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4-Amino-3-(4-hydroxyphenyl)-1*H*-1,2,4-triazol-5(4*H*)-one

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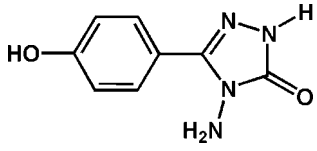
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 10.0.

The molecule of the title compound, $C_8H_8N_4O_2$, is nearly planar, with a dihedral angle between the rings of 1.1 (1)°. Adjacent molecules are linked into a layered structure by hydroxy–oxo O—H···O and triazolyl–hydroxy N—H···O hydrogen bonds. Only one of the H atoms of the pyramidal amino group is engaged in building up the infinite layer. The second H atom of the amino group also shows hydrogen-bonding interactions, linking adjacent layers into a three-dimensional network.

Related literature

For a synthesis of the title compound using CS_2 as a reactant, see: Chande & Singh-Jathar (1998). This product was obtained unexpectedly in the present study.



Experimental

Crystal data

$C_8H_8N_4O_2$
M_r = 192.18
Triclinic, $P\overline{1}$
a = 6.534 (1) Å
b = 7.330 (1) Å
c = 9.804 (1) Å
α = 106.69 (1)°
β = 102.328 (9)°
γ = 106.712 (2)°
V = 407.7 (1) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 295 (2) K
0.25 × 0.16 × 0.04 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: none
3032 measured reflections
1434 independent reflections
1115 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.03
1434 reflections
143 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.86 (1)	1.78 (1)	2.633 (2)	175 (3)
N2—H2···O1^{ii}	0.86 (1)	1.93 (1)	2.789 (2)	173 (2)
N4—H4···O2^{iii}	0.87 (1)	2.24 (1)	3.077 (3)	163 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2089).

References

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Chande, M. S. & Singh-Jathar, K. (1998). *Indian J. Chem. Sect. B*, **37**, 352–357.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2008). *publCIF*. In preparation.

supporting information

Acta Cryst. (2008). E**64**, o2483 [doi:10.1107/S1600536808039688]

4-Amino-3-(4-hydroxyphenyl)-1*H*-1,2,4-triazol-5(4*H*)-one

Kai-Ge Shi, Guang Yang and Seik Weng Ng

S1. Comment

In connection with our work on metal triazolates, we are interested in synthesizing 4-amino-bis(4-hydroxyphenyl)-1,2,4-triazole. The synthesis of this triazole yielded the title compound as an unexpected product. A specific procedure for the synthesis of the title compound is reported in the literature to start from carbonyl sulfide and 4-hydroxybenzohydrazide in potassium hydroxide to give a precursor that was subsequently reacted with hydrazine (Chande & Singh-Jathar, 1998).

S2. Experimental

4-Hydroxybenzoic acid (2.76 g, 0.02 mol) and 80% hydrazine hydrate (1.55 g, 0.02 mol) were heated in a sealed tube at 439 K for three days. After cooling to room temperature, the mixture was centrifuged. The resulting white solid was suspended in water, and 6*M* hydrochloric acid was added until the pH was 3. The white product was collected and recrystallized from a DMSO–water mixture(10:1) to afford colorless crystals in 3% yield. CH&N elemental analysis. C 49.58 (calc. 49.99), H 4.19 (found 4.20), N 29.25% (29.15%).

S3. Refinement

Carbon-bound H atoms were generated geometrically (C–H 0.93 Å), and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C). The amino and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints of N–H = O–H = 0.85±0.01 Å; their temperature factors were freely refined.

