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Ethyl 2-(2-hydroxy-5-nitrophenyl)acetate

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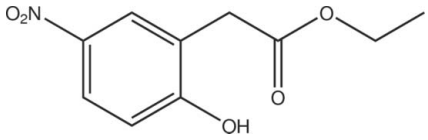
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.057; wR factor = 0.153; data-to-parameter ratio = 13.1.

In the crystal structure of the title compound, $C_{10}H_{11}NO_5$, intermolecular O—H···O hydrogen bonds link the molecules into chains along the b-axis direction. Weak C—H·O hydrogen bonds also occur.

Related literature

For the use of the title compound as a pharmaceutical intermediate and for the preparation, see: Omar *et al.* (2003). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_{11}NO_5$
M_r = 225.20
Monoclinic, $P2_1/c$
a = 11.066 (2) Å
b = 10.860 (2) Å
c = 8.6970 (17) Å
β = 97.85 (3)°
V = 1035.4 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.988
3894 measured reflections
1905 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.153
S = 1.00
1905 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2^{i}	0.82	1.93	2.749 (2)	180
C2—H2A···O3^{ii}	0.97	2.60	3.340 (3)	134
C4—H4A···O4^{iii}	0.97	2.54	3.431 (3)	153
C6—H6A···O5^{iv}	0.93	2.51	3.351 (3)	151
C8—H8A···O4^{v}	0.93	2.59	3.430 (3)	150

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2266).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 EXPRESS*. Enraf–Nonius, Delft. The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Omar, M. M. & Eusebio, J. (2003). *Tetrahedron*, **59**, 4223–4229.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2011). E**67**, o509 [doi:10.1107/S1600536811000389]

Ethyl 2-(2-hydroxy-5-nitrophenyl)acetate

Bing Guo, Ya-Bin Shi, Jin-Hua Yao and Jian-Ning Guan

S1. Comment

The compound, 5-nitrobenzofuran-2(3H)-one, which is an effective intermediate prepared dronedarone, plays an important role in the fields of natural products and medicinal chemistry. The title compound, ethyl 2-(2-hydroxy-5-nitrophenyl)acetate, (I), is a useful pharmaceutical intermediate (Omar *et al.*, 2003).

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. In the crystal structure, intermolecular O-H···O hydrogen bonds (Table 1.) link the molecules forming a stable structure and the other weak C-H···O hydrogen bonds reinforced the packing (Fig. 2).

S2. Experimental

The title compound I was prepared by the literature method (Omar *et al.*, 2003). To a 100 mL flask provided with Dean–Stark tramp and magnetic stirrer was added (2-hydroxy-phenyl)-acetic acid (4.4 g, 29 mmol) in 60 mL of toluene and catalytic amounts of p-TsOH. The mixture was refluxed for 4 h with removal of water and then the residual solvent was removed at reduced pressure to give 3H-benzofuran-2-one in quantitative yield (3.9 g), mp 325K. Then, a mixture of concentrated nitric acid (4 ml) and glacial acetic acid (4 ml) was added drop wise to a solution of 3H-benzofuran-2-one (3.9 g) in acetic anhydride (25 ml) while the temperature was maintained below 293 K. The mixture was stirred and refluxed for 1 hour in ethanol (30 ml). The precipitate (the desired anthranilic acid esters ethyl 2-(2-hydroxy-5-nitrophenyl) acetate) was filtered off and washed with water, yield 80%. Crystals suitable for x-ray analysis were obtained by slow evaporation of an methanol solution.

S3. Refinement

H atoms were positioned geometrically, with O-H =1.92 Å (for OH) and C-H =0.93, 0.98 and 0.96 Å for aromatic, methine and methyl, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

