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2-Hydroxy-*N*-(4-methylphenyl)-benzamideAbdul Rauf Raza,^a Bushra Nisar^a and M. Nawaz Tahir^{b*}^aDepartment of Chemistry, University of Sargodha, Sargodha, Pakistan, and^bDepartment of Physics, University of Sargodha, Sargodha, Pakistan

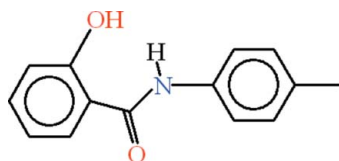
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.059; wR factor = 0.159; data-to-parameter ratio = 17.8.

In the crystal structure of the title compound, $\text{C}_{14}\text{H}_{13}\text{NO}_2$, the molecules are approximately planar, the r.m.s. deviation for all non-H atoms being 0.0435 Å; the dihedral angle between the two rings is 3.45 (12)°. The planarity is accounted for in terms of the presence of intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding, each of which completes an $S(6)$ ring motif. The molecules are stabilized in the form of supramolecular chains extending along the crystallographic c axis due to intermolecular $\text{O}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding; each type leads to an $R_2^1(6)$ ring motif.

Related literature

For related benzamide structures, see: Raza *et al.* (2010*a,b,c*).For graph-set notation, see: Bernstein *et al.* (1995).

Experimental

Crystal data

 $\text{C}_{14}\text{H}_{13}\text{NO}_2$ $M_r = 227.25$ Monoclinic, $P2_1/c$ $a = 19.4067$ (17) Å $b = 4.9122$ (5) Å $c = 12.7261$ (11) Å $\beta = 104.793$ (4)° $V = 1172.96$ (19) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 296$ K $0.34 \times 0.14 \times 0.12$ mm

Data collection

Bruker Kappa APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.979$, $T_{\max} = 0.988$

10416 measured reflections

2771 independent reflections

1243 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.060$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.059$ $wR(F^2) = 0.159$ $S = 0.96$

2771 reflections

156 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^i$	0.82	1.78	2.596 (2)	179
$\text{N1}-\text{H1A}\cdots\text{O1}$	0.86	1.92	2.647 (2)	141
$\text{C3}-\text{H3}\cdots\text{O2}^i$	0.93	2.51	3.179 (3)	129
$\text{C9}-\text{H9}\cdots\text{O2}$	0.93	2.25	2.840 (3)	121

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); 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 for Windows (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

The authors acknowledge the provision of funds for the purchase of the diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Ex-Vice Chancellor, University of Sargodha, Pakistan. ARR also acknowledges the Higher Education Commission, Government of Pakistan, for generous support of a research project (20-819).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2771).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc. Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Raza, A. R., Nisar, B. & Tahir, M. N. (2010*a*). *Acta Cryst.* **E66**, o2435.
- Raza, A. R., Nisar, B., Tahir, M. N. & Shamshad, S. (2010*b*). *Acta Cryst.* **E66**, o2922.
- Raza, A. R., Nisar, B., Tahir, M. N. & Shamshad, S. (2010*c*). *Acta Cryst.* **E66**, o3100.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2011). E67, o2253 [doi:10.1107/S1600536811030716]

2-Hydroxy-*N*-(4-methylphenyl)benzamide

A. R. Raza, B. Nisar and M. N. Tahir

Comment

We have reported the crystal structures of (II) *i.e.* 2-hydroxy-*N*-(3-nitrophenyl)benzamide (Raza *et al.*, 2010a), (III) *i.e.* *N*-(4-chlorophenyl)-2-hydroxybenzamide (Raza *et al.*, 2010b) and (IV) *i.e.* *N*-(3-chlorophenyl)-2-hydroxybenzamide (Raza *et al.*, 2010c). In this connection, the title compound (I, Fig. 1) has been prepared as a precursor for the synthesis of symmetric as well as asymmetric benzoxazepines.

In (I), the 2-hydroxyphenyl group A (C1–C6/O1) and 4-methylanilinic group B (C8–C14/N1) are planar with r.m.s. deviations of 0.0048 and 0.0086 Å, respectively. The dihedral angle between A/B is 3.45 (12)°. There is intramolecular H-bonding of the type N—H···O and C—H···O types (Table 1, Fig. 1), each of which completes a *S*(6) ring motif (Bernstein *et al.*, 1995). There is also intermolecular H-bonding of the type C—H···O and O—H···O (Table 1). These lead to the formation of two $R_2^1(6)$ ring motifs and to supramolecular chains extending along the crystallographic *c*-axis (Fig. 2).

