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Propyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-carboxylate

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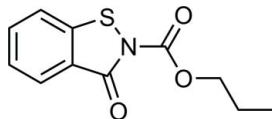
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Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.039; wR factor = 0.099; data-to-parameter ratio = 18.9.

The title compound, $\text{C}_{11}\text{H}_{11}\text{NO}_3\text{S}$, was synthesized by the reaction of benzo[*d*]isothiazol-3(2*H*)-one with propyl carbonochloridate in toluene. The benzoisothiazolone ring system is approximately planar with a maximum deviation from the mean plane of 0.0226 (14) Å for the N atom. Weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding occurs in the crystal structure.

Related literature

For background to the synthesis of benzoisothiazolone derivatives, see: Davis (1972); Elgazwy & Abdel-Sattar (2003). For their biological activity, see: Taubert *et al.* (2002). For related structures, see: Xu *et al.* (2005, 2006); Cavalca *et al.* (1969, 1970).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{11}\text{NO}_3\text{S}$
 $M_r = 237.27$

Monoclinic, $P2_1/c$
 $a = 16.235$ (7) Å

$b = 5.123$ (2) Å
 $c = 12.791$ (6) Å
 $\beta = 90.720$ (7)°
 $V = 1063.7$ (8) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.29$ mm⁻¹
 $T = 153$ K
 $0.35 \times 0.25 \times 0.20$ mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.904$, $T_{\max} = 0.943$

8491 measured reflections
2766 independent reflections
2224 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.099$
 $S = 1.00$
2766 reflections

146 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C}2-\text{H}2\cdots\text{O}2^i$	0.95	2.60	3.437 (3)	148

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

Data collection: *CrystalClear* (Rigaku, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2349).

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supporting information

Acta Cryst. (2011). E67, o2237 [doi:10.1107/S1600536811028613]

Propyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-carboxylate

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S1. Comment

1,2-benzisothiazol-3(2*H*)-ones are a class of compounds with a wide spectrum of biological activities (Davis, 1972), Elgazwy & Abdel-Sattar, 2003). 1, 2-Benzisothiazolone derivatives have been reported to possess high antibacterial and antifungal activity (Taubert *et al.*, 2002). In view of the importance of the 1,2-benzisothiazol-3(2*H*)-ones, the title compound, (I), was synthesized and characterized by X-ray diffraction.

The molecular structure of the title compound is shown in Fig. 1. In the molecule, the benzisothiazolone ring system is approximately planar with a maximum deviation from the mean plane of 0.0226 (14) Å° for the N atom, and the C8—O2—C9—C10 torsion angle is 85.16 (18)°. Weak intermolecular C—H···O hydrogen bonding occurs in the crystal structure (Table 1, Fig. 22)).

S2. Experimental

A toluol solution (20 ml) containing benzo[*d*]isothiazol-3(2*H*)-one (1.51 g, 0.01 mol) was added dropwise to a solution of propyl carbonochloridate (1.22 g, 0.01 mol) in toluol (20 ml) under stirring on an ice-water bath. The reaction mixture was stirred at room temperature for 4.5 h to afford the title compound (1.55 g, yield 65.5%). Single crystals suitable for X-ray measurements were obtained by recrystallization of the title compound from cyclohexane at room temperature.

S3. Refinement

The H atoms were placed at calculated positions and refined in riding mode, with the carrier atom-H distances = 0.95 Å for aryl, 0.99 for methylene, 0.98 Å for the methyl. The *U*_{iso} values were constrained to be 1.5*U*_{eq} of the carrier atom for the methyl H atoms and 1.2*U*_{eq} for the remaining H atoms.

