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3,4-Dimethylbenzaldehyde (3-pyridylcarbonyl)hydrazone monohydrate

Ming-Yang Hao,[a] Yi-Min Feng[b] and Xi-Shi Tai[b]*

[a]Clinical College of Weifang Medical University, Weifang 261042, People's Republic of China, and [b]Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
Correspondence e-mail: taixishi@lzu.edu.cn

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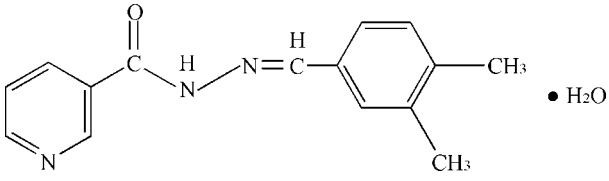
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.132; data-to-parameter ratio = 13.5.

In the crystal structure of the title compound, $C_{15}H_{15}N_3O\cdot H_2O$, O—H···O, O—H···N, N—H···O and C—H···O hydrogen bonds occur.

Related literature

For related literature, see: Tai *et al.* (2003).



Experimental

Crystal data

$C_{15}H_{15}N_3O\cdot H_2O$
M_r = 271.32
Monoclinic, $P2_1/c$
a = 13.4762 (19) Å
b = 6.5174 (14) Å
c = 16.497 (2) Å
β = 101.629 (2)°
V = 1419.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.56 × 0.53 × 0.49 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.953, T_{max} = 0.959
6293 measured reflections
2475 independent reflections
1583 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.132
S = 1.03
2475 reflections
183 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.07	2.897 (2)	162
O2—H2A···O1	0.85	2.03	2.858 (2)	163
O2—H2B···N1^{ii}	0.85	2.11	2.936 (2)	163
C1—H1···O1	0.93	2.48	2.799 (2)	100
C3—H3···O2^{i}	0.93	2.51	3.375 (3)	156
C7—H7···O2^{i}	0.93	2.49	3.297 (3)	145

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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, 2000); 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: AT2330).

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supplementary materials

Acta Cryst. (2007). E**63**, o3252 [**doi:10.1107/S1600536807029212**]

3,4-Dimethylbenzaldehyde (3-pyridylcarbonyl)hydrazone monohydrate

M.-Y. Hao, Y.-M. Feng and X.-S. Tai

Comment

As part of our ongoing studies of the coordination chemistry of aroylhydrazones ligands (Tai *et al.*, 2003), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

In the molecule of (I), both C7—N3 [1.269 (3) Å] and C6—O1 [1.216 (2) Å] are close to double-bond separations, indicating that the Lewis structure shown in the scheme is only an approximation to the electron distribution in the molecule. Otherwise, the geometrical parameters for (I) are normal. The dihedral angle between the pyridine and benzene ring mean planes is 10.69 (10)°, indicating that the molecule is non-planar, which perhaps correlates with the intramolecular O—H···O hydrogen bond (Table 1). An intermolecular N—H···O link also occurs.

Experimental

10 mmol of 3,4-Dimethylbenzaldehyde was added to a solution of nicotinic acid hydrazine (10 mmol) in 10 ml of ethanol. The mixture was continuously stirred for 3 h at refluxing temperature, evaporating some ethanol, then, upon cooling, the solid product was collected by filtration and dried *in vacuo* (yield 68%). Clear blocks of (I) were obtained by evaporation from a methanol solution after five days.

Refinement

The H atoms were placed geometrically (C—H = 0.93 − 0.96 Å, O—H = 0.85 Å, N—H = 0.86 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(C_{methyl} or O_{water}).

