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3,5-Bis(salicylideneamino)-1*H*-1,2,4-triazole methanol solvate

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Key indicators

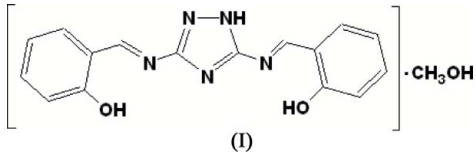
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.065
wR factor = 0.159
Data-to-parameter ratio = 14.1

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

In the crystal structure of the title compound, $C_{16}H_{13}N_5O_2\cdot CH_4O$, there are intra- and intermolecular hydrogen bonds. Molecules form dimers, which are extended to afford a ribbon structure. These ribbons are further packed, forming a three-dimensional grid structure.

Comment

Azole subunits are frequently present in biologically active compounds (Street *et al.*, 1995). Triazole derivatives have been studied as anti-inflammatory drug candidates and also been used as ligands for binding Pt and Ru to form antitumor metal complexes (Komeda *et al.*, 2002). As a consequence, much ongoing effort has been devoted to derivatives of 1,2,4-triazole and their metal complexes for medical use. However, derivatives of 3,5-diamino-1*H*-1,2,4-triazole have not been well studied (El-Hefnawy *et al.*, 1993; Elshani *et al.*, 2005). We report here the crystal structure of the title compound, (I), which is a Schiff base derived from 3,5-diamino-1*H*-1,2,4-triazole.



(I)

In (I), there are intra- and intermolecular hydrogen bonds (Table 1). The two benzene rings (ring 1 = C1–C6 and ring 3 = C11–C16) and the triazole ring (ring 2 = N1/N2/C8/N4/C9) are almost in the same plane (Fig. 1), the angles between rings 1 and 2, and between rings 2 and 3 being 3.7 (2) and 3.3 (2)°, respectively. The bond lengths of the triazole ring are very similar to other 1*H*-1,2,4-triazole derivatives (Claramunt *et al.*, 2001; Zhou *et al.*, 2001). The triazole and methanol molecules are linked through O—H···N and N—H···O hydrogen bonds (Table 1) to form dimers, which are extended by weak intermolecular C—H···O interactions to afford a ribbon structure (Fig. 2). These ribbons are further packed through weak C—H···N hydrogen bonds, forming a three-dimensional grid structure.

Experimental

The title compound, (I), was prepared, in 60% yield, by condensation of 3,5-diamino-1*H*-1,2,4-triazole with salicylaldehyde in a 1:2 ratio in hot methanol. Suitable crystals for X-ray diffraction were obtained by recrystallization from a methanol solution. Analysis found: C 60.15, H 5.15, N 20.58%; calculated for $C_{17}H_{17}N_5O_3$: C 60.17, H 5.05, N 20.64%.

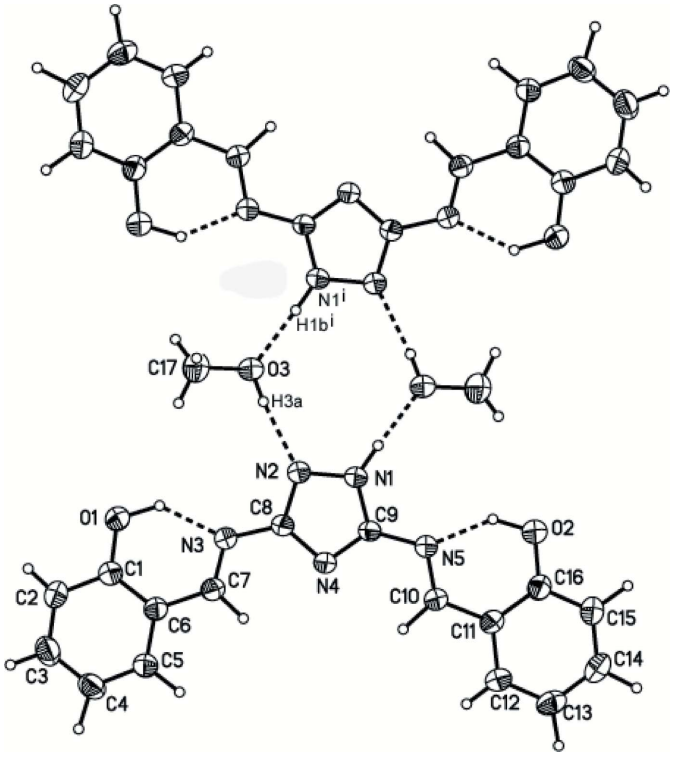


Figure 1
A view of the molecular structure of (I), showing 30% probability displacement ellipsoids. Two molecules form a dimer through N—H···O and O—H···N hydrogen bonds (dashed lines) [symmetry code: (i) $2-x$, $1-y$, $-z$].

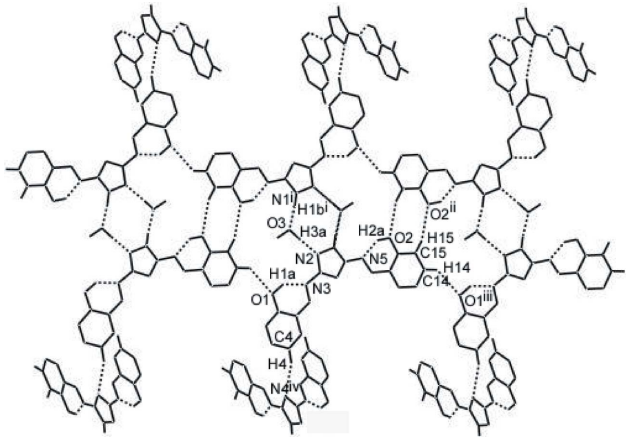


Figure 2
A view of the ribbon structure of (I). The ribbons are arranged in crosslinking directions, forming a three-dimensional grid structure [symmetry codes: (ii) $-x$, $2-y$, $-z$; (iii) $-2+x$, $1+y$, $-z$; (iv) $\frac{3}{2}-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$].

Crystal data

$C_{16}H_{13}N_5O_2\cdot CH_4O$
$M_r = 339.36$
Monoclinic, $P2_1/n$
$a = 5.8562\ (7)$ Å
$b = 9.1651\ (12)$ Å
$c = 31.907\ (4)$ Å
$\beta = 90.332\ (3)^\circ$
$V = 1712.5\ (4)$ Å^3
$Z = 4$
$D_x = 1.316$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 428 reflections
$\theta = 2.3$–17.5°
$\mu = 0.09$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
$0.28 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.97$, $T_{max} = 0.98$
9110 measured reflections
3374 independent reflections
1686 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 26.0^\circ$
$h = -7 \rightarrow 6$
$k = -7 \rightarrow 11$
$l = -39 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.159$
$S = 0.96$
3374 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N5	1.05 (4)	1.72 (4)	2.618 (3)	141 (3)
O1—H1A···N3	1.06 (4)	1.77 (4)	2.645 (3)	137 (3)
O3—H3A···N2	0.89 (4)	1.91 (4)	2.773 (3)	164 (4)
N1—H1B···O3^{i}	0.94 (3)	1.77 (3)	2.702 (3)	172 (3)
C15—H15···O2^{ii}	0.93	2.79	3.694 (4)	164
C14—H14···O1^{iii}	0.93	2.81	3.522 (4)	134
C4—H4···N4^{iv}	0.93	2.64	3.481 (4)	151

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

N- and O-bound H atoms were found in a difference Fourier map and their coordinates were refined with $U_{iso}(H) = 1.2U_{eq}(O,N)$. Other H atoms were placed in calculated positions, with C—H = 0.93–0.96 Å, and included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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