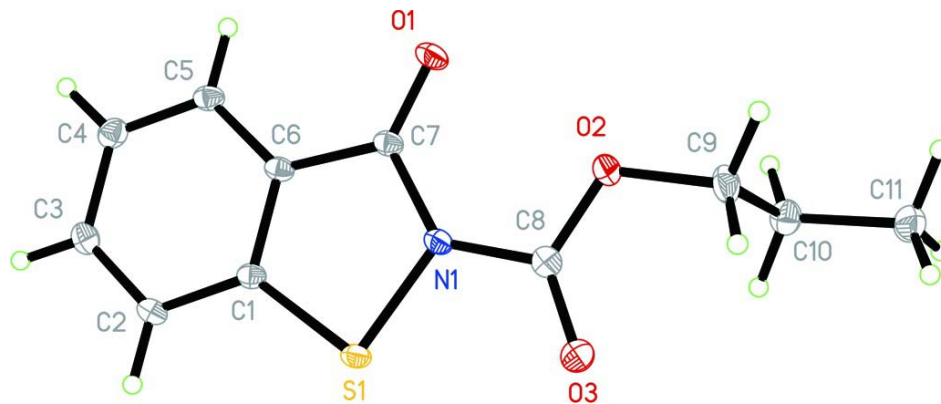
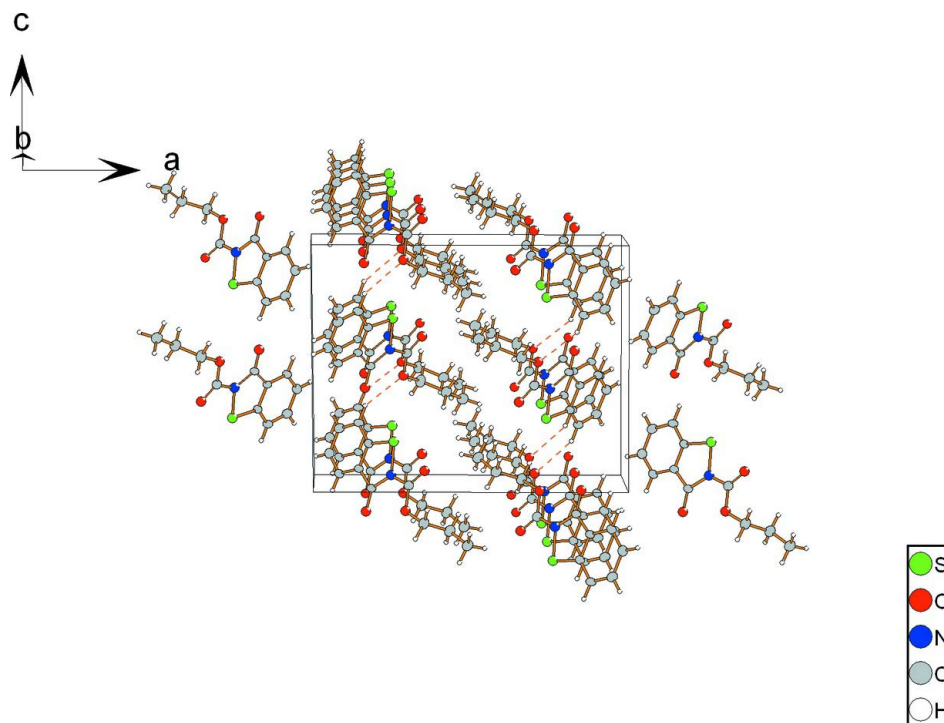


Figure 1

Title molecule showing the 30% probability displacement ellipsoids and the atom numbering scheme.

**Figure 2**

The crystal packing of the title compound, showing a hydrogen-bonded (dashed lines) molecular chain.

Propyl 3-oxo-2,3-dihydro-1,2-benzothiazole-2-carboxylate

Crystal data

$C_{11}H_{11}NO_3S$

$M_r = 237.27$

Monoclinic, $P2_1/c$

$a = 16.235$ (7) Å

$b = 5.123$ (2) Å

$c = 12.791$ (6) Å

$\beta = 90.720$ (7)°

$V = 1063.7$ (8) Å³

$Z = 4$

$F(000) = 496$

$D_x = 1.482$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3253 reflections

$\theta = 3.0$ – 29.1 °

$\mu = 0.29$ mm⁻¹

$T = 153$ K

Block, pink

$0.35 \times 0.25 \times 0.20$ mm

Data collection

Rigaku AFC10/Saturn724+
diffractometer

Radiation source: Rotating Anode

Graphite monochromator

Detector resolution: 28.5714 pixels mm⁻¹

phi and ω scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.904$, $T_{\max} = 0.943$

8491 measured reflections

2766 independent reflections

2224 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 29.1$ °, $\theta_{\min} = 3.2$ °

