Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Nitrobenzaldehyde benzoylhydrazone monohydrate

Huan-Mei Guo

Department of Chemistry, Weifang College, Weifang 261061, People's Republic of China
Correspondence e-mail: huanmeiguo@163.com

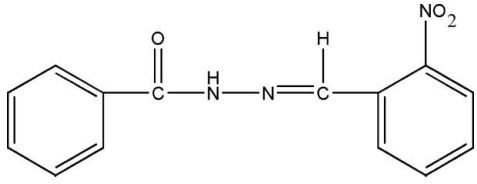
Received 19 April 2007; accepted 24 April 2007

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.056; wR factor = 0.153; data-to-parameter ratio = 13.4.

The title compound, $C_{14}H_{11}N_3O_3$, was prepared by the reaction of 2-nitrobenzophenone and benzoylhydrazine. In the crystal structure, there are some intermolecular N—H···O hydrogen-bond interactions.

Related literature

For related literature, see: Cimerman *et al.* (1997); Sutherland & Hoy (1968); Tucker *et al.* (1975).



Experimental

Crystal data

$C_{14}H_{11}N_3O_3$
M_r = 269.26
Monoclinic, $P2_1/n$
a = 6.9121 (12) Å
b = 25.854 (2) Å
c = 7.6626 (14) Å
β = 111.406 (7)°
V = 1274.9 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
0.22 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.978, T_{max} = 0.990
9812 measured reflections
2499 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.153
S = 1.01
2499 reflections
186 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.92 (2)	1.96 (2)	2.848 (2)	163.8 (19)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); 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: AT2278).

References

Bruker (1997). *SADABS*, *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cimerman, Z., Galic, N. & Bosner, B. (1997). *Anal. Chim. Acta*, **343**, 145–153.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sutherland, H. H. & Hoy, T. G. (1968). *Acta Cryst.* B**24**, 1207–1213.
Tucker, P. A., Hoekstra, A., ten Cate, J. M. & Vos, A. (1975). *Acta Cryst.* B**31**, 733–737.

  doi:10.1107/S1600536807020387

supplementary materials

Acta Cryst. (2007). E**63**, o2736 [**doi:10.1107/S1600536807020387**]

2-Nitrobenzaldehyde benzoylhydrazone monohydrate

H.-M. Guo

Comment

Schiff bases have received considerable attention in the literature. They are attractive from several points of view, such as the possibility of analytical application (Cimerman *et al.*, 1997). As part of our search for new schiff base compounds we synthesized the title compound (I), and describe its structure here.

The C13—N2 distance of 1.269 (4) Å is shorter than the one of 1.287 Å reported by Tucker *et al.* (1975). The C1—O1 distance of 1.240 (2)Å is shorter than the reported distance of 1.298Å by Sutherland & Hoy (1968). The structure of (I) is stabilized by the intermolecular N—H···O hydrogen bonds (Table 1).

Experimental

A mixture of the 2-nitrylbenzophenone (0.1 mol), and benzoyl hydrazine (0.1 mol) was stirred in refluxing ethanol (30 mL) for 5 h to afford the title compound (I) (0.085 mol, yield 85%). Single crystals of (I) suitable for X-ray measurements were obtained by slow evaporation from ethanol at room temperature.

Refinement

The H atom of the NH group were found from a difference Fourier map and refined freely. The other H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

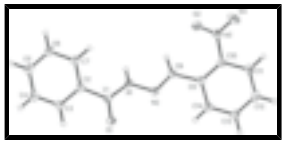


Fig. 1. The structure of the title compound (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

2-Nitrobenzaldehyde benzoylhydrazone monohydrate

Crystal data

$C_{14}H_{11}N_3O_3$	$Z = 4$
$M_r = 269.26$	$F_{000} = 560$
Monoclinic, $P2_1/n$	$D_x = 1.403$ Mg m^{-3}
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 6.9121$ (12) Å	$\theta = 3.0$–26.0º
$b = 25.854$ (2) Å	$\mu = 0.10$ mm^{-1}

c = 7.6626 (14) Å	T = 113 (2) K
β = 111.406 (7)º	Block, colourless
V = 1274.9 (3) Å^3	0.22 × 0.14 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2499 independent reflections
Radiation source: fine-focus sealed tube	1894 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.055
T = 113(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 3.0º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	h = −8→8
T_{min} = 0.978, T_{max} = 0.990	k = −31→30
9812 measured reflections	l = −9→8

