Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[(2-Hydroxybenzylidene)hydrazinocarbonyl]-1*H*-1,2,4-triazole ethanol solvate

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Received 31 January 2005
Accepted 24 February 2005
Online 4 March 2005

Key indicators

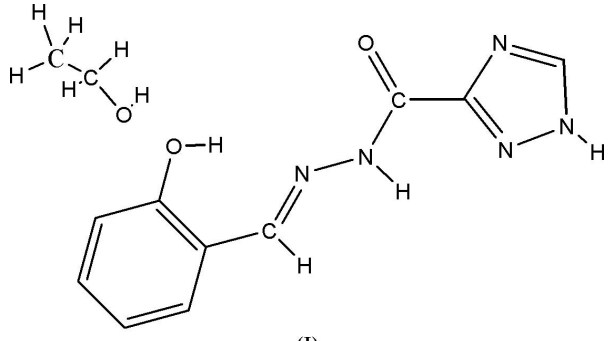
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.076
wR factor = 0.229
Data-to-parameter ratio = 11.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{10}H_9N_5O_2\cdot C_2H_6O$, was synthesized by the reaction of 3-hydrazino-1*H*-1,2,4-triazole with 2-hydroxybenzaldehyde in ethanol. The crystal structure involves intermolecular O—H···O, N—H···O and N—H···N hydrogen bonds; intramolecular O—H···N and N—H···N hydrogen bonds are also found.

Comment

Azole derivatives, such as derivatives of pyrazole, imidazole, triazole (including benzotriazole), tetrazole, indole, *etc*., exhibit extensive biological activities. They have become the central focus in the study of agricultural chemicals, adjustment reagents for plant growth, and so on (Haddock & Hopwood, 1982). A Schiff base is a good type of biologically active substructure and a study of one type of triazole Schiff base has been reported (Sauter *et al.*, 1991). The hydrazinecarbonyl grouping has also been shown to be bioactive (Zhi *et al.*, 2003). Furthermore, some structures of triazoles containing the hydrazinecarbonyl group have been reported (Pan & Yang, 2005*a*,*b*; Yang & Pan, 2004). In a search for more effective antibacterial medicines, we have synthesized the title compound, (I).



(I)

The title molecule (Fig. 1) is essentially planar with an r.m.s. deviation of 0.0012 Å. Bond lengths and angles are unexceptional (Allen *et al.*, 1987).

Intramolecular O—H···N and N—H···N hydrogen bonds are observed, forming six- and five-membered rings, respectively (Fig. 2). Intermolecular N—H···N and N—H···O hydrogen bonds are present (Table 1); these are linked, forming a chain in the *b*-axis direction (Fig. 3)

Experimental

3-Hydrazino-1*H*-1,2,4-triazole (0.02 mol, 2.54 g) was dissolved in anhydrous ethanol (50 ml) at room temperature. 2–Hydroxybenz-

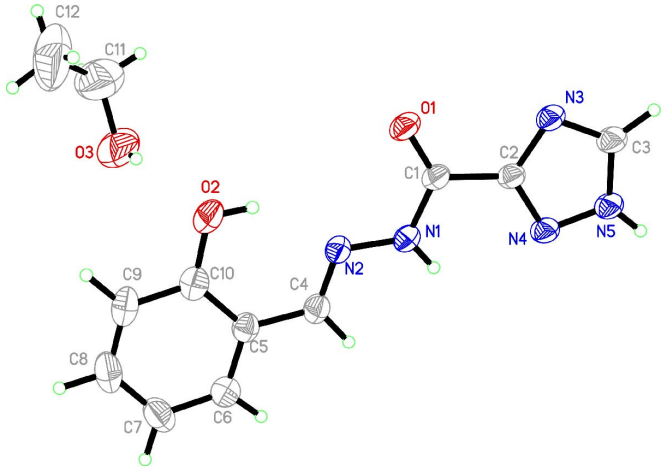


Figure 1
The structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

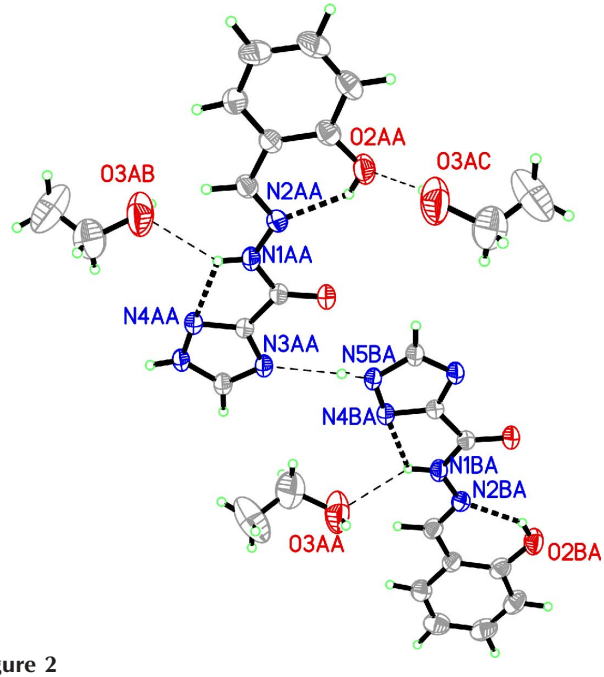


Figure 2
The packing of (I), showing the hydrogen bonds as dashed lines.

