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2-Oxo-3,4-dihydro-1,4-benzoxazine-4-acetic acid

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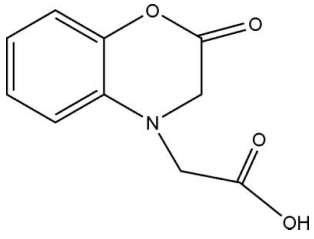
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.053; wR factor = 0.171; data-to-parameter ratio = 12.0.

In the title compound, $C_{10}H_9NO_4$, the 2-oxo-3,4-dihydro-1,4-benzoxazine unit is not planar. Adjacent molecules are linked together to form a two-dimensional supramolecular structure through C—H···O and O—H···O hydrogen bonding.

Related literature

For general background, see: Yagai (2006); Desiraju (1995). For a related structure, see: Desiraju (2003).



Experimental

Crystal data

$C_{10}H_9NO_4$
M_r = 207.18
Monoclinic, $C2/c$
a = 21.782 (3) Å
b = 9.743 (2) Å
c = 9.271 (2) Å
β = 108.178 (2)°
V = 1869.2 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.56 × 0.49 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
4535 measured reflections
1632 independent reflections
887 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.171
S = 1.00
1632 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O3^{i}	0.82	1.87	2.692 (3)	180
C2—H2B···O1^{ii}	0.97	2.55	3.416 (4)	148
C4—H4B···O2^{ii}	0.97	2.42	3.282 (4)	148

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2260).

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supplementary materials

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2-Oxo-3,4-dihydro-1,4-benzoxazine-4-acetic acid

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Comment

Crystal engineering has been widely applied in the design of functional materials and coordination polymers (Yagai, 2006). Because of the various types and a number of supramolecular architectures, hydrogen bonds are widely used in crystal engineering (Desiraju, 2003). Carboxylic acids have strong and directional hydrogen bonds so that they are greatly applied in crystal engineering (Desiraju, 1995). We now report one of carboxylic acids compound, which has two-dimensional network connected through intermolecular hydrogen bonding.

The crystal structure of title compound is shown in Fig. 1. The compound is made up of a benzene ring, a lactonic ring and a acetic acid, which bonds to the N atom of the lactonic ring. The planes of the benzene and lactonic ring are tilted at an angle of 171.4°.

In the crystal structure, the adjacent molecules are connected together to form one-dimensional chain along *b* axis through C2—H2B···O1^{ii} and C4—H4B···O2^{ii} [symmetry code: (ii) 0.5 − *x*,1/2 + *y*,-0.5 − *z*] interactions. The O4—H4···O3 intermolecular H-bonds link the adjacent molecules to form eight-membered rings ($R_2^2(8)$), which join the adjacent chains together to form two-dimensional network (Fig.2).

Experimental

The *o*-amino-phenol (1.09 g, 10 mmol), monochloroacetic acid (1.89 g, 20 mmol) and anhydrous sodium carbonate (2.12 g, 20 mmol) were mixed in water (50 ml). The mixture was refluxed for 3 h and then was adjusted to pH = 1 with hydrochloric acid (6 mol/l). The resulting white precipitate was separeted and dissolved with ethanol. The ethanol solution was slowly evaporization at room temperature. The colourless crystals were obtained after one week.

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms, with C—H = 0.93 (aromatic), 0.97 (methylene) and 0.82 Å (hydroxyl). The U_{iso}(H) values were set at 1.2U_{eq}(C) for all C-bound H atoms, 1.5U_{eq}(0) for O-bound H atoms.

Figures

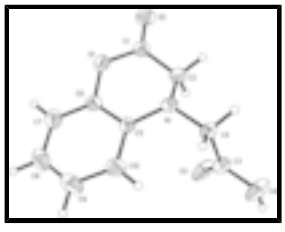

Fig. 1. The molecular structure of the title compound with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

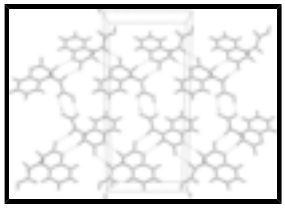

Fig. 2. The two-dimensional net formed by hydrogen-bonding [symmetry codes: (i) −*x* + 1, −*y* + 1, −*z*, (ii) 0.5 − *x*,1/2 + *y*,-0.5 − *z*].

