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N′-(4-Hydroxy-3-methoxybenzylidene)-isonicotinohydrazide methanol solvate

Xia Liu and Xue-Fang Shi*

Department of Chemistry, Tianjin Normal University, People's Republic of China
Correspondence e-mail: xuefangshi@126.com

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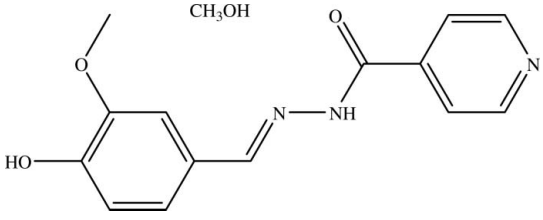
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.155; data-to-parameter ratio = 16.0.

The crystal packing of the title compound, $C_{14}H_{13}N_3O_3\cdot CH_3OH$, is stabilized by intermolecular N—H···O, O—H···N and O—H···O interactions. The dihedral angle between the two aromatic rings is 61.15 (5)°.

Related literature

For related literature, see: Allen *et al.* (1987); Edwards *et al.* (1975); Hadjoudis *et al.* (1987); Kushner *et al.* (1952); Parashar *et al.* (1988); Shi *et al.* (2007); Yu *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{13}N_3O_3\cdot CH_3OH$
M_r = 303.32
Monoclinic, $P2_1/c$
a = 13.2742 (12) Å
b = 6.3787 (7) Å
c = 17.5375 (16) Å
β = 105.151 (6)°
V = 1433.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
0.24 × 0.22 × 0.18 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.982
12902 measured reflections
3380 independent reflections
2324 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.155
S = 1.12
3380 reflections
211 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4^{i}	0.85 (2)	2.10 (2)	2.887 (2)	153 (2)
O4—H4···N1	0.90 (2)	2.32 (2)	3.033 (2)	136.2 (19)
O4—H4···O3	0.90 (2)	2.21 (2)	2.9367 (19)	138 (2)
O1—H1···N3^{ii}	0.92 (3)	1.88 (3)	2.751 (2)	156 (2)

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *CrystalStructure* (Rigaku, 2000).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2627).

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supplementary materials

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N'-(4-Hydroxy-3-methoxybenzylidene)isonicotinohydrazide methanol solvate

X. Liu and X.-F. Shi

Comment

Isonicotinylhydrazine is often used for the treatment of tuberculosis with anti fungicidal activity (Edwards *et al.*, 1975; Kushner *et al.*, 1952) and pharmacological activity (Parashar *et al.*, 1988). Earlier studies have reported that hydrazonecarbonyl compounds have special photochromic properties (Hadjoudis *et al.*, 1987).

The title compound contains one isonicotinic acid (4-hydroxy-3- methoxybenzylidene)hydrazide (IBHZ) molecule and one methanol molecule (Fig. 1). The IBHZ molecule adopts an E configuration with respect to the C=N bond. The bond lengths and angles in IBHZ are within normal ranges (Allen *et al.*, 1987). The C7—C8, C7=N1 and N1—N2 bond lengths are comparable with those observed in some related compounds (Shi *et al.*, 2007, Yu *et al.*, 2005). The dihedral angle between the two aromatic rings is 61.15 (5)°. In the crystal, the O atom of methanol molecule connected with H atom of acylamide group through hydrogen bond (N2—H···O4) as shown in Fig. 2. The molecular structure is stabilized by intermolecular O—H···N and O—H···O hydrogen bonds (Table 1).

Experimental

The title compound was synthesized by the reaction of isonicotinic acid hydrazide (1.37 g, 10 mmol) with 4-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) in methanol (60 ml). The mixture was stirred and refluxed for 5 h, producing a light-yellow precipitate. The product was isolated, recrystallized from methanol and then dried *in vacuo* to give the IBHZ compound in 82% yield. Single crystals suitable for X-ray diffraction were obtained by the slow evaporation of the IBHZ methanol solution.

