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rac-2-Hydroxy-2-(2-nitrophenyl)acetic acid

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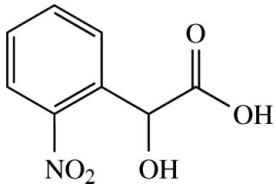
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.126; data-to-parameter ratio = 14.6.

In the racemic title compound, $C_8H_7NO_5$, the nitro group attached to the 2-hydroxyacetic acid (glycolic acid) moiety is tilted by 31.2 (2)° with respect to the aromatic plane. The characteristics of the molecular packing are (i) hydrogen-bonded carboxylic acid dimers; (ii) a trifurcated hydrogen bond with the 2-hydroxy function as the donor and a nitro O atom as the only intermolecular acceptor, both kinds of hydrogen bonds linking the molecules into ribbons along [001].

Related literature

The compound was prepared according to a known procedure (Heller, 1904).



Experimental

Crystal data

$C_8H_7NO_5$
M_r = 197.15
Monoclinic, $P2_1/c$
a = 9.4343 (3) Å
b = 9.7690 (5) Å
c = 9.8842 (5) Å
β = 116.130 (2)°
V = 817.86 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 200 (2) K
0.14 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
3617 measured reflections
1871 independent reflections
1466 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.126
S = 1.04
1871 reflections
128 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H82···O1^{i}	0.87	1.81	2.679 (2)	176
O3—H83···O4^{ii}	0.96	2.05	2.888 (2)	146
O3—H83···O1	0.96	2.25	2.742 (2)	111
O3—H83···O5	0.96	2.44	2.843 (2)	105

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

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2054).

References

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supplementary materials

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rac-2-Hydroxy-2-(2-nitrophenyl)acetic acid

R. Betz, F. Betzler and P. Klüfers

Comment

Racemic 2-hydroxy-2-*ortho*-nitrophenyl acetic acid (I) acid was prepared as a chelating molecule bearing the sterically demanding *ortho*-nitro-phenyl group. In the molecular structure, the best planes of the nitro function and the aromatic ring enclose a tilt angle of 31.2 (2)°. The molecules of (I) are stiffened by intramolecular hydrogen bonds (Fig. 1). The 2-hydroxy function is a hydrogen-bond donor towards – listed in descending order of the bond's stabilities in terms of both the H—A distances and the D—H—A angles – a nitro-O atom in an intermolecular contact, the double-bonded O atom of the carboxylic group, and another nitro-O atom. The latter two contacts in the thus trifurcated bond are intramolecular.

In the crystal structure of (I), each molecule is part of a hydrogen-bonded ribbon and thus has two hydrogen-bonded neighbours. Each individual molecular contact comprises two bonds in a centrosymmetric pattern: the first pattern resembles the common motif of carboxylic-acid dimers (pink hydrogen bonds in Fig. 2); in the second pattern, hydroxy and nitro functions are connected in a similar way (green bonds in Fig. 2). The three-dimensional structure is formed by hydrophobic interaction of the phenyl groups which enclose the ribbons laterally (Fig. 2).

Experimental

The compound was obtained by acidic hydrolysis of *ortho*-nitro-benzaldehyde cyanohydrin according to a known procedure (Heller, 1904). Crystals suitable for X-ray analysis were obtained by recrystallization of the crude reaction product from boiling water.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to U_{iso}(H) = 0.041 (2).

Figures

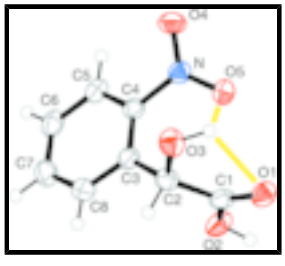

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms. The two intramolecular components of a trifurcated hydrogen bond are shown as yellow bars.

