Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

**Xiang-hui Wang,[a] Jian-xin Yang,[b] Cheng-hang You[b] and Qiang Lin[c,d]*

[a]Institute of Environmental Science and Engineering, Kunming University of Science and Technology, Kunming 650093, People's Republic of China, [b]Hainan University Materials and Chemical Engineering, Haikou 570228, People's Republic of China, [c]Hainan Provincial Fine Chemical Engineering Center, Hainan University, Haikou 570228, People's Republic of China, and [d]College of Chemistry and Chemical Engineering, Hainan Normal University, Haikou 571100, People's Republic of China
Correspondence e-mail: linqiang@hainu.edu.cn

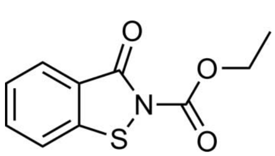
Received 25 May 2011; accepted 19 July 2011

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(2H)-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).

The authors are grateful to the National Natural Science Foundation of China (No. 20962007) and the Creative Talents Plan of the Hainan University 211 Project.

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

References

Cavalca, L., Gaetani, A., Mangia, A. & Pelizzi, G. (1970). *Gazz. Chim. Ital.* **100**, 629–638.
Cavalca, L., Gasparri, G. F., Mangia, A. & Pelizzi, G. (1969). *Acta Cryst.* B**25**, 2349–2354.
Davis, M. (1972). *Adv. Heterocycl. Chem.* **14**, 43–98.
Elgazwy, H. & Abdel-Sattar, S. (2003). *Tetrahedron*, **59**, 7445–7463.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2008). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Taubert, K., Kraus, S. & Schulze, B. (2002). *Sulfur Rep.* **23**, 79–81.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Xu, F.-L., Lin, Q. & Yin, X.-Q. (2006). *Acta Cryst.* E**62**, o496–o497.
Xu, L.-Z., Si, G.-D., Li, Z.-F., Yang, S.-H. & Li, K. (2005). *Acta Cryst.* E**61**, o1329–o1330.

supporting information

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

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

Xiang-hui Wang, Jian-xin Yang, Cheng-hang You and Qiang Lin

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 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)°.

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 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.

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.

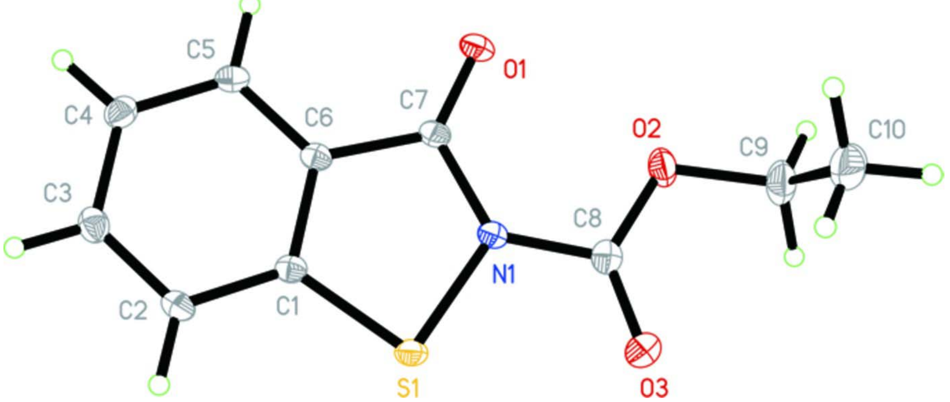


Figure 1

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

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

Crystal data

$C_{10}H_9NO_3S$
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
$F(000)$ = 464
D_x = 1.515 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3063 reflections
θ = 3.2–29.1°
μ = 0.32 mm^{-1}
T = 153 K
Block, colourless
0.39 × 0.33 × 0.32 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} = 0.887, T_{max} = 0.907
8002 measured reflections
2570 independent reflections
2219 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 29.1°, θ_{min} = 3.2°
h = −21→23
k = −6→6
l = −13→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.00
2570 reflections
137 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
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.216P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −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 (Å²)

	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, º)

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)