$h = -20 \rightarrow 22$

$k = -6 \rightarrow 6$

$l = -16 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.099$

$S = 1.00$

2766 reflections

146 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

$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.316P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.35 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.25453 (2)	0.70918 (8)	0.71855 (3)	0.02263 (12)
O1	0.16642 (7)	0.7439 (2)	0.43840 (8)	0.0285 (3)
O2	0.28813 (7)	1.0985 (2)	0.46620 (9)	0.0289 (3)
O3	0.34796 (8)	1.1023 (3)	0.62778 (10)	0.0339 (3)
N1	0.24529 (8)	0.8149 (3)	0.59025 (10)	0.0210 (3)
C1	0.17595 (9)	0.4842 (3)	0.69666 (11)	0.0195 (3)
C2	0.14578 (10)	0.3037 (3)	0.76888 (12)	0.0227 (3)
H2	0.1671	0.2966	0.8383	0.027*
C3	0.08395 (10)	0.1360 (3)	0.73593 (13)	0.0256 (3)
H3	0.0628	0.0116	0.7837	0.031*
C4	0.05157 (10)	0.1449 (3)	0.63348 (13)	0.0258 (3)
H4	0.0088	0.0285	0.6130	0.031*
C5	0.08200 (9)	0.3230 (3)	0.56251 (12)	0.0226 (3)
H5	0.0608	0.3292	0.4930	0.027*
C6	0.14445 (9)	0.4939 (3)	0.59480 (11)	0.0189 (3)
C7	0.18296 (9)	0.6918 (3)	0.52901 (12)	0.0203 (3)
C8	0.29898 (9)	1.0187 (3)	0.56431 (12)	0.0231 (3)
C9	0.34321 (10)	1.3087 (3)	0.43295 (15)	0.0304 (4)
H9A	0.3533	1.4290	0.4923	0.036*
H9B	0.3163	1.4093	0.3760	0.036*
C10	0.42408 (10)	1.2039 (4)	0.39541 (14)	0.0295 (4)
H10A	0.4139	1.0737	0.3395	0.035*
H10B	0.4532	1.1151	0.4539	0.035*
C11	0.47784 (11)	1.4220 (4)	0.35365 (15)	0.0357 (4)
H11A	0.4520	1.4964	0.2907	0.043*

H11B	0.5322	1.3520	0.3364	0.043*
H11C	0.4840	1.5583	0.4070	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0270 (2)	0.0248 (2)	0.01593 (19)	-0.00360 (15)	-0.00484 (14)	0.00027 (15)
O1	0.0359 (7)	0.0350 (7)	0.0145 (5)	-0.0040 (5)	-0.0035 (5)	0.0026 (5)
O2	0.0261 (6)	0.0344 (7)	0.0262 (6)	-0.0044 (5)	-0.0009 (5)	0.0112 (5)
O3	0.0381 (7)	0.0357 (7)	0.0279 (6)	-0.0132 (6)	-0.0037 (5)	-0.0001 (5)
N1	0.0242 (6)	0.0236 (7)	0.0151 (6)	-0.0010 (5)	-0.0010 (5)	0.0009 (5)
C1	0.0213 (7)	0.0193 (7)	0.0178 (7)	0.0021 (5)	-0.0014 (5)	-0.0029 (6)
C2	0.0276 (8)	0.0237 (8)	0.0166 (7)	0.0024 (6)	-0.0013 (6)	0.0017 (6)
C3	0.0299 (8)	0.0225 (8)	0.0245 (8)	0.0000 (6)	0.0042 (6)	0.0010 (6)
C4	0.0248 (8)	0.0254 (8)	0.0270 (8)	-0.0027 (6)	0.0001 (6)	-0.0052 (7)
C5	0.0232 (7)	0.0258 (8)	0.0188 (7)	0.0026 (6)	-0.0025 (6)	-0.0051 (6)
C6	0.0218 (7)	0.0199 (7)	0.0150 (7)	0.0044 (6)	-0.0006 (5)	-0.0030 (6)
C7	0.0226 (7)	0.0227 (8)	0.0156 (7)	0.0024 (6)	-0.0006 (6)	-0.0030 (6)
C8	0.0242 (8)	0.0221 (8)	0.0228 (8)	0.0023 (6)	0.0018 (6)	-0.0005 (6)
C9	0.0274 (8)	0.0260 (9)	0.0378 (10)	0.0003 (7)	0.0037 (7)	0.0136 (7)
C10	0.0300 (9)	0.0278 (9)	0.0308 (9)	-0.0015 (7)	0.0049 (7)	-0.0023 (7)
C11	0.0334 (9)	0.0434 (11)	0.0305 (9)	-0.0095 (8)	0.0042 (7)	0.0008 (8)

Geometric parameters (Å, °)