Experimental

To a well stirred solution of 2-hydroxy benzoic acid (2.76 g, 0.02 mol, 1 equiv.) and SOCl₂ (1.74 mL, 2.84 g, 0.024 mol, 1.2 equiv.) in dry CHCl₃, 4-metylaniline (2.14 g, 0.02 mol, 1 equiv.) and Et₃N (4.16 mL, 3 g, 0.03 mol, 1.5 equiv.) were added slowly at room temperature followed by 3 h reflux. After completion of reaction, the reaction mixture was cooled to room temperature, neutralized with aqueous NaHCO₃ (10 %) and extracted with CHCl₃ (3×25 mL). The organic layers were combined, dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to afford a brown solid. The column chromatographic purification with 1%, 2% and 3% CHCl₃ in petrol (300 mL each) over a silica gel packed column (of 25.5 cm length) afforded white needles of (I) in the 96th-280th fractions (50 mL each).

Refinement

Although H atoms appeared in difference Fourier maps they were positioned geometrically with (O–H = 0.82, N–H = 0.86 and C–H = 0.93-0.96 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for hydroxy- & methyl-H atoms and $x = 1.2$ for other H atoms.

Figures

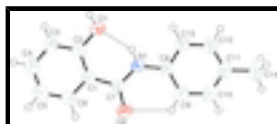


Fig. 1. View of the title compound with the atom numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii. The dotted line indicate the intramolecular H-bonding.

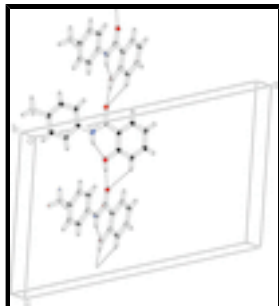


Fig. 2. The partial packing diagram which shows that molecules form supramolecular chains extending along the *c*-axis. The dotted line indicate the intramolecular H-bonding.

2-Hydroxy-*N*-(4-methylphenyl)benzamide

Crystal data

$C_{14}H_{13}NO_2$

$M_r = 227.25$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 19.4067$ (17) Å

$b = 4.9122$ (5) Å

$c = 12.7261$ (11) Å

$\beta = 104.793$ (4)°

$V = 1172.96$ (19) Å³

$Z = 4$

$F(000) = 480$

$D_x = 1.287$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1243 reflections

$\theta = 1.1$ – 27.9 °

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Needle, colorless

$0.34 \times 0.14 \times 0.12$ mm

Data collection

Bruker Kappa APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 7.6 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.979$, $T_{\max} = 0.988$

10416 measured reflections

2771 independent reflections

1243 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.060$

$\theta_{\max} = 27.9$ °, $\theta_{\min} = 1.1$ °

$h = -25 \rightarrow 25$

$k = -4 \rightarrow 6$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.059$

$wR(F^2) = 0.159$

$S = 0.96$

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

$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

2771 reflections	$(\Delta/\sigma)_{\max} < 0.001$
156 parameters	$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