aldehyde (0.02 mol, 2.44 g) was added and the mixture was refluxed for 4 h; the precipitate was collected by filtration and washed with ethanol. The product was recrystallized from ethanol and dried under reduced pressure to give the title compound. The latter (2.0 mmol, 0.46 g) was dissolved in dimethylformamide (30 ml) and was kept at room temperature for 38 d. Block-shaped colourless single crystals formed, which were washed with distilled water.

Crystal data

$C_{10}H_9N_5O_2\cdot C_2H_6O$
M_r = 277.29
Monoclinic, $P2_1/c$
a = 15.554 (6) Å
b = 10.012 (4) Å
c = 8.856 (4) Å
β = 96.217 (8)°
V = 1370.9 (10) Å^3
Z = 4
D_x = 1.344 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 950 reflections
θ = 4.8–42.9°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colourless
0.50 × 0.35 × 0.11 mm

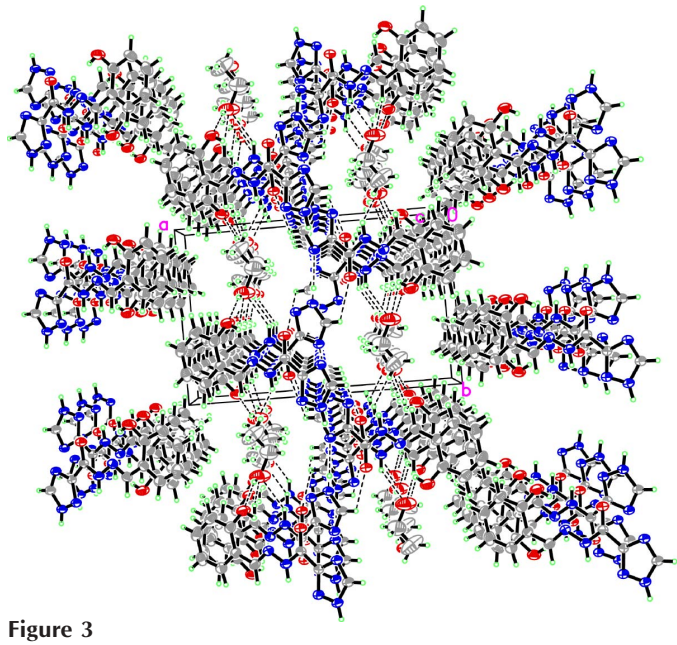


Figure 3
The packing of (I), viewed down the c axis, showing hydrogen-bonded chains (dashed lines).

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.959, T_{max} = 0.989
6982 measured reflections
2541 independent reflections
1287 reflections with $I > 2\sigma(I)$
R_{int} = 0.145
θ_{max} = 25.5°
$h = -18 \rightarrow 18$
$k = -9 \rightarrow 12$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.229
S = 0.96
2541 reflections
219 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0984P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.002 (2)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···N3^{i}	0.85 (4)	2.05 (4)	2.893 (5)	171 (4)
N1—H1···O3^{ii}	0.84 (2)	2.28 (3)	3.005 (5)	145 (4)
O2—H2···N2	0.85 (2)	1.92 (3)	2.625 (4)	140 (4)
N1—H1···N4	0.84 (2)	2.30 (4)	2.739 (4)	113 (3)
O3—H3A···O2	0.82	2.23	3.009 (6)	159

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{5}{2}-z$; (ii) $x, \frac{3}{2}-y, \frac{1}{2}+z$.

H atoms of the triazole molecule were located in a difference map and their parameters were refined: N—H = 0.84 (2) or 0.85 (4) Å and O—H = 0.85 (2) Å, and the C—H distances lie in the range 0.92 (4)–0.99 (4) Å. The H atoms of the solvent molecule were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.96 or 0.97 Å, O—H = 0.82 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl or O). The crystal was unstable and diffracted rather weakly; hence the high value of R_{int} of 0.145. The displacement

parameters of the solvent atoms are large, but a disordered model was not considered.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support by the Zhejiang Provincial Natural Science Foundation of China (No. M203115).

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