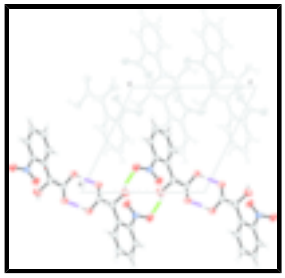

Fig. 2. A hydrogen-bonded ribbon along [001] in a [010]-projection. Pink bonds mark carboxylic-acid dimers, green bonds connect 2-hydroxy and nitro functions. A second ribbon (grey) shows the van-der-Waals packing through the lateral phenyl groups.

rac-2-Hydroxy-2-(2-nitrophenyl)acetic acid

Crystal data

$C_8H_7NO_5$	F_{000} = 408
M_r = 197.15	D_x = 1.601 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 13027 reflections
a = 9.4343 (3) Å	θ = 3.1–27.5º
b = 9.7690 (5) Å	μ = 0.14 mm^{-1}
c = 9.8842 (5) Å	T = 200 (2) K
β = 116.130 (2)º	Block, yellow
V = 817.86 (6) Å^3	0.14 × 0.10 × 0.08 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	1466 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.020
Monochromator: graphite	θ_{max} = 27.5º
T = 200(2) K	θ_{min} = 3.2º
φ/ω scans	h = −12→12
Absorption correction: none	k = −12→12
3617 measured reflections	l = −12→12
1871 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.046	Only H-atom displacement parameters refined
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.4137P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$
1871 reflections	$\Delta\rho_{max}$ = 0.50 e Å^{-3}
128 parameters	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.99600 (16)	0.23800 (15)	0.14149 (16)	0.0418 (4)
H83	1.0642	0.1602	0.1755	0.041 (2)*
O2	0.82437 (14)	0.08167 (15)	0.36283 (14)	0.0402 (4)
H82	0.8597	0.0329	0.4448	0.041 (2)*
O1	1.07440 (14)	0.07928 (14)	0.39310 (14)	0.0378 (3)
O4	0.76077 (16)	−0.05081 (16)	−0.15598 (15)	0.0456 (4)
O5	0.89088 (15)	−0.03814 (15)	0.08572 (14)	0.0408 (4)
N	0.78192 (16)	−0.00364 (15)	−0.03350 (16)	0.0298 (3)
C1	0.9378 (2)	0.1182 (2)	0.32753 (19)	0.0327 (4)
C2	0.8853 (2)	0.2223 (2)	0.2006 (2)	0.0334 (4)
H2	0.8834	0.3123	0.2481	0.041 (2)*
C3	0.7177 (2)	0.20068 (19)	0.07818 (19)	0.0302 (4)
C4	0.66966 (19)	0.09861 (18)	−0.03184 (18)	0.0281 (4)
C5	0.5157 (2)	0.08671 (19)	−0.14383 (19)	0.0323 (4)
H5	0.4882	0.0162	−0.2173	0.041 (2)*
C6	0.4034 (2)	0.1789 (2)	−0.1468 (2)	0.0375 (4)
H6	0.2972	0.1717	−0.2216	0.041 (2)*
C7	0.4467 (2)	0.2817 (2)	−0.0402 (2)	0.0423 (5)
H7	0.3699	0.3455	−0.0419	0.041 (2)*
C8	0.6010 (2)	0.2925 (2)	0.0689 (2)	0.0382 (4)
H8	0.6283	0.3650	0.1400	0.041 (2)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0355 (7)	0.0453 (8)	0.0493 (8)	0.0044 (6)	0.0229 (7)	0.0084 (6)
O2	0.0289 (7)	0.0636 (9)	0.0260 (7)	0.0005 (6)	0.0101 (5)	0.0037 (6)
O1	0.0264 (6)	0.0541 (8)	0.0300 (7)	0.0014 (6)	0.0096 (5)	0.0017 (6)
O4	0.0412 (8)	0.0571 (9)	0.0332 (7)	0.0082 (6)	0.0117 (6)	−0.0134 (6)
O5	0.0384 (7)	0.0454 (8)	0.0307 (7)	0.0119 (6)	0.0079 (6)	0.0031 (6)
N	0.0275 (7)	0.0326 (8)	0.0277 (8)	−0.0007 (6)	0.0108 (6)	−0.0009 (6)
C1	0.0284 (9)	0.0438 (10)	0.0245 (8)	−0.0032 (8)	0.0104 (7)	−0.0069 (7)
C2	0.0283 (9)	0.0373 (10)	0.0336 (9)	0.0001 (7)	0.0128 (8)	−0.0044 (7)
C3	0.0287 (8)	0.0358 (9)	0.0258 (8)	0.0027 (7)	0.0118 (7)	0.0032 (7)
C4	0.0279 (8)	0.0325 (9)	0.0256 (8)	0.0019 (7)	0.0132 (7)	0.0030 (7)