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 esds 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.29367 (9)	-0.2157 (4)	0.22720 (13)	0.0599 (7)
O2	0.29429 (9)	-0.0348 (3)	-0.09273 (12)	0.0598 (7)
N1	0.24804 (9)	0.0722 (4)	0.04697 (14)	0.0438 (7)
C1	0.33925 (11)	-0.2782 (5)	0.07185 (18)	0.0407 (8)
C2	0.33876 (12)	-0.3489 (5)	0.17802 (18)	0.0438 (8)
C3	0.38382 (13)	-0.5515 (5)	0.2327 (2)	0.0516 (9)
C4	0.42941 (13)	-0.6849 (5)	0.1833 (2)	0.0596 (11)
C5	0.43143 (13)	-0.6168 (6)	0.0796 (2)	0.0615 (11)
C6	0.38654 (13)	-0.4161 (5)	0.0249 (2)	0.0539 (10)
C7	0.29213 (12)	-0.0712 (5)	0.00262 (18)	0.0416 (8)
C8	0.19724 (12)	0.2725 (5)	-0.00178 (18)	0.0428 (8)
C9	0.18909 (14)	0.3743 (5)	-0.1055 (2)	0.0564 (10)
C10	0.13704 (14)	0.5697 (6)	-0.1449 (2)	0.0605 (11)
C11	0.09251 (13)	0.6670 (5)	-0.0859 (2)	0.0544 (10)
C12	0.10233 (14)	0.5654 (5)	0.0186 (2)	0.0592 (10)
C13	0.15368 (13)	0.3727 (5)	0.06023 (19)	0.0524 (9)
C14	0.03636 (15)	0.8783 (5)	-0.1313 (2)	0.0751 (11)
H1	0.29314	-0.29364	0.28396	0.0899*
H1A	0.25091	0.03812	0.11424	0.0525*
H3	0.38315	-0.59736	0.30329	0.0619*
H4	0.45896	-0.82174	0.22036	0.0714*
H5	0.46273	-0.70494	0.04648	0.0739*
H6	0.38794	-0.37165	-0.04556	0.0647*
H9	0.21810	0.31275	-0.14848	0.0677*
H10	0.13229	0.63728	-0.21461	0.0726*
H12	0.07355	0.62883	0.06158	0.0711*
H13	0.15915	0.30919	0.13074	0.0628*
H14A	0.03008	0.89281	-0.20839	0.1127*
H14B	0.05114	1.05104	-0.09782	0.1127*
H14C	-0.00787	0.82537	-0.11655	0.1127*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0753 (12)	0.0677 (13)	0.0429 (11)	0.0164 (10)	0.0262 (9)	0.0130 (9)
O2	0.0889 (13)	0.0586 (13)	0.0371 (10)	0.0035 (10)	0.0256 (9)	0.0009 (8)
N1	0.0496 (12)	0.0504 (14)	0.0318 (10)	0.0029 (10)	0.0111 (9)	0.0039 (9)
C1	0.0437 (13)	0.0379 (15)	0.0409 (14)	-0.0064 (11)	0.0117 (11)	-0.0060 (11)
C2	0.0449 (14)	0.0444 (16)	0.0438 (14)	-0.0034 (12)	0.0147 (12)	-0.0036 (12)
C3	0.0500 (14)	0.0479 (18)	0.0534 (16)	-0.0010 (13)	0.0070 (12)	0.0053 (13)
C4	0.0506 (16)	0.0481 (19)	0.077 (2)	0.0041 (13)	0.0109 (15)	0.0031 (14)
C5	0.0525 (16)	0.061 (2)	0.076 (2)	0.0033 (15)	0.0257 (15)	-0.0090 (16)
C6	0.0557 (16)	0.0571 (19)	0.0522 (16)	-0.0014 (14)	0.0197 (13)	-0.0048 (14)
C7	0.0494 (14)	0.0400 (16)	0.0365 (14)	-0.0101 (12)	0.0132 (11)	-0.0050 (11)
C8	0.0474 (14)	0.0383 (15)	0.0400 (14)	-0.0038 (12)	0.0062 (11)	0.0006 (11)
C9	0.0608 (16)	0.0616 (19)	0.0465 (16)	-0.0014 (15)	0.0130 (13)	0.0051 (14)
C10	0.0673 (18)	0.056 (2)	0.0515 (17)	-0.0024 (15)	0.0028 (14)	0.0110 (14)
C11	0.0544 (16)	0.0360 (16)	0.0628 (19)	-0.0060 (13)	-0.0031 (14)	-0.0021 (13)
C12	0.0613 (17)	0.0524 (19)	0.0615 (18)	0.0060 (14)	0.0112 (14)	-0.0038 (14)
C13	0.0599 (16)	0.0563 (18)	0.0397 (14)	0.0043 (14)	0.0105 (12)	0.0046 (12)
C14	0.0718 (19)	0.0511 (19)	0.086 (2)	0.0023 (16)	-0.0097 (16)	0.0010 (16)

Geometric parameters (\AA , $^\circ$)

O1—C2	1.366 (3)	C10—C11	1.368 (4)
O2—C7	1.238 (3)	C11—C12	1.387 (3)
O1—H1	0.8200	C11—C14	1.509 (4)
N1—C7	1.339 (3)	C12—C13	1.379 (4)
N1—C8	1.419 (3)	C3—H3	0.9300
N1—H1A	0.8600	C4—H4	0.9300
C1—C7	1.495 (3)	C5—H5	0.9300
C1—C6	1.392 (3)	C6—H6	0.9300
C1—C2	1.397 (3)	C9—H9	0.9300
C2—C3	1.389 (3)	C10—H10	0.9300
C3—C4	1.375 (4)	C12—H12	0.9300
C4—C5	1.372 (4)	C13—H13	0.9300
C5—C6	1.381 (4)	C14—H14A	0.9600
C8—C13	1.386 (3)	C14—H14B	0.9600
C8—C9	1.382 (3)	C14—H14C	0.9600
C9—C10	1.391 (4)		
C2—O1—H1	109.00	C11—C12—C13	121.6 (2)
C7—N1—C8	128.90 (19)	C8—C13—C12	120.8 (2)
C8—N1—H1A	116.00	C2—C3—H3	120.00
C7—N1—H1A	116.00	C4—C3—H3	120.00
C2—C1—C6	117.5 (2)	C3—C4—H4	120.00
C2—C1—C7	125.8 (2)	C5—C4—H4	120.00
C6—C1—C7	116.7 (2)	C4—C5—H5	120.00
O1—C2—C3	120.6 (2)	C6—C5—H5	120.00

