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D-(−)-2-Azaniumyl-2-(4-hydroxyphenyl)acetate: an orthorhombic polymorph

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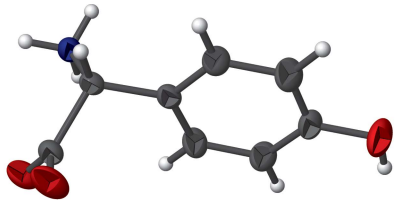
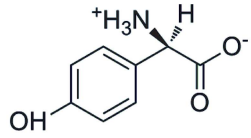
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_8H_9NO_3$, is the zwitterionic form of D-(−)-4-hydroxyphenylglycine. The plane of the hydroxybenzene ring is inclined at an angle of 88.89 (5)° to the best-fit plane through the five non-H atoms of the aminoacetate substituent. In the crystal, N—H···O and O—H···O hydrogen bonds link adjacent molecules, forming a three-dimensional network. Weak C—H···π interactions are also observed.

3D view



Chemical scheme



Structure description

D-(−)-4-hydroxyphenylglycine (D-HPG) is a key intermediate in the preparation of semi-synthetic antibiotics (Rudolph *et al.*, 2001). The crystal structure of a monoclinic polymorph of D-(−)-amino-(4-hydroxyphenyl) acetate has been reported previously in the space group $P2_1$ (Báthori & Bourne, 2009). For the crystal structures of other related compounds, see for example the structures of (*p*-hydroxyphenyl)glycine(−)-4-(2-chlorophenyl)-5,5-dimethyl-2-hydroxy-1,3,2-dioxaphosphorinane 2-oxide (Ten Hoeve & Wynberg, 1985) and D-*p*-hydroxyphenylglycine (−)-1-phenylethanesulfonate (Yoshioka *et al.*, 1994). Here we report the crystal structure of D-(−)-amino-(4-hydroxyphenyl)-acetate in the orthorhombic space group $P2_12_12_1$. Although there is no heavy atom in the structure, the absolute configuration could be well determined by the Flack parameter [0.03 (11)]. However, crystals were grown from a commercial sample of D-(−)-4-hydroxyphenylglycine and the absolute structure assigned on that basis.

As shown in Fig. 1, the reported compound is a zwitterion with the carboxylic acid of the glycine moiety deprotonated and the amino group protonated. The hydroxybenzene ring plane approximately bisects the N1—C7—C8 angle and the plane of the hydroxybenzene ring is inclined at an angle of 88.89 (5) ° to the best fit plane through the five non-hydrogen atoms of the aminoacetate substituent.

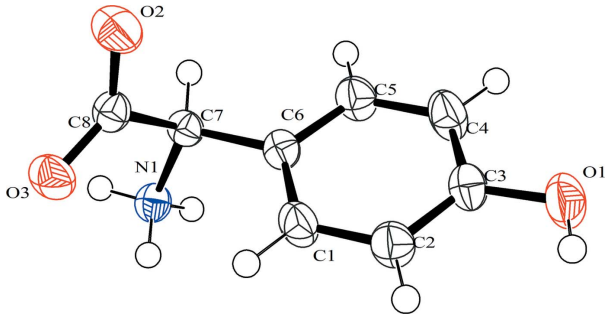


Figure 1
View of the title compound, showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

In the crystal, adjacent molecules are linked by N—H···O and O—H···O hydrogen bonds, and a weak C—H···π interaction into a three-dimensional network, Fig. 2 and Table 1.

Synthesis and crystallization

1.2 g D-(−)-4-hydroxyphenylglycine (Aldrich, 22818–40-2) was stirred into 40 ml of water at 55°C to prepare a saturated solution. Colourless block-like crystals of the title compound were grown by slow evaporation of this solution at 1°C.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors are grateful to the Analysis and Testing Center of Zhengzhou University for use of the single-crystal X-ray diffractometer. This work was supported by the Science and Technology Bureau of Henan through the Cooperation Research Project fund (No. 152107000043 for WL), the Education Bureau of Henan for Major Research Project fund (No. 14 A530007 for WL), National Natural Science Foundation of China (project Nos. 81430085, 21372206 and 81172937 for HML), a PhD Educational Award from the Ministry of Education (No. 20134101130001 for HML), and the Graduate Student Science Research Foundation of Zhengzhou University.

