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(*E*)-*N*′-[1-(4-Aminophenyl)ethylidene]-2-hydroxybenzohydrazide methanol solvate

Xue-Fang Shi[a]* and Zhi-Yong Xing[b]

[a]Department of Chemistry, Tianjin Normal University, Tianjin 300074, People's Republic of China, and [b]College of Chemistry and Pharmacy, Jiamusi University, Jiamusi 154007, People's Republic of China
Correspondence e-mail: xuefangshi@126.com

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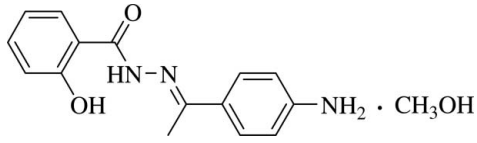
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.137; data-to-parameter ratio = 13.6.

In the crystal structure of the title compound, $C_{15}H_{15}N_3O_2\cdot CH_3OH$, the molecules are held together by N—H···O, O—H···O and O—H···N hydrogen bonds. The dihedral angle between the two aromatic rings is 45.70 (6)°.

Related literature

For related literature, see: Lehn (1990).



Experimental

Crystal data

$C_{16}H_{19}N_3O_3$
M_r = 301.34
Triclinic, $P\overline{1}$
a = 9.532 (2) Å
b = 9.866 (3) Å
c = 10.070 (3) Å
α = 106.877 (4)°
β = 102.705 (4)°
γ = 110.461 (4)°
V = 792.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.22 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.981, T_{max} = 0.989
4104 measured reflections
2765 independent reflections
1998 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.06
2765 reflections
203 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3^{i}	0.82	1.98	2.781 (2)	164
N3—H3A···O3^{ii}	0.87	2.15	3.009 (2)	169
N3—H3B···O2^{iii}	0.90	2.18	3.026 (2)	157
N1—H1A···O1	0.89	1.95	2.675 (2)	138
O3—H3C···O2	0.82	2.03	2.761 (2)	149

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2602).

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lehn, J. M. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 1304–1319.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

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(*E*)-*N*'-[1-(4-Aminophenyl)ethylidene]-2-hydroxybenzohydrazide methanol solvate

X.-F. Shi and Z.-Y. Xing

Comment

Hydrogen bond interaction has been exploited in the design of supramolecular assemblies, due to their important application in the development of new optical, magnetic and electronic systems (Lehn, 1990). (*E*)—*N*'-(1-(4-aminophenyl)ethylidene)-2-hydroxybenzohydrazide, synthesized from 2-hydroxybenzohydrazide and 1-(4-aminophenyl)ethanone in methanol, which is a new kind of schiff base.

The dihedral angle between the two aromatic rings is 45.70 (6)°. There is an intramolecularly hydrogen bond N1—H1A···O1 forming a six-membered ring as shown in Fig. 1. The O atom of hydroxy group connecting with H atom of NH2 group from adjacent molecules forms a one-dimensional hydrogen bonded chain by O1—H1···N3 hydrogen bonds [H1···N3 = 1.984 Å, O1···N3 = 2.781 (2) Å and O1—H1···N3 = 163.8 (3)°]. The methanol molecule act not only as hydrogen bond donor but also as an acceptor (Fig. 2).

Experimental

The title compound was synthesized by the reaction of 2-hydroxybenzohydrazide (0.01 mol, 1.52 g) and 1-(4-aminophenyl)ethanone (0.01 mol, 1.35 g) in methanol. The solution was refluxed for 2 h. The solid material obtained on cooling was filtered, washed with ethanol: ether (1:1), dried and crystallized from methanol (yield 78%). Single crystals suitable for X-ray analysis were obtained by slow evaporation of a methanol solution (Yield 53%).

Refinement

All H atoms were initially located in a difference Fourier map. The C—H atoms were then constrained to an ideal geometry, with C(CH_3)—H distances of 0.96 Å, *C*(phenyl)—H distances of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C). The amino H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with N—H distances in the range 0.89–0.92 Å.