O1—C2—C1	119.2 (2)	C1—C6—H6	119.00
C1—C2—C3	120.3 (2)	C5—C6—H6	119.00
C2—C3—C4	120.6 (2)	C8—C9—H9	120.00
C3—C4—C5	120.2 (2)	C10—C9—H9	120.00
C4—C5—C6	119.3 (2)	C9—C10—H10	118.00
C1—C6—C5	122.1 (2)	C11—C10—H10	118.00
N1—C7—C1	118.03 (19)	C11—C12—H12	119.00
O2—C7—C1	120.5 (2)	C13—C12—H12	119.00
O2—C7—N1	121.5 (2)	C8—C13—H13	120.00
N1—C8—C9	124.5 (2)	C12—C13—H13	120.00
N1—C8—C13	117.0 (2)	C11—C14—H14A	109.00
C9—C8—C13	118.5 (2)	C11—C14—H14B	109.00
C8—C9—C10	119.3 (2)	C11—C14—H14C	109.00
C9—C10—C11	123.1 (2)	H14A—C14—H14B	109.00
C10—C11—C14	121.7 (2)	H14A—C14—H14C	109.00
C10—C11—C12	116.7 (2)	H14B—C14—H14C	109.00
C12—C11—C14	121.6 (2)		
C8—N1—C7—O2	2.0 (4)	C1—C2—C3—C4	0.0 (4)
C8—N1—C7—C1	-178.1 (2)	C2—C3—C4—C5	0.8 (4)
C7—N1—C8—C9	-6.3 (4)	C3—C4—C5—C6	-1.0 (4)
C7—N1—C8—C13	174.3 (2)	C4—C5—C6—C1	0.4 (4)
C6—C1—C2—O1	179.2 (2)	N1—C8—C9—C10	179.6 (2)
C6—C1—C2—C3	-0.5 (4)	C13—C8—C9—C10	-1.0 (4)
C7—C1—C2—O1	-2.3 (4)	N1—C8—C13—C12	-179.2 (2)
C7—C1—C2—C3	177.9 (2)	C9—C8—C13—C12	1.4 (4)
C2—C1—C6—C5	0.3 (4)	C8—C9—C10—C11	-0.4 (4)
C7—C1—C6—C5	-178.3 (2)	C9—C10—C11—C12	1.4 (4)
C2—C1—C7—O2	-176.0 (2)	C9—C10—C11—C14	-179.7 (2)
C2—C1—C7—N1	4.1 (4)	C10—C11—C12—C13	-1.0 (4)
C6—C1—C7—O2	2.5 (3)	C14—C11—C12—C13	-179.9 (2)
C6—C1—C7—N1	-177.5 (2)	C11—C12—C13—C8	-0.4 (4)
O1—C2—C3—C4	-179.8 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots O2 ⁱ	0.82	1.78	2.596 (2)	179
N1—H1A \cdots O1	0.86	1.92	2.647 (2)	141
C3—H3 \cdots O2 ⁱ	0.93	2.51	3.179 (3)	129
C9—H9 \cdots O2	0.93	2.25	2.840 (3)	121

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

Fig. 1

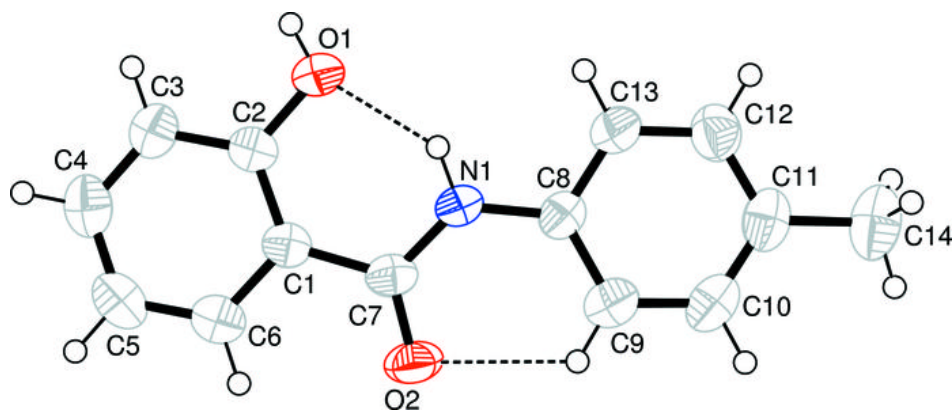


Fig. 2