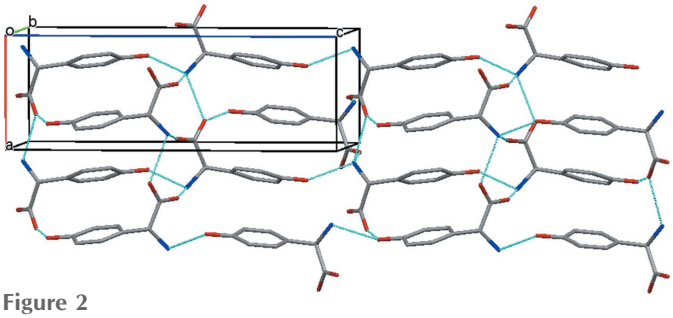


Figure 2
The packing diagram of the title compound, viewed along the *b* axis.

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

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.89	2.02	2.838 (2)	153
N1—H1*B*···O2^{ii}	0.89	1.96	2.811 (2)	160
N1—H1*C*···O3^{iii}	0.89	1.87	2.759 (2)	172
O1—H1*D*···O3^{iv}	0.82	1.81	2.591 (2)	158
C2—H2···*Cg*^{v}	0.93	3.17	3.913 (2)	139

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_9NO_3$
M_r	167.16
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	293
a, *b*, *c* (Å)	5.83624 (17), 8.4245 (3), 17.0381 (5)
V (Å^3)	837.72 (4)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.86
Crystal size (mm)	0.2 × 0.19 × 0.16
Data collection	
Diffractometer	Agilent Xcalibur Eos Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min}, T_{max}	0.861, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	3647, 1613, 1570
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.613
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.096, 1.05
No. of reflections	1613
No. of parameters	112
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.34, −0.17
Absolute structure	Flack *x* determined using 616 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.03 (11)

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

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Crystal data

$C_8H_9NO_3$
M_r = 167.16
Orthorhombic, $P2_12_12_1$
a = 5.83624 (17) Å
b = 8.4245 (3) Å
c = 17.0381 (5) Å
V = 837.72 (4) Å^3
Z = 4
F(000) = 352
D_x = 1.325 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2415 reflections
θ = 5.2–70.8°
μ = 0.86 mm^{-1}
T = 293 K
Block, colourless
0.2 × 0.19 × 0.16 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.2312 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.861, T_{max} = 1.000
3647 measured reflections
1613 independent reflections
1570 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 70.9°, θ_{min} = 5.2°
h = −7→6
k = −10→10
l = −17→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.096
S = 1.05
1613 reflections
112 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.1107P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.076 (5)
Absolute structure: Flack x determined using 616 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.03 (11)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8218 (4)	0.6736 (2)	0.67248 (12)	0.0355 (5)
H1	0.8643	0.7484	0.6351	0.043*
C2	0.7995 (4)	0.7195 (3)	0.75027 (12)	0.0369 (5)
H2	0.8267	0.8242	0.7648	0.044*
C3	0.7367 (4)	0.6088 (3)	0.80590 (11)	0.0329 (5)
C4	0.6892 (5)	0.4543 (3)	0.78378 (12)	0.0424 (6)
H4	0.6415	0.3806	0.8210	0.051*
C5	0.7130 (4)	0.4097 (2)	0.70566 (12)	0.0355 (5)
H5	0.6822	0.3055	0.6911	0.043*
C6	0.7817 (3)	0.5177 (2)	0.64934 (10)	0.0264 (4)
C7	0.8146 (3)	0.4675 (2)	0.56526 (10)	0.0250 (4)
H7	0.7981	0.3519	0.5622	0.030*
C8	1.0554 (3)	0.5127 (2)	0.53564 (11)	0.0270 (4)
N1	0.6394 (3)	0.5416 (2)	0.51327 (9)	0.0276 (4)
H1A	0.6596	0.5079	0.4642	0.033*
H1B	0.6540	0.6467	0.5148	0.033*
H1C	0.5000	0.5145	0.5296	0.033*
O1	0.7186 (4)	0.6457 (2)	0.88365 (8)	0.0478 (5)
H1D	0.7565	0.7384	0.8905	0.072*
O2	1.0735 (3)	0.62801 (19)	0.49116 (10)	0.0396 (4)
O3	1.2178 (3)	0.4283 (2)	0.56048 (10)	0.0445 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0467 (12)	0.0325 (10)	0.0274 (9)	−0.0062 (9)	0.0064 (9)	0.0043 (8)
C2	0.0468 (12)	0.0329 (10)	0.0310 (10)	−0.0058 (9)	0.0005 (9)	−0.0006 (8)
C3	0.0358 (10)	0.0425 (11)	0.0205 (8)	−0.0008 (8)	−0.0037 (8)	0.0010 (8)
C4	0.0586 (14)	0.0416 (12)	0.0268 (10)	−0.0122 (11)	0.0024 (9)	0.0069 (8)
C5	0.0447 (11)	0.0325 (10)	0.0293 (9)	−0.0092 (9)	0.0017 (8)	0.0014 (8)
C6	0.0216 (8)	0.0330 (9)	0.0244 (9)	−0.0005 (7)	0.0003 (7)	0.0028 (7)
C7	0.0216 (8)	0.0274 (8)	0.0260 (8)	−0.0022 (7)	0.0014 (7)	0.0022 (7)
C8	0.0232 (9)	0.0317 (9)	0.0261 (9)	−0.0017 (7)	0.0027 (7)	0.0011 (7)
N1	0.0235 (7)	0.0347 (8)	0.0247 (7)	−0.0002 (6)	0.0002 (6)	0.0000 (6)
O1	0.0744 (12)	0.0473 (9)	0.0216 (7)	−0.0069 (9)	−0.0020 (8)	0.0002 (6)
O2	0.0347 (8)	0.0366 (8)	0.0474 (9)	−0.0045 (7)	0.0063 (7)	0.0119 (6)
O3	0.0237 (7)	0.0589 (10)	0.0510 (9)	0.0039 (7)	0.0020 (6)	0.0199 (7)