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

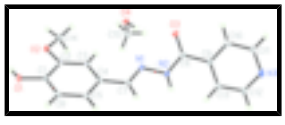

Fig. 1. A perpective view of the title compound. with displacement ellipsoids drawn at the 30% probability level.

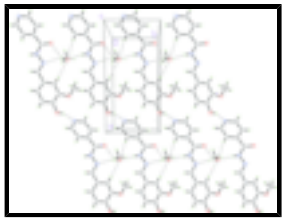

Fig. 2. A packing diagram of the title compound viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

N'-(4-Hydroxy-3-methoxybenzylidene)isonicotinohydrazide methanol solvate

Crystal data

$C_{14}H_{13}N_3O_3 \cdot CH_3OH$	$F_{000} = 640$
$M_r = 303.32$	$D_x = 1.406$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 13.2742$ (12) Å	Cell parameters from 3012 reflections
$b = 6.3787$ (7) Å	$\theta = 2.4$–27.8°
$c = 17.5375$ (16) Å	$\mu = 0.10$ mm^{-1}
$\beta = 105.151$ (6)°	$T = 113$ (2) K
$V = 1433.3$ (2) Å^3	Prism, colorless
$Z = 4$	0.24 × 0.22 × 0.18 mm

Data collection

Rigaku Saturn diffractometer	2324 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.044$
Monochromator: confocal	$\theta_{max} = 27.8^\circ$
$T = 113$(2) K	$\theta_{min} = 1.6^\circ$
ω scans	$h = -17 \rightarrow 17$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -22 \rightarrow 23$
$T_{min} = 0.976$, $T_{max} = 0.982$	$l = -22 \rightarrow 23$
12902 measured reflections	Standard reflections: ?
3380 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.155$	$w = 1/[\sigma^2(F_o^2) + (0.0832P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
3380 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}

211 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.81481 (9)	0.4960 (2)	0.20125 (9)	0.0226 (3)
H1	0.8228 (18)	0.384 (4)	0.1701 (15)	0.034*
O2	0.65087 (10)	0.28240 (19)	0.11681 (8)	0.0203 (3)
O3	0.14534 (10)	0.5921 (2)	0.12390 (10)	0.0324 (4)
O4	0.29362 (10)	0.2973 (2)	0.08392 (8)	0.0212 (3)
H4	0.2811 (18)	0.405 (4)	0.1129 (14)	0.032*
N1	0.33294 (11)	0.7533 (2)	0.13140 (10)	0.0189 (4)
N2	0.24502 (12)	0.8776 (3)	0.12472 (10)	0.0182 (4)
H2	0.2550 (17)	1.010 (4)	0.1275 (13)	0.027*
N3	−0.10337 (12)	1.1838 (3)	0.12720 (12)	0.0275 (4)
C1	0.71810 (13)	0.5725 (3)	0.19374 (11)	0.0163 (4)
C2	0.62897 (14)	0.4672 (3)	0.14833 (11)	0.0166 (4)
C3	0.53122 (13)	0.5516 (3)	0.13846 (11)	0.0158 (4)
H3	0.4715	0.4794	0.1082	0.019*
C4	0.51999 (13)	0.7457 (3)	0.17344 (11)	0.0159 (4)
C5	0.60767 (14)	0.8465 (3)	0.21904 (11)	0.0168 (4)
H5	0.6006	0.9774	0.2429	0.020*
C6	0.70578 (13)	0.7574 (3)	0.23001 (11)	0.0175 (4)
H6	0.7650	0.8253	0.2630	0.021*
C7	0.41905 (14)	0.8478 (3)	0.16264 (11)	0.0179 (4)
H7	0.4170	0.9890	0.1793	0.021*
C8	0.15396 (14)	0.7824 (3)	0.12207 (12)	0.0188 (4)
C9	0.06400 (14)	0.9253 (3)	0.12005 (12)	0.0190 (4)
C10	−0.01499 (15)	0.8525 (3)	0.15132 (13)	0.0249 (5)
H10	−0.0138	0.7131	0.1706	0.030*
C11	−0.09532 (16)	0.9868 (4)	0.15384 (14)	0.0292 (5)
H11	−0.1486	0.9358	0.1761	0.035*
C12	−0.02792 (15)	1.2498 (3)	0.09594 (14)	0.0269 (5)
H12	−0.0324	1.3883	0.0754	0.032*
C13	0.05660 (14)	1.1284 (3)	0.09165 (13)	0.0232 (5)

