Acta Cryst E STRUCTURE REPORTS

OPEN ACCESS

ISSN 1600-5368

Crystal structure of 4-(3-carboxypropanamido)-2-hydroxybenzoic acid monohydrate

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Received 4 November 2014; accepted 9 November 2014

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

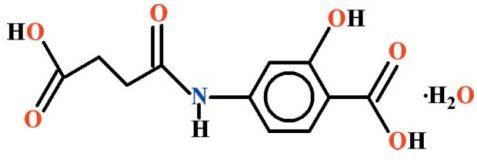
In the title hydrate, $C_{11}H_{11}NO_6\cdot H_2O$, the organic molecule is approximately planar (r.m.s. deviation for the non-H atoms = 0.129 Å) and an intramolecular O—H···O hydrogen bond closes an $S(6)$ ring. In the crystal, the benzoic acid group participates in an O—H···O hydrogen bond to the water molecule and accepts a similar bond from another water molecule. The other $-CO_2H$ group forms a carboxylic acid inversion dimer, thereby forming an $R_2^2(8)$ loop. These bonds, along with N—H···O and C—H···O interactions, generate a three-dimensional network.

Keywords: crystal structure; 2-hydroxybenzoic acid; hydrate; hydrogen bonding.

CCDC reference: 1033346

1. Related literature

For related structures, see: Gowda *et al.* (2009, 2011); Jia *et al.* (2012); Saraswathi *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{11}H_{11}NO_6\cdot H_2O$ M_r = 271.22

Monoclinic, $C2/c$
a = 25.2516 (19) Å
b = 8.4656 (5) Å
c = 12.4732 (10) Å
β = 117.446 (3)°
V = 2366.3 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 K
0.28 × 0.24 × 0.16 mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.964, T_{max} = 0.983
9402 measured reflections
2556 independent reflections
1738 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.122
S = 1.02
2556 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O7	0.82	1.81	2.605 (2)	163
O3—H3···O2	0.82	1.89	2.6099 (19)	146
O5—H5···O6^{i}	0.82	1.80	2.618 (2)	174
N1—H1A···O3^{ii}	0.86	2.18	3.035 (2)	173
C10—H10B···O5^{iii}	0.97	2.53	3.424 (3)	153
O7—H7A···O4^{iv}	0.78 (3)	2.15 (3)	2.854 (2)	151 (3)
O7—H7B···O2^{v}	0.72 (3)	2.17 (3)	2.827 (2)	152 (3)

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

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 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

Acknowledgements

The authors acknowledge the provision of funds for the purchase of diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7310).

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supporting information

Acta Cryst. (2014). E**70**, o1254–o1255 [doi:10.1107/S1600536814024581]

Crystal structure of 4-(3-carboxypropanamido)-2-hydroxybenzoic acid monohydrate

Muhammad Nawaz Tahir, Muhammad Naeem Ahmed, Arshad Farooq Butt and Hazoor Ahmad Shad

S1. Comment

The title compound (I), (Fig. 1) has been synthesized as a potential ligand for forming different metal complexes.

The crystal structures of *N*-Phenylsuccinamic acid (Gowda *et al.*, 2011), 4-((4-Chlorophenyl)amino)-4-oxobutanoic acid (Gowda *et al.*, 2009), 3-[(4-methylphenyl)carbamoyl]propanoic acid (Saraswathi *et al.*, 2011) and 4-[(2-Carboxyethyl)-amino]benzoic acid monohydrate (Jia *et al.*, 2012) have been published which are related to the title compound (I).

