Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Ethyl 2,1,3-benzothiadiazole-5-carboxylate

Xue-Mei Li, Li-Ping Du, Jun Yang and Shu-Sheng Zhang*

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, 266042 Qingdao, Shandong, People's Republic of China

Correspondence e-mail: shushzhang@126.com

Received 4 January 2006
Accepted 6 January 2006

Key indicators

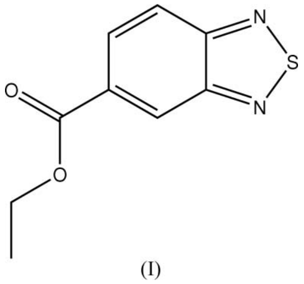
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.105
Data-to-parameter ratio = 14.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the essentially planar molecule of the title compound, $C_9H_8N_2O_2S$, the dihedral angle between the benzene and thiadiaole rings is 0.44 (7)°. In the crystal structure, molecules are stabilized by π–π interactions and van der Waals forces.

Comment

Recently, fluorene-based conjugated polymers have emerged as very promising candidates for polymer light-emitting diodes due to their combination of desirable properties, such as high fluorescence quantum yield, good film-forming and hole-transporting properties. Colour tuning in these polymers can be achieved by incorporating an electron-deficient monomer, benzothiadiazole, into the polymer backbone (Donat-Bouillud *et al.*, 2000). These polymers made from benzothiadiazole exhibited high external quantum efficiencies and high photometric power efficiency (Herguth *et al.*, 2002). In our studies of benzimidazole compounds, the title compound, (I), was obtained unexpectedly. The crystal structure is reported here.



(I)

In the title compound, the whole molecule excluding the H atoms is planar, the dihedral angle between the C4–C9 benzene and thiadiazole rings being 0.44 (7)°. All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The bonds in the benzothiadiazole system show intermediate character between single and double bonds, implying a highly π-conjugated delocalization. In the crystal structure, molecules are stabilized by π-π interactions involving the benzene rings, with $Cg2\cdots Cg2(1 - x, -y, 1 - z)$ = 3.777 Å ($Cg2$ is the centroid of the C4–C9 benzene ring), and by van der Waals forces.

Experimental

To a solution of $SOCl_2$ (10 ml) in CH_2Cl_2 (25 ml) were added 2-nitrobenzoic acid (3.3 g, 20 mmol) and a catalytic amount of dimethylformamide. The reaction mixture was stirred at room temperature for 16 h to give a clear solution. The volatiles were removed *in vacuo* to afford the crude acyl chloride product as a brown oil. A solution of the acyl chloride in CH_2Cl_2 (40 ml) was added dropwise to a solution of ethyl 3,4-diaminobenzoate (3.7 g, 20 mmol) and Et_3N (3.6 ml) in CH_2Cl_2 (20 ml). After the addition was complete, the reaction mixture was stirred at 273 K for 1 h and at room temperature for 3 h. The volatiles were removed *in vacuo* to give an off-white solid. The solid was refluxed in glacial AcOH (50 ml) in the presence of AcONa (1.64 g, 20 mmol) for 15 h. The resulting brown oil was partitioned between CH_2Cl_2 and water. The organic extracts were evaporated *in vacuo* to give a yellow solid. Single crystals were obtained from ethyl acetate/petroleum ether (1:2 *v*/*v*) solution over a period of 5 d.

Crystal data

$C_9H_8N_2O_2S$
$M_r = 208.23$
Monoclinic, $P2_1/c$
$a = 7.3545$ (6) Å
$b = 15.0337$ (12) Å
$c = 8.7923$ (7) Å
$\beta = 104.467$ (1)°
$V = 941.30$ (13) Å^3
$Z = 4$

$D_x = 1.469$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2739 reflections
$\theta = 2.7$–$26.0°$
$\mu = 0.32$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.34 × 0.20 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.900$, $T_{\max} = 0.972$
5276 measured reflections

1852 independent reflections
1647 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\max} = 26.0°$
$h = -9 \rightarrow 9$
$k = -18 \rightarrow 12$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.105$
$S = 1.04$
1852 reflections
127 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.1742P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—N2	1.6083 (15)	O1—C2	1.4608 (19)
S1—N1	1.6097 (14)	O2—C3	1.2034 (19)
O1—C3	1.3370 (19)		

All H atoms were located in difference Fourier maps, idealized, and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$ or $1.5U_{\rm eq}$(methyl C).

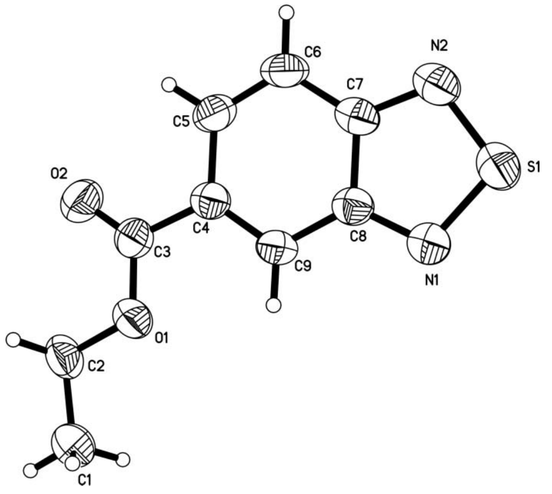


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

This project was supported by the Project of Educational Administration of Shandong Province (No. J04B12) and the Outstanding Adult-Young Scientific Research Encouraging Foundation of Shandong Province (No. 2005BS04007).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, S1–19.
Donat-Bouillud, A., Blondin, P., Ranger, M., Bouchard, J. & Leclerc, M. (2000). *Chem. Mater.* **12**, 1931–1936.
Herguth, P., Jiang, X., Liu, M. S. & Jen, A. K.-Y. (2002). *Macromolecules*, **35**, 6094-6100.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Acta Cryst. (2006). E**62**, o568–o569 Li *et al.* • $C_9H_8N_2O_2S$ **o569**