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A new polymorph of 2′-(4-methoxybenzylidene)isonicotinohydrazide monohydrate

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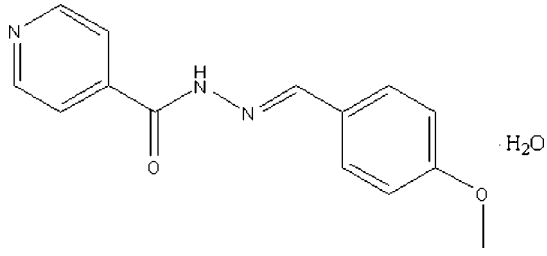
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.051; wR factor = 0.135; data-to-parameter ratio = 9.7.

The title compound, $C_{14}H_{13}N_3O_2\cdot H_2O$, is a new orthorhombic polymorph of a structure reported previously [Jing, Fan, Yu, Chen & Deng (2005). *Acta Cryst.* E**61**, o3208–o3209]. The crystal structure is stabilized by a network of N—H···O and O—H···N hydrogen bonds linking three molecules to each water molecule, forming layers in the *bc* plane.

Related literature

For a polymorph of the title compound, see: Jing *et al.* (2005). For other isonicotinohydrazide derivatives, see: Maurya *et al.* (2005); Qiu *et al.* (2006); Yang *et al.* (2006); Yin *et al.* (2005).



Experimental

Crystal data

$C_{14}H_{13}N_3O_2\cdot H_2O$
M_r = 273.29
Orthorhombic, $P2_12_12_1$
a = 7.415 (5) Å
b = 12.621 (8) Å
c = 14.4387 (13) Å
V = 1351.1 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.40 × 0.30 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer
Absorption correction: ψ scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.962, T_{max} = 0.976
12498 measured reflections
1768 independent reflections
1130 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.136
S = 1.09
1768 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O3	0.86	2.13	2.957 (4)	162
O3—H3WB···O1^{i}	0.96	1.96	2.889 (4)	161
O3—H3WA···N1^{ii}	0.99	1.92	2.901 (4)	170

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

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2327).

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supplementary materials

Acta Cryst. (2007). E**63**, o3974 [**doi:10.1107/S1600536807042067**]

A new polymorph of 2'-(4-methoxybenzylidene)isonicotinohydrazide monohydrate

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Comment

As part of a study of substituted isonicotinohydrazides and their complexes, a new polymorph of 2'-(4-Methoxybenzylidene)isonicotinohydrazide monohydrate, I, was synthesized in our group and its structure is reported here, Fig. 1. A polymorph of this molecule II has also been reported (Jing *et al.*, 2005) and the two compounds show similar molecular configurations, bond lengths and angles. In I, the system (C7–C14/N2/N3/O2) is planar, the r.m.s. deviation of fitted atoms being 0.0481 Å, and the pyridine ring(C1–C6/N1) is also planar with an r.m.s. deviation of 0.0059 Å. The dihedral angle between the two planes is 43.20 (9)°, while the corresponding angle in polymorph II is 36.78 (3)°.

There are three hydrogen bonds for every solvent water molecule in the crystal structure, Table 1. These link the molecules of I and the water solvate into layers in the *bc* plane, Fig 2.

Experimental

Pyridine-4-carboxylic acid hydrazide (10 mmol, 1.37 g) was dissolved in anhydrous ethanol (10 ml) and the mixture was stirred for several minutes at 351 K. 4-methoxybenzaldehyde (10 mmol, 1.36 g) in ethanol (10 ml) was added dropwise and the mixture refluxed for 3 h. The solid product was filtered off and recrystallized from a methanol solution. Single crystals of (I) were obtained after 7 d.

Refinement

The H Atoms in water molecule were located in a difference Fourier maps, and then allowed to ride on the oxygen atom with U_{eq} = 1.5U_{eq}(O). The other H atoms were placed in idealized positions and treated as riding with C—H = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic 0.96\ %A, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms and 0.86 Å, U_{iso} = 1.2U_{eq}(O) for the NH groups.

In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Figures

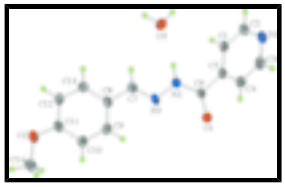

Fig. 1. The molecular structure of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

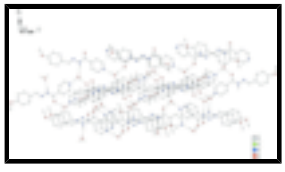

Fig. 2. A plot illustrating extended three-dimensional structure of (I), hydrogen bonds are drawn as dashed lines. H atoms not-involved in hydrogen bonding have been omitted.