In (I) the moieties of 4-aminosalicylic acid A (C1—C7/N1/O1/O2/O3) and propanal B (C8—C10/O4) are planar with r.m.s. deviation of 0.0440 and 0.0122 Å, respectively. The dihedral angle between A/B is 5.729 (129)°. The carboxylate moiety C (C11/O5/O6) is of course planar. The dihedral angle between B/C is 13.446 (351)°. In (I), *S*(6) ring motif is present due to H-bonding of O—H···O type (Table 1, Fig. 2). The molecules are dimerized due to H-bondings of O—H···O type (Table 1, Fig. 2) from carboxyl groups formed from succinic anhydride. The dimers are further interlinked due to N—H···O, O—H···O bondings of water molecules and carboxyl group of aminoacid moiety. The molecules are stabilized in the form of three dimensional polymeric network.

S2. Experimental

Equimolar quantities of 4-aminosalicylic acid and succenic anhydride were stirred in ethylacetate for 4 h. The resulting mixture with white precipitate was placed to dry for 48 h. A colourless plate was selected for data collection.

S3. Refinement

The coordinates of H7A and H7B of water were refined. The H-atoms were positioned geometrically (O–H = 0.82, N–H = 0.86, C–H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C, N, O), where x = 1.5 for hydroxy & x = 1.2 for other H-atoms.

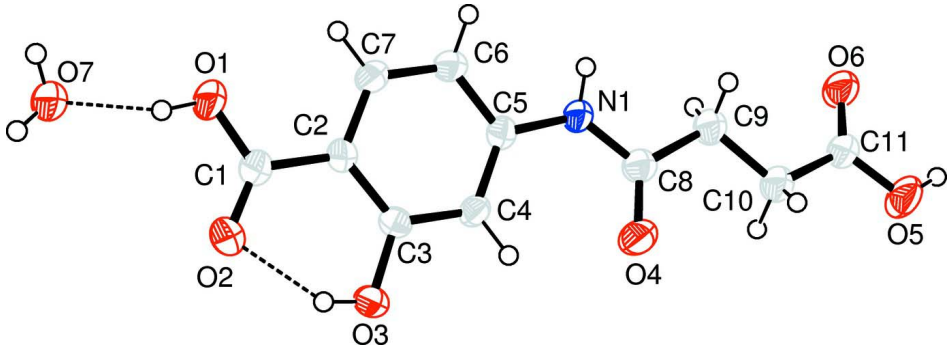

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level.