Figures

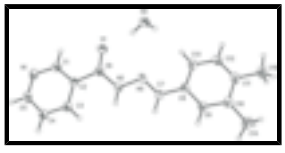

Fig. 1. The molecular structure of (I), showing 30% displacement ellipsoids

3,4-Dimethylbenzaldehyde (3-pyridylcarbonyl)hydrazone monohydrate

Crystal data

$C_{15}H_{15}N_3O·H_2O$	F_{000} = 576
M_r = 271.32	D_x = 1.270 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2 y b c	Cell parameters from 1686 reflections

a = 13.4762 (19) Å	θ = 2.7–26.9º
b = 6.5174 (14) Å	μ = 0.09 mm^{-1}
c = 16.497 (2) Å	T = 298 K
β = 101.629 (2)º	Block, colourless
V = 1419.2 (4) Å^3	0.56 × 0.53 × 0.49 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2475 independent reflections
Radiation source: fine-focus sealed tube	1583 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.5º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −15→15
T_{min} = 0.953, T_{max} = 0.959	k = −7→7
6293 measured reflections	l = −13→19

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-atom parameters constrained
$wR(F^2)$ = 0.132	$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.2072P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2475 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
183 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *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	0.32848 (12)	0.7209 (2)	−0.01437 (8)	0.0570 (6)
N1	0.40161 (16)	1.0572 (3)	−0.20590 (10)	0.0613 (7)
N2	0.30314 (13)	1.0062 (3)	0.05661 (9)	0.0478 (6)
N3	0.26026 (13)	0.8926 (3)	0.11142 (10)	0.0472 (6)
C1	0.37042 (16)	0.9576 (3)	−0.14497 (12)	0.0508 (8)
C2	0.37106 (14)	1.0398 (3)	−0.06760 (11)	0.0374 (6)
C3	0.40561 (17)	1.2374 (3)	−0.05342 (12)	0.0499 (8)
C4	0.44022 (17)	1.3419 (4)	−0.11451 (13)	0.0565 (8)
C5	0.43719 (17)	1.2463 (4)	−0.18867 (13)	0.0571 (9)
C6	0.33334 (15)	0.9064 (3)	−0.00641 (11)	0.0411 (7)
C7	0.23315 (15)	0.9945 (3)	0.16883 (12)	0.0468 (7)
C8	0.18329 (15)	0.8997 (3)	0.22987 (11)	0.0424 (7)
C9	0.17182 (15)	1.0139 (3)	0.29861 (12)	0.0467 (7)
C10	0.12531 (15)	0.9365 (4)	0.35975 (12)	0.0469 (8)
C11	0.08658 (15)	0.7372 (4)	0.35100 (12)	0.0477 (8)
C12	0.09697 (16)	0.6241 (4)	0.28210 (12)	0.0505 (8)
C13	0.14457 (16)	0.7015 (3)	0.22211 (12)	0.0491 (8)
C14	0.11591 (19)	1.0666 (4)	0.43276 (14)	0.0676 (10)
C15	0.03416 (19)	0.6467 (4)	0.41503 (14)	0.0705 (10)
O2	0.32861 (13)	0.4266 (2)	0.11411 (8)	0.0642 (6)
H1	0.34670	0.82420	−0.15520	0.0610*
H2	0.31050	1.13680	0.06230	0.0570*
H3	0.40560	1.30010	−0.00280	0.0600*
H4	0.46520	1.47480	−0.10560	0.0680*
H5	0.46140	1.31750	−0.22950	0.0690*
H7	0.24560	1.13490	0.17200	0.0560*
H9	0.19640	1.14760	0.30370	0.0560*
H12	0.07100	0.49160	0.27610	0.0610*
H13	0.15070	0.62150	0.17670	0.0590*
H14A	0.15010	1.19480	0.42990	0.1010*
H14B	0.14600	0.99650	0.48290	0.1010*
H14C	0.04560	1.09170	0.43230	0.1010*
H15A	0.01530	0.50740	0.40050	0.1060*
H15B	−0.02540	0.72530	0.41740	0.1060*
H15C	0.07920	0.64950	0.46810	0.1060*
H2A	0.33440	0.53020	0.08410	0.0770*
H2B	0.35320	0.45640	0.16430	0.0770*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0900 (12)	0.0355 (10)	0.0492 (9)	−0.0060 (8)	0.0226 (8)	−0.0005 (7)
N1	0.0875 (14)	0.0591 (14)	0.0439 (10)	−0.0084 (11)	0.0289 (10)	−0.0033 (10)
N2	0.0710 (12)	0.0351 (11)	0.0436 (9)	−0.0046 (9)	0.0266 (9)	0.0025 (8)