Figures

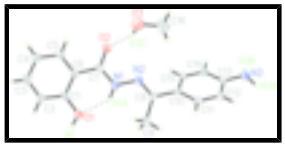

Fig. 1. The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

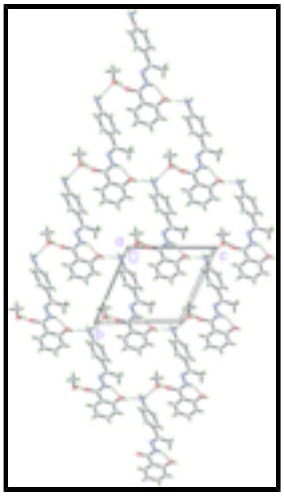

Fig. 2. Showing the hydrogen bonded network, viewed down the *a* axis.

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Crystal data

$C_{16}H_{19}N_3O_3$	$Z = 2$
$M_r = 301.34$	$F_{000} = 320$
Triclinic, $P\overline{1}$	$D_x = 1.263$ Mg m^{-3}
$a = 9.532$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.866$ (3) Å	Cell parameters from 1758 reflections
$c = 10.070$ (3) Å	$\theta = 2.4$–26.3°
$\alpha = 106.877$ (4)°	$\mu = 0.09$ mm^{-1}
$\beta = 102.705$ (4)°	$T = 293$ (2) K
$\gamma = 110.461$ (4)°	Black, colorless
$V = 792.6$ (3) Å^3	0.22 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2765 independent reflections
Radiation source: fine-focus sealed tube	1998 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.017$
$T = 293$(2) K	$\theta_{max} = 25.0^\circ$
phi and ω scans	$\theta_{min} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 11$
$T_{min} = 0.981$, $T_{max} = 0.989$	$k = -11 \rightarrow 11$
4104 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.137$	$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2765 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
203 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
4 restraints	Extinction correction: SHELXL
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2589 (2)	1.11169 (19)	0.69922 (16)	0.0553 (4)
H1	0.2580	1.1367	0.7840	0.083*
O2	0.14206 (18)	0.99148 (17)	0.24078 (15)	0.0518 (4)
N1	0.2377 (2)	0.93249 (19)	0.43197 (17)	0.0423 (4)
H1A	0.2568	0.9544	0.5277	0.051*
N2	0.2353 (2)	0.79924 (19)	0.33350 (18)	0.0428 (4)
N3	0.2016 (2)	0.1382 (2)	−0.03749 (19)	0.0495 (5)
H3A	0.2530	0.0931	0.0013	0.059*
H3B	0.0959	0.0770	−0.0894	0.059*
C1	0.2088 (2)	1.1987 (2)	0.6347 (2)	0.0400 (5)
C2	0.1960 (3)	1.3305 (3)	0.7190 (2)	0.0522 (6)
H2	0.2216	1.3594	0.8209	0.063*
C3	0.1459 (3)	1.4181 (3)	0.6538 (3)	0.0584 (6)
H3	0.1371	1.5052	0.7116	0.070*
C4	0.1085 (3)	1.3772 (3)	0.5024 (3)	0.0566 (6)
H4	0.0745	1.4363	0.4580	0.068*
C5	0.1223 (3)	1.2482 (2)	0.4182 (2)	0.0474 (5)
H5	0.0983	1.2220	0.3167	0.057*
C6	0.1712 (2)	1.1555 (2)	0.4803 (2)	0.0371 (4)
C7	0.1811 (2)	1.0198 (2)	0.3753 (2)	0.0387 (5)
C8	0.3019 (2)	0.7255 (2)	0.3898 (2)	0.0382 (4)