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0874P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.056	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.153	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
2499 reflections	Extinction correction: none
186 parameters	
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 esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.40173 (19)	0.30647 (5)	0.67476 (18)	0.0299 (4)
O2	0.0596 (3)	0.06885 (6)	0.4061 (2)	0.0443 (4)
O3	0.2749 (3)	0.00827 (6)	0.5525 (2)	0.0509 (5)
N1	0.1914 (2)	0.24328 (6)	0.4954 (2)	0.0250 (4)
N2	0.2363 (2)	0.21033 (6)	0.6474 (2)	0.0250 (4)
N3	0.1778 (3)	0.04839 (7)	0.5520 (3)	0.0341 (5)
C1	0.2827 (3)	0.29049 (8)	0.5203 (2)	0.0233 (4)
C2	0.2344 (3)	0.32216 (7)	0.3462 (3)	0.0240 (5)
C3	0.2281 (3)	0.37602 (8)	0.3620 (3)	0.0275 (5)
H3	0.2490	0.3914	0.4802	0.033*
C4	0.1913 (3)	0.40687 (8)	0.2055 (3)	0.0325 (5)
H4	0.1845	0.4434	0.2158	0.039*
C5	0.1644 (3)	0.38448 (9)	0.0339 (3)	0.0352 (5)
H5	0.1400	0.4058	−0.0729	0.042*
C6	0.1727 (3)	0.33112 (9)	0.0163 (3)	0.0333 (5)
H6	0.1546	0.3161	−0.1017	0.040*
C7	0.2077 (3)	0.29990 (8)	0.1728 (3)	0.0269 (5)
H7	0.2135	0.2634	0.1617	0.032*
C8	0.1707 (3)	0.16388 (8)	0.6035 (3)	0.0252 (5)
H8	0.1034	0.1541	0.4760	0.030*
C9	0.2021 (3)	0.12596 (7)	0.7554 (3)	0.0235 (5)
C10	0.2048 (3)	0.07208 (8)	0.7335 (3)	0.0270 (5)
C11	0.2366 (3)	0.03809 (8)	0.8819 (3)	0.0337 (5)
H11	0.2405	0.0019	0.8629	0.040*
C12	0.2625 (3)	0.05726 (9)	1.0573 (3)	0.0381 (6)
H12	0.2814	0.0343	1.1590	0.046*
C13	0.2607 (3)	0.11027 (8)	1.0839 (3)	0.0316 (5)
H13	0.2782	0.1236	1.2043	0.038*
C14	0.2337 (3)	0.14395 (8)	0.9368 (3)	0.0257 (5)
H14	0.2367	0.1802	0.9589	0.031*
H1	0.090 (3)	0.2336 (8)	0.385 (3)	0.042 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0265 (7)	0.0331 (8)	0.0229 (8)	−0.0030 (6)	0.0004 (6)	−0.0015 (6)
O2	0.0554 (10)	0.0454 (10)	0.0318 (9)	−0.0092 (8)	0.0156 (8)	−0.0059 (7)
O3	0.0606 (11)	0.0348 (10)	0.0656 (12)	0.0028 (8)	0.0329 (10)	−0.0128 (8)
N1	0.0195 (8)	0.0317 (10)	0.0181 (9)	−0.0038 (7)	0.0002 (7)	0.0029 (7)
N2	0.0177 (7)	0.0308 (10)	0.0231 (9)	0.0001 (6)	0.0036 (6)	0.0035 (7)
N3	0.0361 (10)	0.0327 (11)	0.0392 (12)	−0.0095 (8)	0.0206 (9)	−0.0067 (8)
C1	0.0158 (8)	0.0305 (11)	0.0214 (10)	0.0022 (7)	0.0043 (7)	0.0009 (8)
C2	0.0131 (8)	0.0331 (11)	0.0234 (11)	0.0001 (7)	0.0037 (7)	0.0030 (8)
C3	0.0181 (9)	0.0313 (12)	0.0312 (11)	0.0013 (8)	0.0067 (8)	0.0016 (9)

C4	0.0201 (9)	0.0346 (12)	0.0386 (13)	0.0018 (8)	0.0058 (9)	0.0088 (9)
C5	0.0223 (10)	0.0454 (14)	0.0332 (12)	−0.0032 (9)	0.0046 (9)	0.0140 (10)
C6	0.0225 (9)	0.0499 (14)	0.0246 (11)	−0.0055 (9)	0.0051 (8)	0.0021 (10)
C7	0.0176 (9)	0.0351 (12)	0.0261 (11)	−0.0021 (8)	0.0057 (8)	−0.0016 (9)
C8	0.0180 (9)	0.0333 (12)	0.0220 (10)	−0.0008 (8)	0.0047 (8)	−0.0002 (9)
C9	0.0136 (8)	0.0290 (11)	0.0262 (11)	−0.0003 (7)	0.0052 (7)	0.0012 (8)
C10	0.0223 (9)	0.0315 (11)	0.0292 (11)	−0.0021 (8)	0.0116 (8)	−0.0026 (9)
C11	0.0333 (11)	0.0300 (12)	0.0393 (13)	0.0040 (9)	0.0151 (10)	0.0061 (10)
C12	0.0359 (12)	0.0434 (14)	0.0345 (13)	0.0040 (10)	0.0123 (10)	0.0119 (10)
C13	0.0262 (10)	0.0412 (13)	0.0251 (11)	0.0005 (9)	0.0066 (9)	0.0024 (9)
C14	0.0164 (8)	0.0312 (11)	0.0265 (11)	0.0002 (7)	0.0043 (8)	0.0012 (8)