2'-(4-methoxybenzylidene)isonicotinohydrazide monohydrate

Crystal data

$C_{14}H_{13}N_3O_2 \cdot H_2O$	$F_{000} = 576$
$M_r = 273.29$	$D_x = 1.343$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 12498 reflections
$a = 7.415$ (5) Å	$\theta = 3.1$–27.5º
$b = 12.621$ (8) Å	$\mu = 0.10$ mm^{-1}
$c = 14.4387$ (13) Å	$T = 293$ (2) K
$V = 1351.1$ (13) Å^3	Prism, colourless
$Z = 4$	0.40 × 0.30 × 0.25 mm

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer	1768 independent reflections
Radiation source: fine-focus sealed tube	1130 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.075$
$T = 293$(2) K	$\theta_{max} = 27.5$º
ω scans	$\theta_{min} = 3.1$º
Absorption correction: ψ scan (TEXRAY; Molecular Structure Corporation, 1999)	$h = -9 \rightarrow 9$
$T_{min} = 0.962$, $T_{max} = 0.976$	$k = -15 \rightarrow 16$
12498 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.136$	$\Delta\rho_{max} = 0.21$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
1768 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
183 parameters	Extinction coefficient: 0.010 (3)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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}
N1	0.1918 (4)	0.3865 (3)	0.0147 (2)	0.0568 (8)
N2	0.1289 (5)	0.2584 (2)	0.34455 (19)	0.0499 (7)
H2B	0.1506	0.3254	0.3441	0.060*
N3	0.1091 (4)	0.2054 (2)	0.42850 (18)	0.0499 (7)
O1	0.0859 (4)	0.10855 (18)	0.25897 (17)	0.0588 (7)
O2	0.1211 (4)	0.1269 (2)	0.87090 (17)	0.0633 (7)
C1	0.0711 (5)	0.3734 (3)	0.1690 (2)	0.0464 (8)
H1A	0.0083	0.4050	0.2174	0.056*
C2	0.1005 (5)	0.4270 (3)	0.0870 (3)	0.0538 (9)
H2A	0.0543	0.4952	0.0815	0.065*
C3	0.2553 (6)	0.2881 (3)	0.0242 (3)	0.0593 (10)
H3A	0.3193	0.2587	−0.0249	0.071*
C4	0.2308 (6)	0.2268 (3)	0.1036 (2)	0.0553 (10)
H4A	0.2758	0.1581	0.1068	0.066*
C5	0.1382 (5)	0.2703 (2)	0.1774 (2)	0.0432 (8)
C6	0.1143 (5)	0.2054 (3)	0.2645 (2)	0.0453 (8)
C7	0.1430 (5)	0.2620 (3)	0.4997 (3)	0.0519 (9)
H7A	0.1759	0.3324	0.4910	0.062*
C8	0.1328 (5)	0.2214 (3)	0.5944 (2)	0.0481 (8)
C9	0.0858 (6)	0.1177 (3)	0.6160 (2)	0.0585 (10)
H9A	0.0580	0.0707	0.5685	0.070*
C10	0.0795 (6)	0.0829 (3)	0.7076 (3)	0.0589 (10)
H10A	0.0489	0.0130	0.7208	0.071*
C11	0.1190 (5)	0.1526 (3)	0.7790 (2)	0.0494 (9)
C12	0.1615 (6)	0.2564 (3)	0.7585 (3)	0.0578 (10)
H12A	0.1840	0.3040	0.8063	0.069*
C13	0.1708 (5)	0.2900 (3)	0.6680 (3)	0.0568 (10)
H13A	0.2029	0.3598	0.6555	0.068*
C14	0.0980 (7)	0.0184 (3)	0.8971 (3)	0.0709 (12)
H14A	0.1091	0.0120	0.9631	0.106*
H14B	0.1886	−0.0241	0.8675	0.106*
H14C	−0.0194	−0.0054	0.8782	0.106*
O3	0.1336 (5)	0.4908 (2)	0.3704 (2)	0.0803 (10)

