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Crystal structure of 4-benzamido-2-hydroxybenzoic acid

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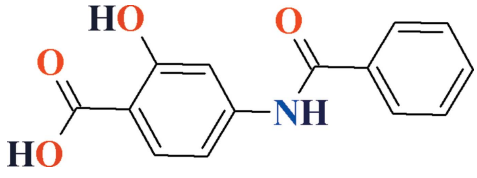
In the title compound, $C_{14}H_{11}NO_4$, the dihedral angle between the mean planes of the aromatic rings is 3.96 (12)° and an intramolecular O—H···O hydrogen bond closes an $S(6)$ ring. A short intramolecular C—H···O contact is also seen. In the crystal, carboxylic acid inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(8)$ loops. Conversely, the N—H group does not form a hydrogen bond. Aromatic π–π interactions exist at a centroid–centroid distance of 3.8423 (15) Å between the benzene rings. An extremely weak C—H···π interaction also is present.

Keywords: crystal structure; hydrogen bonding; π–π interactions.

CCDC reference: 1400009

1. Related literature

For related structures, see: Gibson *et al.* (2010); Júnior *et al.* (2013); Montis & Hursthouse (2012).



2. Experimental

2.1. Crystal data

$C_{14}H_{11}NO_4$
M_r = 257.24
Monoclinic, $P2_1/c$
a = 5.6689 (5) Å
b = 32.039 (3) Å
c = 6.6413 (5) Å
β = 103.530 (5)°
V = 1172.74 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.38 × 0.30 × 0.16 mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.960, T_{max} = 0.984
9560 measured reflections
2582 independent reflections
1602 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
S = 1.04
2582 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

*Cg*2 is the centroid of the C9–C14 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.83	2.6470 (19)	176
O3—H3···O2	0.82	1.88	2.601 (2)	146
C4—H4···O4	0.93	2.23	2.828 (3)	122
C12—H12···*Cg*2^{ii}	0.93	2.95	3.773 (3)	142

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

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: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7423).

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supporting information

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Crystal structure of 4-benzamido-2-hydroxybenzoic acid

Muhammad Shahid, Muhammad Aziz Choudhary, Arshad Farooq Butt, Muhammad Nawaz Tahir and Muhammad Salim

S1. Comment

The crystal structures of methyl 4-(isonicotinoylamino)-2-methoxybenzoate (Gibson, 2010), 4-acetamido-2-hydroxy-benzoic acid (Montis & Hursthouse, 2012) and 2-([(4-carboxy-3-hydroxyphenyl)iminiumyl]methyl)phenolate (Junior *et al.*, 2013) have been published which are related to the title compound (I, Fig. 1). (I) is synthesized for the biological studies and for the complexation with different metals.

In (I), the parts of 4-aminosalicylic acid *A* (C1–C7/O1/O2/O3) and benzaldehyde *B* (C8—C14/O4) are planar with r. m. s. deviation of 0.0189 Å and 0.0524 Å, respectively. The dihedral angle between A/B is 5.86 (10)°. All heavy atoms of the compound form roughly a plane with r. m. s. devation of 0.0997 Å. In this plane the maximumu deviation is for O4-atom which is 0.321 (2) Å from the mean square plane. There exist intermolecular H-bonding of O—H···O type (Table 1, Fig. 2) forming *S* (6) loop. The molecules are dimerized due to inversion and O—H···O type of H-bonding (Table 1, Fig. 2) completing $R_2^2(8)$ rings motifs (Table 1, Fig. 2). The dimers are interlinked due to C—H···O interactions (Table 1, Fig. 2). There exist strong π–π interactions at a distance of 3.8423 (15) Å between the centeroids of *Cg*1—*Cg*2^{i} and *Cg*2—*Cg*1^{ii} [i = *x*, *y*, -1 + *z*: ii = *x*, *y*, 1 + *z*], where *Cg*1 and *Cg*2 are the centroids of benzene rings *C* (C2—C7) and *D* (C9—C14), respectively. There also exist a C—H···π interaction (Table 1) and may have important role in stabilizing the molecules.

S2. Experimental

4-Aminosalicylic acid was dissolved in ethylacetate and equimolar benzoyl chloride was added to the solution under stirring. The mixture was stired for 5 h. Light orange plates were obtained after 48 h.

S3. Refinement

The H atoms were positioned geometrically (C–H = 0.93 Å, N—H= 0.86 Å, O—H= 0.82 Å) and refined as riding with U_{iso}(H) = xU_{eq} (C, N, O), where x = 1.5 for hydroxy and x = 1.2 for other H-atoms.