2-Oxo-3,4-dihydro-1,4-benzoxazine-4-acetic acid

Crystal data

$C_{10}H_9NO_4$	F_{000} = 864
M_r = 207.18	D_x = 1.472 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 878 reflections
a = 21.782 (3) Å	θ = 3.0–25.7º
b = 9.743 (2) Å	μ = 0.12 mm^{-1}
c = 9.271 (2) Å	*T* = 298 (2) K
β = 108.178 (2)º	Block, colourless
V = 1869.2 (6) Å^3	0.56 × 0.49 × 0.20 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	887 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.069
Monochromator: graphite	θ_{max} = 25.0º
T = 298(2) K	θ_{min} = 2.0º
φ and ω scans	*h* = −23→25
Absorption correction: none	*k* = −11→11
4535 measured reflections	*l* = −10→11
1632 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.053	H-atom parameters constrained
$wR(F^2)$ = 0.171	$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max} < 0.001$
1632 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
136 parameters	$\Delta\rho_{min}$ = −0.24 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.

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 $F^2 > 2\text{sigma}(F^2)$ 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}
N1	0.32873 (10)	0.2976 (3)	0.0161 (3)	0.0388 (7)
O1	0.28240 (10)	0.0464 (2)	−0.1180 (2)	0.0483 (6)
O2	0.21904 (11)	0.1524 (2)	−0.3167 (3)	0.0583 (7)
O3	0.44418 (11)	0.3817 (3)	−0.0316 (3)	0.0690 (8)
O4	0.44513 (11)	0.5747 (3)	0.0959 (3)	0.0738 (9)
H4	0.4789	0.5876	0.0762	0.111*
C1	0.26313 (15)	0.1613 (4)	−0.2008 (4)	0.0432 (8)
C2	0.29897 (14)	0.2909 (4)	−0.1473 (3)	0.0471 (9)
H2A	0.3323	0.3009	−0.1956	0.057*
H2B	0.2694	0.3675	−0.1793	0.057*
C3	0.42010 (15)	0.4568 (4)	0.0387 (4)	0.0451 (9)
C4	0.35799 (13)	0.4265 (3)	0.0716 (3)	0.0421 (8)
H4A	0.3664	0.4292	0.1806	0.051*
H4B	0.3273	0.4989	0.0275	0.051*
C5	0.35776 (13)	0.1763 (3)	0.0847 (3)	0.0364 (8)
C6	0.33470 (13)	0.0503 (4)	0.0168 (3)	0.0405 (8)
C7	0.35902 (16)	−0.0716 (4)	0.0774 (4)	0.0546 (10)
H7	0.3425	−0.1528	0.0274	0.066*
C8	0.40803 (19)	−0.0760 (5)	0.2126 (5)	0.0639 (11)
H8	0.4257	−0.1594	0.2541	0.077*
C9	0.43049 (17)	0.0447 (5)	0.2854 (4)	0.0646 (12)
H9	0.4624	0.0423	0.3793	0.078*
C10	0.40682 (15)	0.1697 (4)	0.2226 (4)	0.0515 (10)
H10	0.4239	0.2503	0.2730	0.062*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0322 (13)	0.0429 (18)	0.0430 (15)	−0.0036 (12)	0.0140 (11)	−0.0002 (12)
O1	0.0438 (13)	0.0383 (15)	0.0635 (15)	−0.0034 (10)	0.0179 (12)	−0.0015 (12)
O2	0.0514 (14)	0.0624 (19)	0.0551 (15)	−0.0046 (12)	0.0081 (12)	−0.0097 (12)

