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(*E*)-2-(Isonicotinoylhydrazonomethyl)-benzoic acid methanol monosolvate

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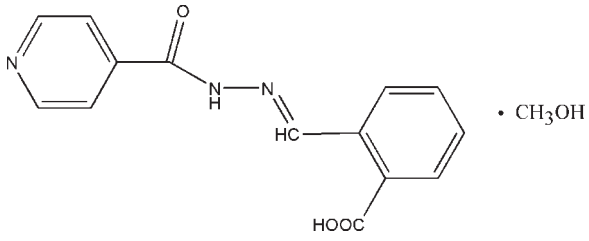
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.141; data-to-parameter ratio = 12.6.

The title compound, $C_{14}H_{11}N_3O_3\cdot CH_4O$, was synthesized by the condensation reaction of isonicotinohydrazide with an equimolar quantity of 2-formylbenzoic acid in methanol. The hydrazone molecule displays an *E* configuration about the C=N bond. The dihedral angel between the pyridine and the benzene rings is 12.04 (5)°. In the crystal structure, molecules are linked by O—H···N, O—H···O and N—H···O hydrogen-bonding interactions.

Related literature

For general background to hydrazones, see: Dhande *et al.* (2007). For a related structure, see: Zhang *et al.* (2009).



Experimental

Crystal data

$C_{14}H_{11}N_3O_3\cdot CH_4O$
M_r = 301.30
Monoclinic, $P2_1/n$
a = 6.9768 (11) Å
b = 12.2103 (13) Å
c = 17.2650 (19) Å
β = 95.497 (1)°
V = 1464.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.43 × 0.19 × 0.15 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.985
7290 measured reflections
2508 independent reflections
1233 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.141
S = 0.99
2508 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 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
N1—H1···O4^{i}	0.86	2.13	2.891 (3)	148
O4—H4···O1	0.82	2.14	2.864 (4)	148
O2—H2···N3^{ii}	0.82	1.76	2.565 (3)	165

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2160).

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supporting information

Acta Cryst. (2009). E**65**, o2578 [doi:10.1107/S1600536809038902]

(*E*)-2-(Isonicotinoylhydrazonomethyl)benzoic acid methanol monosolvate

Wenkuan Li, Handong Yin, Liyuan Wen, Jichun Cui and Daqi Wang

S1. Comment

Hydrazones have been attracted significant attention because of their physiological activity, coordinative capability, and applications in analytical chemistry (Dhande *et al.* 2007). Recently, a large number of hydrazone compounds have been reported (Zhang *et al.* 2009). As a contribution to the chemistry of hydrazone, we report here the synthesis and crystal structure of the title compound (I).

The crystal structure of (I) is built up of hydrazone and methanol molecules (Fig.1). The dihedral angel between the pyridine and the benzene rings is 12.04 (5) °. The hydrazone molecule crystallizes in E conformation. In the crystal structure, three kinds of intermolecular O—H···N, O—H···O and N—H···O hydrogen bonding interactions are observed and the crystal packing is stabilized by these intermolecular interactions. (Table 1. and Fig. 2).

S2. Experimental

Isonicotinohydrazide (10 mmol) was dissolved in ethanol (40 ml), then 2-formylbenzoic acid (10 mmol) was added into the solution. The reaction mixture was heated under reflux for 2 h. After the solution had cooled to room white sediment appeared. The product was crystallized from methanol. Anal. Calcd (%) for [($C_{14}H_{11}N_3O_3$).($C_1H_4O_1$)] (Mr = 301.30): C, 59.79; H, 5.02; N, 13.95; O, 21.24 Found (%): C, 59.83; H, 5.00; N, 13.92; O, 21.25

S3. Refinement

The imino H atom was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.86 Å. Other H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93 (aromatic and methylene) and 0.96(methyl), O—H = 0.82, and with U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C15 and O).

