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The crystal structure of ethyl 2-(3-(2-ethoxy-2-oxoethyl)benzo[*d*] thiazol-2(3*H*)-ylidene)acetate, $C_{15}H_{17}NO_4S$

Table 1: Data collection and handling.

Crystal:	Colorless prismatic
Size:	0.20 × 0.16 × 0.13 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.22 mm ⁻¹
Diffractometer, scan mode:	APEX CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, 97%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16103, 2745, 0.047
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1985
$N(\text{param})_{\text{refined}}$:	220
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

$C_{15}H_{17}NO_4S$, orthorhombic, *Pbca* (no. 61), $a = 9.9890(11)$ Å, $b = 14.4529(16)$ Å, $c = 21.635(2)$ Å, $V = 3123.4(6)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0565$, $wR_{\text{ref}}(F^2) = 0.1656$, $T = 293(2)$ K.

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The molecular structure is shown in the figure (Hydrogen atoms are omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared according to published method [5], and obtained pale yellow solid. ¹H NMR (Bruker Avance 500 MHz, CDCl₃) δ ppm: 7.43–7.42 (m, 1H), 7.21–7.18 (m, 1H), 7.06–7.03 (m, 1H), 6.84–6.83 (m, 1H), 5.09 (s, 1H), 4.50 (s, 2H), 4.2–4.13 (m, 4H), 1.25–1.19 (m, 6H); ¹³C NMR (Bruker

Avance 500 MHz, CDCl₃) δ ppm: 168.6, 166.6, 160.8, 140.3, 126.5, 126.2, 122.5, 122.0, 108.8, 79.7, 62.2, 59.5, 46.6, 14.6, 14.1. Single crystal suitable for X-ray diffraction cultured from a slow evaporation of a mixture of acetone and petroleum ether.

Experimental details

H atoms bonded to C atoms were positioned geometrically and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms. All the non-hydrogen atoms were refined anisotropically.

Comment

In recent decades, benzothiazole derivatives have been found to bear a variety of biological activity and widely used in the field of industry [6, 7], agriculture [8], and medicine [9, 10]. Thus, the synthesis and crystal structures of various benzothiazole derivatives have attracted great attention [5, 11, 12]. In the title compound, the benzo and the thiazole moiety are in the same plane. This planar parent structure is present in previously reported benzothiazolylacetate [12]. There are three kinds of C–H···O hydrogen bonds, such as C(10)–H(10A)···O(1) hydrogen bond (2.477 Å), C(8)–H(8)···O(3) hydrogen bond (2.590 Å), and C(12)–H(12B)···O(3) hydrogen bond (2.559 Å). Although the benzo and the thiazole ring are coplanar, there is no π – π stacking interaction but a kind of aliphatic C(12)–H(12A)··· π (benzo moiety) bond (2.741 Å). This is should be ascribed to the the presence of the torsion angle (85.2°) between the *N*-substituted group