O3	0.0620 (16)	0.072 (2)	0.091 (2)	−0.0323 (14)	0.0506 (15)	−0.0346 (15)
O4	0.0589 (16)	0.072 (2)	0.103 (2)	−0.0343 (13)	0.0433 (15)	−0.0387 (15)
C1	0.0378 (18)	0.044 (2)	0.051 (2)	0.0023 (15)	0.0188 (16)	−0.0058 (17)
C2	0.0428 (19)	0.051 (2)	0.046 (2)	−0.0036 (16)	0.0106 (15)	0.0031 (16)
C3	0.0396 (18)	0.049 (2)	0.0473 (19)	−0.0109 (16)	0.0149 (16)	−0.0048 (17)
C4	0.0391 (17)	0.040 (2)	0.0482 (19)	−0.0076 (15)	0.0158 (15)	−0.0048 (15)
C5	0.0312 (16)	0.037 (2)	0.0481 (19)	0.0005 (14)	0.0222 (14)	0.0042 (15)
C6	0.0291 (16)	0.048 (2)	0.0487 (19)	0.0018 (15)	0.0191 (15)	0.0051 (17)
C7	0.052 (2)	0.046 (3)	0.076 (3)	0.0068 (17)	0.035 (2)	0.0116 (19)
C8	0.057 (2)	0.064 (3)	0.080 (3)	0.020 (2)	0.035 (2)	0.027 (2)
C9	0.048 (2)	0.094 (4)	0.055 (2)	0.021 (2)	0.0195 (18)	0.024 (2)
C10	0.0415 (19)	0.061 (3)	0.054 (2)	−0.0002 (17)	0.0173 (17)	0.0057 (18)

Geometric parameters (Å, °)

N1—C5	1.397 (4)	C4—H4A	0.9700
N1—C4	1.429 (4)	C4—H4B	0.9700
N1—C2	1.451 (4)	C5—C10	1.389 (4)
O1—C1	1.348 (4)	C5—C6	1.399 (4)
O1—C6	1.404 (3)	C6—C7	1.350 (5)
O2—C1	1.200 (3)	C7—C8	1.370 (5)
O3—C3	1.204 (4)	C7—H7	0.9300
O4—C3	1.310 (4)	C8—C9	1.368 (6)
O4—H4	0.8200	C8—H8	0.9300
C1—C2	1.486 (4)	C9—C10	1.379 (5)
C2—H2A	0.9700	C9—H9	0.9300
C2—H2B	0.9700	C10—H10	0.9300
C3—C4	1.506 (4)		
C5—N1—C4	119.5 (2)	C3—C4—H4B	108.4
C5—N1—C2	115.3 (3)	H4A—C4—H4B	107.5
C4—N1—C2	114.8 (3)	C10—C5—N1	124.5 (3)
C1—O1—C6	120.4 (3)	C10—C5—C6	116.0 (3)
C3—O4—H4	109.5	N1—C5—C6	119.3 (3)
O2—C1—O1	118.0 (3)	C7—C6—C5	123.1 (3)
O2—C1—C2	123.4 (3)	C7—C6—O1	116.8 (3)
O1—C1—C2	118.5 (3)	C5—C6—O1	120.1 (3)
N1—C2—C1	113.8 (3)	C6—C7—C8	120.0 (4)
N1—C2—H2A	108.8	C6—C7—H7	120.0
C1—C2—H2A	108.8	C8—C7—H7	120.0
N1—C2—H2B	108.8	C9—C8—C7	118.8 (4)
C1—C2—H2B	108.8	C9—C8—H8	120.6
H2A—C2—H2B	107.7	C7—C8—H8	120.6
O3—C3—O4	123.7 (3)	C8—C9—C10	121.5 (4)
O3—C3—C4	124.1 (3)	C8—C9—H9	119.3
O4—C3—C4	112.1 (3)	C10—C9—H9	119.3
N1—C4—C3	115.3 (3)	C9—C10—C5	120.5 (4)
N1—C4—H4A	108.4	C9—C10—H10	119.7
C3—C4—H4A	108.4	C5—C10—H10	119.7
N1—C4—H4B	108.4		