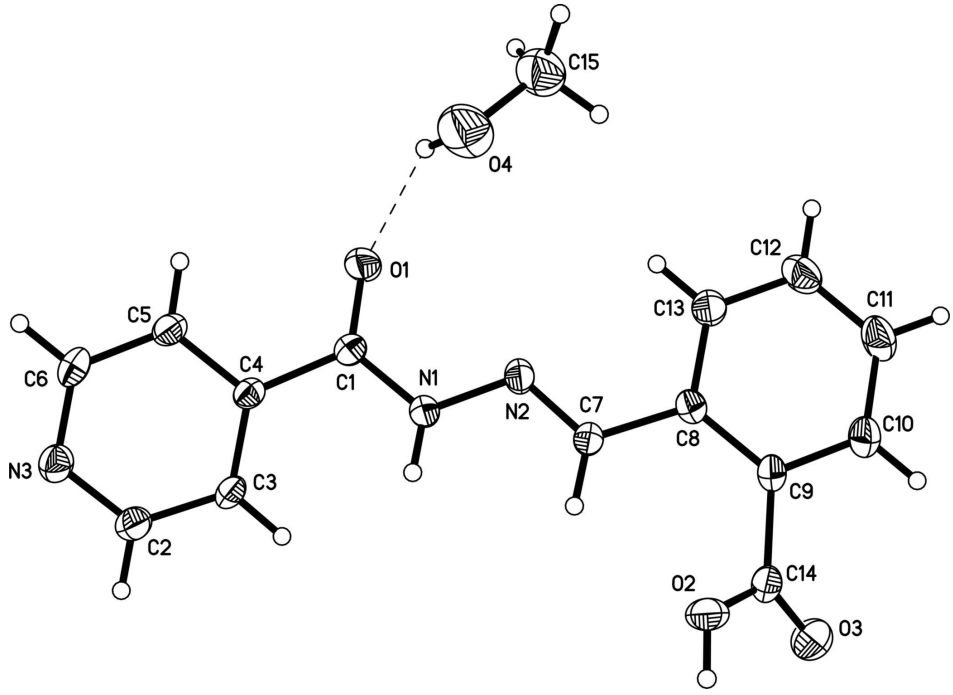

Figure 1

The molecular structure (I) with 50% probability displacement ellipsoids. O—H···O hydrogen bond is shown in dashed line.

