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2-Hydroxy-5-nitrobenzamide

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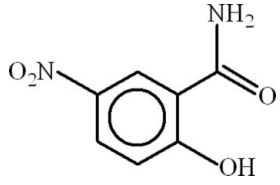
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.112; data-to-parameter ratio = 14.4.

In the title compound, $C_7H_6N_2O_4$, an intramolecular O—H···O hydrogen bond generates an $S(6)$ ring. In the crystal, inversion dimers linked by pairs of N—H···O hydrogen bonds occur. Weak C—H···O links consolidate the packing, leading to $R_2^1(7)$ and $R_2^2(10)$ loops within (100) polymeric sheets.

Related literature

For related structures, see: Pertlik (1990); Raza *et al.* (2009).



Experimental

Crystal data

$C_7H_6N_2O_4$
M_r = 182.14
Monoclinic, $P2_1/n$
a = 5.1803 (3) Å
b = 11.1037 (8) Å
c = 13.7214 (10) Å
β = 100.642 (4)°
V = 775.69 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 K
0.28 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.970, T_{max} = 0.976
4581 measured reflections
1799 independent reflections
1434 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.112
S = 1.05
1799 reflections
125 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	1.79	2.5196 (16)	148
N1—H1A···O2^{i}	0.914 (19)	1.969 (19)	2.8807 (17)	174.9 (18)
N1—H1B···O3^{ii}	0.88 (2)	2.167 (19)	3.0193 (17)	164.6 (15)
C4—H4···O1^{iii}	0.93	2.49	3.3915 (18)	164
C6—H6···O3^{ii}	0.93	2.47	3.3826 (16)	169

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5244).

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supplementary materials

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Comment

The title compound (I, Fig. 1) is an intermediate for various derivatives. We have reported the preparation and crystal structure of (II) 2-hydroxy-3-nitrobenzamide (Raza *et al.*, 2009) which is isomer of (I). The crystal structures of (III) 2-Hydroxybenzamide (Pertlik, 1990) has been published also.

In the asymmetric unit of (I), the benzene ring A (C1—C6) is of course planar. The nitro group B (N2/O3/O4) and the amide group C (C7/N1/O2) make dihedral angle of 8.49 (13)° and 8.48 (21)° respectively, with the benzene ring. The dihedral angle between B/C is 14.51 (22)°. There exist an intramolecular H-bonding of O–H···O type forming S(6) ring motif (Bernstein *et al.*, 1995). The molecules of the title compound are dimerised forming a $R_2^2(10)$ and two $R_2^1(7)$ ring motifs (Table 1, Fig. 2). The dimers are interlinked each other forming polymeric network and the dimers are surounded by six $R_5^4(16)$ ring motifs.

Experimental

A solution of 2-hydroxy-benzamide (1.37 g, 0.01 mol) in ethyl acetate (25 ml) was added as drops to a nitrating mixture of HNO_3 (3 ml, 1.89 g, 0.03 mol) and H_2SO_4 (2 ml, 1.96 g, 0.02 mol), with constant stirring, while the temperature was kept below 278 K. The reaction mixture was stirred at room temperature for 4–5 h, refluxed for 1 h, cooled, neutralized with aqueous $NaHCO_3$ (10%) and extracted with EtOAc (3 × 25 ml). The organic layer was combined, dried over anhydrous Na_2SO_4, filtered and rotary concentrated to afford light yellowish solid. The column chromatographic purification with 0, 2.5, and 5% EtOAc in petrol (0.5 l each) over a silica gel packed column (25.5 cm height) afforded the title compound.

Refinement

The coordintes of H-atoms of NH_2 group were refined. The other H-atoms were positioned geometrically (O–H = 0.82, C–H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C, N, O).

Figures

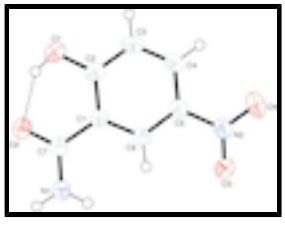

Fig. 1. View of (I) with displacement ellipsoids drawn at the 50% probability level. The dashed line represents the intramolecular H-bond.

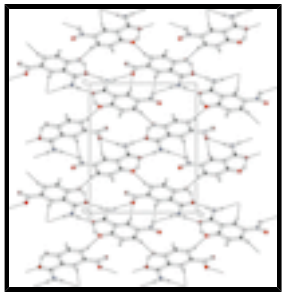

Fig. 2. The projectional view of the title compound showing that molecules are dimerized and dimers are linked in the formation of two dimensional polymeric sheets with various ring motifs.

