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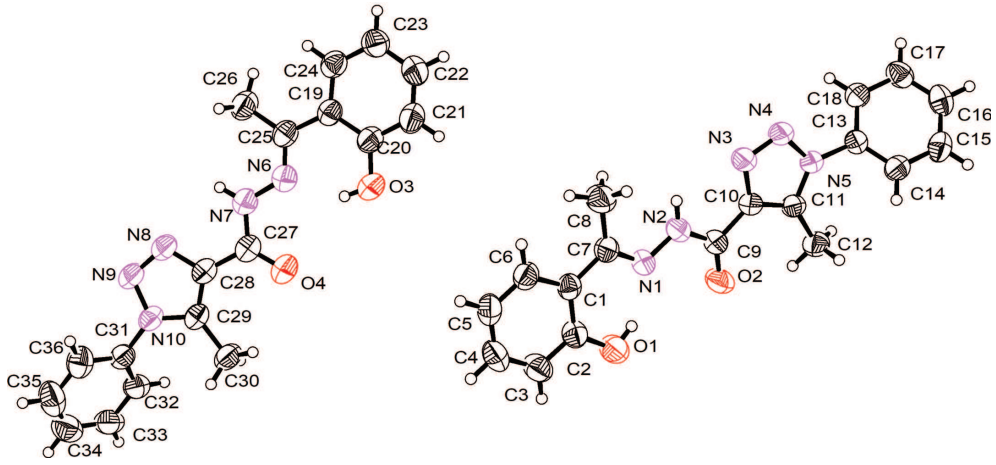
Crystal structure of *N′*-(1-(2-hydroxyphenyl) ethylidene)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carbohydrazide, $C_{18}H_{17}N_5O_2$



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Abstract

$C_{18}H_{17}N_5O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 20.3702(7)$ Å, $b = 7.3482(2)$ Å, $c = 23.2504(10)$ Å, $\beta = 106.507(4)°$, $V = 3336.8(2)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0571$, $wR_{ref}(F^2) = 0.1507$, $T = 296(2)$ K.

CCDC no.: 1875460

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.37 × 0.20 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	32673, 8345, 0.033
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5134
$N(param)_{refined}$:	458
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], WinGX, ORTEP [4]

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The asymmetric unit, containing two crystallographically independent molecules, of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized based on a literature procedure [5] and recrystallized from dimethylformamide to give colourless block crystals (86%).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model. N—H bonds were fixed at