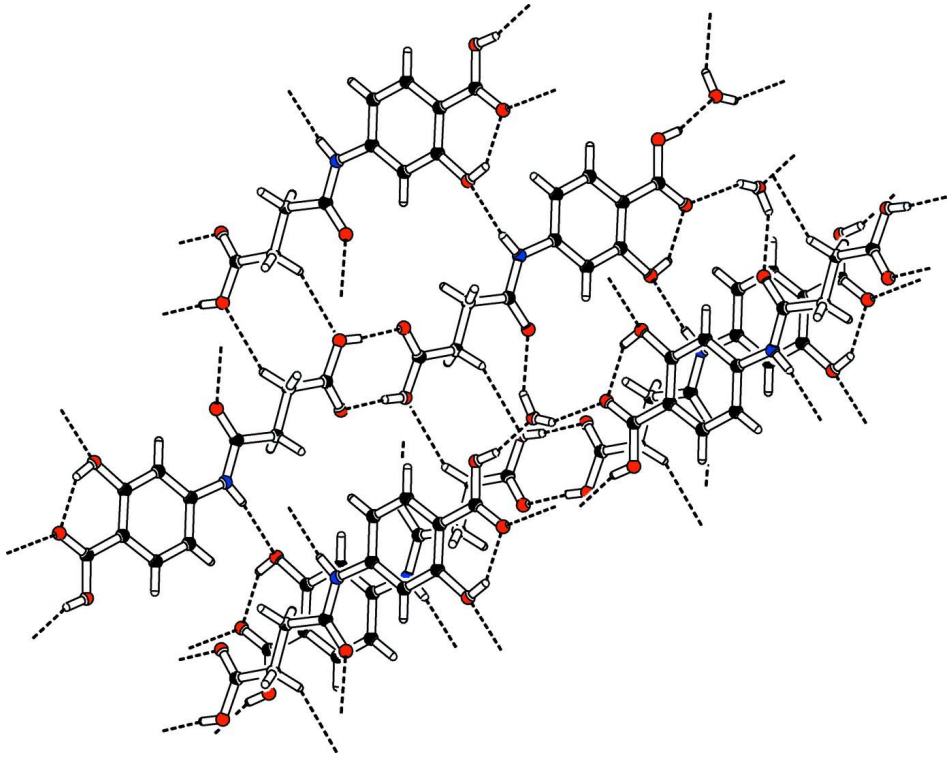

Figure 2

The partial packing (*PLATON*; Spek, 2009), which shows that molecules form dimers which are interlinked to three-dimensional polymeric network.

4-(3-Carboxypropanamido)–2-hydroxybenzoic acid monohydrate

Crystal data

$C_{11}H_{11}NO_6·H_2O$
M_r = 271.22
Monoclinic, *C*2/*c*
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b = 8.4656 (5) Å
c = 12.4732 (10) Å
β = 117.446 (3)°
V = 2366.3 (3) Å^3
Z = 8
F(000) = 1136
D_x = 1.523 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 1738 reflections
θ = 2.6–27.0°

μ = 0.13 mm^{-1}
T = 296 K

Plate, colourless
0.28 × 0.24 × 0.16 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.80 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.964, T_{max} = 0.983

9402 measured reflections
2556 independent reflections
1738 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.0°, θ_{min} = 2.6°
h = −32→32
k = −10→10
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.122
S = 1.02
2556 reflections
181 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.9039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 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	0.45356 (6)	−0.2827 (2)	0.78347 (15)	0.0561 (5)
H1	0.4765	−0.3404	0.8379	0.084*
O2	0.39232 (6)	−0.35291 (17)	0.85809 (13)	0.0452 (4)
O3	0.28556 (6)	−0.22986 (16)	0.76254 (12)	0.0378 (4)
H3	0.3133	−0.2845	0.8098	0.057*
O4	0.14489 (7)	0.0630 (2)	0.42736 (15)	0.0607 (5)
O5	−0.01362 (6)	0.3599 (2)	0.08989 (17)	0.0658 (6)
H5	−0.0287	0.4249	0.0352	0.099*
O6	0.06897 (6)	0.43838 (18)	0.08601 (14)	0.0494 (4)
N1	0.22758 (6)	0.07905 (19)	0.40306 (14)	0.0340 (4)
H1A	0.2411	0.1192	0.3571	0.041*
C1	0.40152 (9)	−0.2813 (2)	0.78284 (18)	0.0347 (5)

