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

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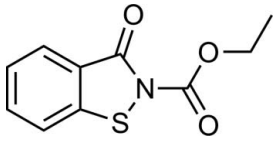
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.101; data-to-parameter ratio = 18.8.

The title compound, $C_{10}H_9NO_3S$, was synthesized by the reaction of benzo[*d*]isothiazol-3(2*H*)-one with ethyl carbonochloridate in toluol. The benzisothiazolone ring system is approximately planar, with a maximum deviation from the mean plane of 0.020 (1) Å for the N atom.

Related literature

For background to the sythesis of benzisothiazolone derivatives, see: Davis (1972); Elgazwy & Abdel-Sattar (2003). For details of 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

$C_{10}H_9NO_3S$
M_r = 223.24
Monoclinic, $P2_1/c$
a = 16.904 (5) Å
b = 4.8912 (13) Å
c = 12.676 (4) Å
β = 110.929 (4)°
V = 979.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 153 K
0.39 × 0.33 × 0.32 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.887, T_{max} = 0.907
8002 measured reflections
2570 independent reflections
2219 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.00
2570 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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 *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2180).

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supplementary materials

Acta Cryst. (2011). E**67**, o2238 [**doi:10.1107/S1600536811029199**]

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

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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 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 0.020 (1) A ° for the N atom, and the C8—O2—C9—C10 torsion angle is -85.9 (2)°.

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 ethyl carbonochloridate (1.08 g, 0.01 mol) in toluol (20 ml) under stirring on an ice-water bath. The reaction mixture was stirred at room temperature for 3.5 h to afford the title compound (1.65 g, yield 72%).. Single crystals suitable for X-ray measurements were obtained by recrystallization of the title compound from cyclohexane at room temperature.

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.

Figures

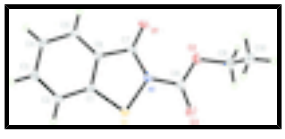

Fig. 1. Title molecule showing the 50% probability displacement ellipsoids and the atom-numbering scheme.

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

Crystal data

$C_{10}H_9NO_3S$	$F(000) = 464$
$M_r = 223.24$	$D_x = 1.515$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 16.904$ (5) Å	Cell parameters from 3063 reflections

b = 4.8912 (13) Å	θ = 3.2–29.1°
c = 12.676 (4) Å	μ = 0.32 mm^{-1}
β = 110.929 (4)°	T = 153 K
V = 979.0 (5) Å^3	Block, colourless
Z = 4	0.39 × 0.33 × 0.32 mm

Data collection

Rigaku AFC10/Saturn724+ diffractometer	2570 independent reflections
Radiation source: Rotating Anode	2219 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.027
Detector resolution: 28.5714 pixels mm^{-1}	θ_{max} = 29.1°, θ_{min} = 3.2°
phi and ω scans	h = −21→23
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	k = −6→6
T_{min} = 0.887, T_{max} = 0.907	l = −13→17
8002 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.038	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.101	H-atom parameters constrained
S = 1.00	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.216P]$ where $P = (F_o^2 + 2F_c^2)/3$
2570 reflections	$(\Delta/\sigma)_{max} < 0.001$
137 parameters	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.23 e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}

S1	0.27279 (2)	0.29920 (8)	0.34210 (3)	0.02398 (12)
O1	0.18146 (7)	0.2458 (2)	0.01861 (9)	0.0304 (3)
O2	0.31201 (7)	−0.1043 (2)	0.10660 (10)	0.0330 (3)
O3	0.37405 (7)	−0.0967 (2)	0.29680 (10)	0.0345 (3)
N1	0.26376 (8)	0.1848 (2)	0.20927 (10)	0.0221 (3)
C1	0.18919 (9)	0.5239 (3)	0.28158 (11)	0.0206 (3)
C2	0.15658 (10)	0.7092 (3)	0.33898 (12)	0.0248 (3)
H2	0.1792	0.7220	0.4190	0.030*
C3	0.09010 (10)	0.8740 (3)	0.27524 (13)	0.0272 (3)
H3	0.0672	1.0030	0.3125	0.033*
C4	0.05574 (10)	0.8556 (3)	0.15727 (13)	0.0275 (3)
H4	0.0095	0.9691	0.1158	0.033*
C5	0.08898 (9)	0.6725 (3)	0.10098 (12)	0.0238 (3)
H5	0.0666	0.6606	0.0209	0.029*
C6	0.15598 (9)	0.5061 (3)	0.16423 (11)	0.0203 (3)
C7	0.19790 (9)	0.3036 (3)	0.11718 (12)	0.0217 (3)
C8	0.32227 (9)	−0.0189 (3)	0.20969 (13)	0.0249 (3)
C9	0.37419 (11)	−0.3059 (4)	0.10007 (17)	0.0422 (5)
H9A	0.3489	−0.4204	0.0319	0.051*
H9B	0.3901	−0.4269	0.1670	0.051*
C10	0.45105 (12)	−0.1664 (5)	0.0952 (2)	0.0548 (6)
H10A	0.4349	−0.0427	0.0301	0.066*
H10B	0.4910	−0.3033	0.0876	0.066*
H10C	0.4778	−0.0613	0.1647	0.066*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0276 (2)	0.02520 (19)	0.01661 (18)	0.00118 (14)	0.00484 (13)	0.00023 (13)
O1	0.0399 (6)	0.0342 (6)	0.0176 (5)	0.0038 (5)	0.0110 (5)	−0.0003 (4)
O2	0.0273 (6)	0.0384 (6)	0.0333 (6)	0.0050 (5)	0.0110 (5)	−0.0109 (5)
O3	0.0350 (6)	0.0345 (6)	0.0318 (6)	0.0075 (5)	0.0092 (5)	0.0049 (5)
N1	0.0247 (6)	0.0243 (6)	0.0174 (6)	0.0004 (5)	0.0078 (5)	−0.0002 (4)
C1	0.0235 (6)	0.0188 (6)	0.0195 (7)	−0.0034 (5)	0.0078 (5)	0.0013 (5)
C2	0.0309 (7)	0.0252 (7)	0.0197 (7)	−0.0036 (6)	0.0109 (6)	−0.0024 (5)
C3	0.0321 (8)	0.0234 (7)	0.0312 (8)	−0.0002 (6)	0.0174 (6)	−0.0003 (6)
C4	0.0275 (7)	0.0267 (7)	0.0288 (8)	0.0027 (6)	0.0108 (6)	0.0064 (6)
C5	0.0253 (7)	0.0254 (7)	0.0198 (7)	−0.0026 (5)	0.0072 (5)	0.0034 (5)
C6	0.0236 (6)	0.0195 (6)	0.0189 (7)	−0.0049 (5)	0.0089 (5)	0.0006 (5)
C7	0.0254 (7)	0.0229 (6)	0.0188 (6)	−0.0023 (5)	0.0102 (5)	0.0019 (5)
C8	0.0233 (7)	0.0231 (7)	0.0299 (8)	−0.0022 (6)	0.0113 (6)	0.0001 (6)
C9	0.0322 (9)	0.0456 (10)	0.0462 (11)	0.0092 (8)	0.0109 (8)	−0.0155 (8)
C10	0.0375 (10)	0.0769 (16)	0.0567 (13)	0.0176 (10)	0.0249 (9)	0.0170 (11)