H13	0.1090	1.1838	0.0694	0.028*
C14	0.56812 (15)	0.1851 (3)	0.05950 (12)	0.0236 (5)
H14A	0.5112	0.1511	0.0834	0.035*
H14B	0.5937	0.0561	0.0406	0.035*
H14C	0.5425	0.2809	0.0150	0.035*
C15	0.26698 (18)	0.3322 (4)	0.00129 (13)	0.0347 (5)
H15A	0.1940	0.3771	−0.0165	0.052*
H15B	0.2764	0.2021	−0.0258	0.052*
H15C	0.3122	0.4416	−0.0110	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0129 (6)	0.0250 (8)	0.0297 (8)	0.0042 (5)	0.0052 (6)	−0.0058 (6)
O2	0.0177 (6)	0.0174 (7)	0.0251 (8)	0.0019 (5)	0.0045 (6)	−0.0054 (6)
O3	0.0203 (7)	0.0156 (7)	0.0613 (12)	−0.0007 (5)	0.0105 (7)	0.0000 (7)
O4	0.0247 (7)	0.0167 (7)	0.0235 (8)	0.0006 (5)	0.0083 (6)	−0.0023 (6)
N1	0.0156 (7)	0.0177 (8)	0.0241 (9)	0.0046 (6)	0.0066 (7)	0.0014 (7)
N2	0.0139 (7)	0.0124 (7)	0.0286 (10)	0.0031 (6)	0.0057 (7)	−0.0016 (7)
N3	0.0146 (8)	0.0262 (9)	0.0417 (12)	0.0007 (7)	0.0078 (7)	−0.0075 (8)
C1	0.0137 (8)	0.0195 (9)	0.0174 (10)	0.0021 (7)	0.0069 (7)	0.0026 (7)
C2	0.0178 (9)	0.0127 (8)	0.0207 (10)	0.0016 (7)	0.0078 (7)	0.0027 (7)
C3	0.0148 (8)	0.0157 (9)	0.0171 (10)	−0.0013 (7)	0.0045 (7)	0.0013 (7)
C4	0.0158 (8)	0.0171 (9)	0.0164 (10)	0.0011 (7)	0.0070 (7)	0.0031 (7)
C5	0.0224 (9)	0.0145 (9)	0.0160 (10)	−0.0001 (7)	0.0095 (8)	−0.0009 (7)
C6	0.0151 (8)	0.0203 (9)	0.0168 (10)	−0.0025 (7)	0.0038 (7)	−0.0001 (7)
C7	0.0190 (9)	0.0152 (9)	0.0222 (11)	0.0020 (7)	0.0101 (8)	0.0029 (7)
C8	0.0171 (9)	0.0176 (9)	0.0212 (11)	−0.0011 (7)	0.0041 (8)	−0.0011 (8)
C9	0.0150 (8)	0.0180 (9)	0.0247 (11)	−0.0018 (7)	0.0063 (8)	−0.0038 (8)
C10	0.0217 (10)	0.0229 (10)	0.0313 (13)	−0.0014 (8)	0.0088 (9)	0.0005 (9)
C11	0.0205 (10)	0.0335 (12)	0.0381 (14)	−0.0026 (8)	0.0157 (9)	−0.0025 (10)
C12	0.0184 (9)	0.0196 (10)	0.0429 (14)	0.0012 (8)	0.0085 (9)	−0.0010 (9)
C13	0.0152 (9)	0.0209 (10)	0.0340 (13)	0.0004 (7)	0.0071 (8)	0.0006 (9)
C14	0.0225 (10)	0.0191 (10)	0.0264 (12)	0.0007 (7)	0.0015 (8)	−0.0040 (8)
C15	0.0366 (12)	0.0462 (14)	0.0219 (13)	−0.0004 (10)	0.0088 (10)	−0.0003 (10)