C6—O1—C1—O2	178.5 (2)	C10—C5—C6—C7	−1.7 (4)
C6—O1—C1—C2	0.8 (4)	N1—C5—C6—C7	−178.4 (3)
C5—N1—C2—C1	41.0 (3)	C10—C5—C6—O1	176.0 (2)
C4—N1—C2—C1	−174.1 (2)	N1—C5—C6—O1	−0.7 (4)
O2—C1—C2—N1	154.2 (3)	C1—O1—C6—C7	−167.6 (3)
O1—C1—C2—N1	−28.2 (4)	C1—O1—C6—C5	14.6 (4)
C5—N1—C4—C3	70.7 (3)	C5—C6—C7—C8	0.9 (5)
C2—N1—C4—C3	−72.7 (3)	O1—C6—C7—C8	−176.9 (3)
O3—C3—C4—N1	1.5 (5)	C6—C7—C8—C9	1.4 (5)
O4—C3—C4—N1	−178.4 (3)	C7—C8—C9—C10	−2.8 (5)
C4—N1—C5—C10	13.0 (4)	C8—C9—C10—C5	1.9 (5)
C2—N1—C5—C10	156.2 (3)	N1—C5—C10—C9	176.8 (3)
C4—N1—C5—C6	−170.6 (2)	C6—C5—C10—C9	0.3 (4)
C2—N1—C5—C6	−27.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O3^{i}	0.82	1.87	2.692 (3)	180
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Symmetry codes: (i) −*x*+1, −*y*+1, −*z*; (ii) −*x*+1/2, *y*+1/2, −*z*−1/2.

Fig. 1

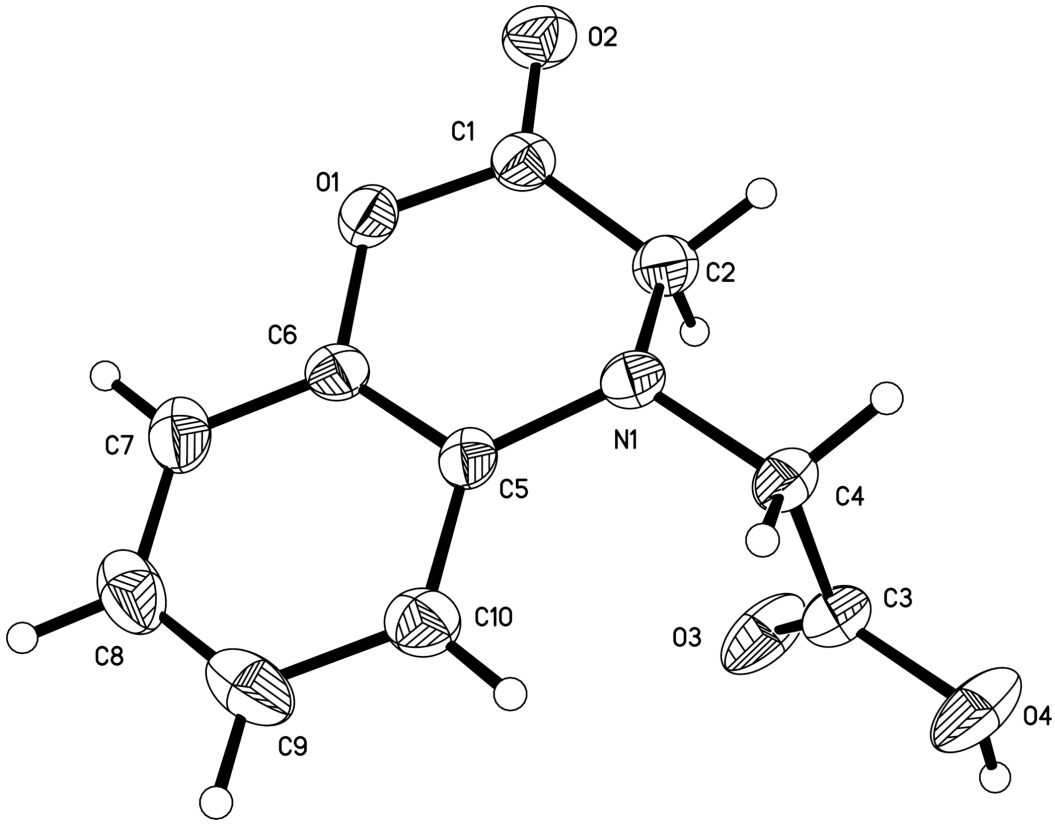

Fig. 2