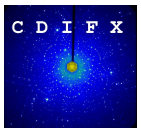
Electronic Supplementary Material (ESI) for Physical Chemistry Chemical Physics.
This journal is © the Owner Societies 2014



LaTeXreport created by T.R. [CDIFX/ISCR] - Date: 25-10-2013 at 15:15:55
Working directory: x:\structures\2012\JL_Fillaut\PS144_150K_06mars12
Input CIF file : archive.cif
CRYSCAL version : Sept. 2013 [T. Roisnel (CDIFX / ISCR UMR6226 - Rennes)]

PS144: Crystal structure report

X-ray crystallographic study

(C_{16} H_{16} N_4); $M = 264.33$. APEXII, Bruker-AXS diffractometer, $Mo - K_\alpha$ radiation (λ = 0.71073 Å), $T = 150(2)K$; monoclinic $P2_1/n$ (I.T.#14), $a = 5.7857(5), b = 16.921(2), c = 13.7060(17)$ Å, $\beta; = 98.453(4)^\circ$, $V = 1327.2(3)$ Å^3, $Z = 4$, $d = 1.323$ $g.cm^{-3}$, $\mu = 0.082$ mm^{-1}. The structure was solved by direct methods using the SIR97 program [1], and then refined with full-matrix least-square methods based on F^2 (SHELXL-97) [2] with the aid of the WINGX [3] program. All non-hydrogen atoms were refined with anisotropic atomic displacement parameters. H atoms were finally included in their calculated positions. A final refinement on F^2 with 3038 unique intensities and 183 parameters converged at $\omega R(F^2) = 0.1066$ ($RF = 0.0444$) for 2515 observed reflections with ($I > 2\sigma$).

1. A. Altomare, M. C. Burla, M. Camalli, G. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna, J. Appl. Cryst. (1999) 32, 115-119
2. Sheldrick G.M., Acta Cryst. A64 (2008), 112-122
3. L. J. Farrugia, J. Appl. Cryst., 2012, 45, 849-854

Structural data

Empirical formula	$C_{16}H_{16}N_4$
Formula weight	264.33
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system, space group	monoclinic, $P\ 2_1/n$
Unit cell dimensions	a = 5.7857(5) Å, α = 90 °
	b = 16.921(2) Å, β = 98.453(4) °
	c = 13.7060(17) Å, γ = 90 °
Volume	1327.2(3) $Å^3$
Z, Calculated density	4 , 1.323 ($g.cm^{-1}$)
Absorption coefficient	0.082 mm^{-1}
F(000)	560
Crystal size	0.58 x 0.25 x 0.08 mm
Crystal color	colourless
Theta range for data collection	3.76 to 27.48 °
h_min, h_max	-4 , 7
k_min, k_max	-21 , 21
l_min, l_max	-17 , 17
Reflections collected / unique	11435 / 3038 [^{a}R(int) = 0.0529]
Reflections [$I > 2\sigma$]	2515
Completeness to theta_max	0.994
Absorption correction type	multi-scan
Max. and min. transmission	0.993 , 0.878
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3038 / 0 / 183
^{b}Goodness-of-fit	1.042
Final R indices [$I > 2\sigma$]	$^cR_1$ = 0.0444, $^dwR_2$ = 0.1066
R indices (all data)	$^cR_1$ = 0.0546, $^dwR_2$ = 0.1144
Largest diff. peak and hole	0.251 and -0.278 $\overline{e}.Å^{-3}$

$^aR_{int} = \frac{\sum |F_o^2 - \langle F_o^2 \rangle|}{\sum F_o^2}$

$^bS = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{n-p} \right\}^{1/2}$

$^cR_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$

$^dwR_2 = \left\{ \frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right\}^{1/2}$