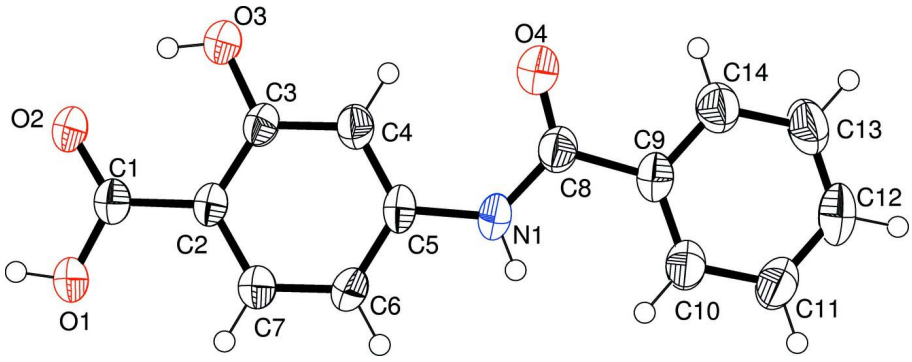

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted line show intramolecular H-bonding.

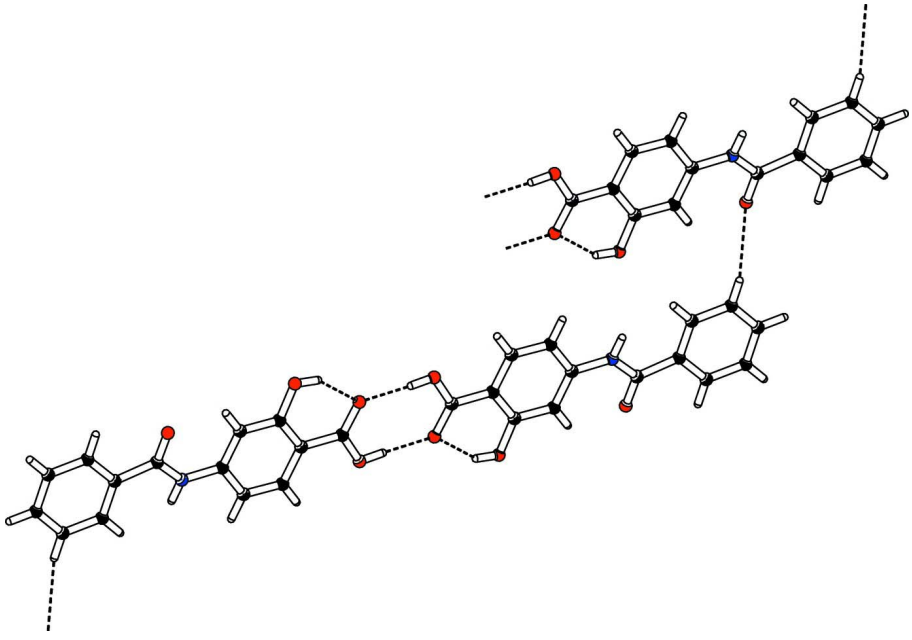

Figure 2

The partial packing, which shows that molecules form dimers and which are interlinked with each othere.

4-Benzamido-2-hydroxybenzoic acid

Crystal data

$C_{14}H_{11}NO_4$
$M_r = 257.24$
Monoclinic, $P2_1/c$
$a = 5.6689$ (5) Å
$b = 32.039$ (3) Å
$c = 6.6413$ (5) Å
$\beta = 103.530$ (5)°
$V = 1172.74$ (18) Å^3
$Z = 4$

$F(000) = 536$
$D_x = 1.457$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1602 reflections
$\theta = 3.2$–27.1°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Plate, light orange
0.38 × 0.30 × 0.16 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.70 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.960, T_{max} = 0.984
9560 measured reflections
2582 independent reflections
1602 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 27.1°, θ_{min} = 3.2°
h = −7→7
k = −41→24
l = −8→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.147
S = 1.04
2582 reflections
174 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.2313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.7252 (3)	0.01068 (6)	−0.2615 (2)	0.0515 (5)
H1	0.6964	−0.0023	−0.3708	0.077*
O2	0.3434 (3)	0.03147 (5)	−0.3868 (2)	0.0478 (4)
O3	0.1427 (3)	0.08122 (6)	−0.1660 (2)	0.0554 (5)
H3	0.1498	0.0670	−0.2670	0.083*
O4	0.2383 (3)	0.14573 (7)	0.4823 (3)	0.0723 (6)
N1	0.6221 (3)	0.12432 (6)	0.4934 (2)	0.0457 (5)
H1A	0.7682	0.1253	0.5677	0.055*
C1	0.5318 (4)	0.03152 (7)	−0.2456 (3)	0.0379 (5)
C2	0.5512 (4)	0.05479 (7)	−0.0538 (3)	0.0339 (5)
C3	0.3563 (4)	0.07877 (7)	−0.0233 (3)	0.0367 (5)
C4	0.3735 (4)	0.10154 (7)	0.1581 (3)	0.0415 (6)
H4	0.2419	0.1170	0.1775	0.050*
C5	0.5876 (4)	0.10094 (7)	0.3091 (3)	0.0378 (5)
C6	0.7835 (4)	0.07703 (8)	0.2805 (3)	0.0439 (6)
H6	0.9273	0.0766	0.3826	0.053*