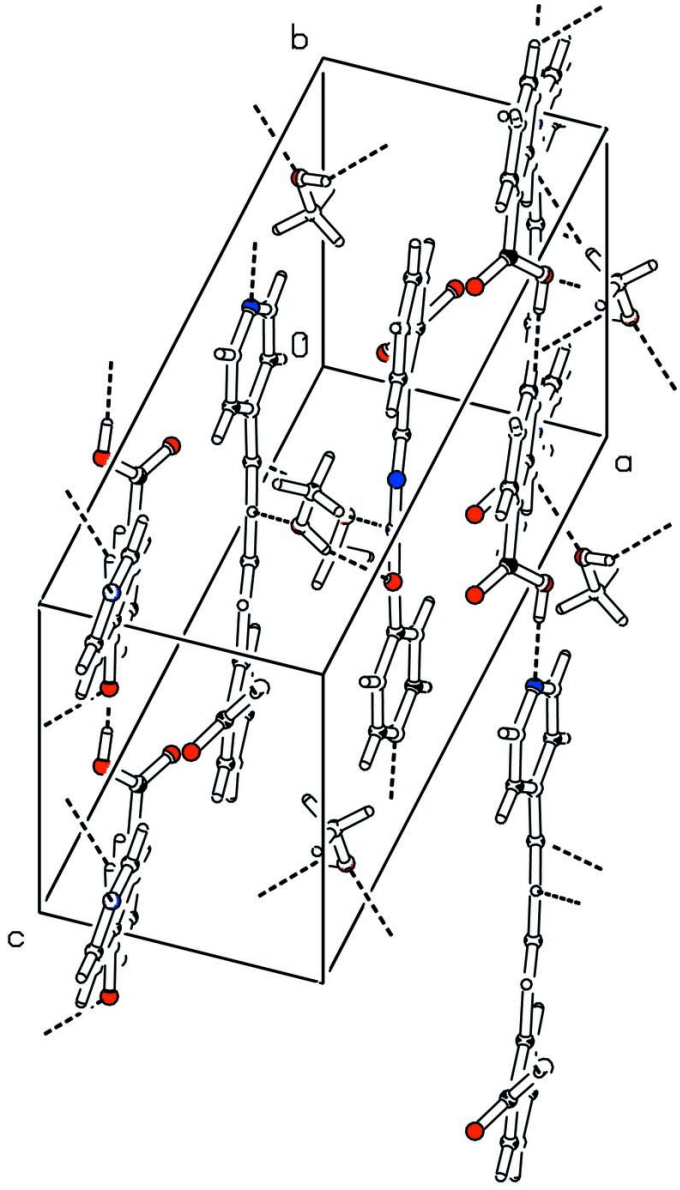

Figure 2

The molecular packing of the title compound. Hydrogen bonding is shown in dashed lines.

(*E*)-2-(Isonicotinoylhydrazonomethyl)benzoic acid methanol monosolvate

Crystal data

$C_{14}H_{11}N_3O_3 \cdot CH_4O$
$M_r = 301.30$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.9768\ (11)$ Å
$b = 12.2103\ (13)$ Å
$c = 17.2650\ (19)$ Å
$\beta = 95.497\ (1)°$
$V = 1464.0\ (3)$ Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1170 reflections
$\theta = 2.4–21.5°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Block, yellow
0.43 × 0.19 × 0.15 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.985
7290 measured reflections
2508 independent reflections
1233 reflections with $I > 2\sigma(I)$
R_{int} = 0.076
θ_{max} = 25.0°, θ_{min} = 2.1°
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 14$
$l = -19 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.141
S = 0.99
2508 reflections
199 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 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}
N1	0.2762 (4)	0.52906 (18)	0.52873 (12)	0.0371 (7)
H1	0.3076	0.5854	0.5570	0.045*
N2	0.2993 (4)	0.42528 (18)	0.55868 (14)	0.0390 (7)
N3	0.1111 (4)	0.8638 (2)	0.36585 (14)	0.0438 (7)
O1	0.1643 (3)	0.46359 (16)	0.41058 (12)	0.0537 (7)
O2	0.5588 (3)	0.46550 (16)	0.77984 (11)	0.0523 (7)
H2	0.5752	0.5125	0.8137	0.078*
O3	0.4040 (4)	0.38081 (18)	0.86917 (13)	0.0654 (8)
O4	0.4701 (4)	0.3132 (2)	0.38940 (15)	0.0876 (10)
H4	0.3697	0.3467	0.3775	0.131*
C1	0.2034 (5)	0.5407 (2)	0.45415 (17)	0.0361 (8)
C2	0.1221 (5)	0.8462 (3)	0.44169 (18)	0.0474 (9)
H2A	0.1103	0.9057	0.4745	0.057*
C3	0.1501 (5)	0.7440 (2)	0.47459 (17)	0.0406 (9)
H3	0.1551	0.7347	0.5282	0.049*
C4	0.1704 (4)	0.6558 (2)	0.42630 (15)	0.0312 (7)
C5	0.1561 (5)	0.6736 (2)	0.34726 (16)	0.0380 (8)
H5	0.1660	0.6155	0.3131	0.046*
C6	0.1271 (5)	0.7785 (3)	0.31942 (18)	0.0429 (9)
H6	0.1183	0.7900	0.2659	0.052*
C7	0.3532 (4)	0.4180 (2)	0.63048 (16)	0.0347 (8)
H7	0.3768	0.4809	0.6602	0.042*
C8	0.3783 (4)	0.3093 (2)	0.66650 (17)	0.0322 (8)
C9	0.4202 (5)	0.2943 (2)	0.74724 (17)	0.0353 (8)
C10	0.4338 (5)	0.1888 (2)	0.77718 (19)	0.0456 (9)