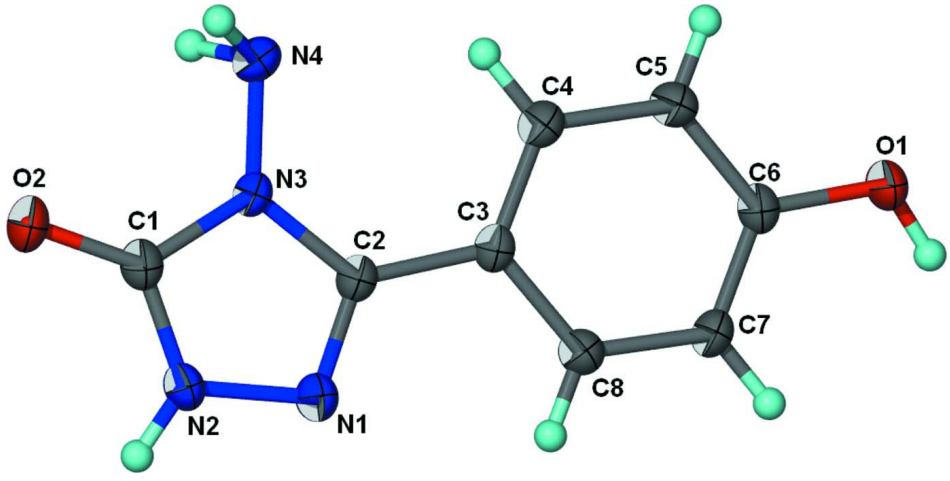

Figure 1

Thermal ellipsoid (Barbour, 2001) plot of $C_{18}H_8N_4O_2$ at the 70% probability level.

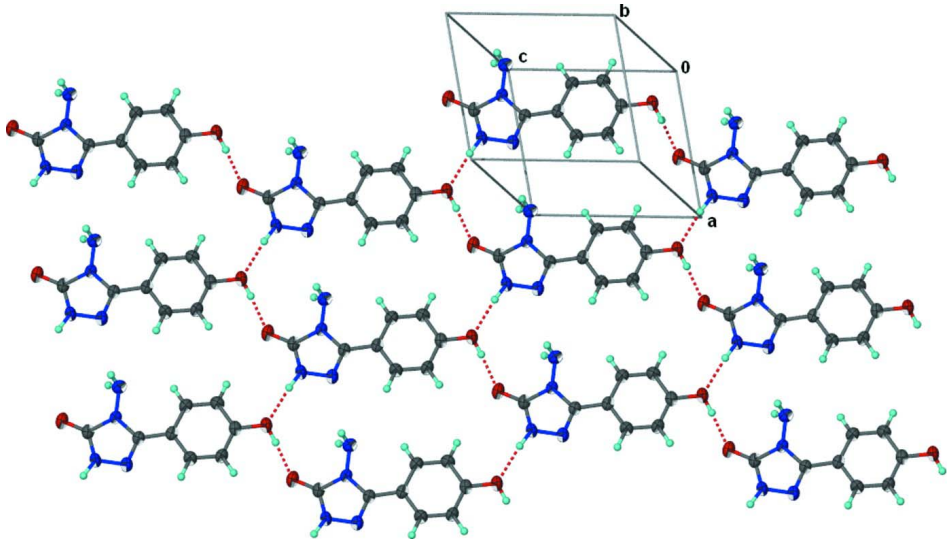

Figure 2

Thermal ellipsoid (Barbour, 2001) plot of the layered structure arising from *O*–$H_{hydroxy}$ and N–$H_{triazolyl}$ hydrogen bonds.