2-Hydroxy-5-nitrobenzamide

Crystal data

$C_7H_6N_2O_4$	$F(000) = 376$
$M_r = 182.14$	$D_x = 1.560$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1799 reflections
$a = 5.1803$ (3) Å	$\theta = 2.4$–$27.8°$
$b = 11.1037$ (8) Å	$\mu = 0.13$ mm^{-1}
$c = 13.7214$ (10) Å	$T = 296$ K
$\beta = 100.642$ (4)°	Prisms, light yellow
$V = 775.69$ (9) Å^3	0.28 × 0.20 × 0.18 mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD diffractometer	1799 independent reflections
Radiation source: fine-focus sealed tube	1434 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.018$
Detector resolution: 7.50 pixels mm^{-1}	$\theta_{max} = 27.8°$, $\theta_{min} = 2.4°$
ω scans	$h = -6 \rightarrow 6$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$k = -14 \rightarrow 13$
$T_{min} = 0.970$, $T_{max} = 0.976$	$l = -17 \rightarrow 16$
4581 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.037$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.112$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.0918P]$ where $P = (F_o^2 + 2F_c^2)/3$

1799 reflections	$(\Delta/\sigma)_{max} < 0.001$
125 parameters	$\Delta\rho_{max} = 0.23$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

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 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 $F^2 > \sigma(F^2)$ 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	0.5646 (2)	0.40418 (9)	0.31899 (9)	0.0527 (4)
O2	0.2337 (2)	0.46688 (9)	0.42269 (9)	0.0538 (4)
O3	0.1375 (2)	−0.09501 (9)	0.42368 (9)	0.0604 (4)
O4	0.4447 (2)	−0.15349 (9)	0.34804 (9)	0.0599 (4)
N1	0.0539 (3)	0.33482 (11)	0.51407 (10)	0.0505 (4)
N2	0.3188 (2)	−0.07405 (10)	0.37931 (8)	0.0425 (4)
C1	0.3227 (2)	0.26057 (11)	0.39880 (9)	0.0352 (3)
C2	0.5024 (3)	0.28970 (12)	0.33611 (10)	0.0393 (4)
C3	0.6220 (3)	0.19813 (14)	0.28969 (11)	0.0460 (4)
C4	0.5642 (3)	0.07915 (13)	0.30371 (10)	0.0427 (4)
C5	0.3840 (2)	0.05179 (11)	0.36410 (9)	0.0363 (4)
C6	0.2636 (2)	0.13973 (11)	0.41112 (9)	0.0351 (3)
C7	0.1992 (3)	0.36003 (11)	0.44683 (10)	0.0390 (4)
H1	0.48184	0.44981	0.34877	0.0632*
H1A	−0.032 (4)	0.3969 (16)	0.5380 (14)	0.0606*
H1B	0.028 (3)	0.2603 (18)	0.5307 (13)	0.0606*
H3	0.74118	0.21799	0.24920	0.0552*
H4	0.64399	0.01822	0.27341	0.0513*
H6	0.14354	0.11839	0.45083	0.0421*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0632 (7)	0.0386 (5)	0.0644 (7)	−0.0088 (5)	0.0331 (5)	0.0035 (5)
O2	0.0678 (7)	0.0286 (5)	0.0737 (7)	0.0004 (4)	0.0362 (5)	0.0027 (4)
O3	0.0834 (8)	0.0346 (5)	0.0760 (8)	−0.0045 (5)	0.0481 (6)	0.0018 (5)
O4	0.0723 (7)	0.0372 (6)	0.0751 (8)	0.0129 (5)	0.0267 (6)	−0.0080 (5)
N1	0.0732 (8)	0.0274 (6)	0.0610 (8)	0.0076 (5)	0.0389 (7)	0.0020 (5)
N2	0.0535 (7)	0.0333 (6)	0.0433 (6)	0.0050 (5)	0.0156 (5)	−0.0021 (4)
C1	0.0383 (6)	0.0321 (6)	0.0378 (6)	0.0010 (5)	0.0135 (5)	0.0011 (5)

C2	0.0412 (6)	0.0381 (7)	0.0411 (7)	−0.0044 (5)	0.0143 (5)	0.0029 (5)
C3	0.0454 (7)	0.0500 (8)	0.0490 (8)	−0.0033 (6)	0.0254 (6)	−0.0015 (6)
C4	0.0435 (7)	0.0433 (7)	0.0458 (7)	0.0044 (5)	0.0199 (5)	−0.0056 (5)
C5	0.0407 (6)	0.0315 (6)	0.0389 (7)	0.0024 (5)	0.0133 (5)	−0.0015 (5)
C6	0.0390 (6)	0.0319 (6)	0.0382 (6)	0.0023 (5)	0.0170 (5)	0.0007 (5)
C7	0.0451 (7)	0.0298 (6)	0.0452 (7)	0.0008 (5)	0.0161 (5)	0.0002 (5)

Geometric parameters (Å, °)

