Mohamadi, F., Richards, N. G. J., Guida, W. C., Liskamp, R., Lipton, M., Caufield, C., Chang, G., Hendrickson, T. & Still, W. C. (1990). *J. Comput. Chem.* **11**, 440–467.

Sharp, K. A. & Nicholls, A. (1989). *DelPhi Manual*. Version 3.0. Department of Biochemistry and Molecular Biophysics, Columbia University, New York, USA.

Acta Cryst. (1997). C**53**, 143–144

5-(4-Hydroxyphenyl)tetrazole–Water (2/3)

Hugo Gallardo, Ieda M. Begnini and Ivo Vencato

Departamento Química, UFSC, 88040-900 Florianópolis, SC, Brazil. E-mail: vencato@qmc.ufsc.br

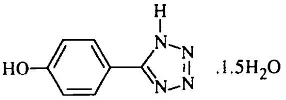
(Received 21 June 1996; accepted 8 October 1996)

Abstract

The title compound, $C_7H_6N_4O.1.5H_2O$, is a key intermediate in the synthesis of mesogens, which are derivatives of phenyltetrazole. The tetrazole and phenyl rings are planar to within 0.007 (2) Å, with a dihedral angle of 14.8 (1)° between the planes.

Comment

As part of our synthetic and structural studies of conventional liquid crystals and metallomesogens which contain a heterocyclic ring (Gallardo & Merlo, 1993; Gallardo & Favarin, 1993; Gallardo & Begnini, 1995), the structure of the title compound, (I), has been determined and is presented here.



(I)

A rather small number of structures with a tetrazole ring have been described (Destro & Soave, 1995; Gowda, Rudman & Acharya, 1982; Bray & White, 1979; Bradbury *et al.*, 1992). The bond distances and angles in the title molecule are similar to the values found in another phenyltetrazole (Gallardo, Meyer & Vencato, 1995). In the tetrazole ring, the N2—N3 distance of 1.286 (2) Å is clearly a double bond, significantly shorter than the N1—N2 and N3—N4 single bonds of 1.360 (2) and 1.344 (2) Å, respectively. These values are consistent with those observed previously for the tetrazole ring.

The structure consists of almost planar molecules joined by hydrogen bonds to water molecules, resulting in layers extending along the *bc* face of the unit cell (Table 3). The r.m.s. deviation of atoms C1–C7, O1 and N1–N4 from the least-squares plane through them is 0.106 Å. The hydrogen-bond geometry around the O*W*2 water molecule is quite planar, as can likewise be seen by the r.m.s. deviation of 0.120 Å from the plane through O*W*1, O*W*2, N2^{iv} and N4^{ii} (see Table 3 for symmetry codes). The hydrogen bonds to the O*W*1 water molecule are in an approximately tetrahedral configuration.

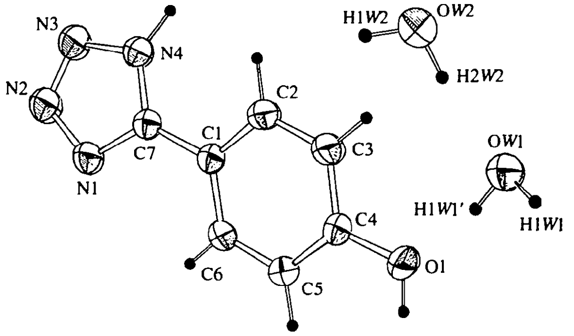


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title molecule was prepared by treating 4-hydroxybenzonitrile with NaN_3 in NH_4Cl/DMF. The reaction mixture was then heated and refluxed for 3 h, cooled and poured into 200 ml of water and crushed ice. The crude tetrazole was separated as a solid and recrystallized from a 1:1 mixture of benzene and ethanol. ^1H NMR (200 MHz, DMSO-d_6): δ 7.05 (*d*, 3H, J = 8.4 Hz, aromatic system), 7.95 (*d*, 2H, J = 8.4 Hz, aromatic system).

Crystal data

$C_7H_6N_4O.1.5H_2O$
$M_r = 189.18$
Monoclinic
$C2/c$
$a = 14.852\,(1)$ Å
$b = 9.910\,(1)$ Å
$c = 13.036\,(2)$ Å
$\beta = 113.749\,(8)°$
$V = 1756.2\,(3)$ Å^3
$Z = 8$
$D_x = 1.431$ Mg m^{-3}
$D_m = 1.42\,(2)$ Mg m^{-3}
D_m measured by flotation in CCl_4/benzene

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.59–13.92°$
$\mu = 0.112$ mm^{-1}
$T = 293\,(2)$ K
Prismatic
0.60 × 0.60 × 0.25 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1625 measured reflections
1552 independent reflections
1334 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0123$
$\theta_{\rm max} = 25°$
$h = -17 \rightarrow 16$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: −1.5%