C9	0.3890 (3)	0.7790 (3)	0.5534 (2)	0.0554 (6)
H9A	0.4602	0.8898	0.5953	0.083*
H9B	0.4498	0.7212	0.5671	0.083*
H9C	0.3126	0.7601	0.6022	0.083*
C10	0.2813 (2)	0.5740 (2)	0.2816 (2)	0.0370 (4)
C11	0.3929 (2)	0.5133 (2)	0.3002 (2)	0.0440 (5)
H11	0.4863	0.5696	0.3841	0.053*
C12	0.3678 (3)	0.3709 (3)	0.1963 (2)	0.0465 (5)
H12	0.4453	0.3344	0.2106	0.056*
C13	0.2279 (2)	0.2820 (2)	0.0711 (2)	0.0395 (5)
C14	0.1155 (2)	0.3420 (2)	0.0516 (2)	0.0436 (5)
H14	0.0216	0.2851	−0.0317	0.052*
C15	0.1420 (2)	0.4843 (2)	0.1540 (2)	0.0414 (5)
H15	0.0655	0.5218	0.1382	0.050*
O3	0.3330 (3)	0.9427 (3)	0.0800 (3)	0.0916 (7)
H3C	0.2748	0.9244	0.1281	0.137*
C16	0.3621 (4)	0.8161 (4)	0.0251 (4)	0.1017 (11)
H16A	0.4096	0.8271	−0.0483	0.153*
H16B	0.2630	0.7211	−0.0197	0.153*
H16C	0.4341	0.8107	0.1044	0.153*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0806 (11)	0.0635 (10)	0.0355 (8)	0.0457 (9)	0.0231 (8)	0.0194 (7)
O2	0.0691 (10)	0.0563 (9)	0.0352 (8)	0.0365 (8)	0.0174 (7)	0.0153 (7)
N1	0.0550 (11)	0.0442 (10)	0.0318 (8)	0.0281 (8)	0.0169 (8)	0.0121 (8)
N2	0.0521 (10)	0.0434 (10)	0.0360 (9)	0.0265 (8)	0.0170 (8)	0.0124 (8)
N3	0.0632 (12)	0.0526 (11)	0.0428 (10)	0.0382 (10)	0.0185 (9)	0.0174 (9)
C1	0.0427 (12)	0.0410 (11)	0.0380 (10)	0.0187 (9)	0.0186 (9)	0.0147 (9)
C2	0.0692 (15)	0.0494 (13)	0.0418 (11)	0.0285 (12)	0.0285 (11)	0.0141 (10)
C3	0.0813 (17)	0.0502 (14)	0.0632 (15)	0.0388 (13)	0.0445 (13)	0.0229 (12)
C4	0.0780 (17)	0.0547 (14)	0.0630 (15)	0.0425 (13)	0.0376 (13)	0.0325 (12)
C5	0.0587 (14)	0.0509 (13)	0.0433 (11)	0.0296 (11)	0.0249 (10)	0.0217 (10)
C6	0.0357 (11)	0.0369 (11)	0.0393 (10)	0.0154 (9)	0.0171 (8)	0.0141 (9)
C7	0.0383 (11)	0.0413 (11)	0.0352 (10)	0.0172 (9)	0.0144 (8)	0.0132 (9)
C8	0.0371 (11)	0.0412 (11)	0.0355 (10)	0.0164 (9)	0.0133 (8)	0.0154 (9)
C9	0.0637 (15)	0.0501 (13)	0.0400 (12)	0.0248 (11)	0.0064 (10)	0.0115 (10)
C10	0.0401 (11)	0.0408 (11)	0.0353 (10)	0.0199 (9)	0.0165 (9)	0.0174 (9)
C11	0.0376 (11)	0.0500 (13)	0.0417 (11)	0.0215 (10)	0.0079 (9)	0.0169 (10)
C12	0.0495 (13)	0.0570 (13)	0.0475 (12)	0.0363 (11)	0.0186 (10)	0.0236 (11)
C13	0.0507 (12)	0.0451 (12)	0.0347 (10)	0.0290 (10)	0.0202 (9)	0.0189 (9)
C14	0.0449 (12)	0.0503 (12)	0.0339 (10)	0.0269 (10)	0.0088 (9)	0.0115 (9)
C15	0.0434 (12)	0.0499 (12)	0.0379 (10)	0.0296 (10)	0.0134 (9)	0.0168 (10)
O3	0.1202 (18)	0.1157 (17)	0.1053 (16)	0.0813 (15)	0.0766 (14)	0.0696 (14)
C16	0.125 (3)	0.087 (2)	0.125 (3)	0.059 (2)	0.080 (2)	0.043 (2)