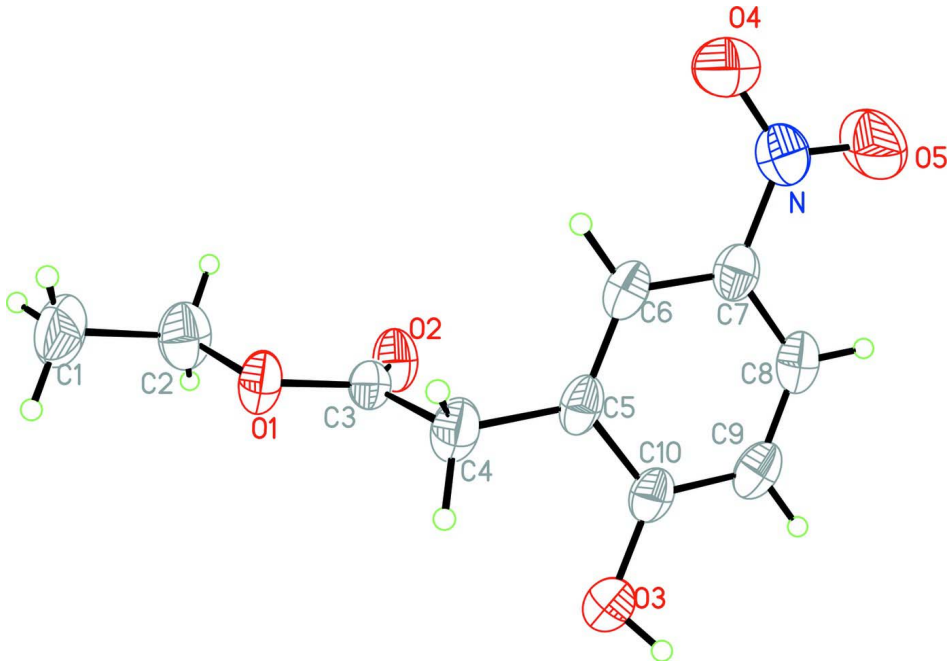


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

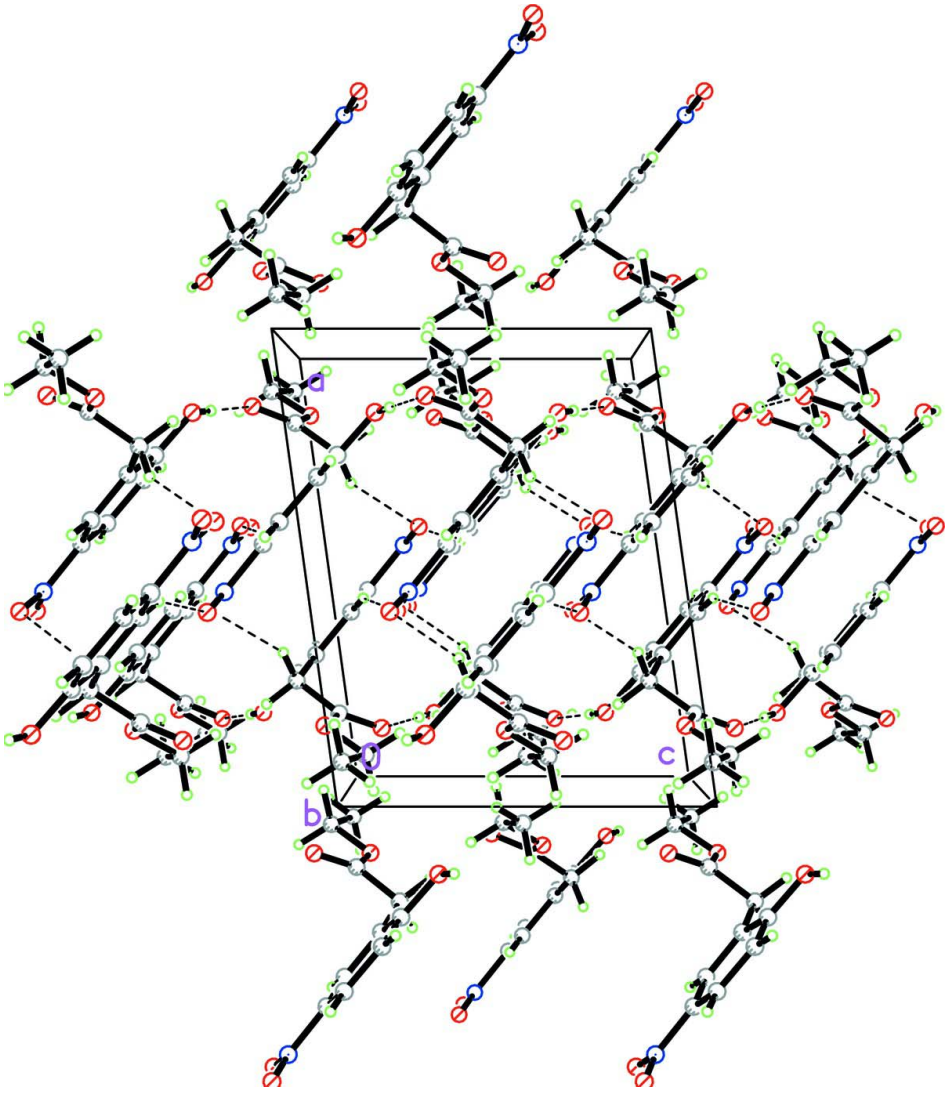


Figure 2

A packing diagram of (I). Hydrogen bond is shown as dashed line.