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

Refinement

Refinement on F^2
$R(F) = 0.0335$
$wR(F^2) = 0.0916$
$S = 1.031$
1551 reflections
152 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.9685P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.034$
$\Delta\rho_{\max} = 0.169$ e Å^{-3}
$\Delta\rho_{\min} = -0.178$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.038 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.60988 (9)	0.03455 (12)	0.64819 (9)	0.0466 (4)
N1	0.65007 (10)	0.23416 (13)	0.19492 (10)	0.0402 (4)
N2	0.65184 (11)	0.33591 (14)	0.12573 (11)	0.0462 (4)
N3	0.64398 (11)	0.45117 (14)	0.16673 (11)	0.0470 (4)
N4	0.63653 (10)	0.42559 (13)	0.26429 (10)	0.0398 (4)
C1	0.63337 (10)	0.22477 (14)	0.37749 (11)	0.0311 (3)
C2	0.60061 (11)	0.2934 (2)	0.44894 (13)	0.0380 (4)
C3	0.59357 (12)	0.2288 (2)	0.53898 (13)	0.0394 (4)
C4	0.61844 (10)	0.09348 (15)	0.55846 (11)	0.0339 (4)
C5	0.65219 (11)	0.0242 (2)	0.48882 (12)	0.0365 (4)
C6	0.66007 (11)	0.08935 (15)	0.39946 (12)	0.0360 (4)
C7	0.64051 (10)	0.29221 (14)	0.28151 (11)	0.0325 (3)
O*W*1	1/2	0.1661 (2)	3/4	0.0446 (4)
O*W*2	0.37968 (13)	0.36321 (14)	0.61262 (12)	0.0699 (5)

Table 2. *Selected geometric parameters (Å, °)*

O1—C4	1.358 (2)	N3—N4	1.344 (2)
N1—C7	1.325 (2)	N4—C7	1.338 (2)
N1—N2	1.360 (2)	C1—C7	1.459 (2)
N2—N3	1.286 (2)		
C7—N1—N2	106.3 (1)	O1—C4—C5	122.4 (1)
N3—N2—N1	110.7 (1)	O1—C4—C3	117.6 (1)
N2—N3—N4	106.4 (1)	N1—C7—N4	107.4 (1)
C7—N4—N3	109.3 (1)	N1—C7—C1	127.0 (1)
C2—C1—C7	121.1 (1)	N4—C7—C1	125.6 (1)
C6—C1—C7	120.2 (1)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O1—HO1···N1$^{\rm i}$	0.85 (2)	1.91 (2)	2.743 (2)	166 (2)
N4—HN4···O*W*2$^{\rm ii}$	0.96 (2)	1.75 (2)	2.706 (2)	178 (2)
O*W*1—H1*W*1···O1$^{\rm iii}$	0.88 (2)	1.94 (2)	2.805 (1)	167 (2)
O*W*2—H1*W*2···N2$^{\rm iv}$	0.79 (2)	2.18 (2)	2.962 (2)	172 (2)
O*W*2—H2*W*2···O*W*1	0.85 (2)	1.92 (2)	2.764 (2)	169 (2)

Symmetry codes: (i) $x, -y, \frac{1}{2}+z$; (ii) $1-x, 1-y, 1-z$; (iii) $1-x, y, \frac{3}{2}-z$; (iv) $1-x, y, \frac{1}{2}-z$.

One water molecule was placed in a special position. All H atoms were located from $\Delta\rho$ maps and refined isotropically with a common displacement factor.

Data collection: *CAD*-4 *Express* (Enraf–Nonius, 1992). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93. The calculations were performed on a DEC 3000 AXP and PC/486 computer.

This work was supported by grants from PADCT, CNPq, FINEP and CAPES.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1405). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bradbury, R. H., Allot, C. P., Dennis, M., Fisher, E., Major, J. S., Masek, B. B., Pearce, R. J., Rankine, N., Revill, J. M., Roberts, D. A. & Russell, S. T. (1992). *J. Med. Chem.* **35**, 4027–4038.

Bray, D. D. & White, J. G. (1979). *Acta Cryst.* **B35**, 3089–3091.

Destro, R. & Soave, R. (1995). *Acta Cryst.* **C51**, 1383–1385.

Enraf–Nonius (1992). *CAD*-4 *Express Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Gallardo, H. & Begnini, I. M. (1995). *Mol. Cryst. Liq. Cryst.* **258**, 85–94.

Gallardo, H. & Favarin, I. E. (1993). *Liq. Cryst.* **13**, 115–125.

Gallardo, H. & Merlo, A. (1993). *Synth. Commun.* **23**, 2159–2169.

Gallardo, H., Meyer, E. & Vencato, I. (1995). *Acta Cryst.* **C51**, 2430–2432.

Gowda, D. S. S., Rudman, R. & Acharya, K. R. (1982). *Acta Cryst.* **B38**, 2487–2490.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1997). **C53**, 144–146

1,2-Dihydronaphtho[3,4-*b*]cyclopenteno-[1,2-*e*]-4*H*-pyran-4-one

ZHENGBAI CAO,[a] XUEGIN CAO,[a] DE-CHUN ZHANG,[a] FAN XU[a] AND KAIBEI YU[b]

[a]*Department of Chemistry, Suzhou University, Suzhou 215006, People's Republic of China, and* [b]*Center of Analysis and Determination, Chengdu Branch, Academic Sinica, Chengdu 610041, People's Republic of China*

(*Received 26 July 1996; accepted 10 October 1996*)

Abstract

In the title compound, 2,3,5,6-tetrahydrocyclopenta[1,2-*e*]naphtho[1,2-*b*]pyran-4(1*H*)-one, $C_{16}H_{14}O_2$, the planar pyrone ring makes dihedral angles of less than 10° with the best planes of each of the other three rings. The maximum deviation for each of the planes through the non-H atoms is less than 0.225 (4) Å. Molecules in the crystal are packed in columns. Molecular-dynamics cal-