Geometric parameters (Å, °)

O1—C1	1.347 (2)	C5—C6	1.387 (2)
O1—H1	0.92 (3)	C5—H5	0.9500
O2—C2	1.365 (2)	C6—H6	0.9500
O2—C14	1.423 (2)	C7—H7	0.9500
O3—C8	1.221 (2)	C8—C9	1.495 (2)
O4—C15	1.417 (3)	C9—C13	1.382 (3)
O4—H4	0.90 (2)	C9—C10	1.384 (3)
N1—C7	1.282 (2)	C10—C11	1.378 (3)
N1—N2	1.390 (2)	C10—H10	0.9500
N2—C8	1.343 (2)	C11—H11	0.9500
N2—H2	0.85 (2)	C12—C13	1.381 (3)

N3—C12	1.330 (3)	C12—H12	0.9500
N3—C11	1.335 (3)	C13—H13	0.9500
C1—C6	1.370 (3)	C14—H14A	0.9800
C1—C2	1.412 (2)	C14—H14B	0.9800
C2—C3	1.373 (2)	C14—H14C	0.9800
C3—C4	1.407 (3)	C15—H15A	0.9800
C3—H3	0.9500	C15—H15B	0.9800
C4—C5	1.386 (2)	C15—H15C	0.9800
C4—C7	1.457 (2)		
C1—O1—H1	118.5 (14)	O3—C8—C9	121.82 (16)
C2—O2—C14	117.13 (14)	N2—C8—C9	115.55 (16)
C15—O4—H4	115.1 (15)	C13—C9—C10	117.86 (17)
C7—N1—N2	113.77 (16)	C13—C9—C8	124.13 (17)
C8—N2—N1	118.26 (15)	C10—C9—C8	117.96 (17)
C8—N2—H2	125.0 (15)	C11—C10—C9	118.47 (19)
N1—N2—H2	116.4 (15)	C11—C10—H10	120.8
C12—N3—C11	116.27 (17)	C9—C10—H10	120.8
O1—C1—C6	119.51 (16)	N3—C11—C10	124.47 (19)
O1—C1—C2	121.32 (16)	N3—C11—H11	117.8
C6—C1—C2	119.17 (16)	C10—C11—H11	117.8
O2—C2—C3	125.58 (16)	N3—C12—C13	123.69 (19)
O2—C2—C1	113.80 (15)	N3—C12—H12	118.2
C3—C2—C1	120.62 (17)	C13—C12—H12	118.2
C2—C3—C4	119.64 (16)	C12—C13—C9	119.22 (18)
C2—C3—H3	120.2	C12—C13—H13	120.4
C4—C3—H3	120.2	C9—C13—H13	120.4
C5—C4—C3	119.41 (16)	O2—C14—H14A	109.5
C5—C4—C7	118.23 (17)	O2—C14—H14B	109.5
C3—C4—C7	122.35 (16)	H14A—C14—H14B	109.5
C4—C5—C6	120.41 (17)	O2—C14—H14C	109.5
C4—C5—H5	119.8	H14A—C14—H14C	109.5
C6—C5—H5	119.8	H14B—C14—H14C	109.5
C1—C6—C5	120.66 (17)	O4—C15—H15A	109.5
C1—C6—H6	119.7	O4—C15—H15B	109.5
C5—C6—H6	119.7	H15A—C15—H15B	109.5
N1—C7—C4	122.20 (17)	O4—C15—H15C	109.5
N1—C7—H7	118.9	H15A—C15—H15C	109.5
C4—C7—H7	118.9	H15B—C15—H15C	109.5
O3—C8—N2	122.58 (17)		
C7—N1—N2—C8	−155.06 (18)	C5—C4—C7—N1	−169.62 (18)
C14—O2—C2—C3	10.1 (3)	C3—C4—C7—N1	11.3 (3)
C14—O2—C2—C1	−169.42 (17)	N1—N2—C8—O3	−1.3 (3)
O1—C1—C2—O2	2.4 (3)	N1—N2—C8—C9	176.27 (16)
C6—C1—C2—O2	−178.45 (16)	O3—C8—C9—C13	−157.0 (2)
O1—C1—C2—C3	−177.20 (17)	N2—C8—C9—C13	25.4 (3)
C6—C1—C2—C3	2.0 (3)	O3—C8—C9—C10	25.5 (3)
O2—C2—C3—C4	−179.00 (17)	N2—C8—C9—C10	−152.07 (19)
C1—C2—C3—C4	0.5 (3)	C13—C9—C10—C11	−1.2 (3)