H10	0.4572	0.1786	0.8306	0.055*
C11	0.4133 (5)	0.0993 (3)	0.7289 (2)	0.0548 (10)
H11	0.4255	0.0290	0.7496	0.066*
C12	0.3750 (5)	0.1135 (3)	0.6503 (2)	0.0551 (10)
H12	0.3624	0.0530	0.6175	0.066*
C13	0.3551 (5)	0.2177 (2)	0.61980 (18)	0.0434 (9)
H13	0.3254	0.2264	0.5665	0.052*
C14	0.4585 (5)	0.3846 (3)	0.80510 (18)	0.0416 (9)
C15	0.4316 (6)	0.2034 (3)	0.3937 (2)	0.0742 (13)
H15A	0.4264	0.1828	0.4471	0.111*
H15B	0.5314	0.1626	0.3720	0.111*
H15C	0.3101	0.1879	0.3648	0.111*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0488 (19)	0.0284 (14)	0.0325 (15)	0.0004 (13)	−0.0045 (13)	0.0009 (11)
N2	0.0479 (19)	0.0308 (14)	0.0365 (15)	−0.0022 (13)	−0.0049 (13)	0.0049 (12)
N3	0.046 (2)	0.0452 (16)	0.0389 (16)	0.0059 (14)	−0.0003 (13)	0.0045 (13)
O1	0.076 (2)	0.0369 (12)	0.0436 (13)	0.0013 (12)	−0.0172 (12)	−0.0072 (11)
O2	0.078 (2)	0.0419 (13)	0.0375 (13)	−0.0119 (13)	0.0066 (12)	−0.0091 (10)
O3	0.099 (2)	0.0622 (16)	0.0371 (14)	0.0029 (15)	0.0188 (14)	0.0043 (12)
O4	0.091 (3)	0.0691 (19)	0.096 (2)	−0.0088 (17)	−0.0259 (17)	−0.0136 (15)
C1	0.041 (2)	0.0362 (18)	0.0302 (17)	0.0006 (16)	−0.0025 (15)	−0.0011 (14)
C2	0.063 (3)	0.045 (2)	0.0339 (19)	0.0114 (18)	0.0027 (16)	−0.0026 (15)
C3	0.055 (3)	0.0425 (19)	0.0232 (17)	0.0076 (17)	0.0002 (16)	0.0029 (14)
C4	0.030 (2)	0.0366 (17)	0.0266 (16)	0.0033 (14)	0.0013 (13)	−0.0005 (13)
C5	0.041 (2)	0.0438 (19)	0.0284 (17)	0.0048 (16)	0.0018 (15)	−0.0019 (14)
C6	0.046 (2)	0.054 (2)	0.0286 (18)	0.0026 (18)	−0.0005 (16)	0.0094 (16)
C7	0.041 (2)	0.0315 (17)	0.0309 (17)	−0.0005 (15)	−0.0007 (14)	0.0031 (13)
C8	0.028 (2)	0.0290 (17)	0.0388 (18)	−0.0009 (14)	0.0007 (14)	0.0024 (14)
C9	0.034 (2)	0.0332 (17)	0.0374 (18)	0.0021 (15)	−0.0006 (15)	0.0079 (14)
C10	0.051 (2)	0.041 (2)	0.044 (2)	0.0005 (17)	0.0033 (17)	0.0115 (16)
C11	0.061 (3)	0.0333 (19)	0.069 (3)	−0.0039 (18)	0.001 (2)	0.0131 (18)
C12	0.067 (3)	0.032 (2)	0.066 (2)	−0.0038 (18)	0.001 (2)	−0.0039 (18)
C13	0.048 (3)	0.0372 (19)	0.0430 (19)	−0.0029 (17)	−0.0059 (17)	−0.0002 (15)
C14	0.050 (2)	0.0410 (19)	0.0329 (18)	0.0089 (18)	0.0001 (16)	0.0054 (15)
C15	0.085 (4)	0.058 (3)	0.077 (3)	−0.005 (2)	−0.004 (2)	−0.007 (2)