C7	0.7651 (4)	0.05425 (7)	0.1036 (3)	0.0416 (6)
H7	0.8959	0.0382	0.0873	0.050*
C8	0.4525 (4)	0.14561 (8)	0.5685 (3)	0.0449 (6)
C9	0.5423 (4)	0.16900 (7)	0.7674 (3)	0.0417 (6)
C10	0.7710 (5)	0.16503 (9)	0.8943 (3)	0.0550 (7)
H10	0.8831	0.1472	0.8569	0.066*
C11	0.8349 (5)	0.18742 (9)	1.0775 (4)	0.0620 (8)
H11	0.9896	0.1846	1.1626	0.074*
C12	0.6713 (6)	0.21360 (9)	1.1334 (4)	0.0639 (8)
H12	0.7149	0.2287	1.2561	0.077*
C13	0.4429 (5)	0.21765 (9)	1.0090 (4)	0.0648 (8)
H13	0.3319	0.2356	1.0473	0.078*
C14	0.3771 (5)	0.19531 (8)	0.8274 (4)	0.0533 (7)
H14	0.2210	0.1979	0.7446	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0514 (10)	0.0617 (12)	0.0387 (9)	0.0102 (9)	0.0050 (7)	−0.0200 (8)
O2	0.0507 (9)	0.0585 (11)	0.0312 (8)	0.0042 (8)	0.0034 (7)	−0.0140 (7)
O3	0.0487 (10)	0.0754 (14)	0.0379 (9)	0.0111 (9)	0.0016 (7)	−0.0185 (8)
O4	0.0574 (12)	0.1049 (18)	0.0502 (10)	0.0122 (11)	0.0037 (8)	−0.0290 (10)
N1	0.0492 (11)	0.0537 (13)	0.0324 (9)	−0.0019 (10)	0.0059 (8)	−0.0152 (9)
C1	0.0473 (13)	0.0353 (13)	0.0319 (10)	−0.0030 (11)	0.0111 (9)	−0.0027 (9)
C2	0.0411 (12)	0.0335 (13)	0.0275 (10)	−0.0052 (10)	0.0090 (8)	−0.0033 (8)
C3	0.0414 (12)	0.0412 (14)	0.0271 (10)	−0.0043 (10)	0.0072 (8)	−0.0029 (9)
C4	0.0461 (13)	0.0452 (15)	0.0346 (11)	−0.0001 (11)	0.0120 (9)	−0.0068 (10)
C5	0.0511 (13)	0.0366 (14)	0.0276 (10)	−0.0068 (11)	0.0133 (9)	−0.0064 (9)
C6	0.0442 (13)	0.0548 (16)	0.0298 (11)	0.0008 (12)	0.0031 (9)	−0.0073 (10)
C7	0.0440 (13)	0.0462 (15)	0.0345 (11)	0.0025 (11)	0.0090 (9)	−0.0057 (10)
C8	0.0515 (15)	0.0474 (16)	0.0347 (11)	0.0022 (12)	0.0079 (10)	−0.0041 (10)
C9	0.0570 (14)	0.0368 (14)	0.0323 (11)	−0.0025 (11)	0.0124 (10)	−0.0026 (9)
C10	0.0643 (16)	0.0577 (17)	0.0404 (13)	0.0090 (13)	0.0070 (11)	−0.0099 (11)
C11	0.0696 (18)	0.066 (2)	0.0430 (14)	0.0040 (15)	−0.0008 (12)	−0.0123 (13)
C12	0.089 (2)	0.0590 (19)	0.0442 (14)	−0.0075 (16)	0.0173 (14)	−0.0202 (12)
C13	0.0700 (18)	0.064 (2)	0.0639 (17)	0.0005 (15)	0.0223 (14)	−0.0274 (14)
C14	0.0570 (15)	0.0515 (17)	0.0525 (14)	−0.0018 (13)	0.0152 (11)	−0.0103 (12)

Geometric parameters (Å, º)