C5	0.0289 (9)	0.0393 (10)	0.0261 (9)	−0.0010 (7)	0.0097 (7)	0.0045 (7)
C6	0.0270 (9)	0.0491 (11)	0.0331 (10)	0.0052 (8)	0.0101 (7)	0.0102 (8)
C7	0.0353 (10)	0.0507 (12)	0.0422 (11)	0.0148 (9)	0.0182 (9)	0.0086 (9)
C8	0.0395 (10)	0.0422 (11)	0.0331 (10)	0.0079 (8)	0.0161 (8)	−0.0006 (8)

Geometric parameters (Å, °)

O3—C2	1.412 (2)	C3—C8	1.391 (3)
O3—H83	0.9560	C3—C4	1.396 (2)
O2—C1	1.313 (2)	C4—C5	1.390 (2)
O2—H82	0.8701	C5—C6	1.381 (3)
O1—C1	1.220 (2)	C5—H5	0.9500
O4—N	1.2269 (19)	C6—C7	1.381 (3)
O5—N	1.2214 (18)	C6—H6	0.9500
N—C4	1.461 (2)	C7—C8	1.382 (3)
C1—C2	1.518 (3)	C7—H7	0.9500
C2—C3	1.524 (2)	C8—H8	0.9500
C2—H2	1.0000		
C2—O3—H83	105.9	C4—C3—C2	125.80 (15)
C1—O2—H82	111.9	C5—C4—C3	123.17 (16)
O5—N—O4	123.39 (15)	C5—C4—N	116.24 (15)
O5—N—C4	118.95 (14)	C3—C4—N	120.59 (14)
O4—N—C4	117.66 (14)	C6—C5—C4	119.02 (17)
O1—C1—O2	124.62 (18)	C6—C5—H5	120.5
O1—C1—C2	122.08 (16)	C4—C5—H5	120.5
O2—C1—C2	113.20 (15)	C7—C6—C5	119.45 (17)
O3—C2—C1	112.52 (14)	C7—C6—H6	120.3
O3—C2—C3	112.57 (14)	C5—C6—H6	120.3
C1—C2—C3	114.21 (15)	C6—C7—C8	120.45 (18)
O3—C2—H2	105.5	C6—C7—H7	119.8
C1—C2—H2	105.5	C8—C7—H7	119.8
C3—C2—H2	105.5	C7—C8—C3	122.22 (18)
C8—C3—C4	115.67 (16)	C7—C8—H8	118.9
C8—C3—C2	118.48 (16)	C3—C8—H8	118.9
O1—C1—C2—O3	16.5 (2)	O5—N—C4—C5	−148.82 (16)
O2—C1—C2—O3	−166.89 (15)	O4—N—C4—C5	30.9 (2)
O1—C1—C2—C3	146.46 (17)	O5—N—C4—C3	30.7 (2)
O2—C1—C2—C3	−37.0 (2)	O4—N—C4—C3	−149.49 (17)
O3—C2—C3—C8	−121.79 (18)	C3—C4—C5—C6	−0.6 (3)
C1—C2—C3—C8	108.29 (19)	N—C4—C5—C6	178.92 (15)
O3—C2—C3—C4	55.5 (2)	C4—C5—C6—C7	0.9 (3)
C1—C2—C3—C4	−74.4 (2)	C5—C6—C7—C8	−0.1 (3)
C8—C3—C4—C5	−0.5 (3)	C6—C7—C8—C3	−1.1 (3)
C2—C3—C4—C5	−177.85 (17)	C4—C3—C8—C7	1.3 (3)
C8—C3—C4—N	180.00 (16)	C2—C3—C8—C7	178.90 (17)
C2—C3—C4—N	2.6 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H82···O1^{i}	0.87	1.81	2.679 (2)	176
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O3—H83···O5	0.96	2.44	2.843 (2)	105