$w = 1./[\sigma(F_o^2) + (aP)^2 + bP]$ with $P = [2F_c^2 + Max(F_o^2, 0)]/3$

Atomic coordinates, site occupancy (%) and equivalent isotropic displacement parameters ($A^2 \times 10^3$). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	occ.	U(eq)
C1	0.4721(2)	0.19391(8)	-0.18914(10)	1	0.0245(3)
H1	0.3275	0.1681	-0.1861	1	0.029
C2	0.4939(2)	0.24414(9)	-0.26737(10)	1	0.0290(3)
H2	0.3645	0.2525	-0.3176	1	0.035
C3	0.7052(2)	0.28224(8)	-0.27234(10)	1	0.0279(3)
H3	0.7193	0.3178	-0.3247	1	0.033
C4	0.8947(2)	0.26782(8)	-0.20029(10)	1	0.0258(3)
H4	1.0403	0.2926	-0.2045	1	0.031
C5	0.8738(2)	0.21764(7)	-0.12220(10)	1	0.0227(3)
H5	1.0052	0.2081	-0.0733	1	0.027
C6	0.6608(2)	0.18105(7)	-0.11494(9)	1	0.0196(3)
C7	0.6310(2)	0.13195(7)	-0.02905(9)	1	0.0190(3)
N8	0.41426(19)	0.11726(7)	-0.00460(8)	1	0.0229(3)
N9	0.43670(19)	0.07473(7)	0.07642(8)	1	0.0230(3)
N10	0.66811(18)	0.06246(6)	0.10475(8)	1	0.0191(2)
C11	0.7938(2)	0.09688(7)	0.04070(9)	1	0.0203(3)
H11	0.9588	0.0968	0.0433	1	0.024
C12	0.7471(2)	0.02002(7)	0.19363(9)	1	0.0196(3)
C13	0.9560(2)	-0.02172(8)	0.20460(10)	1	0.0224(3)
H13	1.0481	-0.0223	0.1526	1	0.027
C14	1.0303(2)	-0.06245(8)	0.29102(10)	1	0.0232(3)
H14	1.1727	-0.0912	0.2974	1	0.028
C15	0.8992(2)	-0.06213(7)	0.36969(9)	1	0.0220(3)
C16	0.6868(2)	-0.02038(8)	0.35562(10)	1	0.0245(3)
H16	0.5921	-0.0201	0.4067	1	0.029
C17	0.6124(2)	0.02035(8)	0.26909(10)	1	0.0229(3)
H17	0.4689	0.0485	0.2615	1	0.027
N18	0.9792(2)	-0.09865(7)	0.45877(9)	1	0.0290(3)
C19	1.1574(3)	-0.15858(11)	0.46515(12)	1	0.0423(4)
H19A	1.0904	-0.2074	0.4346	1	0.063
H19B	1.2184	-0.1686	0.5346	1	0.063
H19C	1.2845	-0.1406	0.4306	1	0.063
C20	0.8266(3)	-0.10311(9)	0.53346(10)	1	0.0308(3)
H20A	0.7749	-0.0498	0.5481	1	0.046
H20B	0.9114	-0.1268	0.5936	1	0.046
H20C	0.6903	-0.1358	0.5092	1	0.046

Anisotropic displacement parameters (Å^2)

The anisotropic displacement factor exponent takes the form: $-2\pi[h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12}]$

Atom	U11	U22	U33	U23	U13	U12
C1	0.0200(6)	0.0286(7)	0.0246(7)	0.0008(5)	0.0024(5)	-0.0009(5)
C2	0.0267(7)	0.0351(8)	0.0244(7)	0.0053(6)	0.0014(6)	0.0036(6)
C3	0.0328(8)	0.0266(7)	0.0260(7)	0.0060(5)	0.0100(6)	0.0043(6)
C4	0.0248(7)	0.0235(7)	0.0308(7)	0.0002(5)	0.0100(6)	-0.0011(5)
C5	0.0210(6)	0.0222(6)	0.0248(7)	-0.0010(5)	0.0029(5)	0.0009(5)
C6	0.0217(6)	0.0171(6)	0.0205(6)	-0.0019(5)	0.0045(5)	0.0022(5)
C7	0.0189(6)	0.0174(6)	0.0206(6)	-0.0030(5)	0.0023(5)	0.0002(5)
N8	0.0207(5)	0.0252(6)	0.0230(6)	0.0019(4)	0.0041(4)	0.0011(4)
N9	0.0188(5)	0.0266(6)	0.0235(6)	0.0026(4)	0.0030(4)	0.0021(4)
N10	0.0172(5)	0.0198(5)	0.0202(5)	-0.0002(4)	0.0027(4)	0.0002(4)
C11	0.0195(6)	0.0207(6)	0.0211(6)	-0.0008(5)	0.0045(5)	-0.0017(5)
C12	0.0211(6)	0.0184(6)	0.0189(6)	0.0003(5)	0.0019(5)	-0.0010(5)
C13	0.0213(6)	0.0253(6)	0.0213(6)	-0.0015(5)	0.0051(5)	0.0010(5)
C14	0.0196(6)	0.0253(7)	0.0243(6)	-0.0012(5)	0.0015(5)	0.0034(5)
C15	0.0237(7)	0.0207(6)	0.0210(6)	-0.0002(5)	0.0012(5)	-0.0014(5)
C16	0.0248(7)	0.0266(7)	0.0234(7)	0.0016(5)	0.0081(5)	0.0014(5)
C17	0.0207(6)	0.0234(6)	0.0250(7)	0.0010(5)	0.0044(5)	0.0026(5)
N18	0.0319(7)	0.0328(7)	0.0225(6)	0.0050(5)	0.0048(5)	0.0082(5)
C19	0.0444(10)	0.0482(10)	0.0349(9)	0.0155(7)	0.0074(7)	0.0201(8)
C20	0.0312(8)	0.0371(8)	0.0241(7)	0.0079(6)	0.0038(6)	-0.0019(6)

