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3-[*N*′-(4-Hydroxy-3-methoxybenzylidene)-hydrazinocarbonyl]-1*H*-1,2,4-triazole

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Key indicators

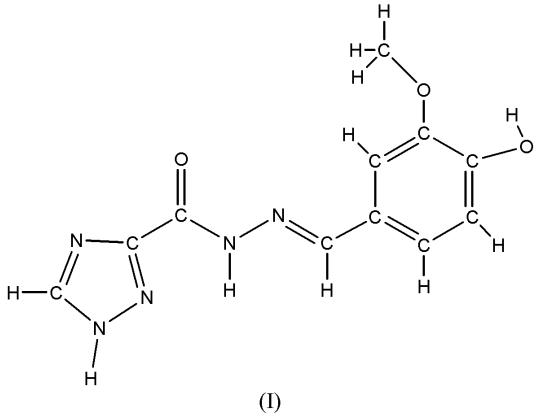
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.140
Data-to-parameter ratio = 12.3

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

The title compound, $C_{11}H_{11}N_5O_3$, was synthesized by the reaction of 3-(1*H*)-1,2,4-triazole hydrazine with 4-hydroxy-3-methoxybenzaldehyde in ethanol. In the crystal structure, intermolecular N—H···O, O—H···N and N—H···N hydrogen bonds are observed. An intramolecular O—H···O hydrogen bond is also found.

Comment

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



(I)

In (I), there are two independent molecules in the asymmetric unit (Fig. 1). The methoxy groups at C9 and C20 are rotated slightly around the C9—O2 and C20—O5 bonds; the torsion angles C11—O2—C9—C10 and C22—O5—C20—C21 are −11.8 (4) and −6.8(0.4)°, respectively. The bond lengths and angles are unexceptional (Allen *et al.*, 1987). Intermolecular N—H···O, N—H···N and O—H···N hydrogen bonds are observed (Table 2), which result in seven-membered and five-membered hydrogen-bonded rings (Fig. 2). In addition, an intramolecular O—H···O hydrogen bond forming a five-membered ring is observed.

Experimental

1*H*-1,2,4-Triazol-3-ylhydrazine (0.02 mol, 2.54 g) was dissolved in 50 ml of anhydrous ethanol at room temperature. 4-Hydroxy-3-methoxybenzaldehyde (0.02 mol, 3.04 g) was added and the mixture was refluxed for 2 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 compound (2.0 mmol, 0.52 g) was dissolved in 30 ml DMF and was kept at room temperature for 30 days. Brown spike-shaped single crystals formed; they were washed with distilled water.

Crystal data

$C_{11}H_{11}N_5O_3$
M_r = 261.25
Triclinic, $P\overline{1}$
a = 4.473 (1) Å
b = 14.772 (5) Å
c = 18.263 (2) Å
α = 79.38 (2)°
β = 88780 (1)°
γ = 86.33 (2)°
V = 1183.6 (5) Å^3
Z = 4
D_x = 1.466 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.8–14.8°
μ = 0.11 mm^{-1}
T = 293 (2) K
Spike, brown
0.40 × 0.20 × 0.10 mm

Data collection

Bruker SMART four-circle diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS* (Bruker, 2002)
T_{min} = 0.957, T_{max} = 0.989
4819 measured reflections
4271 independent reflections
2357 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.2°
$h = -5 \rightarrow 5$
$k = -17 \rightarrow 1$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.140
S = 1.01
4271 reflections
347 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 0.0981P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.217 (3)	O5—C20	1.363 (3)
O2—C9	1.362 (3)	O5—C22	1.427 (3)
O2—C11	1.432 (3)	O6—C19	1.354 (3)
O3—C8	1.361 (3)	N4—C3	1.343 (3)
O4—C14	1.213 (3)	N10—C15	1.263 (3)
C11—O2—C9—C10	−11.8 (4)	C22—O5—C20—C21	−6.7 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.82	2.18	2.642 (2)	116
O3—H3···N8	0.82	2.25	2.868 (3)	133
O6—H6*A*···N3^{i}	0.82	2.00	2.742 (3)	149
O6—H6*A*···O5	0.82	2.22	2.665 (3)	115
N2—H2···N6^{ii}	0.86	1.97	2.823 (3)	172
N2—H2···O4^{ii}	0.86	2.56	3.016 (3)	115
N4—H4···O6^{i}	0.86	2.25	3.089 (3)	166
N7—H7···O1^{iii}	0.86	1.96	2.815 (3)	174
N9—H9···O3	0.86	2.08	2.932 (3)	170

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

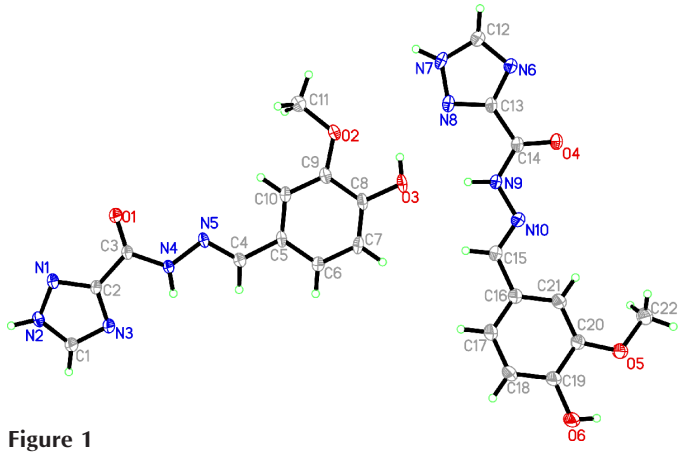


Figure 1
The structure of the asymmetric unit of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

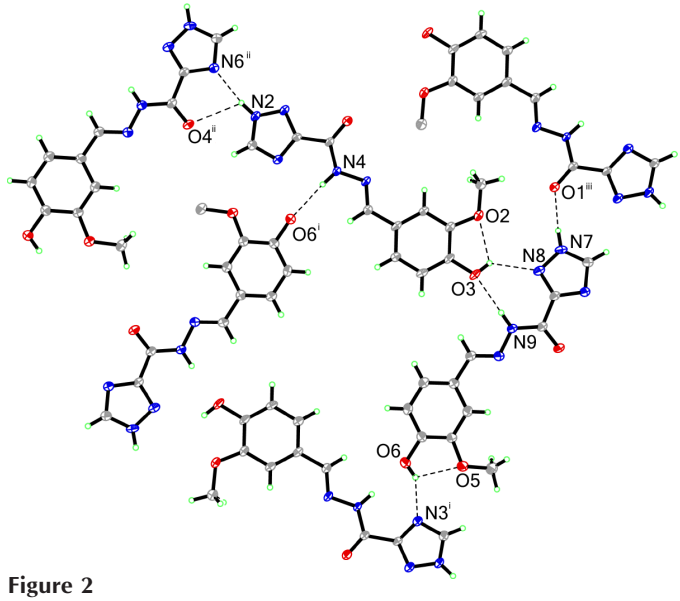


Figure 2
Packing of (I), showing selected inter- and intramolecular hydrogen bonds as dashed lines. Some of the H atoms have been omitted for clarity. (Symmetry codes are as in Table 2.)

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.93–0.96 Å (C—H), 0.86 Å (N—H) and 0.82 Å (O—H), with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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