Ethyl 2-(2-hydroxy-5-nitrophenyl)acetate

Crystal data

$C_{10}H_{11}NO_5$
$M_r = 225.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.066$ (2) Å
$b = 10.860$ (2) Å
$c = 8.6970$ (17) Å
$\beta = 97.85$ (3)°
$V = 1035.4$ (4) Å^3
$Z = 4$
$F(000) = 472$
$D_x = 1.445$ Mg m^{-3}
Melting point: 423 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–12°
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Block, yellow
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.988
3894 measured reflections
1905 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 25.4°, θ_{min} = 1.9°
h = 0→13
k = −13→13
l = −10→10
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.153
S = 1.00
1905 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.099P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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.

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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N	0.44757 (17)	0.77034 (17)	−0.2435 (2)	0.0459 (5)
O1	0.84749 (14)	0.35380 (12)	0.03010 (16)	0.0410 (4)
C1	0.9150 (3)	0.1519 (2)	−0.0185 (3)	0.0683 (9)
H1A	0.9544	0.1015	−0.0874	0.102*
H1B	0.8344	0.1209	−0.0133	0.102*
H1C	0.9614	0.1503	0.0832	0.102*
O2	0.86074 (14)	0.51755 (12)	−0.11869 (19)	0.0463 (4)
C2	0.9070 (2)	0.2789 (2)	−0.0764 (3)	0.0514 (6)
H2A	0.9880	0.3106	−0.0830	0.062*
H2B	0.8604	0.2813	−0.1792	0.062*
O3	0.83631 (14)	0.75835 (12)	0.23336 (18)	0.0447 (4)
H3A	0.8435	0.8252	0.2774	0.067*
C3	0.82574 (18)	0.46958 (16)	−0.0074 (2)	0.0328 (5)
O4	0.40609 (16)	0.67242 (16)	−0.2982 (2)	0.0621 (5)
C4	0.7546 (2)	0.53250 (18)	0.1045 (2)	0.0428 (6)

H4A	0.6900	0.4778	0.1272	0.051*
H4B	0.8082	0.5467	0.2008	0.051*
C5	0.69929 (19)	0.65258 (17)	0.0481 (2)	0.0344 (5)
O5	0.40459 (18)	0.86915 (17)	−0.2870 (2)	0.0748 (7)
C6	0.60137 (19)	0.65579 (17)	−0.0694 (2)	0.0357 (5)
H6A	0.5693	0.5828	−0.1137	0.043*
C7	0.55121 (18)	0.76775 (18)	−0.1209 (2)	0.0360 (5)
C8	0.5979 (2)	0.87793 (19)	−0.0587 (3)	0.0396 (5)
H8A	0.5650	0.9525	−0.0968	0.048*
C9	0.6931 (2)	0.87561 (18)	0.0595 (2)	0.0391 (5)
H9A	0.7243	0.9491	0.1032	0.047*
C10	0.74381 (18)	0.76365 (17)	0.1149 (2)	0.0332 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0430 (11)	0.0481 (11)	0.0477 (12)	0.0049 (9)	0.0104 (9)	0.0045 (9)
O1	0.0551 (10)	0.0265 (7)	0.0436 (9)	0.0072 (6)	0.0143 (7)	0.0031 (6)
C1	0.100 (2)	0.0364 (13)	0.0666 (18)	0.0172 (13)	0.0056 (16)	−0.0105 (12)
O2	0.0520 (9)	0.0380 (8)	0.0525 (10)	0.0080 (7)	0.0201 (7)	0.0112 (7)
C2	0.0547 (15)	0.0494 (13)	0.0546 (15)	0.0148 (11)	0.0232 (12)	−0.0012 (11)
O3	0.0476 (9)	0.0418 (9)	0.0440 (9)	0.0038 (7)	0.0040 (7)	−0.0097 (7)
C3	0.0372 (11)	0.0253 (9)	0.0353 (11)	0.0013 (8)	0.0026 (8)	0.0022 (8)
O4	0.0566 (11)	0.0569 (11)	0.0692 (12)	−0.0035 (9)	−0.0048 (9)	−0.0059 (9)
C4	0.0644 (15)	0.0314 (11)	0.0344 (11)	0.0096 (10)	0.0134 (10)	0.0023 (8)
C5	0.0467 (12)	0.0283 (10)	0.0318 (10)	0.0064 (8)	0.0181 (9)	−0.0006 (8)
O5	0.0697 (13)	0.0553 (11)	0.0918 (15)	0.0148 (9)	−0.0167 (11)	0.0160 (10)
C6	0.0454 (12)	0.0273 (10)	0.0371 (11)	0.0010 (8)	0.0162 (9)	−0.0039 (8)
C7	0.0378 (11)	0.0347 (10)	0.0381 (12)	0.0027 (8)	0.0149 (9)	−0.0021 (8)
C8	0.0487 (13)	0.0310 (10)	0.0424 (12)	0.0093 (9)	0.0178 (10)	0.0040 (9)
C9	0.0527 (13)	0.0268 (10)	0.0407 (12)	−0.0027 (9)	0.0169 (10)	−0.0037 (8)
C10	0.0372 (11)	0.0352 (10)	0.0307 (10)	0.0036 (8)	0.0171 (9)	−0.0032 (8)