Geometric parameters (Å, º)

C1—H1	0.9300	C6—C7	1.506 (3)
C1—C2	1.387 (3)	C7—H7	0.9800
C1—C6	1.391 (3)	C7—C8	1.541 (2)
C2—H2	0.9300	C7—N1	1.490 (2)
C2—C3	1.379 (3)	C8—O2	1.237 (2)
C3—C4	1.383 (3)	C8—O3	1.258 (3)
C3—O1	1.365 (2)	N1—H1A	0.8900
C4—H4	0.9300	N1—H1B	0.8900
C4—C5	1.390 (3)	N1—H1C	0.8900
C5—H5	0.9300	O1—H1D	0.8200
C5—C6	1.382 (3)		
C2—C1—H1	119.4	C5—C6—C7	120.84 (18)
C2—C1—C6	121.21 (19)	C6—C7—H7	108.5
C6—C1—H1	119.4	C6—C7—C8	111.00 (15)
C1—C2—H2	120.2	C8—C7—H7	108.5
C3—C2—C1	119.6 (2)	N1—C7—C6	111.12 (16)
C3—C2—H2	120.2	N1—C7—H7	108.5
C2—C3—C4	120.14 (18)	N1—C7—C8	109.12 (14)
O1—C3—C2	122.26 (19)	O2—C8—C7	118.21 (16)
O1—C3—C4	117.60 (18)	O2—C8—O3	125.84 (18)
C3—C4—H4	120.2	O3—C8—C7	115.94 (16)
C3—C4—C5	119.67 (19)	C7—N1—H1A	109.5
C5—C4—H4	120.2	C7—N1—H1B	109.5
C4—C5—H5	119.5	C7—N1—H1C	109.5
C6—C5—C4	121.1 (2)	H1A—N1—H1B	109.5
C6—C5—H5	119.5	H1A—N1—H1C	109.5
C1—C6—C7	120.89 (17)	H1B—N1—H1C	109.5
C5—C6—C1	118.26 (18)	C3—O1—H1D	109.5
C1—C2—C3—C4	−2.0 (4)	C4—C5—C6—C7	178.0 (2)
C1—C2—C3—O1	178.0 (2)	C5—C6—C7—C8	−126.0 (2)
C1—C6—C7—C8	53.4 (2)	C5—C6—C7—N1	112.4 (2)
C1—C6—C7—N1	−68.2 (2)	C6—C1—C2—C3	0.1 (4)
C2—C1—C6—C5	1.6 (3)	C6—C7—C8—O2	−104.3 (2)
C2—C1—C6—C7	−177.8 (2)	C6—C7—C8—O3	74.7 (2)
C2—C3—C4—C5	2.3 (4)	N1—C7—C8—O2	18.4 (2)
C3—C4—C5—C6	−0.6 (4)	N1—C7—C8—O3	−162.55 (18)
C4—C5—C6—C1	−1.4 (4)	O1—C3—C4—C5	−177.8 (2)

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.89	2.02	2.838 (2)	153
N1—H1*B*···O2^{ii}	0.89	1.96	2.811 (2)	160

N1—H1*C*···O3^{iii}	0.89	1.87	2.759 (2)	172
O1—H1*D*···O3^{iv}	0.82	1.81	2.591 (2)	158
C2—H2···*Cg*^{v}	0.93	3.17	3.913 (2)	139

Symmetry codes: (i) $-x+3/2, -y+1, z-1/2$; (ii) $x-1/2, -y+3/2, -z+1$; (iii) $x-1, y, z$; (iv) $-x+2, y+1/2, -z+3/2$; (v) $-x, y+1/2, -z+3/2$.