H3WB	0.0422	0.5313	0.3391	0.121*
H3WA	0.2055	0.5323	0.4153	0.121*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.071 (2)	0.066 (2)	0.0328 (16)	−0.0101 (18)	0.0001 (14)	0.0050 (15)
N2	0.0706 (19)	0.0437 (14)	0.0354 (16)	−0.0038 (15)	−0.0052 (15)	0.0037 (11)
N3	0.0684 (19)	0.0549 (16)	0.0263 (14)	0.0020 (17)	−0.0028 (14)	0.0058 (12)
O1	0.0901 (19)	0.0451 (13)	0.0413 (15)	−0.0018 (14)	−0.0058 (14)	0.0007 (10)
O2	0.0882 (19)	0.0667 (16)	0.0350 (14)	0.0010 (17)	−0.0002 (14)	0.0037 (12)
C1	0.0572 (19)	0.0500 (18)	0.0321 (17)	−0.0063 (17)	−0.0004 (16)	−0.0049 (15)
C2	0.064 (2)	0.0531 (19)	0.044 (2)	−0.0012 (19)	−0.0031 (19)	0.0052 (16)
C3	0.068 (2)	0.073 (3)	0.038 (2)	−0.002 (2)	0.0084 (18)	−0.0040 (19)
C4	0.069 (2)	0.053 (2)	0.043 (2)	0.0079 (19)	−0.0014 (18)	−0.0059 (16)
C5	0.0503 (17)	0.0480 (18)	0.0314 (17)	−0.0069 (17)	−0.0030 (15)	0.0015 (14)
C6	0.058 (2)	0.0463 (17)	0.0313 (18)	0.0036 (18)	−0.0044 (17)	−0.0003 (14)
C7	0.062 (2)	0.0528 (19)	0.0412 (19)	0.0022 (19)	−0.0040 (17)	0.0019 (14)
C8	0.057 (2)	0.0530 (19)	0.0342 (18)	0.0043 (19)	−0.0057 (16)	0.0033 (14)
C9	0.081 (3)	0.055 (2)	0.040 (2)	−0.004 (2)	−0.008 (2)	−0.0054 (16)
C10	0.083 (3)	0.0517 (19)	0.042 (2)	−0.005 (2)	−0.002 (2)	−0.0010 (16)
C11	0.053 (2)	0.058 (2)	0.0368 (19)	0.0042 (18)	0.0012 (16)	0.0037 (15)
C12	0.079 (3)	0.056 (2)	0.038 (2)	0.000 (2)	−0.0064 (19)	−0.0045 (17)
C13	0.074 (2)	0.0494 (19)	0.047 (2)	−0.0031 (19)	−0.0084 (19)	−0.0005 (18)
C14	0.092 (3)	0.070 (3)	0.050 (3)	0.006 (3)	0.015 (2)	0.0141 (19)
O3	0.119 (3)	0.0526 (14)	0.070 (2)	0.0047 (18)	−0.037 (2)	−0.0100 (13)

Geometric parameters (Å, °)

N1—C3	1.335 (5)	C7—C8	1.461 (5)
N1—C2	1.345 (5)	C7—H7A	0.9300
N2—C6	1.341 (4)	C8—C9	1.389 (5)
N2—N3	1.392 (4)	C8—C13	1.401 (5)
N2—H2B	0.8600	C9—C10	1.395 (5)
N3—C7	1.277 (5)	C9—H9A	0.9300
O1—C6	1.242 (4)	C10—C11	1.386 (5)
O2—C11	1.367 (4)	C10—H10A	0.9300
O2—C14	1.430 (5)	C11—C12	1.379 (5)
C1—C2	1.380 (5)	C12—C13	1.376 (5)
C1—C5	1.398 (5)	C12—H12A	0.9300
C1—H1A	0.9300	C13—H13A	0.9300
C2—H2A	0.9300	C14—H14A	0.9600
C3—C4	1.395 (5)	C14—H14B	0.9600
C3—H3A	0.9300	C14—H14C	0.9600
C4—C5	1.381 (5)	O3—H3WB	0.9612
C4—H4A	0.9300	O3—H3WA	0.9891
C5—C6	1.512 (5)		
C3—N1—C2	116.9 (3)	C8—C7—H7A	118.4