S1—N1	1.7328 (15)	C4—C5	1.383 (2)
S1—C1	1.7393 (17)	C4—H4	0.9500
O1—C7	1.2162 (19)	C5—C6	1.398 (2)
O2—C8	1.329 (2)	C5—H5	0.9500
O2—C9	1.466 (2)	C6—C7	1.463 (2)
O3—C8	1.208 (2)	C9—C10	1.503 (2)
N1—C8	1.403 (2)	C9—H9A	0.9900
N1—C7	1.4195 (19)	C9—H9B	0.9900
C1—C6	1.395 (2)	C10—C11	1.519 (3)
C1—C2	1.400 (2)	C10—H10A	0.9900
C2—C3	1.383 (2)	C10—H10B	0.9900
C2—H2	0.9500	C11—H11A	0.9800
C3—C4	1.407 (2)	C11—H11B	0.9800
C3—H3	0.9500	C11—H11C	0.9800
N1—S1—C1	90.00 (7)	O1—C7—C6	127.56 (14)
C8—O2—C9	115.18 (13)	N1—C7—C6	107.29 (12)
C8—N1—C7	129.86 (13)	O3—C8—O2	127.15 (15)
C8—N1—S1	114.20 (10)	O3—C8—N1	120.72 (15)
C7—N1—S1	115.87 (11)	O2—C8—N1	112.13 (13)
C6—C1—C2	120.86 (14)	O2—C9—C10	111.65 (14)
C6—C1—S1	112.73 (12)	O2—C9—H9A	109.3
C2—C1—S1	126.40 (12)	C10—C9—H9A	109.3

C3—C2—C1	117.89 (14)	O2—C9—H9B	109.3
C3—C2—H2	121.1	C10—C9—H9B	109.3
C1—C2—H2	121.1	H9A—C9—H9B	108.0
C2—C3—C4	121.68 (15)	C9—C10—C11	110.97 (16)
C2—C3—H3	119.2	C9—C10—H10A	109.4
C4—C3—H3	119.2	C11—C10—H10A	109.4
C5—C4—C3	120.04 (15)	C9—C10—H10B	109.4
C5—C4—H4	120.0	C11—C10—H10B	109.4
C3—C4—H4	120.0	H10A—C10—H10B	108.0
C4—C5—C6	118.88 (15)	C10—C11—H11A	109.5
C4—C5—H5	120.6	C10—C11—H11B	109.5
C6—C5—H5	120.6	H11A—C11—H11B	109.5
C1—C6—C5	120.65 (14)	C10—C11—H11C	109.5
C1—C6—C7	114.07 (13)	H11A—C11—H11C	109.5
C5—C6—C7	125.28 (14)	H11B—C11—H11C	109.5
O1—C7—N1	125.15 (15)		
C1—S1—N1—C8	178.88 (12)	S1—N1—C7—O1	178.47 (13)
C1—S1—N1—C7	1.45 (12)	C8—N1—C7—C6	-179.01 (14)
N1—S1—C1—C6	-0.32 (12)	S1—N1—C7—C6	-2.07 (16)
N1—S1—C1—C2	178.34 (14)	C1—C6—C7—O1	-178.77 (16)
C6—C1—C2—C3	0.0 (2)	C5—C6—C7—O1	1.8 (3)
S1—C1—C2—C3	-178.55 (12)	C1—C6—C7—N1	1.79 (18)
C1—C2—C3—C4	-0.3 (2)	C5—C6—C7—N1	-177.67 (14)
C2—C3—C4—C5	0.6 (2)	C9—O2—C8—O3	0.9 (2)
C3—C4—C5—C6	-0.6 (2)	C9—O2—C8—N1	-179.00 (13)
C2—C1—C6—C5	-0.1 (2)	C7—N1—C8—O3	178.88 (15)
S1—C1—C6—C5	178.67 (12)	S1—N1—C8—O3	1.9 (2)
C2—C1—C6—C7	-179.57 (14)	C7—N1—C8—O2	-1.2 (2)
S1—C1—C6—C7	-0.82 (17)	S1—N1—C8—O2	-178.19 (10)
C4—C5—C6—C1	0.4 (2)	C8—O2—C9—C10	85.16 (18)
C4—C5—C6—C7	179.80 (14)	O2—C9—C10—C11	175.72 (15)
C8—N1—C7—O1	1.5 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots O2 ⁱ	0.95	2.60	3.437 (3)	148

Symmetry code: (i) $x, -y+3/2, z+1/2$.