C2—C3—C4—C5	−1.6 (3)	C8—C9—C10—C11	176.39 (18)
C2—C3—C4—C7	177.49 (17)	C12—N3—C11—C10	0.5 (3)
C3—C4—C5—C6	0.2 (3)	C9—C10—C11—N3	0.8 (3)
C7—C4—C5—C6	−178.93 (17)	C11—N3—C12—C13	−1.4 (3)
O1—C1—C6—C5	175.78 (17)	N3—C12—C13—C9	1.0 (3)
C2—C1—C6—C5	−3.4 (3)	C10—C9—C13—C12	0.4 (3)
C4—C5—C6—C1	2.4 (3)	C8—C9—C13—C12	−177.05 (18)
N2—N1—C7—C4	−178.82 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4^{i}	0.85 (2)	2.10 (2)	2.887 (2)	153 (2)
O4—H4···N1	0.90 (2)	2.32 (2)	3.033 (2)	136.2 (19)
O4—H4···O3	0.90 (2)	2.21 (2)	2.9367 (19)	138 (2)
O1—H1···N3^{ii}	0.92 (3)	1.88 (3)	2.751 (2)	156 (2)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*+1, *y*−1, *z*.

Fig. 1

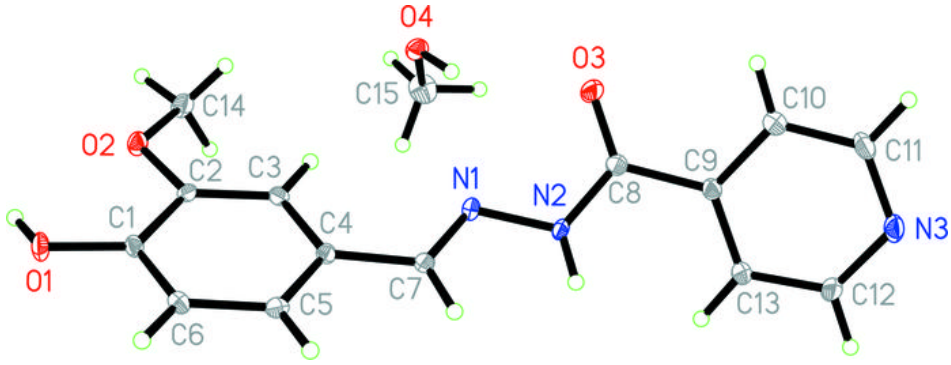
O4
C15
O3
C10
C14
O2
C2
C3
N1
N2
C8
C9
C11
C1
C4
N3
O1
C7
C5
C13
C12
C6

Fig. 2