C6—N2—N3	120.1 (3)	C9—C8—C13	117.5 (3)
C6—N2—H2B	119.9	C9—C8—C7	123.6 (3)
N3—N2—H2B	119.9	C13—C8—C7	118.8 (3)
C7—N3—N2	114.3 (3)	C8—C9—C10	121.3 (3)
C11—O2—C14	118.9 (3)	C8—C9—H9A	119.4
C2—C1—C5	118.3 (3)	C10—C9—H9A	119.4
C2—C1—H1A	120.8	C11—C10—C9	119.8 (3)
C5—C1—H1A	120.8	C11—C10—H10A	120.1
N1—C2—C1	123.9 (3)	C9—C10—H10A	120.1
N1—C2—H2A	118.0	O2—C11—C12	115.5 (3)
C1—C2—H2A	118.0	O2—C11—C10	125.0 (3)
N1—C3—C4	123.6 (3)	C12—C11—C10	119.5 (3)
N1—C3—H3A	118.2	C13—C12—C11	120.5 (4)
C4—C3—H3A	118.2	C13—C12—H12A	119.7
C5—C4—C3	118.6 (3)	C11—C12—H12A	119.7
C5—C4—H4A	120.7	C12—C13—C8	121.3 (4)
C3—C4—H4A	120.7	C12—C13—H13A	119.3
C4—C5—C1	118.7 (3)	C8—C13—H13A	119.3
C4—C5—C6	119.0 (3)	O2—C14—H14A	109.5
C1—C5—C6	122.3 (3)	O2—C14—H14B	109.5
O1—C6—N2	124.1 (3)	H14A—C14—H14B	109.5
O1—C6—C5	120.0 (3)	O2—C14—H14C	109.5
N2—C6—C5	115.9 (3)	H14A—C14—H14C	109.5
N3—C7—C8	123.1 (3)	H14B—C14—H14C	109.5
N3—C7—H7A	118.4	H3WB—O3—H3WA	114.0
C6—N2—N3—C7	173.6 (4)	N2—N3—C7—C8	−179.1 (4)
C3—N1—C2—C1	0.6 (6)	N3—C7—C8—C9	0.2 (6)
C5—C1—C2—N1	−0.8 (6)	N3—C7—C8—C13	−179.3 (4)
C2—N1—C3—C4	0.3 (6)	C13—C8—C9—C10	−1.0 (6)
N1—C3—C4—C5	−1.0 (6)	C7—C8—C9—C10	179.5 (4)
C3—C4—C5—C1	0.8 (5)	C8—C9—C10—C11	0.6 (6)
C3—C4—C5—C6	−178.8 (4)	C14—O2—C11—C12	−173.3 (4)
C2—C1—C5—C4	0.1 (5)	C14—O2—C11—C10	6.2 (7)
C2—C1—C5—C6	179.6 (3)	C9—C10—C11—O2	−178.5 (4)
N3—N2—C6—O1	−1.1 (6)	C9—C10—C11—C12	1.0 (6)
N3—N2—C6—C5	−180.0 (3)	O2—C11—C12—C13	177.3 (4)
C4—C5—C6—O1	−37.8 (5)	C10—C11—C12—C13	−2.2 (6)
C1—C5—C6—O1	142.7 (4)	C11—C12—C13—C8	1.8 (6)
C4—C5—C6—N2	141.0 (4)	C9—C8—C13—C12	−0.2 (6)
C1—C5—C6—N2	−38.5 (5)	C7—C8—C13—C12	179.3 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···O3	0.86	2.13	2.957 (4)	162
O3—H3WB···O1^{i}	0.96	1.96	2.889 (4)	161
O3—H3WA···N1^{ii}	0.99	1.92	2.901 (4)	170

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2; (ii) −*x*+1/2, −*y*+1, *z*+1/2.

Fig. 1

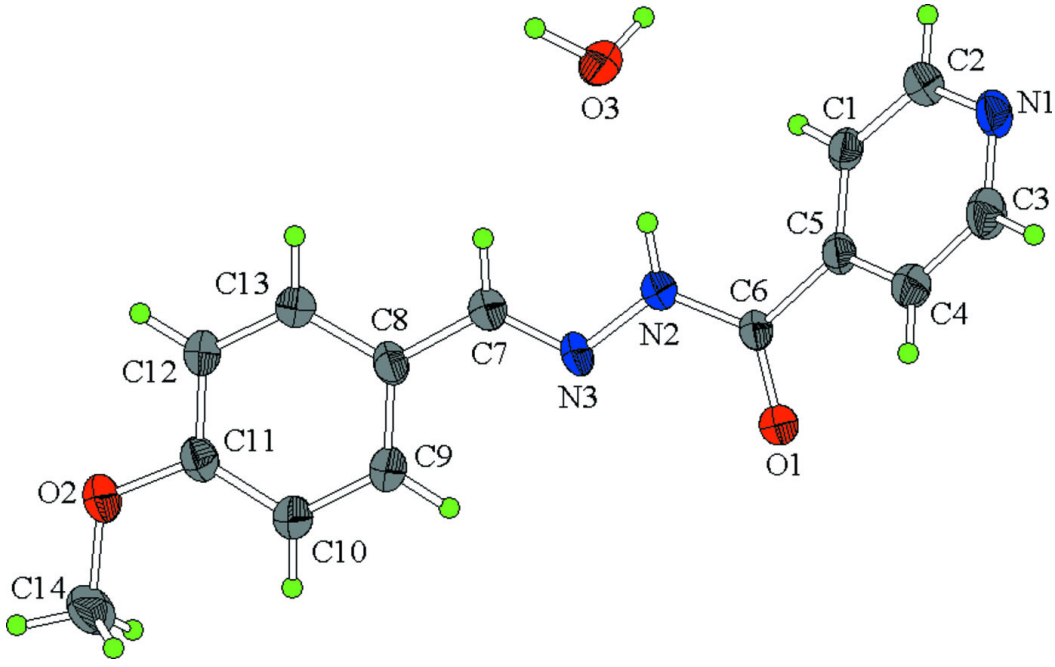

Fig. 2