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

Fig. 1

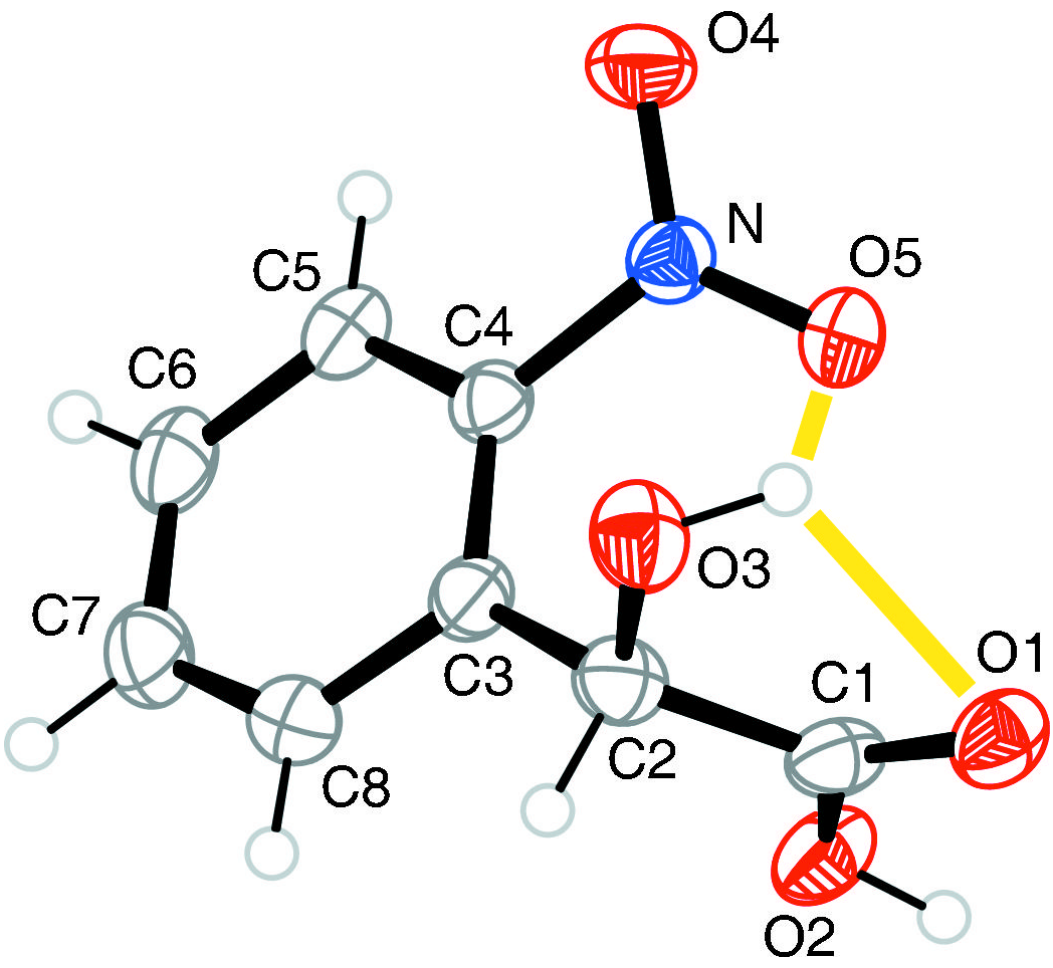

Fig. 2