O1—C2	1.3427 (17)	C1—C2	1.4167 (19)
O2—C7	1.2535 (16)	C1—C6	1.3935 (17)
O3—N2	1.2321 (15)	C2—C3	1.403 (2)
O4—N2	1.2210 (15)	C3—C4	1.376 (2)
O1—H1	0.8200	C4—C5	1.3914 (19)
N1—C7	1.324 (2)	C5—C6	1.3811 (17)
N2—C5	1.4615 (16)	C3—H3	0.9300
N1—H1A	0.914 (19)	C4—H4	0.9300
N1—H1B	0.88 (2)	C6—H6	0.9300
C1—C7	1.4896 (18)		
C2—O1—H1	109.00	C3—C4—C5	118.70 (13)
O3—N2—C5	117.94 (10)	N2—C5—C4	119.47 (11)
O4—N2—C5	119.21 (10)	N2—C5—C6	118.23 (10)
O3—N2—O4	122.86 (11)	C4—C5—C6	122.30 (12)
C7—N1—H1A	117.9 (12)	C1—C6—C5	119.72 (10)
C7—N1—H1B	121.1 (11)	O2—C7—C1	119.41 (13)
H1A—N1—H1B	120.8 (16)	N1—C7—C1	119.83 (11)
C6—C1—C7	122.57 (11)	O2—C7—N1	120.76 (13)
C2—C1—C6	118.51 (11)	C2—C3—H3	120.00
C2—C1—C7	118.91 (11)	C4—C3—H3	120.00
O1—C2—C3	117.79 (13)	C3—C4—H4	121.00
C1—C2—C3	120.33 (12)	C5—C4—H4	121.00
O1—C2—C1	121.89 (12)	C1—C6—H6	120.00
C2—C3—C4	120.43 (14)	C5—C6—H6	120.00
O3—N2—C5—C4	−171.90 (12)	C2—C1—C7—N1	−172.50 (13)
O3—N2—C5—C6	8.36 (17)	C6—C1—C7—O2	−170.64 (13)
O4—N2—C5—C4	8.18 (18)	C6—C1—C7—N1	9.1 (2)
O4—N2—C5—C6	−171.57 (12)	O1—C2—C3—C4	−179.42 (14)
C6—C1—C2—O1	178.56 (12)	C1—C2—C3—C4	0.7 (2)
C6—C1—C2—C3	−1.51 (19)	C2—C3—C4—C5	0.4 (2)
C7—C1—C2—O1	0.12 (19)	C3—C4—C5—N2	179.58 (12)
C7—C1—C2—C3	−179.95 (15)	C3—C4—C5—C6	−0.7 (2)
C2—C1—C6—C5	1.28 (17)	N2—C5—C6—C1	179.54 (11)
C7—C1—C6—C5	179.66 (12)	C4—C5—C6—C1	−0.20 (18)
C2—C1—C7—O2	7.73 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.79	2.5196 (16)	148

N1—H1A···O2^{i}	0.914 (19)	1.969 (19)	2.8807 (17)	174.9 (18)
N1—H1B···O3^{ii}	0.88 (2)	2.167 (19)	3.0193 (17)	164.6 (15)
C4—H4···O1^{iii}	0.93	2.49	3.3915 (18)	164
C6—H6···O3^{ii}	0.93	2.47	3.3826 (16)	169

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

Fig. 1

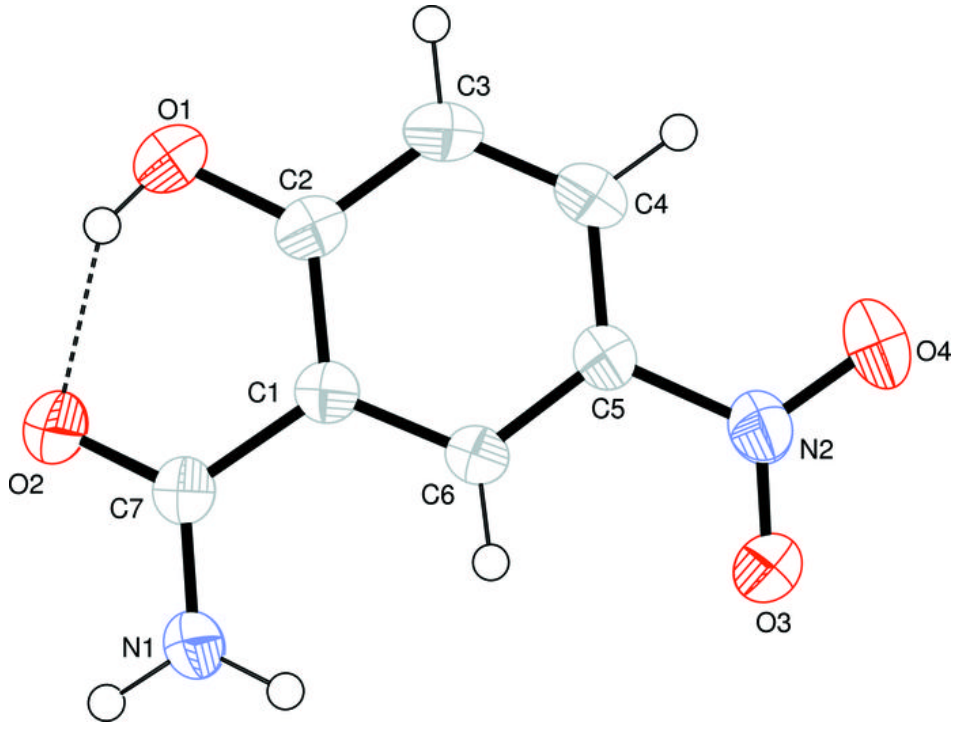

Fig. 2