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4-Amino-3-(4-hydroxyphenyl)-1*H*-1,2,4-triazol-5(4*H*)-oneKai-Ge Shi,^a Guang Yang^a and Seik Weng Ng^{b*}^aDepartment of Chemistry, Zhengzhou University, Zhengzhou 450052, People's Republic of China, and ^bDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

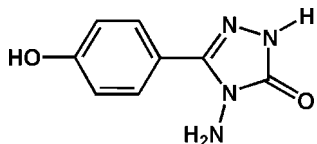
Received 11 November 2008; accepted 25 November 2008

Key indicators: single-crystal X-ray study; *T* = 295 K; mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$; *R* factor = 0.039; *wR* factor = 0.109; data-to-parameter ratio = 10.0.

The molecule of the title compound, $\text{C}_8\text{H}_8\text{N}_4\text{O}_2$, is nearly planar, with a dihedral angle between the rings of $1.1 (1)^\circ$. Adjacent molecules are linked into a layered structure by hydroxy-oxo $\text{O}-\text{H}\cdots\text{O}$ and triazolyl-hydroxy $\text{N}-\text{H}\cdots\text{O}$ 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

$\text{C}_8\text{H}_8\text{N}_4\text{O}_2$
 $M_r = 192.18$
 Triclinic, *P* $\bar{1}$

$a = 6.534 (1) \text{ \AA}$
 $b = 7.330 (1) \text{ \AA}$
 $c = 9.804 (1) \text{ \AA}$

$\alpha = 106.69 (1)^\circ$
 $\beta = 102.328 (9)^\circ$
 $\gamma = 106.712 (2)^\circ$
 $V = 407.7 (1) \text{ \AA}^3$
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.12 \text{ mm}^{-1}$
 $T = 295 (2) \text{ K}$
 $0.25 \times 0.16 \times 0.04 \text{ mm}$

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: none
 3032 measured reflections

1434 independent reflections
 1115 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

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

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
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: *SAINTE* (Bruker, 2007); data reduction: *SAINTE*; 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: *pubCIF* (Westrip, 2008).

We thank the Education Department of Henan Province, Zhengzhou University and the University of Malaya for supporting this work.

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

References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
 Bruker (2007). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chande, M. S. & Singh-Jathar, K. (1998). *Indian J. Chem. Sect. B*, **37**, 352–357.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2008). *pubCIF*. In preparation.

supplementary materials

Acta Cryst. (2008). E64, o2483 [doi:10.1107/S1600536808039688]

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

K.-G. Shi, G. Yang and S. W. Ng

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).

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 6M 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%).

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 $U(\text{H})$ set to $1.2U_{\text{eq}}(\text{C})$. The amino and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints of $\text{N–H} = \text{O–H} = 0.85 \pm 0.01$ Å; their temperature factors were freely refined.

Figures

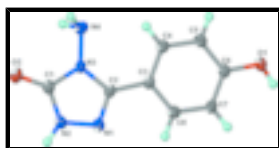


Fig. 1. Thermal ellipsoid (Barbour, 2001) plot of $\text{C}_{18}\text{H}_8\text{N}_4\text{O}_2$ at the 70% probability level.

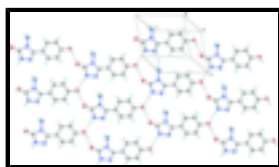


Fig. 2. Thermal ellipsoid (Barbour, 2001) plot of the layered structure arising from $\text{O–H}_{\text{hydroxy}}$ and $\text{N–H}_{\text{triazolyl}}$ hydrogen bonds.

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

Crystal data

$\text{C}_8\text{H}_8\text{N}_4\text{O}_2$
 $M_r = 192.18$

$Z = 2$
 $F_{000} = 200$

supplementary materials

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.534$ (1) Å

$b = 7.330$ (1) Å

$c = 9.804$ (1) Å

$\alpha = 106.69$ (1)°

$\beta = 102.328$ (9)°

$\gamma = 106.712$ (2)°

$V = 407.7$ (1) Å³

$D_x = 1.565$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 986 reflections

$\theta = 2.3$ – 26.7 °

$\mu = 0.12$ mm⁻¹

$T = 295$ (2) K

Block, colorless

$0.25 \times 0.16 \times 0.04$ mm

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 295$ (2) K

φ and ω scans

Absorption correction: None

3032 measured reflections

1434 independent reflections

1115 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.3$ °

$h = -7 \rightarrow 7$

$k = -8 \rightarrow 8$

$l = -11 \rightarrow 11$

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 $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.14$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Extinction correction: none

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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 (\AA , $^\circ$)

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)		

supplementary materials

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—H4O	109.5 (19)	O1—C6—C7	121.86 (17)
H4—N4—H4O	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 (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots O2 ⁱ	0.86 (1)	1.78 (1)	2.633 (2)	175 (3)
N2—H2 \cdots O1 ⁱⁱ	0.86 (1)	1.93 (1)	2.789 (2)	173 (2)
N4—H4 \cdots 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) $-x+1, -y+2, -z+2$.

Fig. 1

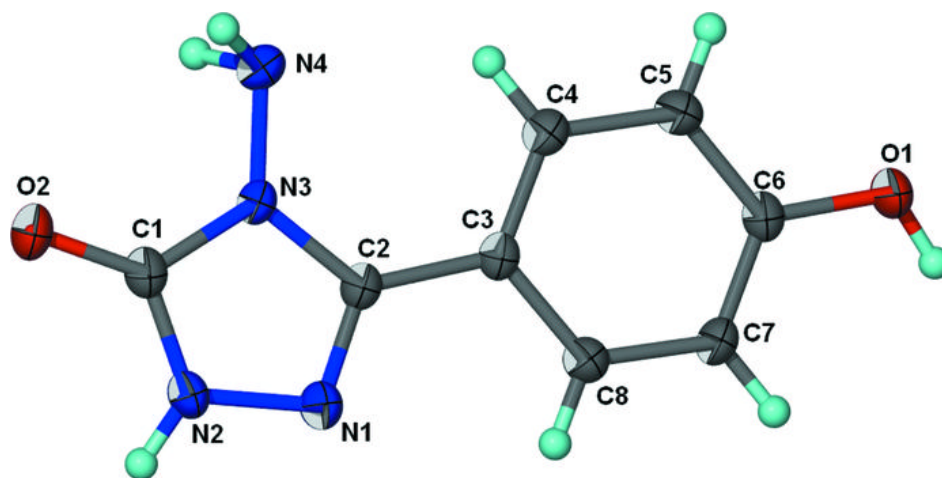


Fig. 2