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.12057(9)	0.8874(2)	0.47514(9)	0.0489(4)
C2	0.08609(10)	0.8835(3)	0.41350(9)	0.0565(5)
C3	0.12032(12)	0.9311(3)	0.37181(10)	0.0689(6)
H3	0.097213	0.927515	0.331097	0.083*
C4	0.18760(12)	0.9833(3)	0.38990(11)	0.0691(6)
H4	0.210018	1.013592	0.361514	0.083*
C5	0.22207(10)	0.9909(3)	0.44999(11)	0.0644(6)
H5	0.267610	1.027692	0.462396	0.077*
C6	0.18894(10)	0.9440(3)	0.49135(10)	0.0582(5)
H6	0.212782	0.950030	0.531862	0.070*
C7	0.08730(9)	0.8324(3)	0.52096(9)	0.0509(4)
C8	0.12478(12)	0.8414(4)	0.58642(10)	0.0779(7)
H8A	0.102238	0.926203	0.605873	0.117*
H8B	0.171001	0.880668	0.591325	0.117*
H8C	0.125129	0.723117	0.604032	0.117*
C9	−0.07306(10)	0.6544(3)	0.52256(9)	0.0519(5)
C10	−0.10264(9)	0.6101(3)	0.57162(8)	0.0480(4)
C11	−0.16850(9)	0.5589(2)	0.56824(8)	0.0466(4)
C12	−0.22810(10)	0.5189(3)	0.51571(9)	0.0580(5)
H12A	−0.252997	0.417098	0.524717	0.087*
H12B	−0.212234	0.490555	0.481621	0.087*
H12C	−0.257561	0.623413	0.506853	0.087*
C13	−0.22141(9)	0.5069(2)	0.65369(8)	0.0466(4)
C14	−0.28706(9)	0.5689(3)	0.62847(9)	0.0563(5)
H14	−0.297984	0.632300	0.592378	0.068*
C15	−0.33675(10)	0.5359(3)	0.65739(11)	0.0658(6)
H15	−0.381453	0.575041	0.640188	0.079*
C16	−0.32030(11)	0.4458(3)	0.71119(11)	0.0650(6)
H16	−0.353582	0.426215	0.730834	0.078*
C17	−0.25458(11)	0.3844(3)	0.73618(10)	0.0611(5)
H17	−0.243494	0.323191	0.772692	0.073*
C18	−0.20502(10)	0.4134(3)	0.70715(9)	0.0554(5)
H18	−0.160779	0.369865	0.723658	0.066*
C19	0.49738(9)	0.2062(3)	0.61744(10)	0.0540(5)
C20	0.42966(10)	0.2652(3)	0.59025(10)	0.0621(5)
C21	0.38566(11)	0.2931(3)	0.62507(12)	0.0749(7)
H21	0.340885	0.329834	0.606804	0.090*
C22	0.40661(12)	0.2679(3)	0.68561(12)	0.0724(6)
H22	0.376246	0.288004	0.708201	0.087*
C23	0.47271(11)	0.2125(3)	0.71350(11)	0.0678(6)
H23	0.487254	0.195850	0.754838	0.081*
C24	0.51682(10)	0.1823(3)	0.67939(10)	0.0607(5)
H24	0.561273	0.144453	0.698396	0.073*
C25	0.54578(10)	0.1731(3)	0.58201(10)	0.0573(5)
C26	0.61401(10)	0.0825(3)	0.60817(11)	0.0732(6)
H26A	0.617815	−0.021000	0.584101	0.110*
H26B	0.617676	0.043331	0.648354	0.110*
H26C	0.650059	0.167180	0.608726	0.110*
C27	0.53785(10)	0.2418(3)	0.43069(10)	0.0608(5)
C28	0.58545(10)	0.2273(3)	0.39434(9)	0.0567(5)
C29	0.57247(9)	0.2507(3)	0.33380(9)	0.0540(5)
C30	0.50873(10)	0.3001(3)	0.28653(10)	0.0689(6)
H30A	0.520297	0.367360	0.255371	0.103*
H30B	0.480210	0.373509	0.303775	0.103*
H30C	0.484577	0.191345	0.269959	0.103*
C31	0.65374(10)	0.2345(3)	0.26991(10)	0.0561(5)
C32	0.61490(11)	0.1478(3)	0.21909(10)	0.0640(5)
H32	0.575629	0.084315	0.219738	0.077*
C33	0.63476(13)	0.1562(3)	0.16747(11)	0.0741(6)
H33	0.608371	0.099877	0.132765	0.089*
C34	0.69322(15)	0.2471(4)	0.16680(13)	0.0838(8)
H34	0.706720	0.250872	0.131821	0.101*
C35	0.73196(14)	0.3326(4)	0.21768(15)	0.0876(8)
H35	0.771589	0.394283	0.216972	0.105*
C36	0.71239(11)	0.3275(3)	0.27001(12)	0.0734(6)
H36	0.738326	0.385813	0.304504	0.088*
N1	0.02520(8)	0.7749(2)	0.50145(7)	0.0536(4)
N2	−0.00829(8)	0.7204(2)	0.54201(7)	0.0564(4)
H2	0.011791	0.728044	0.579840	0.068*
N3	−0.06542(8)	0.6244(3)	0.63013(7)	0.0591(4)
N4	−0.10436(8)	0.5859(3)	0.66399(7)	0.0599(4)
N5	−0.16782(7)	0.5458(2)	0.62651(7)	0.0476(4)
N6	0.52485(9)	0.2239(2)	0.52699(9)	0.0629(5)
N7	0.56550(9)	0.2020(3)	0.48976(8)	0.0663(5)
H7	0.607043	0.164449	0.503194	0.080*
N8	0.65296(8)	0.1891(3)	0.41904(8)	0.0667(5)
N9	0.68330(8)	0.1874(3)	0.37689(8)	0.0685(5)
N10	0.63434(8)	0.2248(2)	0.32432(8)	0.0570(4)
O1	0.01960(8)	0.8355(3)	0.39170(7)	0.0796(5)
H1	0.005693	0.795392	0.419006	0.119*
O2	−0.10394(7)	0.6361(2)	0.46992(6)	0.0719(4)
O3	0.40414(8)	0.2961(3)	0.53053(8)	0.0874(5)
H3A	0.435081	0.288670	0.514508	0.131*
O4	0.47790(8)	0.2840(3)	0.41023(8)	0.0838(5)

0.86 Å (AFIX 43 instruction in SHELXL [4]), with displacement parameters 1.2 times U_{eq}(N). Aromatic C—H distances were set to 0.93 Å (AFIX 43) and their U_{iso}(H) set to 1.2 times the U_{eq}(C). Methyl C—H distances were set to 0.96 Å and their U_{iso}(H) to 1.5 times the U_{eq}(C) with the groups allowed to rotate about the C—C bonds (AFIX 137). O—H bonds were fixed at 0.82 Å (AFIX 147), with displacement parameters 1.2 times U_{eq}(O).

Comment

Various 1*H*-1,2,3-triazoles show miscellaneous biological activities [6–10]. Recently, we have reported the crystal structures for two 1*H*-1,2,3-triazole-4-carbohydrazides [11, 12].

The asymmetric unit consists of two crystallographically independent molecules (*cf.* the figure). Bond lengths and angles excellently fit with those of our previous experiments [11, 12]. The twist angles between the planes through the hydroxyphenyl and the triazolyl groups are 3.37(6)° for molecule (C1—C18, **A**) and 6.02(6)° for the molecule (C19—C36, **B**). The angles between the planes through the triazolyl and phenyl groups are 37.05(6)° for **A** and 46.14(6)° for **B**.

The molecules are stacked along [010] in the crystal structure with **A** and **B** forming separate columns.

Interactions of π—π type occur between hydroxyphenyl and triazolyl groups of neighbouring molecules related by inversion symmetry within the columns. The associated centroid-to-centroid distances are 3.97 Å and 3.66 Å for molecules **A** and **B** respectively. Intramolecular O—H···N hydrogen bonds are observed with O···N distances of 2.561(2) Å and 2.540(2) Å and O—H··· N angles of 144.3° and 143.8° for **A** and **B** respectively.

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