Geometric parameters (Å, °)

O1—C1	1.240 (2)	C5—H5	0.9500
O2—N3	1.237 (2)	C6—C7	1.391 (3)
O3—N3	1.235 (2)	C6—H6	0.9500
N1—C1	1.355 (2)	C7—H7	0.9500
N1—N2	1.384 (2)	C8—C9	1.476 (3)
N1—H1	0.92 (2)	C8—H8	0.9500
N2—C8	1.284 (2)	C9—C10	1.404 (3)
N3—C10	1.468 (3)	C9—C14	1.405 (3)
C1—C2	1.496 (3)	C10—C11	1.390 (3)
C2—C7	1.397 (3)	C11—C12	1.381 (3)
C2—C3	1.400 (3)	C11—H11	0.9500
C3—C4	1.384 (3)	C12—C13	1.386 (3)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.386 (3)	C13—C14	1.382 (3)
C4—H4	0.9500	C13—H13	0.9500
C5—C6	1.389 (3)	C14—H14	0.9500
C1—N1—N2	119.09 (15)	C6—C7—C2	120.10 (19)
C1—N1—H1	122.3 (14)	C6—C7—H7	120.0
N2—N1—H1	118.3 (14)	C2—C7—H7	120.0
C8—N2—N1	113.94 (16)	N2—C8—C9	118.56 (17)
O3—N3—O2	122.77 (18)	N2—C8—H8	120.7
O3—N3—C10	117.79 (18)	C9—C8—H8	120.7
O2—N3—C10	119.44 (17)	C10—C9—C14	116.27 (17)
O1—C1—N1	123.17 (17)	C10—C9—C8	124.70 (18)
O1—C1—C2	121.55 (17)	C14—C9—C8	119.02 (18)
N1—C1—C2	115.26 (15)	C11—C10—C9	122.33 (19)
C7—C2—C3	119.67 (18)	C11—C10—N3	116.04 (18)
C7—C2—C1	122.13 (17)	C9—C10—N3	121.62 (17)
C3—C2—C1	118.10 (17)	C12—C11—C10	119.7 (2)
C4—C3—C2	120.0 (2)	C12—C11—H11	120.2
C4—C3—H3	120.0	C10—C11—H11	120.2
C2—C3—H3	120.0	C11—C12—C13	119.5 (2)
C3—C4—C5	120.0 (2)	C11—C12—H12	120.2
C3—C4—H4	120.0	C13—C12—H12	120.2
C5—C4—H4	120.0	C14—C13—C12	120.6 (2)

C4—C5—C6	120.70 (19)	C14—C13—H13	119.7
C4—C5—H5	119.6	C12—C13—H13	119.7
C6—C5—H5	119.6	C13—C14—C9	121.61 (19)
C5—C6—C7	119.5 (2)	C13—C14—H14	119.2
C5—C6—H6	120.2	C9—C14—H14	119.2
C7—C6—H6	120.2		
C1—N1—N2—C8	−168.80 (16)	N2—C8—C9—C14	21.7 (2)
N2—N1—C1—O1	−1.9 (3)	C14—C9—C10—C11	0.1 (3)
N2—N1—C1—C2	176.60 (15)	C8—C9—C10—C11	179.52 (17)
O1—C1—C2—C7	143.21 (18)	C14—C9—C10—N3	−178.78 (16)
N1—C1—C2—C7	−35.3 (2)	C8—C9—C10—N3	0.7 (3)
O1—C1—C2—C3	−33.1 (2)	O3—N3—C10—C11	−33.6 (2)
N1—C1—C2—C3	148.40 (17)	O2—N3—C10—C11	146.05 (17)
C7—C2—C3—C4	1.3 (3)	O3—N3—C10—C9	145.36 (18)
C1—C2—C3—C4	177.71 (15)	O2—N3—C10—C9	−35.0 (3)
C2—C3—C4—C5	−1.1 (3)	C9—C10—C11—C12	1.4 (3)
C3—C4—C5—C6	0.4 (3)	N3—C10—C11—C12	−179.72 (17)
C4—C5—C6—C7	0.2 (3)	C10—C11—C12—C13	−1.3 (3)
C5—C6—C7—C2	0.0 (3)	C11—C12—C13—C14	−0.1 (3)
C3—C2—C7—C6	−0.7 (3)	C12—C13—C14—C9	1.6 (3)
C1—C2—C7—C6	−176.98 (16)	C10—C9—C14—C13	−1.6 (3)
N1—N2—C8—C9	−177.42 (14)	C8—C9—C14—C13	178.96 (16)
N2—C8—C9—C10	−157.67 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.92 (2)	1.96 (2)	2.848 (2)	163.8 (19)

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

Fig. 1