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2-(2-Ethoxy-2-oxoacetamido)benzoic acid

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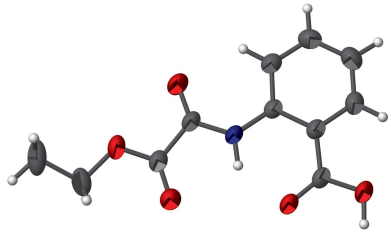
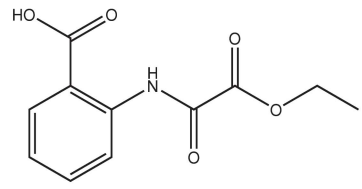
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{11}H_{11}NO_5$, has a nearly planar geometry. In the crystal, the molecules are assembled into chains parallel to the [$\bar{1}$11] direction by O—H···O and C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Oxamide derivatives are of considerable current interest because of their DNA-binding properties and cytotoxic activity (Martínez-Martínez *et al.*, 1998; Li *et al.*, 2012; Yue *et al.*, 2012; Zheng *et al.*, 2012). As part of our studies in this area, we report the structure of the title compound herein.

The asymmetric unit contains one title compound adopting the expected *transoid* conformation (Fig. 1). The molecule has an almost planar geometry except for the terminal methyl group; atom C11 deviates by 0.388 (3) Å from the mean plane of other atoms. Intramolecular N—H···O and C—H···O hydrogen bonds occur (Table 1).

In crystal, pairs of molecules are linked by O—H···O hydrogen bonds (Table 1) into inversion dimers characterized by an $R_2^2(8)$ motif (Fig. 2). The dimers are linked by further C—H···O hydrogen bonds with an $R_2^2(12)$ motif, giving rise to a chain extending along the [$\bar{1}$11] direction.

Synthesis and crystallization

The title compound was synthesized using a literature method (Matović, 2005). Ethyl chlorooxoacetate (10 mmol, 1.13 ml) in 10 ml of tetrahydrofuran (THF) was added dropwise to a 10 ml THF solution containing 2-aminobenzoic acid (10 mmol, 1.37 g) at 273 K. The resulting solution was stirred for 2 h. The solution was concentrated under vacuum and the compound was precipitated as a yellowish powder then washed with ether and dried under vacuum. Well-shaped yellowish single crystals were obtained by slow evaporation of an ethanol solution of the recrystallized product. Yield: 55%.

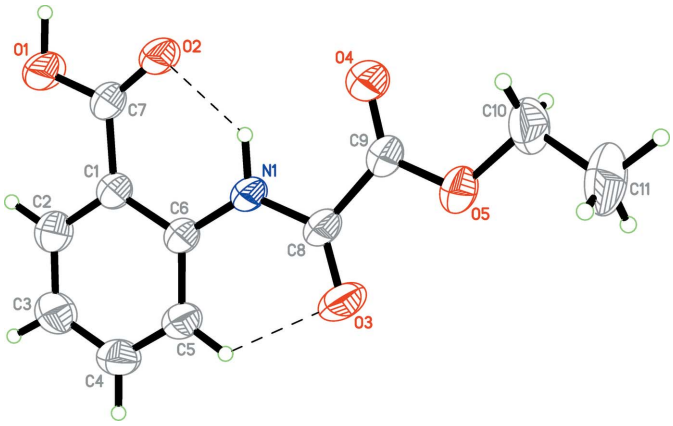


Figure 1
Molecular structure of the title compound with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds (Table 1) are indicated by dashed lines.

Analysis calculated for $C_{11}H_{11}NO_5$: C, 55.70; H, 4.67; N, 5.90%. Found: C, 55.77; H, 4.64; N, 5.92%.

Refinement

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

Funding information

This work was supported by Shandong Province Key Research and Development Project (2018GSF118174), Qingdao People's Livelihood Science and Technology Project (18–6-1–95-nsh), and the NSFC–Shandong Joint Fund for Marine Science Research Centers (grant No. U1606403).