C2	0.35583 (8)	−0.1892 (2)	0.68505 (17)	0.0301 (4)
C3	0.29976 (8)	−0.1667 (2)	0.67907 (16)	0.0281 (4)
C4	0.25635 (8)	−0.0780 (2)	0.58744 (16)	0.0292 (4)
H4	0.2194	−0.0624	0.5852	0.035*
C5	0.26845 (8)	−0.0126 (2)	0.49906 (17)	0.0288 (4)
C6	0.32402 (9)	−0.0373 (2)	0.50317 (19)	0.0378 (5)
H6	0.3320	0.0051	0.4433	0.045*
C7	0.36652 (9)	−0.1230 (2)	0.59436 (19)	0.0383 (5)
H7	0.4035	−0.1379	0.5964	0.046*
C8	0.17030 (9)	0.1135 (2)	0.37201 (19)	0.0364 (5)
C9	0.13998 (9)	0.2187 (2)	0.26307 (18)	0.0381 (5)
H9A	0.1433	0.1717	0.1955	0.046*
H9B	0.1600	0.3203	0.2800	0.046*
C10	0.07507 (9)	0.2429 (2)	0.22904 (19)	0.0412 (5)
H10A	0.0548	0.1417	0.2065	0.049*
H10B	0.0720	0.2808	0.2993	0.049*
C11	0.04396 (9)	0.3559 (2)	0.12818 (18)	0.0367 (5)
O7	0.54172 (8)	−0.4497 (2)	0.94112 (17)	0.0565 (5)
H7A	0.5638 (13)	−0.464 (3)	0.914 (3)	0.085*
H7B	0.5480 (14)	−0.501 (4)	0.991 (3)	0.085*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0331 (8)	0.0803 (12)	0.0558 (11)	0.0224 (8)	0.0212 (8)	0.0336 (9)
O2	0.0410 (9)	0.0564 (9)	0.0380 (9)	0.0118 (7)	0.0181 (7)	0.0180 (7)
O3	0.0376 (8)	0.0478 (8)	0.0336 (8)	0.0106 (7)	0.0213 (7)	0.0120 (6)
O4	0.0362 (9)	0.0880 (12)	0.0641 (11)	0.0175 (8)	0.0284 (8)	0.0389 (10)
O5	0.0295 (9)	0.0899 (14)	0.0719 (13)	0.0136 (8)	0.0181 (9)	0.0463 (10)
O6	0.0346 (8)	0.0602 (10)	0.0530 (10)	0.0090 (7)	0.0199 (8)	0.0229 (8)
N1	0.0254 (9)	0.0424 (9)	0.0339 (10)	0.0059 (7)	0.0133 (8)	0.0122 (8)
C1	0.0309 (11)	0.0383 (11)	0.0343 (11)	0.0058 (9)	0.0147 (9)	0.0034 (9)
C2	0.0281 (10)	0.0325 (10)	0.0283 (10)	0.0038 (8)	0.0118 (8)	0.0032 (8)
C3	0.0316 (10)	0.0289 (9)	0.0257 (10)	−0.0002 (8)	0.0148 (8)	−0.0014 (8)
C4	0.0235 (10)	0.0335 (10)	0.0313 (11)	0.0024 (8)	0.0133 (9)	−0.0013 (8)
C5	0.0255 (10)	0.0290 (10)	0.0294 (10)	0.0022 (8)	0.0105 (8)	0.0018 (8)
C6	0.0328 (11)	0.0473 (12)	0.0385 (12)	0.0067 (9)	0.0207 (10)	0.0135 (10)
C7	0.0295 (11)	0.0479 (12)	0.0421 (12)	0.0088 (9)	0.0203 (10)	0.0128 (10)
C8	0.0335 (11)	0.0388 (11)	0.0385 (12)	0.0020 (9)	0.0180 (10)	0.0058 (9)
C9	0.0335 (11)	0.0411 (11)	0.0379 (12)	0.0055 (9)	0.0149 (10)	0.0085 (9)
C10	0.0325 (11)	0.0482 (13)	0.0416 (12)	0.0067 (10)	0.0159 (10)	0.0106 (10)
C11	0.0295 (11)	0.0424 (12)	0.0371 (12)	0.0045 (9)	0.0143 (10)	0.0039 (9)
O7	0.0420 (10)	0.0775 (13)	0.0557 (11)	0.0260 (9)	0.0273 (9)	0.0329 (9)

Geometric parameters (Å, º)

O1—C1	1.310 (2)	C4—C5	1.388 (3)
O1—H1	0.8200	C4—H4	0.9300

O2—C1	1.226 (2)	C5—C6	1.396 (3)
O3—C3	1.357 (2)	C6—C7	1.359 (3)
O3—H3	0.8200	C6—H6	0.9300
O4—C8	1.217 (2)	C7—H7	0.9300
O5—C11	1.305 (2)	C8—C9	1.505 (3)
O5—H5	0.8200	C9—C10	1.506 (3)
O6—C11	1.212 (2)	C9—H9A	0.9700
N1—C8	1.346 (2)	C9—H9B	0.9700
N1—C5	1.400 (2)	C10—C11	1.485 (3)
N1—H1A	0.8600	C10—H10A	0.9700
C1—C2	1.459 (3)	C10—H10B	0.9700
C2—C7	1.396 (3)	O7—H7A	0.78 (3)
C2—C3	1.396 (3)	O7—H7B	0.72 (3)
C3—C4	1.385 (3)		