4-Amino-3-(4-hydroxyphenyl)-1H-1,2,4-triazol-5(4H)-one

Crystal data

$C_8H_8N_4O_2$
M_r = 192.18
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.534 (1) Å
b = 7.330 (1) Å
c = 9.804 (1) Å
α = 106.69 (1)°
β = 102.328 (9)°
γ = 106.712 (2)°
V = 407.7 (1) Å^3
Z = 2
$F(000)$ = 200
D_x = 1.565 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 986 reflections
θ = 2.3–26.7°
μ = 0.12 mm^{-1}
T = 295 K
Block, colorless
0.25 × 0.16 × 0.04 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
3032 measured reflections
1434 independent reflections
1115 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.0°, θ_{min} = 2.3°
h = −7→7
k = −8→8
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.03
1434 reflections
143 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.1018P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.3462 (2)	0.3381 (2)	0.06978 (15)	0.0481 (4)
O2	0.6706 (2)	1.2219 (2)	1.00449 (15)	0.0456 (4)
N1	0.8848 (3)	1.0137 (3)	0.72942 (18)	0.0423 (5)
N2	0.9057 (3)	1.1419 (3)	0.87015 (19)	0.0444 (5)
N3	0.5523 (2)	0.9858 (2)	0.75552 (16)	0.0319 (4)
N4	0.3171 (3)	0.9141 (3)	0.7256 (2)	0.0420 (5)
C1	0.7075 (3)	1.1281 (3)	0.8902 (2)	0.0361 (5)
C2	0.6672 (3)	0.9197 (3)	0.6610 (2)	0.0314 (4)
C3	0.5739 (3)	0.7693 (3)	0.5056 (2)	0.0302 (4)
C4	0.3450 (3)	0.6652 (3)	0.4264 (2)	0.0373 (5)
H4A	0.2398	0.6910	0.4719	0.045*
C5	0.2715 (3)	0.5241 (3)	0.2810 (2)	0.0402 (5)
H5	0.1177	0.4571	0.2289	0.048*
C6	0.4261 (3)	0.4820 (3)	0.2127 (2)	0.0342 (5)
C7	0.6551 (3)	0.5851 (3)	0.2893 (2)	0.0387 (5)
H7	0.7598	0.5593	0.2432	0.046*
C8	0.7269 (3)	0.7262 (3)	0.4342 (2)	0.0386 (5)
H8	0.8808	0.7942	0.4854	0.046*
H1	0.450 (3)	0.301 (4)	0.043 (3)	0.067 (8)*
H2	1.037 (2)	1.204 (3)	0.937 (2)	0.054 (7)*
H4	0.294 (5)	0.881 (4)	0.801 (2)	0.076 (9)*
H40	0.273 (4)	1.013 (3)	0.732 (3)	0.078 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0338 (8)	0.0580 (10)	0.0293 (8)	0.0155 (7)	0.0038 (6)	−0.0101 (7)
O2	0.0431 (9)	0.0556 (9)	0.0288 (8)	0.0213 (7)	0.0126 (6)	−0.0008 (7)
N1	0.0305 (9)	0.0498 (10)	0.0295 (9)	0.0122 (8)	0.0071 (7)	−0.0045 (8)
N2	0.0288 (9)	0.0540 (11)	0.0268 (9)	0.0114 (8)	0.0029 (7)	−0.0095 (8)
N3	0.0271 (8)	0.0396 (9)	0.0245 (8)	0.0135 (7)	0.0099 (6)	0.0032 (7)
N4	0.0295 (9)	0.0559 (12)	0.0343 (10)	0.0172 (9)	0.0124 (8)	0.0053 (9)
C1	0.0347 (11)	0.0406 (11)	0.0262 (10)	0.0156 (9)	0.0081 (8)	0.0025 (9)
C2	0.0301 (10)	0.0349 (10)	0.0259 (10)	0.0132 (8)	0.0097 (8)	0.0051 (8)
C3	0.0312 (10)	0.0325 (10)	0.0255 (10)	0.0135 (8)	0.0103 (8)	0.0061 (8)
C4	0.0309 (10)	0.0442 (12)	0.0320 (11)	0.0165 (9)	0.0111 (8)	0.0042 (9)
C5	0.0264 (10)	0.0472 (12)	0.0343 (11)	0.0120 (9)	0.0056 (8)	0.0023 (9)
C6	0.0339 (11)	0.0380 (11)	0.0246 (10)	0.0136 (9)	0.0075 (8)	0.0043 (8)
C7	0.0316 (11)	0.0476 (12)	0.0297 (11)	0.0153 (9)	0.0124 (8)	0.0019 (9)
C8	0.0276 (10)	0.0450 (12)	0.0310 (11)	0.0110 (9)	0.0067 (8)	0.0017 (9)