Bond length [Å]

```
C1   - C2       = 1.3884(19)
C1   - C6       = 1.3955(18)
C1   - H1       = 0.95
C2   - C3       = 1.393(2)
C2   - H2       = 0.95
C3   - C4       = 1.385(2)
C3   - H3       = 0.95
C4   - C5       = 1.3855(18)
C4   - H4       = 0.95
C5   - C6       = 1.3958(18)
C5   - H5       = 0.95
C6   - C7       = 1.4715(17)
C7   - N8       = 1.3676(16)
C7   - C11      = 1.3742(18)
N8   - N9       = 1.3135(15)
N9   - N10      = 1.3541(15)
N10  - C11      = 1.3515(16)
N10  - C12      = 1.4300(16)
C11  - H11      = 0.95
C12  - C17      = 1.3833(18)
C12  - C13      = 1.3890(18)
C13  - C14      = 1.3832(18)
C13  - H13      = 0.95
C14  - C15      = 1.4063(18)
C14  - H14      = 0.95
C15  - N18      = 1.3858(16)
C15  - C16      = 1.4062(18)
C16  - C17      = 1.3844(18)
C16  - H16      = 0.95
C17  - H17      = 0.95
N18  - C19      = 1.4393(19)
N18  - C20      = 1.4487(18)
C19  - H19A     = 0.98
C19  - H19B     = 0.98
C19  - H19C     = 0.98
C20  - H20A     = 0.98
C20  - H20B     = 0.98
C20  - H20C     = 0.98
```

Angles [°]

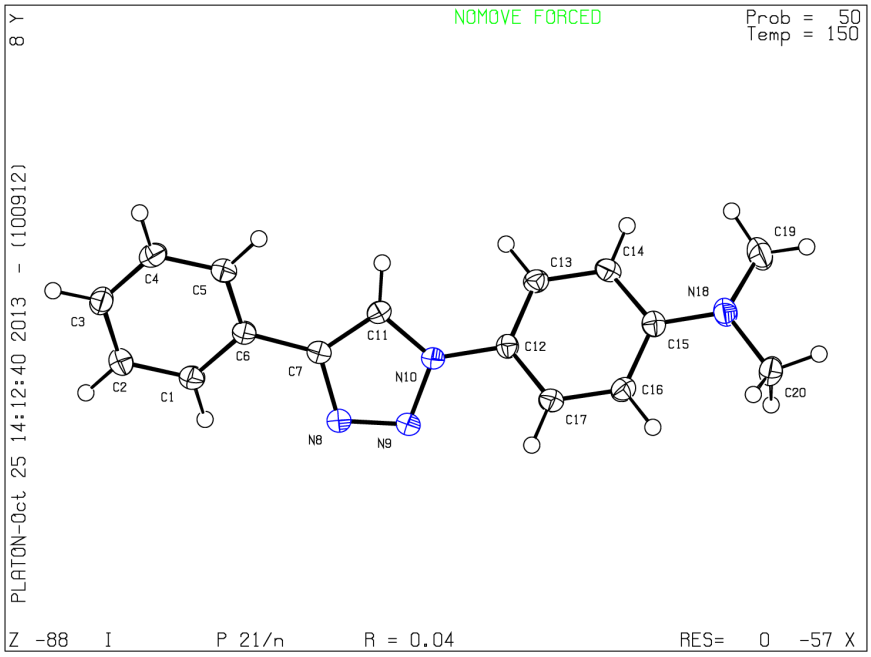
```
C2      - C1      - C6      = 120.54(13)
C2      - C1      - H1      = 119.7
C6      - C1      - H1      = 119.7
C1      - C2      - C3      = 120.08(13)
C1      - C2      - H2      = 120
C3      - C2      - H2      = 120
C4      - C3      - C2      = 119.49(13)
C4      - C3      - H3      = 120.3
C2      - C3      - H3      = 120.3
C3      - C4      - C5      = 120.59(12)
C3      - C4      - H4      = 119.7
C5      - C4      - H4      = 119.7
C4      - C5      - C6      = 120.34(12)
C4      - C5      - H5      = 119.8
C6      - C5      - H5      = 119.8
C1      - C6      - C5      = 118.91(12)
C1      - C6      - C7      = 119.99(11)
C5      - C6      - C7      = 121.06(11)
N8      - C7      - C11     = 108.09(11)
N8      - C7      - C6      = 121.21(11)
C11     - C7      - C6      = 130.64(12)
N9      - N8      - C7      = 109.06(10)
N8      - N9      - N10     = 107.28(10)
C11     - N10     - N9      = 110.57(10)
C11     - N10     - C12     = 129.41(11)
N9      - N10     - C12     = 119.99(10)
N10     - C11     - C7      = 104.99(11)
N10     - C11     - H11     = 127.5
C7      - C11     - H11     = 127.5
C17     - C12     - C13     = 119.87(12)
C17     - C12     - N10     = 119.37(11)
C13     - C12     - N10     = 120.76(11)
C14     - C13     - C12     = 120.13(12)
C14     - C13     - H13     = 119.9
C12     - C13     - H13     = 119.9
C13     - C14     - C15     = 121.34(12)
C13     - C14     - H14     = 119.3
C15     - C14     - H14     = 119.3
N18     - C15     - C16     = 121.12(12)
N18     - C15     - C14     = 121.73(12)
C16     - C15     - C14     = 117.11(12)
C17     - C16     - C15     = 121.53(12)
C17     - C16     - H16     = 119.2
C15     - C16     - H16     = 119.2
C12     - C17     - C16     = 120.02(12)
C12     - C17     - H17     = 120
C16     - C17     - H17     = 120
C15     - N18     - C19     = 120.74(12)
C15     - N18     - C20     = 119.10(12)
C19     - N18     - C20     = 115.20(12)
N18     - C19     - H19A    = 109.5
N18     - C19     - H19B    = 109.5
H19A    - C19     - H19B    = 109.5
N18     - C19     - H19C    = 109.5
H19A    - C19     - H19C    = 109.5
H19B    - C19     - H19C    = 109.5
N18     - C20     - H20A    = 109.5
N18     - C20     - H20B    = 109.5
H20A    - C20     - H20B    = 109.5
N18     - C20     - H20C    = 109.5
```

```
H20A     - C20      - H20C     = 109.5
H20B     - C20      - H20C     = 109.5
```