Geometric parameters (Å, °)

S1—N1	1.7286 (13)	C3—H3	0.9500
S1—C1	1.7374 (15)	C4—C5	1.383 (2)
O1—C7	1.2125 (18)	C4—H4	0.9500

O2—C8	1.3232 (19)	C5—C6	1.393 (2)
O2—C9	1.465 (2)	C5—H5	0.9500
O3—C8	1.1999 (18)	C6—C7	1.463 (2)
N1—C8	1.4025 (19)	C9—C10	1.488 (3)
N1—C7	1.4182 (18)	C9—H9A	0.9900
C1—C6	1.3924 (19)	C9—H9B	0.9900
C1—C2	1.393 (2)	C10—H10A	0.9800
C2—C3	1.384 (2)	C10—H10B	0.9800
C2—H2	0.9500	C10—H10C	0.9800
C3—C4	1.400 (2)		
N1—S1—C1	89.99 (6)	C1—C6—C7	114.11 (12)
C8—O2—C9	115.15 (13)	C5—C6—C7	125.01 (13)
C8—N1—C7	129.73 (12)	O1—C7—N1	125.20 (14)
C8—N1—S1	114.18 (10)	O1—C7—C6	127.71 (13)
C7—N1—S1	116.07 (10)	N1—C7—C6	107.09 (12)
C6—C1—C2	120.98 (13)	O3—C8—O2	127.15 (14)
C6—C1—S1	112.72 (11)	O3—C8—N1	120.68 (14)
C2—C1—S1	126.30 (11)	O2—C8—N1	112.17 (12)
C3—C2—C1	117.65 (13)	O2—C9—C10	110.40 (16)
C3—C2—H2	121.2	O2—C9—H9A	109.6
C1—C2—H2	121.2	C10—C9—H9A	109.6
C2—C3—C4	121.76 (14)	O2—C9—H9B	109.6
C2—C3—H3	119.1	C10—C9—H9B	109.6
C4—C3—H3	119.1	H9A—C9—H9B	108.1
C5—C4—C3	120.16 (14)	C9—C10—H10A	109.5
C5—C4—H4	119.9	C9—C10—H10B	109.5
C3—C4—H4	119.9	H10A—C10—H10B	109.5
C4—C5—C6	118.56 (14)	C9—C10—H10C	109.5
C4—C5—H5	120.7	H10A—C10—H10C	109.5
C6—C5—H5	120.7	H10B—C10—H10C	109.5
C1—C6—C5	120.88 (13)		
C1—S1—N1—C8	−179.94 (11)	C8—N1—C7—O1	−0.7 (2)
C1—S1—N1—C7	−1.47 (11)	S1—N1—C7—O1	−178.93 (12)
N1—S1—C1—C6	0.71 (11)	C8—N1—C7—C6	179.92 (13)
N1—S1—C1—C2	−178.46 (13)	S1—N1—C7—C6	1.74 (15)
C6—C1—C2—C3	0.1 (2)	C1—C6—C7—O1	179.53 (14)
S1—C1—C2—C3	179.22 (11)	C5—C6—C7—O1	−0.9 (2)
C1—C2—C3—C4	0.5 (2)	C1—C6—C7—N1	−1.16 (16)
C2—C3—C4—C5	−1.1 (2)	C5—C6—C7—N1	178.43 (13)
C3—C4—C5—C6	0.9 (2)	C9—O2—C8—O3	−3.4 (2)
C2—C1—C6—C5	−0.2 (2)	C9—O2—C8—N1	176.56 (13)
S1—C1—C6—C5	−179.46 (11)	C7—N1—C8—O3	−179.58 (14)
C2—C1—C6—C7	179.38 (13)	S1—N1—C8—O3	−1.37 (18)
S1—C1—C6—C7	0.15 (15)	C7—N1—C8—O2	0.5 (2)
C4—C5—C6—C1	−0.3 (2)	S1—N1—C8—O2	178.70 (10)
C4—C5—C6—C7	−179.85 (13)	C8—O2—C9—C10	−85.9 (2)

Fig. 1