Geometric parameters (Å, º)

O1—C6	1.368 (2)	C2—C3	1.470 (2)
O1—H1	0.861 (10)	C3—C4	1.389 (3)
O2—C1	1.247 (2)	C3—C8	1.394 (3)
N1—C2	1.308 (2)	C4—C5	1.381 (3)
N1—N2	1.381 (2)	C4—H4A	0.9300
N2—C1	1.331 (3)	C5—C6	1.383 (3)
N2—H2	0.860 (10)	C5—H5	0.9300
N3—C1	1.374 (2)	C6—C7	1.385 (3)
N3—C2	1.380 (2)	C7—C8	1.379 (3)
N3—N4	1.407 (2)	C7—H7	0.9300
N4—H4	0.868 (10)	C8—H8	0.9300
N4—H40	0.846 (10)		
C6—O1—H1	112.3 (18)	C4—C3—C2	124.64 (17)
C2—N1—N2	104.87 (15)	C8—C3—C2	117.37 (17)
C1—N2—N1	112.93 (16)	C5—C4—C3	120.93 (18)
C1—N2—H2	127.2 (16)	C5—C4—H4A	119.5
N1—N2—H2	119.0 (16)	C3—C4—H4A	119.5
C1—N3—C2	108.48 (15)	C4—C5—C6	120.17 (18)
C1—N3—N4	124.53 (15)	C4—C5—H5	119.9
C2—N3—N4	126.90 (16)	C6—C5—H5	119.9
N3—N4—H4	105.8 (19)	O1—C6—C5	118.31 (17)
N3—N4—H40	109.5 (19)	O1—C6—C7	121.86 (17)
H4—N4—H40	104 (3)	C5—C6—C7	119.83 (17)
O2—C1—N2	128.18 (18)	C8—C7—C6	119.58 (18)
O2—C1—N3	127.92 (18)	C8—C7—H7	120.2
N2—C1—N3	103.90 (16)	C6—C7—H7	120.2
N1—C2—N3	109.81 (15)	C7—C8—C3	121.49 (18)
N1—C2—C3	121.80 (16)	C7—C8—H8	119.3
N3—C2—C3	128.39 (16)	C3—C8—H8	119.3
C4—C3—C8	117.99 (17)		
C2—N1—N2—C1	0.4 (2)	N3—C2—C3—C4	−0.2 (3)
N1—N2—C1—O2	179.29 (19)	N1—C2—C3—C8	1.5 (3)
N1—N2—C1—N3	−0.6 (2)	N3—C2—C3—C8	−179.08 (18)
C2—N3—C1—O2	−179.4 (2)	C8—C3—C4—C5	−0.3 (3)
N4—N3—C1—O2	−2.7 (3)	C2—C3—C4—C5	−179.17 (18)
C2—N3—C1—N2	0.5 (2)	C3—C4—C5—C6	0.9 (3)
N4—N3—C1—N2	177.19 (19)	C4—C5—C6—O1	178.66 (18)
N2—N1—C2—N3	−0.1 (2)	C4—C5—C6—C7	−1.4 (3)
N2—N1—C2—C3	179.42 (17)	O1—C6—C7—C8	−178.87 (18)
C1—N3—C2—N1	−0.2 (2)	C5—C6—C7—C8	1.2 (3)
N4—N3—C2—N1	−176.86 (19)	C6—C7—C8—C3	−0.5 (3)
C1—N3—C2—C3	−179.71 (18)	C4—C3—C8—C7	0.1 (3)
N4—N3—C2—C3	3.7 (3)	C2—C3—C8—C7	179.05 (18)
N1—C2—C3—C4	−179.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.86 (1)	1.78 (1)	2.633 (2)	175 (3)
N2—H2···O1^{ii}	0.86 (1)	1.93 (1)	2.789 (2)	173 (2)
N4—H4···O2^{iii}	0.87 (1)	2.24 (1)	3.077 (3)	163 (2)

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