N3	0.0601 (11)	0.0424 (11)	0.0436 (10)	−0.0037 (9)	0.0215 (8)	0.0064 (8)
C1	0.0706 (15)	0.0408 (14)	0.0438 (12)	−0.0054 (11)	0.0179 (11)	−0.0053 (10)
C2	0.0438 (11)	0.0353 (12)	0.0338 (10)	−0.0004 (9)	0.0092 (8)	0.0001 (9)
C3	0.0635 (14)	0.0491 (15)	0.0400 (11)	−0.0101 (11)	0.0171 (10)	−0.0062 (10)
C4	0.0690 (15)	0.0501 (15)	0.0541 (13)	−0.0172 (12)	0.0212 (11)	−0.0038 (11)
C5	0.0675 (15)	0.0621 (18)	0.0476 (13)	−0.0086 (13)	0.0254 (11)	0.0073 (12)
C6	0.0480 (12)	0.0398 (14)	0.0358 (11)	−0.0027 (10)	0.0090 (9)	0.0014 (9)
C7	0.0574 (13)	0.0394 (13)	0.0472 (12)	−0.0044 (11)	0.0189 (10)	0.0020 (10)
C8	0.0445 (12)	0.0422 (14)	0.0429 (11)	−0.0020 (10)	0.0145 (9)	0.0031 (9)
C9	0.0497 (13)	0.0406 (13)	0.0525 (12)	−0.0036 (10)	0.0168 (10)	−0.0026 (10)
C10	0.0455 (12)	0.0555 (15)	0.0413 (12)	0.0025 (11)	0.0126 (9)	−0.0002 (10)
C11	0.0442 (12)	0.0571 (16)	0.0441 (12)	0.0003 (11)	0.0143 (9)	0.0074 (11)
C12	0.0510 (13)	0.0455 (14)	0.0566 (14)	−0.0101 (11)	0.0150 (11)	0.0029 (11)
C13	0.0551 (13)	0.0490 (15)	0.0463 (12)	−0.0040 (11)	0.0175 (10)	−0.0030 (10)
C14	0.0730 (16)	0.0771 (19)	0.0589 (14)	−0.0036 (14)	0.0279 (12)	−0.0122 (13)
C15	0.0730 (17)	0.082 (2)	0.0650 (15)	−0.0105 (14)	0.0343 (13)	0.0083 (14)
O2	0.1121 (13)	0.0394 (10)	0.0449 (8)	−0.0104 (9)	0.0250 (9)	0.0012 (7)

Geometric parameters (Å, °)

O1—C6	1.216 (2)	C10—C14	1.499 (3)
O2—H2A	0.8500	C11—C12	1.385 (3)
O2—H2B	0.8500	C11—C15	1.505 (3)
N1—C1	1.334 (3)	C12—C13	1.379 (3)
N1—C5	1.332 (3)	C1—H1	0.9300
N2—N3	1.382 (2)	C3—H3	0.9300
N2—C6	1.357 (3)	C4—H4	0.9300
N3—C7	1.269 (3)	C5—H5	0.9300
N2—H2	0.8600	C7—H7	0.9300
C1—C2	1.383 (3)	C9—H9	0.9300
C2—C3	1.373 (3)	C12—H12	0.9300
C2—C6	1.497 (3)	C13—H13	0.9300
C3—C4	1.374 (3)	C14—H14B	0.9600
C4—C5	1.366 (3)	C14—H14C	0.9600
C7—C8	1.456 (3)	C14—H14A	0.9600
C8—C9	1.391 (3)	C15—H15C	0.9600
C8—C13	1.389 (3)	C15—H15A	0.9600
C9—C10	1.386 (3)	C15—H15B	0.9600
C10—C11	1.396 (4)		
O1···O2	2.858 (2)	C14···H15B	2.9100
O1···N3	2.678 (2)	C15···H14B	2.8400
O1···C3^{i}	3.420 (3)	C15···H14C	2.9200
O1···C14^{ii}	3.387 (3)	H1···O1	2.4800
O1···C10^{ii}	3.251 (3)	H1···C13^{ii}	3.0500
O2···N3	3.171 (2)	H1···C8^{ii}	2.9800
O2···C7^{i}	3.297 (3)	H2···O2^{v}	2.0700
O2···N2^{i}	2.897 (2)	H2···C3	2.5900