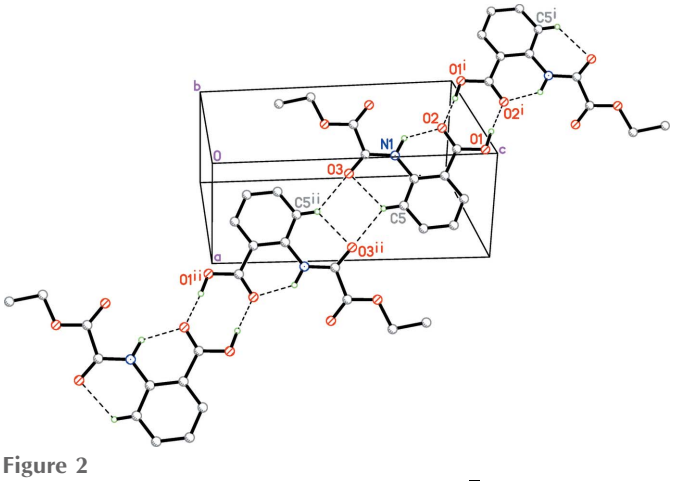


Figure 2
A one-dimensional hydrogen-bonded chain along [$\bar{1}$11]. Hydrogen bonds (Table 1) are indicated by dashed lines. H atoms not involved in hydrogen bonding are omitted for clarity. Symmetry codes: (i) $-x, 1-y, 2-z$; (ii) $1-x, -y, 1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.96 (3)	1.67 (3)	2.6274 (16)	176 (2)
N1—H1A···O2	0.93 (2)	1.92 (2)	2.6574 (15)	135.1 (16)
C5—H5···O3	0.948 (19)	2.230 (18)	2.866 (2)	123.7 (14)
C5—H5···O3^{ii}	0.948 (19)	2.36 (2)	3.1107 (19)	135.9 (14)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{11}NO_5$
M_r	237.21
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	296
a, b, c (Å)	4.8774 (13), 9.470 (3), 12.719 (3)
α, β, γ (°)	106.784 (7), 97.222 (7), 92.444 (8)
V (Å^3)	556.1 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.73 × 0.24 × 0.19
Data collection	
Diffractometer	Bruker *APEX* area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2002)
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8969, 2557, 2016
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.653
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.138, 1.06
No. of reflections	2557
No. of parameters	198
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.27

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *XP* in *SHELXTL* and *SHELXS97* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

References

Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, X.-W., Li, X., Li, Y.-T., Wu, Z.-Y. & Yan, C.-W. (2012). *J. Organomet. Chem.* **700**, 48–57.
Martínez-Martínez, F. J., Padilla-Martínez, I. I., Brito, M. A., Geniz, E. D., Rojas, R. C., Saavedra, J. B. R., Höpfl, H., Tlahuextl, M. & Contreras, R. (1998). *J. Chem. Soc. Perkin Trans. 2*, pp. 401–406.
Matović, Z. D. V. D., Miletić, V. D., Samardžić, G., Pelosi, G., Ianelli, S. & Trifunović, S. (2005). *Inorg. Chim. Acta*, **358**, 3135–3144.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Yue, X.-T., Li, X.-W. & Wu, Z.-Y. (2012). *Acta Cryst.* E**68**, o8.
Zheng, Y.-J., Zheng, K., Wu, Z.-Y. & Li, Y. (2012). *Acta Cryst.* E**68**, o895.

full crystallographic data

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Crystal data

$C_{11}H_{11}NO_5$
$M_r = 237.21$
Triclinic, $P\overline{1}$
$a = 4.8774$ (13) Å
$b = 9.470$ (3) Å
$c = 12.719$ (3) Å
$\alpha = 106.784$ (7)°
$\beta = 97.222$ (7)°
$\gamma = 92.444$ (8)°
$V = 556.1$ (3) Å^3
$Z = 2$
$F(000) = 248$
$D_x = 1.417$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4983 reflections
$\theta = 2.4$–27.7°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Plate, light yellow
0.73 × 0.24 × 0.19 mm

Data collection

Bruker APEX area detector diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)

8969 measured reflections

2557 independent reflections
2016 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.7°$, $\theta_{min} = 3.2°$
$h = -6 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.138$
$S = 1.06$
2557 reflections
198 parameters
0 restraints

Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.0917P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ 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. All H atoms were found in a difference Fourier map and refined freely.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2576 (3)	0.38020 (16)	1.03091 (9)	0.0681 (4)
O2	0.0551 (2)	0.41447 (12)	0.87522 (8)	0.0535 (3)
O3	0.2481 (3)	0.12887 (17)	0.50560 (9)	0.0830 (5)
O4	−0.2254 (3)	0.38059 (15)	0.60001 (9)	0.0660 (4)
O5	−0.1328 (2)	0.27460 (13)	0.42790 (8)	0.0557 (3)
N1	0.1791 (2)	0.24475 (13)	0.68396 (9)	0.0422 (3)
C1	0.3864 (3)	0.23438 (16)	0.86482 (11)	0.0420 (3)
C2	0.5692 (3)	0.1711 (2)	0.92768 (13)	0.0551 (4)
C3	0.7340 (4)	0.0645 (2)	0.87854 (15)	0.0602 (4)
C4	0.7185 (3)	0.01962 (18)	0.76469 (14)	0.0533 (4)
C5	0.5376 (3)	0.07796 (17)	0.70010 (12)	0.0463 (3)
C6	0.3673 (3)	0.18576 (15)	0.74833 (10)	0.0385 (3)
C7	0.2187 (3)	0.35013 (17)	0.92252 (11)	0.0456 (3)
C8	0.1306 (3)	0.21176 (16)	0.57239 (10)	0.0451 (3)
C9	−0.1000 (3)	0.29967 (16)	0.53589 (11)	0.0446 (3)
C10	−0.3469 (4)	0.3514 (2)	0.38191 (15)	0.0622 (5)
C11	−0.3098 (6)	0.3297 (3)	0.26382 (17)	0.0828 (7)
H1	0.139 (5)	0.455 (3)	1.061 (2)	0.099 (8)*
H1A	0.065 (4)	0.311 (2)	0.7222 (17)	0.070 (6)*
H2	0.581 (4)	0.206 (2)	1.0049 (19)	0.078 (6)*
H3	0.856 (4)	0.025 (2)	0.9243 (18)	0.081 (6)*
H4	0.836 (4)	−0.0499 (19)	0.7304 (16)	0.058 (5)*
H5	0.532 (4)	0.046 (2)	0.6220 (16)	0.054 (4)*
H10A	−0.321 (4)	0.452 (2)	0.4258 (18)	0.077 (6)*
H10B	−0.517 (4)	0.307 (2)	0.3905 (17)	0.075 (6)*
H11A	−0.121 (6)	0.375 (3)	0.260 (2)	0.114 (9)*
H11B	−0.326 (5)	0.229 (3)	0.224 (2)	0.109 (9)*
H11C	−0.457 (5)	0.377 (3)	0.230 (2)	0.096 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0815 (8)	0.0954 (9)	0.0244 (5)	0.0438 (7)	0.0077 (5)	0.0081 (5)
O2	0.0659 (7)	0.0677 (7)	0.0261 (5)	0.0287 (5)	0.0102 (4)	0.0077 (4)
O3	0.1083 (10)	0.1073 (10)	0.0320 (6)	0.0657 (9)	0.0163 (6)	0.0079 (6)
O4	0.0726 (8)	0.0869 (9)	0.0377 (6)	0.0387 (6)	0.0092 (5)	0.0122 (6)
O5	0.0683 (7)	0.0699 (7)	0.0289 (5)	0.0181 (5)	0.0034 (5)	0.0144 (5)
N1	0.0479 (6)	0.0527 (7)	0.0251 (5)	0.0165 (5)	0.0087 (4)	0.0070 (5)
C1	0.0421 (7)	0.0520 (8)	0.0306 (7)	0.0093 (6)	0.0066 (5)	0.0089 (6)
C2	0.0592 (9)	0.0720 (10)	0.0338 (7)	0.0177 (7)	0.0036 (6)	0.0150 (7)
C3	0.0601 (10)	0.0710 (10)	0.0518 (9)	0.0246 (8)	0.0020 (7)	0.0218 (8)
C4	0.0539 (9)	0.0549 (9)	0.0525 (9)	0.0201 (7)	0.0124 (7)	0.0136 (7)
C5	0.0509 (8)	0.0510 (8)	0.0364 (7)	0.0127 (6)	0.0110 (6)	0.0089 (6)
C6	0.0401 (6)	0.0452 (7)	0.0299 (6)	0.0072 (5)	0.0073 (5)	0.0093 (5)
C7	0.0493 (8)	0.0588 (8)	0.0261 (6)	0.0117 (6)	0.0062 (5)	0.0072 (6)

C8	0.0543 (8)	0.0521 (8)	0.0269 (6)	0.0143 (6)	0.0090 (5)	0.0060 (5)
C9	0.0510 (8)	0.0505 (8)	0.0294 (6)	0.0078 (6)	0.0043 (5)	0.0076 (6)
C10	0.0689 (11)	0.0728 (12)	0.0470 (9)	0.0097 (9)	−0.0051 (8)	0.0260 (8)
C11	0.1075 (18)	0.0977 (17)	0.0459 (10)	0.0012 (14)	−0.0099 (10)	0.0354 (11)

Geometric parameters (Å, º)