Geometric parameters (Å, º)

N1—C1	1.346 (3)	C5—C6	1.376 (4)
N1—N2	1.372 (3)	C5—H5	0.9300
N1—H1	0.8600	C6—H6	0.9300
N2—C7	1.264 (3)	C7—C8	1.469 (4)
N3—C2	1.322 (4)	C7—H7	0.9300
N3—C6	1.325 (4)	C8—C13	1.380 (4)
O1—C1	1.219 (3)	C8—C9	1.409 (4)

O2—C14	1.309 (4)	C9—C10	1.388 (4)
O2—H2	0.8200	C9—C14	1.495 (4)
O3—C14	1.205 (3)	C10—C11	1.374 (4)
O4—C15	1.371 (4)	C10—H10	0.9300
O4—H4	0.8200	C11—C12	1.369 (4)
C1—O1	1.219 (3)	C11—H11	0.9300
C1—C4	1.496 (4)	C12—C13	1.378 (4)
C2—C3	1.377 (4)	C12—H12	0.9300
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.377 (4)	C15—H15A	0.9600
C3—H3	0.9300	C15—H15B	0.9600
C4—C5	1.376 (4)	C15—H15C	0.9600

C1—N1—N2	118.6 (2)	C8—C7—H7	120.3
C1—N1—H1	120.7	C13—C8—C9	118.3 (3)
N2—N1—H1	120.7	C13—C8—C7	118.9 (3)
C7—N2—N1	116.6 (2)	C9—C8—C7	122.8 (3)
C2—N3—C6	118.1 (3)	C10—C9—C8	119.3 (3)
C14—O2—H2	109.5	C10—C9—C14	115.7 (3)
C15—O4—H4	109.5	C8—C9—C14	124.9 (3)
O1—C1—N1	123.4 (3)	C11—C10—C9	120.9 (3)
O1—C1—N1	123.4 (3)	C11—C10—H10	119.6
O1—C1—C4	120.6 (3)	C9—C10—H10	119.6
O1—C1—C4	120.6 (3)	C12—C11—C10	120.0 (3)
N1—C1—C4	116.0 (3)	C12—C11—H11	120.0
N3—C2—C3	123.3 (3)	C10—C11—H11	120.0
N3—C2—H2A	118.4	C11—C12—C13	119.9 (3)
C3—C2—H2A	118.4	C11—C12—H12	120.0
C2—C3—C4	118.5 (3)	C13—C12—H12	120.0
C2—C3—H3	120.7	C12—C13—C8	121.6 (3)
C4—C3—H3	120.7	C12—C13—H13	119.2
C5—C4—C3	118.3 (3)	C8—C13—H13	119.2
C5—C4—C1	117.5 (2)	O3—C14—O2	124.1 (3)
C3—C4—C1	124.2 (2)	O3—C14—C9	122.2 (3)
C6—C5—C4	119.2 (3)	O2—C14—C9	113.7 (3)
C6—C5—H5	120.4	O4—C15—H15A	109.5
C4—C5—H5	120.4	O4—C15—H15B	109.5
N3—C6—C5	122.5 (3)	H15A—C15—H15B	109.5
N3—C6—H6	118.7	O4—C15—H15C	109.5
C5—C6—H6	118.7	H15A—C15—H15C	109.5
N2—C7—C8	119.4 (3)	H15B—C15—H15C	109.5
N2—C7—H7	120.3		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{i}	0.86	2.13	2.891 (3)	148

O4—H4···O1	0.82	2.14	2.864 (4)	148
O2—H2···N3^{ii}	0.82	1.76	2.565 (3)	165

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1/2, -y+3/2, z+1/2$.