O2···C3^{i}	3.375 (3)	H2···H7	2.1600
O2···O1	2.858 (2)	H2···H3	2.1200
O2···N1^{iii}	2.936 (2)	H2A···N3	2.6400
O1···H2A	2.0300	H2A···O1	2.0300
O1···H1	2.4800	H2A···C4^{iv}	3.0900
O1···H14B^{ii}	2.8300	H2A···C6	2.8700
O2···H3^{i}	2.5100	H2A···H3^{i}	2.4000
O2···H7^{i}	2.4900	H2B···H7^{i}	2.5700
O2···H4^{iv}	2.8800	H2B···C5^{iii}	2.7900
O2···H2^{i}	2.0700	H2B···N1^{iii}	2.1100
N1···O2^{ii}	2.936 (2)	H3···N2	2.6600
N2···O2^{v}	2.897 (2)	H3···O2^{v}	2.5100
N3···O1	2.678 (2)	H3···H2A^{v}	2.4000
N3···O2	3.171 (2)	H3···H2	2.1200
N1···H5^{vi}	2.7900	H4···O2^{iv}	2.8800
N1···H2B^{ii}	2.1100	H5···N1^{vii}	2.7900
N2···H3	2.6600	H7···H2B^{v}	2.5700
N3···H2A	2.6400	H7···O2^{v}	2.4900
N3···H13	2.6700	H7···H2	2.1600
C1···C13^{ii}	3.529 (3)	H7···H9	2.4000
C3···O1^{v}	3.420 (3)	H9···H7	2.4000
C3···O2^{v}	3.375 (3)	H9···H14A	2.3100
C3···C6^{iv}	3.582 (3)	H12···H15A	2.3300
C6···C3^{iv}	3.582 (3)	H13···N3	2.6700
C7···O2^{v}	3.297 (3)	H14A···H9	2.3100
C10···O1^{iii}	3.251 (3)	H14B···C15	2.8400
C13···C1^{iii}	3.529 (3)	H14B···H15C	2.4300
C14···O1^{iii}	3.387 (3)	H14B···O1^{iii}	2.8300
C3···H2	2.5900	H14C···C15	2.9200
C4···H2A^{iv}	3.0900	H14C···H15B	2.5700
C5···H2B^{ii}	2.7900	H15A···H12	2.3300
C6···H2A	2.8700	H15B···C14	2.9100
C8···H1^{iii}	2.9800	H15B···H14C	2.5700
C13···H1^{iii}	3.0500	H15C···C14	2.8400
C14···H15C	2.8400	H15C···H14B	2.4300
H2A—O2—H2B	109.00	C2—C1—H1	118.00
C1—N1—C5	116.36 (18)	N1—C1—H1	118.00
N3—N2—C6	118.30 (18)	C2—C3—H3	120.00
N2—N3—C7	115.41 (18)	C4—C3—H3	120.00
N3—N2—H2	121.00	C3—C4—H4	121.00
C6—N2—H2	121.00	C5—C4—H4	121.00
N1—C1—C2	124.26 (19)	C4—C5—H5	118.00
C3—C2—C6	125.41 (17)	N1—C5—H5	118.00