O1—C1	1.309 (3)	C6—C7	1.366 (3)
O1—H1	0.8200	C6—H6	0.9300
O2—C1	1.245 (2)	C7—H7	0.9300
O3—C3	1.354 (2)	C8—C9	1.501 (3)
O3—H3	0.8200	C9—C10	1.377 (3)
O4—C8	1.215 (3)	C9—C14	1.386 (3)
N1—C8	1.365 (3)	C10—C11	1.386 (3)
N1—C5	1.409 (2)	C10—H10	0.9300

N1—H1A	0.8600	C11—C12	1.365 (4)
C1—C2	1.458 (3)	C11—H11	0.9300
C2—C3	1.399 (3)	C12—C13	1.369 (4)
C2—C7	1.404 (3)	C12—H12	0.9300
C3—C4	1.392 (3)	C13—C14	1.377 (3)
C4—C5	1.382 (3)	C13—H13	0.9300
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.398 (3)		

C1—O1—H1	109.5	C6—C7—H7	119.6
C3—O3—H3	109.5	C2—C7—H7	119.6
C8—N1—C5	128.08 (19)	O4—C8—N1	122.7 (2)
C8—N1—H1A	116.0	O4—C8—C9	120.6 (2)
C5—N1—H1A	116.0	N1—C8—C9	116.6 (2)
O2—C1—O1	121.71 (18)	C10—C9—C14	118.9 (2)
O2—C1—C2	122.3 (2)	C10—C9—C8	124.8 (2)
O1—C1—C2	115.96 (18)	C14—C9—C8	116.4 (2)
C3—C2—C7	118.20 (18)	C9—C10—C11	120.3 (2)
C3—C2—C1	120.46 (18)	C9—C10—H10	119.8
C7—C2—C1	121.3 (2)	C11—C10—H10	119.8
O3—C3—C4	116.45 (19)	C12—C11—C10	120.1 (2)
O3—C3—C2	122.51 (17)	C12—C11—H11	119.9
C4—C3—C2	121.04 (18)	C10—C11—H11	119.9
C5—C4—C3	119.5 (2)	C11—C12—C13	120.1 (2)
C5—C4—H4	120.3	C11—C12—H12	120.0
C3—C4—H4	120.3	C13—C12—H12	120.0
C4—C5—C6	119.99 (18)	C12—C13—C14	120.2 (3)
C4—C5—N1	122.9 (2)	C12—C13—H13	119.9
C6—C5—N1	117.12 (19)	C14—C13—H13	119.9
C7—C6—C5	120.4 (2)	C13—C14—C9	120.4 (2)
C7—C6—H6	119.8	C13—C14—H14	119.8
C5—C6—H6	119.8	C9—C14—H14	119.8
C6—C7—C2	120.9 (2)		

O2—C1—C2—C3	−1.0 (3)	C3—C2—C7—C6	−0.9 (3)
O1—C1—C2—C3	178.68 (19)	C1—C2—C7—C6	178.3 (2)
O2—C1—C2—C7	179.8 (2)	C5—N1—C8—O4	2.4 (4)
O1—C1—C2—C7	−0.5 (3)	C5—N1—C8—C9	−178.3 (2)
C7—C2—C3—O3	−179.9 (2)	O4—C8—C9—C10	168.2 (2)
C1—C2—C3—O3	0.8 (3)	N1—C8—C9—C10	−11.1 (4)
C7—C2—C3—C4	0.0 (3)	O4—C8—C9—C14	−9.7 (3)
C1—C2—C3—C4	−179.2 (2)	N1—C8—C9—C14	171.0 (2)
O3—C3—C4—C5	−179.1 (2)	C14—C9—C10—C11	−0.8 (4)
C2—C3—C4—C5	1.0 (3)	C8—C9—C10—C11	−178.6 (3)
C3—C4—C5—C6	−1.1 (3)	C9—C10—C11—C12	0.0 (4)
C3—C4—C5—N1	177.6 (2)	C10—C11—C12—C13	0.4 (5)
C8—N1—C5—C4	10.2 (4)	C11—C12—C13—C14	0.1 (5)
C8—N1—C5—C6	−171.0 (2)	C12—C13—C14—C9	−0.9 (4)

C4—C5—C6—C7	0.2 (3)	C10—C9—C14—C13	1.2 (4)
N1—C5—C6—C7	−178.6 (2)	C8—C9—C14—C13	179.3 (2)
C5—C6—C7—C2	0.9 (4)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C9–C14 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.83	2.6470 (19)	176
O3—H3···O2	0.82	1.88	2.601 (2)	146
C4—H4···O4	0.93	2.23	2.828 (3)	122
C12—H12···*Cg*2^{ii}	0.93	2.95	3.773 (3)	142

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