Geometric parameters (Å, º)

N—O5	1.213 (2)	C3—C4	1.498 (3)
N—O4	1.228 (2)	C4—C5	1.495 (3)
N—C7	1.455 (3)	C4—H4A	0.9700
O1—C3	1.313 (2)	C4—H4B	0.9700
O1—C2	1.456 (2)	C5—C6	1.385 (3)
C1—C2	1.466 (3)	C5—C10	1.399 (3)
C1—H1A	0.9600	C6—C7	1.385 (3)
C1—H1B	0.9600	C6—H6A	0.9300
C1—H1C	0.9600	C7—C8	1.384 (3)
O2—C3	1.208 (2)	C8—C9	1.369 (3)
C2—H2A	0.9700	C8—H8A	0.9300
C2—H2B	0.9700	C9—C10	1.397 (3)
O3—C10	1.351 (3)	C9—H9A	0.9300

O3—H3A	0.8200		
O5—N—O4	122.4 (2)	C3—C4—H4A	108.7
O5—N—C7	118.79 (18)	C5—C4—H4B	108.7
O4—N—C7	118.85 (17)	C3—C4—H4B	108.7
C3—O1—C2	117.42 (16)	H4A—C4—H4B	107.6
C2—C1—H1A	109.5	C6—C5—C10	118.72 (17)
C2—C1—H1B	109.5	C6—C5—C4	120.57 (18)
H1A—C1—H1B	109.5	C10—C5—C4	120.70 (19)
C2—C1—H1C	109.5	C5—C6—C7	119.96 (18)
H1A—C1—H1C	109.5	C5—C6—H6A	120.0
H1B—C1—H1C	109.5	C7—C6—H6A	120.0
O1—C2—C1	108.58 (19)	C8—C7—C6	121.4 (2)
O1—C2—H2A	110.0	C8—C7—N	118.96 (18)
C1—C2—H2A	110.0	C6—C7—N	119.64 (18)
O1—C2—H2B	110.0	C9—C8—C7	119.06 (18)
C1—C2—H2B	110.0	C9—C8—H8A	120.5
H2A—C2—H2B	108.4	C7—C8—H8A	120.5
C10—O3—H3A	109.5	C8—C9—C10	120.47 (18)
O2—C3—O1	122.96 (19)	C8—C9—H9A	119.8
O2—C3—C4	125.33 (17)	C10—C9—H9A	119.8
O1—C3—C4	111.71 (17)	O3—C10—C9	121.81 (18)
C5—C4—C3	114.36 (17)	O3—C10—C5	117.85 (17)
C5—C4—H4A	108.7	C9—C10—C5	120.34 (19)
C3—O1—C2—C1	−176.2 (2)	O4—N—C7—C8	−179.8 (2)
C2—O1—C3—O2	−5.6 (3)	O5—N—C7—C6	179.8 (2)
C2—O1—C3—C4	174.94 (19)	O4—N—C7—C6	0.5 (3)
O2—C3—C4—C5	15.6 (3)	C6—C7—C8—C9	−2.1 (3)
O1—C3—C4—C5	−164.98 (18)	N—C7—C8—C9	178.19 (18)
C3—C4—C5—C6	71.4 (3)	C7—C8—C9—C10	1.0 (3)
C3—C4—C5—C10	−109.9 (2)	C8—C9—C10—O3	−178.73 (19)
C10—C5—C6—C7	1.4 (3)	C8—C9—C10—C5	1.3 (3)
C4—C5—C6—C7	−179.79 (18)	C6—C5—C10—O3	177.52 (17)
C5—C6—C7—C8	0.9 (3)	C4—C5—C10—O3	−1.3 (3)
C5—C6—C7—N	−179.43 (17)	C6—C5—C10—C9	−2.5 (3)
O5—N—C7—C8	−0.4 (3)	C4—C5—C10—C9	178.75 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2^{i}	0.82	1.93	2.749 (2)	180
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Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) −*x*+2, −*y*+1, −*z*; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+1, *y*−1/2, −*z*−1/2; (v) −*x*+1, *y*+1/2, −*z*−1/2.