Torsion angles [°]

C6	- C1	- C2	- C3	= 0.0(2)
C1	- C2	- C3	- C4	= 1.9(2)
C2	- C3	- C4	- C5	= -1.8(2)
C3	- C4	- C5	- C6	= -0.2(2)
C2	- C1	- C6	- C5	= -1.91(19)
C2	- C1	- C6	- C7	= 175.88(12)
C4	- C5	- C6	- C1	= 2.02(19)
C4	- C5	- C6	- C7	= -175.75(12)
C1	- C6	- C7	- N8	= -19.20(18)
C5	- C6	- C7	- N8	= 158.54(12)
C1	- C6	- C7	- C11	= 163.96(13)
C5	- C6	- C7	- C11	= -18.3(2)
C11	- C7	- N8	- N9	= -0.22(14)
C6	- C7	- N8	- N9	= -177.69(11)
C7	- N8	- N9	- N10	= 0.46(14)
N8	- N9	- N10	- C11	= -0.55(14)
N8	- N9	- N10	- C12	= 177.66(10)
N9	- N10	- C11	- C7	= 0.41(14)
C12	- N10	- C11	- C7	= -177.58(11)
N8	- C7	- C11	- N10	= -0.12(14)
C6	- C7	- C11	- N10	= 177.04(12)
C11	- N10	- C12	- C17	= 150.94(13)
N9	- N10	- C12	- C17	= -26.89(17)
C11	- N10	- C12	- C13	= -29.46(19)
N9	- N10	- C12	- C13	= 152.71(12)
C17	- C12	- C13	- C14	= -0.45(19)
N10	- C12	- C13	- C14	= 179.95(11)
C12	- C13	- C14	- C15	= -0.6(2)
C13	- C14	- C15	- N18	= -175.91(12)
C13	- C14	- C15	- C16	= 1.63(19)
N18	- C15	- C16	- C17	= 175.97(12)
C14	- C15	- C16	- C17	= -1.59(19)
C13	- C12	- C17	- C16	= 0.49(19)
N10	- C12	- C17	- C16	= -179.90(12)
C15	- C16	- C17	- C12	= 0.6(2)
C16	- C15	- N18	- C19	= 162.51(14)
C14	- C15	- N18	- C19	= -20.0(2)
C16	- C15	- N18	- C20	= 8.63(19)
C14	- C15	- N18	- C20	= -173.92(12)

Structure visualisation



This structural report has been created through CRYSCAL (Sept. 2013). Please report bugs and problems to cdifx@univ-rennes1.fr