Geometric parameters (Å, °)

O1—C1	1.364 (2)	C8—C10	1.488 (3)
O1—H1	0.8200	C8—C9	1.507 (3)
O2—C7	1.240 (2)	C9—H9A	0.9600
N1—C7	1.354 (2)	C9—H9B	0.9600
N1—N2	1.387 (2)	C9—H9C	0.9600
N1—H1A	0.8855	C10—C11	1.396 (3)
N2—C8	1.288 (2)	C10—C15	1.402 (3)
N3—C13	1.415 (2)	C11—C12	1.388 (3)
N3—H3A	0.8720	C11—H11	0.9300
N3—H3B	0.9011	C12—C13	1.392 (3)
C1—C2	1.394 (3)	C12—H12	0.9300
C1—C6	1.410 (3)	C13—C14	1.398 (3)
C2—C3	1.374 (3)	C14—C15	1.377 (3)
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.386 (3)	C15—H15	0.9300
C3—H3	0.9300	O3—C16	1.360 (3)
C4—C5	1.376 (3)	O3—H3C	0.8200
C4—H4	0.9300	C16—H16A	0.9600
C5—C6	1.396 (3)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C6—C7	1.493 (3)		
C1—O1—H1	109.5	C8—C9—H9A	109.5
C7—N1—N2	118.14 (16)	C8—C9—H9B	109.5
C7—N1—H1A	117.0	H9A—C9—H9B	109.5
N2—N1—H1A	123.9	C8—C9—H9C	109.5
C8—N2—N1	117.11 (16)	H9A—C9—H9C	109.5
C13—N3—H3A	111.1	H9B—C9—H9C	109.5
C13—N3—H3B	110.7	C11—C10—C15	117.13 (18)
H3A—N3—H3B	115.5	C11—C10—C8	123.66 (18)
O1—C1—C2	121.08 (18)	C15—C10—C8	119.20 (16)
O1—C1—C6	119.39 (16)	C12—C11—C10	121.49 (18)
C2—C1—C6	119.52 (18)	C12—C11—H11	119.3
C3—C2—C1	121.0 (2)	C10—C11—H11	119.3
C3—C2—H2	119.5	C11—C12—C13	120.74 (17)
C1—C2—H2	119.5	C11—C12—H12	119.6
C2—C3—C4	120.2 (2)	C13—C12—H12	119.6
C2—C3—H3	119.9	C12—C13—C14	118.10 (18)
C4—C3—H3	119.9	C12—C13—N3	121.54 (17)
C5—C4—C3	119.4 (2)	C14—C13—N3	120.31 (18)
C5—C4—H4	120.3	C15—C14—C13	120.90 (19)
C3—C4—H4	120.3	C15—C14—H14	119.6
C4—C5—C6	122.02 (19)	C13—C14—H14	119.6
C4—C5—H5	119.0	C14—C15—C10	121.63 (17)
C6—C5—H5	119.0	C14—C15—H15	119.2
C5—C6—C1	117.92 (17)	C10—C15—H15	119.2
C5—C6—C7	115.95 (17)	C16—O3—H3C	109.5