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.60644(8)	0.22712(6)	0.58873(3)	0.0654(3)
N1	0.5023(2)	0.19535(14)	0.69547(10)	0.0503(6)
O1	0.4639(3)	0.37876(17)	0.54489(10)	0.0888(7)
O2	0.3299(2)	0.46485(15)	0.60470(10)	0.0744(6)
O3	0.6185(2)	0.30863(16)	0.78006(10)	0.0765(6)
C1	0.6022(3)	0.12826(18)	0.68930(13)	0.0537(7)
C2	0.6380(3)	0.06146(19)	0.73131(16)	0.0668(8)
H2	0.5950	0.0572	0.7693	0.080*
C3	0.7391(4)	0.0011(2)	0.7159(2)	0.0839(11)
H3	0.7637	−0.0448	0.7438	0.101*
C4	0.8038(4)	0.0071(2)	0.6606(2)	0.0918(12)
H4	0.8718	−0.0347	0.6514	0.110*
C5	0.7698(3)	0.0742(2)	0.61819(17)	0.0783(10)
H5	0.8141	0.0782	0.5805	0.094*
C6	0.6683(3)	0.1355(2)	0.63294(13)	0.0603(7)
C7	0.4935(2)	0.25685(17)	0.64751(12)	0.0488(6)
C8	0.4119(3)	0.33077(18)	0.64578(13)	0.0544(7)
H8	0.3574	0.3432	0.6796	0.065*
C9	0.4066(3)	0.3909(2)	0.59328(14)	0.0610(7)
C10	0.3194(5)	0.5333(3)	0.55617(18)	0.0973(12)
H10A	0.4074	0.5463	0.5392	0.117*
H10B	0.2630	0.5101	0.5231	0.117*
C11	0.2632(8)	0.6154(3)	0.5813(2)	0.151(2)
H11A	0.1927	0.5995	0.6095	0.227*
H11B	0.2277	0.6527	0.5484	0.227*
H11C	0.3313	0.6494	0.6028	0.227*
C12	0.4344(2)	0.21128(17)	0.75308(12)	0.0508(6)
H12A	0.4228	0.1530	0.7746	0.061*
H12B	0.3463	0.2369	0.7450	0.061*
C13	0.5122(3)	0.27675(19)	0.79307(13)	0.0569(7)
O4 ^a	0.4419(8)	0.3038(4)	0.8397(3)	0.0710(13)
C14 ^a	0.5084(6)	0.3689(5)	0.8811(2)	0.0747(12)
H14A ^a	0.4960	0.4319	0.8669	0.090*
H14B ^a	0.6036	0.3560	0.8833	0.090*
C15 ^a	0.4408(9)	0.3543(6)	0.9452(3)	0.0953(18)
H15A ^a	0.3476	0.3698	0.9425	0.143*
H15B ^a	0.4832	0.3934	0.9753	0.143*
H15C ^a	0.4501	0.2908	0.9575	0.143*
O4 ^b	0.4570(13)	0.2754(7)	0.8524(5)	0.0772(16)
C14 ^b	0.5272(10)	0.3168(7)	0.9073(5)	0.0861(15)
H14C ^b	0.5281	0.2733	0.9415	0.103*
H14D ^b	0.6188	0.3325	0.8969	0.103*
C15 ^b	0.4561(14)	0.3956(8)	0.9232(6)	0.106(2)
H15D ^b	0.4505	0.4360	0.8881	0.158*
H15E ^b	0.5009	0.4269	0.9564	0.158*
H15F ^b	0.3675	0.3786	0.9361	0.158*

^aOccupancy: 0.6, ^bOccupancy: 0.4.

and benzothiazole ring, which causes that there is no overlap between the aromatic cycles in the upper and lower molecules.

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References

1. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, WI, USA (2007).
2. Sheldrick, G. M.: SHELXT – integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. Takayama, W.; Shirasaki, Y.; Sakai, Y.; Nakajima, E.; Fujita, S.; Sakamoto-Mizutani, K.; Inoue, J.: Synthesis and PDF inhibitory activities of novel benzothiazolylidenehydroxamic acid derivatives. *Bioorg. Med. Chem. Lett.* **13** (2003) 3273–3276.
6. Zhang, S.; Fan, J.; Li, Z.; Hao, N.; Cao, J.; Wu, T.; Wang, J.; Peng, X.: A bright red fluorescent cyanine dye for live-cell nucleic acid imaging, with high photostability and a large Stokes shift. *J. Mater. Chem. B* **2** (2014) 2688–2693.
7. Lanke, S. K.; Sekar, N.: Coumarin push-pull NLOphores with red emission: solvatochromic and theoretical approach. *J. Fluoresc.* **26** (2016) 949–962.
8. Graham, J.; Wong, C. E.; Day, J.; McFaddin, E.; Ochsner, U.; Hoang, T.; Young, C. L.; Ribble, W.; DeGroot, M. A.; Jarvis, T.; Sun, X.: Discovery of benzothiazole amides as potent antimycobacterial agents. *Bioorg. Med. Chem. Lett.* **28** (2018) 3177–3181.
9. Ahmed, K.; Naseer, A. K. M.; Sultana, F.; Methuku, K. R.: Recent advances on structural modifications of benzothiazoles and their conjugate systems as potential chemotherapeutics. *Expert Opin. Investig. Drugs* **21** (2012) 619–635.
10. Vellasco Jr, W. T.; Gomes, C. R. B.; Vasconcelos, T. R. A.: Chemistry and biological activities of 1,3-benzoxathiol-2-ones. *Mini-Rev. Org. Chem.* **8** (2011) 103–109.
11. Zhu, N.; Zhang, Z.; Gao, M.; Han, L.; Suo, Q.; Hong, H.: New progress in the synthesis of 2-substituent-benzothiazole derivatives. *Chin. J. Org. Chem.* **33** (2013) 1423–1436.
12. Pi, H.-J.; Liu, H.; Du, W.; Deng, W.-P.: The facile synthesis of benzothiazolylideneacetates and 1,4-benzothiazines through a highly controllable oxidation of benzothiazolylacetates. *Tetrahedron Lett.* **50** (2009) 4529–4531.