C1—C2—C3	117.35 (18)	N3—C7—H7	119.00
C1—C2—C6	117.24 (17)	C8—C7—H7	119.00
C2—C3—C4	119.58 (19)	C10—C9—H9	119.00
C3—C4—C5	118.5 (2)	C8—C9—H9	119.00
N1—C5—C4	123.9 (2)	C11—C12—H12	119.00
N2—C6—C2	115.69 (17)	C13—C12—H12	119.00
O1—C6—N2	122.78 (18)	C12—C13—H13	120.00
O1—C6—C2	121.52 (17)	C8—C13—H13	120.00
N3—C7—C8	122.44 (18)	C10—C14—H14A	109.00
C7—C8—C9	118.65 (18)	C10—C14—H14B	109.00
C7—C8—C13	123.22 (17)	H14A—C14—H14B	109.00
C9—C8—C13	118.12 (18)	H14A—C14—H14C	110.00
C8—C9—C10	122.66 (19)	C10—C14—H14C	109.00
C11—C10—C14	121.25 (19)	H14B—C14—H14C	109.00
C9—C10—C11	118.55 (19)	C11—C15—H15B	110.00
C9—C10—C14	120.2 (2)	C11—C15—H15C	109.00
C10—C11—C12	118.8 (2)	C11—C15—H15A	109.00
C10—C11—C15	120.71 (19)	H15A—C15—H15C	109.00
C12—C11—C15	120.5 (2)	H15B—C15—H15C	109.00
C11—C12—C13	122.2 (2)	H15A—C15—H15B	109.00
C8—C13—C12	119.63 (19)		
C5—N1—C1—C2	1.4 (3)	N3—C7—C8—C9	169.1 (2)
C1—N1—C5—C4	−2.0 (4)	N3—C7—C8—C13	−12.3 (3)
C6—N2—N3—C7	−179.72 (18)	C7—C8—C9—C10	179.93 (19)
N3—N2—C6—O1	−3.4 (3)	C13—C8—C9—C10	1.2 (3)
N3—N2—C6—C2	175.06 (16)	C7—C8—C13—C12	−179.0 (2)
N2—N3—C7—C8	177.64 (17)	C9—C8—C13—C12	−0.3 (3)
N1—C1—C2—C3	0.4 (3)	C8—C9—C10—C11	−1.4 (3)
N1—C1—C2—C6	−180.0 (2)	C8—C9—C10—C14	179.5 (2)
C1—C2—C3—C4	−1.8 (3)	C9—C10—C11—C12	0.6 (3)
C6—C2—C3—C4	178.6 (2)	C9—C10—C11—C15	−179.2 (2)
C1—C2—C6—O1	19.6 (3)	C14—C10—C11—C12	179.7 (2)
C1—C2—C6—N2	−158.97 (18)	C14—C10—C11—C15	−0.1 (3)
C3—C2—C6—O1	−160.9 (2)	C10—C11—C12—C13	0.2 (3)
C3—C2—C6—N2	20.6 (3)	C15—C11—C12—C13	−180.0 (2)
C2—C3—C4—C5	1.3 (3)	C11—C12—C13—C8	−0.4 (3)
C3—C4—C5—N1	0.7 (4)		

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) *x*, −*y*+3/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*+1/2; (iv) −*x*+1, −*y*+2, −*z*; (v) *x*, *y*+1, *z*; (vi) −*x*+1, *y*−1/2, −*z*−1/2; (vii) −*x*+1, *y*+1/2, −*z*−1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{v}	0.86	2.07	2.897 (2)	162
O2—H2A···O1	0.85	2.03	2.858 (2)	163
O2—H2B···N1^{iii}	0.85	2.11	2.936 (2)	163
C1—H1···O1	0.93	2.48	2.799 (2)	100
C3—H3···O2^{v}	0.93	2.51	3.375 (3)	156

C7—H7···O2^{v}	0.93	2.49	3.297 (3)	145

Symmetry codes: (v) *x*, *y*+1, *z*; (iii) *x*, −*y*+3/2, *z*+1/2.

Fig. 1