O1—C7	1.3116 (17)	C2—H2	0.94 (2)
O1—H1	0.96 (3)	C3—C4	1.377 (2)
O2—C7	1.2258 (17)	C3—H3	0.94 (2)
O3—C8	1.2007 (18)	C4—C5	1.371 (2)
O4—C9	1.1963 (18)	C4—H4	0.939 (18)
O5—C9	1.3122 (16)	C5—C6	1.3955 (19)
O5—C10	1.456 (2)	C5—H5	0.948 (19)
N1—C8	1.3493 (17)	C8—C9	1.532 (2)
N1—C6	1.3944 (18)	C10—C11	1.491 (3)
N1—H1A	0.93 (2)	C10—H10A	0.95 (2)
C1—C2	1.392 (2)	C10—H10B	0.95 (2)
C1—C6	1.4084 (18)	C11—H11A	1.01 (3)
C1—C7	1.474 (2)	C11—H11B	0.93 (3)
C2—C3	1.374 (2)	C11—H11C	0.98 (3)
C7—O1—H1	108.9 (15)	C5—C6—C1	118.68 (13)
C9—O5—C10	116.37 (13)	O2—C7—O1	121.64 (13)
C8—N1—C6	128.24 (12)	O2—C7—C1	124.02 (12)
C8—N1—H1A	115.5 (12)	O1—C7—C1	114.35 (12)
C6—N1—H1A	116.1 (12)	O3—C8—N1	127.84 (14)
C2—C1—C6	119.03 (13)	O3—C8—C9	121.08 (13)
C2—C1—C7	118.80 (12)	N1—C8—C9	111.06 (11)
C6—C1—C7	122.16 (12)	O4—C9—O5	126.54 (14)
C3—C2—C1	121.37 (14)	O4—C9—C8	122.79 (12)
C3—C2—H2	121.1 (13)	O5—C9—C8	110.66 (12)
C1—C2—H2	117.5 (13)	O5—C10—C11	106.64 (18)
C2—C3—C4	119.29 (15)	O5—C10—H10A	106.7 (13)
C2—C3—H3	118.4 (13)	C11—C10—H10A	113.3 (13)
C4—C3—H3	122.3 (13)	O5—C10—H10B	105.1 (12)
C5—C4—C3	120.89 (14)	C11—C10—H10B	113.6 (13)
C5—C4—H4	119.0 (11)	H10A—C10—H10B	110.7 (18)
C3—C4—H4	120.1 (11)	C10—C11—H11A	109.9 (15)
C4—C5—C6	120.72 (14)	C10—C11—H11B	111.3 (16)
C4—C5—H5	119.4 (11)	H11A—C11—H11B	109 (2)
C6—C5—H5	119.9 (11)	C10—C11—H11C	108.4 (15)
N1—C6—C5	121.55 (12)	H11A—C11—H11C	110 (2)
N1—C6—C1	119.77 (11)	H11B—C11—H11C	108 (2)
C6—C1—C2—C3	1.2 (2)	C2—C1—C7—O2	178.82 (14)
C7—C1—C2—C3	−178.93 (16)	C6—C1—C7—O2	−1.3 (2)
C1—C2—C3—C4	0.2 (3)	C2—C1—C7—O1	−1.2 (2)

C2—C3—C4—C5	−1.2 (3)	C6—C1—C7—O1	178.69 (13)
C3—C4—C5—C6	0.8 (3)	C6—N1—C8—O3	−3.3 (3)
C8—N1—C6—C5	−0.1 (2)	C6—N1—C8—C9	178.43 (13)
C8—N1—C6—C1	−179.63 (13)	C10—O5—C9—O4	−1.1 (2)
C4—C5—C6—N1	−178.99 (14)	C10—O5—C9—C8	179.88 (14)
C4—C5—C6—C1	0.5 (2)	O3—C8—C9—O4	178.26 (18)
C2—C1—C6—N1	178.03 (13)	N1—C8—C9—O4	−3.3 (2)
C7—C1—C6—N1	−1.9 (2)	O3—C8—C9—O5	−2.7 (2)
C2—C1—C6—C5	−1.5 (2)	N1—C8—C9—O5	175.70 (12)
C7—C1—C6—C5	178.62 (13)	C9—O5—C10—C11	169.35 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.96 (3)	1.67 (3)	2.6274 (16)	176 (2)
N1—H1*A*···O2	0.93 (2)	1.92 (2)	2.6574 (15)	135.1 (16)
C5—H5···O3	0.948 (19)	2.230 (18)	2.866 (2)	123.7 (14)
C5—H5···O3^{ii}	0.948 (19)	2.36 (2)	3.1107 (19)	135.9 (14)

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