C1—O1—H1	109.5	C5—C6—H6	119.9
C3—O3—H3	109.5	C6—C7—C2	121.31 (18)
C11—O5—H5	109.5	C6—C7—H7	119.3
C8—N1—C5	129.57 (16)	C2—C7—H7	119.3
C8—N1—H1A	115.2	O4—C8—N1	122.57 (19)
C5—N1—H1A	115.2	O4—C8—C9	122.59 (18)
O2—C1—O1	122.28 (18)	N1—C8—C9	114.84 (17)
O2—C1—C2	123.24 (18)	C8—C9—C10	111.69 (17)
O1—C1—C2	114.48 (17)	C8—C9—H9A	109.3
C7—C2—C3	118.18 (17)	C10—C9—H9A	109.3
C7—C2—C1	121.20 (17)	C8—C9—H9B	109.3
C3—C2—C1	120.62 (17)	C10—C9—H9B	109.3
O3—C3—C4	117.26 (16)	H9A—C9—H9B	107.9
O3—C3—C2	121.68 (16)	C11—C10—C9	114.12 (17)
C4—C3—C2	121.05 (16)	C11—C10—H10A	108.7
C3—C4—C5	119.45 (17)	C9—C10—H10A	108.7
C3—C4—H4	120.3	C11—C10—H10B	108.7
C5—C4—H4	120.3	C9—C10—H10B	108.7
C4—C5—C6	119.77 (17)	H10A—C10—H10B	107.6
C4—C5—N1	123.71 (16)	O6—C11—O5	122.79 (19)
C6—C5—N1	116.52 (17)	O6—C11—C10	124.11 (18)
C7—C6—C5	120.22 (18)	O5—C11—C10	113.09 (18)
C7—C6—H6	119.9	H7A—O7—H7B	111 (3)

O2—C1—C2—C7	−173.4 (2)	C8—N1—C5—C6	175.30 (19)
O1—C1—C2—C7	6.1 (3)	C4—C5—C6—C7	−1.0 (3)
O2—C1—C2—C3	5.8 (3)	N1—C5—C6—C7	179.09 (18)
O1—C1—C2—C3	−174.74 (18)	C5—C6—C7—C2	0.5 (3)
C7—C2—C3—O3	178.72 (17)	C3—C2—C7—C6	0.8 (3)
C1—C2—C3—O3	−0.5 (3)	C1—C2—C7—C6	180.0 (2)
C7—C2—C3—C4	−1.6 (3)	C5—N1—C8—O4	−1.2 (4)
C1—C2—C3—C4	179.23 (17)	C5—N1—C8—C9	179.15 (18)
O3—C3—C4—C5	−179.20 (16)	O4—C8—C9—C10	−3.5 (3)

C2—C3—C4—C5	1.1 (3)	N1—C8—C9—C10	176.14 (17)
C3—C4—C5—C6	0.2 (3)	C8—C9—C10—C11	175.35 (17)
C3—C4—C5—N1	−179.88 (17)	C9—C10—C11—O6	−9.3 (3)
C8—N1—C5—C4	−4.6 (3)	C9—C10—C11—O5	171.72 (19)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O7	0.82	1.81	2.605 (2)	163
O3—H3···O2	0.82	1.89	2.6099 (19)	146
O5—H5···O6^{i}	0.82	1.80	2.618 (2)	174
N1—H1*A*···O3^{ii}	0.86	2.18	3.035 (2)	173
C10—H10*B*···O5^{iii}	0.97	2.53	3.424 (3)	153
O7—H7*A*···O4^{iv}	0.78 (3)	2.15 (3)	2.854 (2)	151 (3)
O7—H7*B*···O2^{v}	0.72 (3)	2.17 (3)	2.827 (2)	152 (3)

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