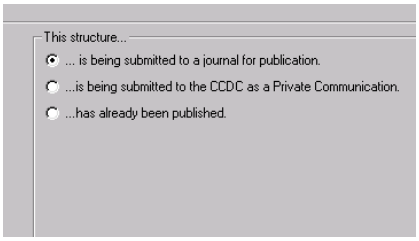
http://www.ccdc.cam.ac.uk/free_services/encifer/

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.

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;
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)
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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.

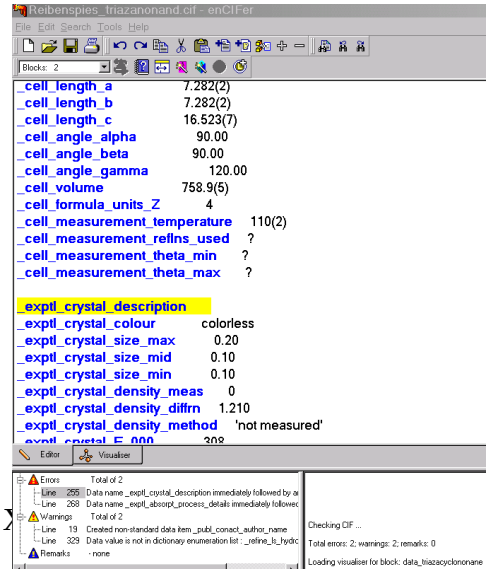
Chaudhuri, P., Wieghardt, K. (1987) Prog. Inorg. Chem. 35, 329-436

;
_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.

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

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Location of coordinate table (affects preprint only)
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Coordinates printed in main body
All coordinates (including H atoms) in main body

PREVIEW 1

30 May 2006

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

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

J. H. Reibenspies

Department of Chemistry, Texas A & M University, College Station, Texas 77843-3255. E-mail: j-reibenspies@tamu.edu

Abstract

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

Comment

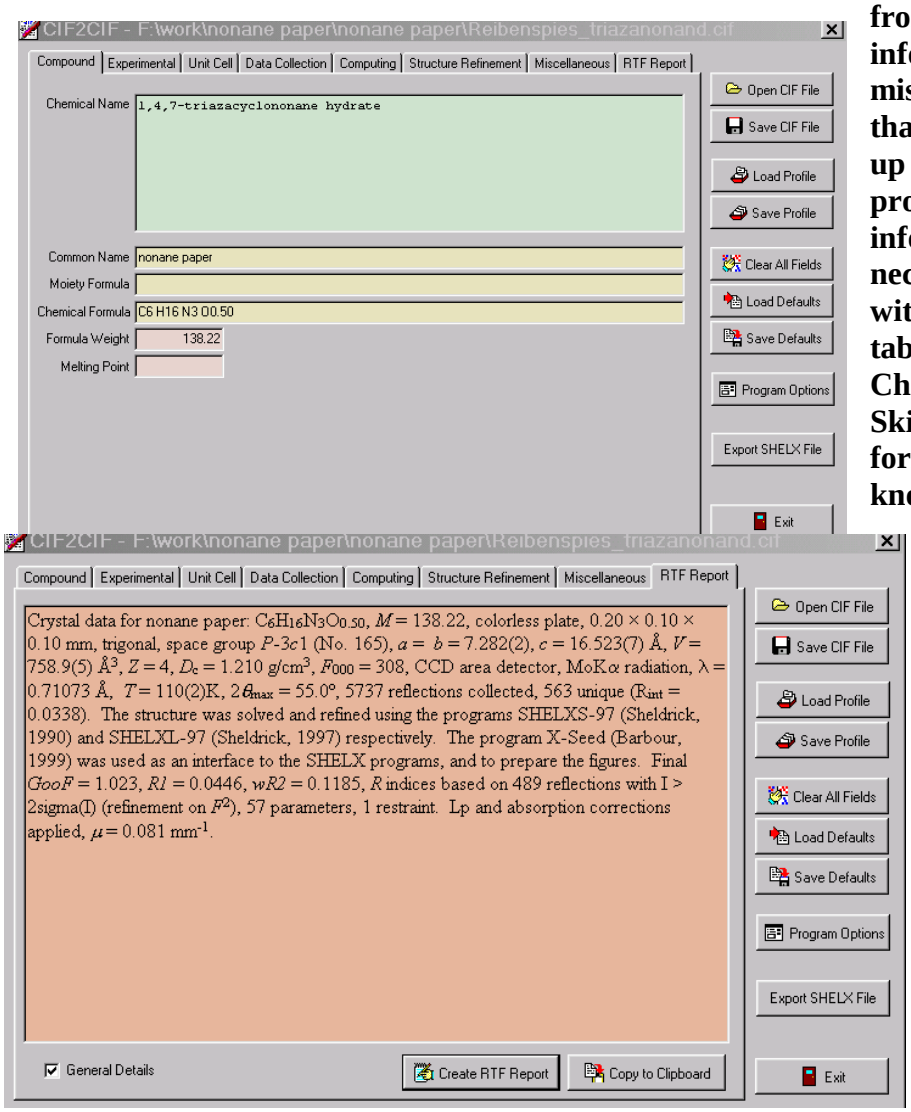
1,4,7-triazacyclononane ([9]aneN$_3$) forms a variety of complexes with metallic and nonmetallic elements and has been extensively reviewed (Chaudhuri & Wieghardt, 1987). Only 1/3 of the [9]aneN$_3$ molecule is unique with the remaining atoms generated by inherent symmetry. The structure of [9]aneN$_3$, reported in this manuscript, displays the triangular [333-B] (600) ring conformation (Dale, 1973; Ribeiro-Claro, Amado, Marques & Teixeira-Dias, 1996; Meyer) and is similar to the ring conformations seen in the protonated 1,4,7-triazacyclononane [333-A] ([H$_3$[9]aneN$_3$] [tr(H$_2$O)$_4$Cl$_2$]SO$_4$) (Flensburg, S imonsen & Skov, 1993). The results reported herin confirm the molecular orbital *ab initio* calculations that predicts the [333-B] conformation for ([9]aneN$_3$) (Dalsoni-Gindrey, Leconte & Guilard, 1998). An included water molecule (1/2 mole per mole of [9]aneN$_3$) is observed and forms a hydrogen bond with the N atom of the [9]-aneN$_3$ [N3···O1 2.69 (2) Å]. The H atom on the nitrogen was located in a difference fourier map and is directed away from the center of the ring systems and toward the the included water (600 conformation). However because of the high symmetry of the molecule and the site symmetry, a disorder in the hydrogen position with one of the three symmetrical H atoms of the N atoms may be directed inward [N3···N3 2.86 (2) 3_665] (h10 conformation) as predicted by the molecular orbital calculations (Dalsoni-Gindrey, Leconte & Guilard, 1998).

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 260K. 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, $2\theta_{max}$ = 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.