C1—C6—C7	126.12 (17)	O3—C16—H16A	109.5
O2—C7—N1	121.48 (17)	O3—C16—H16B	109.5
O2—C7—C6	120.40 (17)	H16A—C16—H16B	109.5
N1—C7—C6	118.09 (16)	O3—C16—H16C	109.5
N2—C8—C10	115.30 (17)	H16A—C16—H16C	109.5
N2—C8—C9	124.50 (18)	H16B—C16—H16C	109.5
C10—C8—C9	120.13 (17)		
C7—N1—N2—C8	174.29 (17)	C1—C6—C7—N1	−3.1 (3)
O1—C1—C2—C3	179.8 (2)	N1—N2—C8—C10	173.68 (15)
C6—C1—C2—C3	0.5 (3)	N1—N2—C8—C9	−3.4 (3)
C1—C2—C3—C4	−0.5 (4)	N2—C8—C10—C11	150.28 (19)
C2—C3—C4—C5	−0.1 (4)	C9—C8—C10—C11	−32.5 (3)
C3—C4—C5—C6	0.8 (3)	N2—C8—C10—C15	−30.6 (3)
C4—C5—C6—C1	−0.7 (3)	C9—C8—C10—C15	146.6 (2)
C4—C5—C6—C7	−179.79 (19)	C15—C10—C11—C12	0.5 (3)
O1—C1—C6—C5	−179.19 (18)	C8—C10—C11—C12	179.64 (18)
C2—C1—C6—C5	0.1 (3)	C10—C11—C12—C13	−1.4 (3)
O1—C1—C6—C7	−0.3 (3)	C11—C12—C13—C14	1.3 (3)
C2—C1—C6—C7	179.04 (18)	C11—C12—C13—N3	178.68 (18)
N2—N1—C7—O2	−6.7 (3)	C12—C13—C14—C15	−0.5 (3)
N2—N1—C7—C6	175.08 (16)	N3—C13—C14—C15	−177.91 (18)
C5—C6—C7—O2	−2.3 (3)	C13—C14—C15—C10	−0.3 (3)
C1—C6—C7—O2	178.73 (19)	C11—C10—C15—C14	0.3 (3)
C5—C6—C7—N1	175.89 (17)	C8—C10—C15—C14	−178.86 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3^{i}	0.82	1.98	2.781 (2)	164
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Symmetry codes: (i) *x*, *y*+1, *z*+1; (ii) *x*, *y*−1, *z*; (iii) −*x*, −*y*+1, −*z*.

Fig. 1

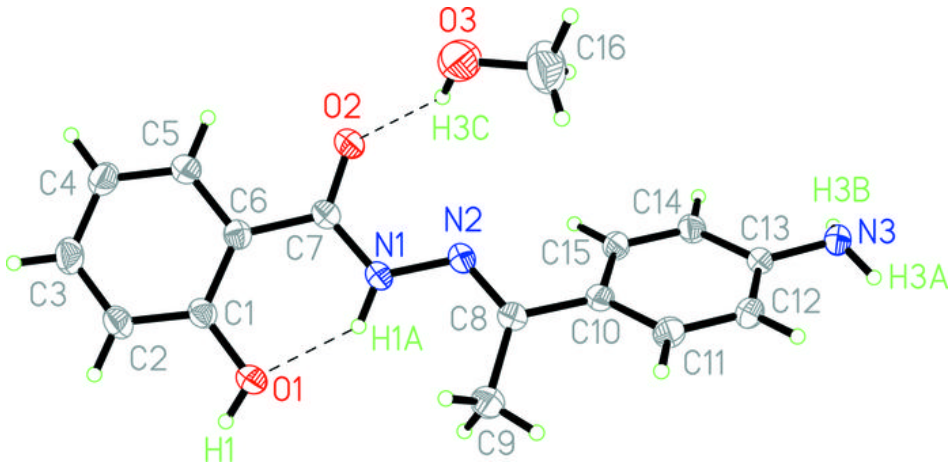
O3
C16
O2
H3C
C5
C4
C6
C7
N1
N2
C14
H3B
C13
N3
C15
H3A
C3
C1
C8
C10
C12
H1A
C2
O1
C11
H1
C9

Fig. 2