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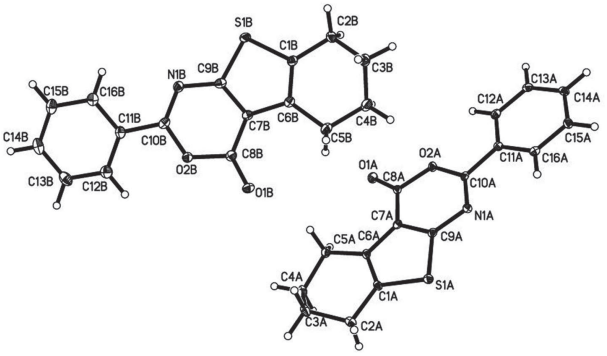
Crystal structure of 2-phenyl-5,6,7,8-tetrahydro-4*H*-benzo[4,5]thieno[2,3-d][1,3]oxazin-4-one, $C_{16}H_{13}NO_2S$



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Abstract

$C_{16}H_{13}NO_2S$, $P\bar{1}$ (no. 2), $a = 8.0873(4)$ Å, $b = 12.7820(7)$ Å, $c = 13.5574(7)$ Å, $\alpha = 74.880(2)°$, $\beta = 75.421(2)°$, $\gamma = 76.391(2)°$, $V = 1287.61(12)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0413$, $wR_{ref}(F^2) = 0.1075$, $T = 100$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of 4,5,6,7-tetrahydrobenzo[*b*]thiophene-2-amino-3-carboxylic acid (0.01 mol) in dry pyridine (30 mL) and benzoyl chloride (0.02 mol) was stirred for 1 h in an ice bath. After cooling, the reaction mixture was acidified with ice-cold hydrochloric acid. The solid formed after complete

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Table 1: Data collection and handling.

Crystal:	Yellow, needle, size 0.050×0.125×0.425 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.51 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	28481, 4534
$N(param)_{refined}$:	361
Programs:	Bruker programs [16], SHELX [17]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2AA)	2*i*	0.2537	0.7054	0.1062	0.019
H(2AB)	2*i*	0.0492	0.7041	0.1331	0.019
H(3AA)	2*i*	0.1659	0.6081	−0.0001	0.021
H(3AB)	2*i*	0.3208	0.5371	0.0565	0.021
H(4AA)	2*i*	0.1043	0.4303	0.0764	0.020
H(4AB)	2*i*	−0.0376	0.5167	0.1345	0.020
H(5AA)	2*i*	0.0382	0.3781	0.2688	0.019
H(5AB)	2*i*	0.2400	0.3620	0.2145	0.019
H(12A)	2*i*	0.2641	0.2634	0.7382	0.017
H(13A)	2*i*	0.3316	0.2352	0.9016	0.016
H(14A)	2*i*	0.4142	0.3752	0.9490	0.018
H(15A)	2*i*	0.4292	0.5455	0.8319	0.017
H(16A)	2*i*	0.3621	0.5742	0.6686	0.016
H(2BA)	2*i*	0.8921	−0.0281	0.4850	0.024
H(2BB)	2*i*	0.6862	−0.0209	0.5260	0.024
H(3BA)	2*i*	0.7593	0.1342	0.5580	0.023
H(3BB)	2*i*	0.8602	0.1659	0.4393	0.023
H(4BA)	2*i*	0.5753	0.2773	0.4686	0.025
H(4BB)	2*i*	0.4922	0.1684	0.4984	0.025
H(5BA)	2*i*	0.4712	0.2351	0.3283	0.026
H(5BB)	2*i*	0.6566	0.2724	0.2963	0.026
H(12B)	2*i*	0.5987	0.1016	−0.1372	0.025
H(13B)	2*i*	0.6188	0.0361	−0.2858	0.029
H(14B)	2*i*	0.8066	−0.1281	−0.3122	0.029
H(15B)	2*i*	0.9762	−0.2268	−0.1893	0.028
H(16B)	2*i*	0.9488	−0.1649	−0.0375	0.023

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1A)	2*i*	0.22167(7)	0.66481(4)	0.33083(4)	0.0156(3)	0.0126(3)	0.0127(3)	−0.0030(2)	−0.0034(2)	−0.0024(2)
O(1A)	2*i*	0.1546(2)	0.2726(1)	0.4557(1)	0.0239(9)	0.0135(8)	0.0194(9)	−0.0058(7)	−0.0066(7)	−0.0054(7)
O(2A)	2*i*	0.2217(2)	0.3553(1)	0.5625(1)	0.0180(8)	0.0115(8)	0.0134(8)	−0.0043(6)	−0.0044(6)	−0.0037(7)
N(1A)	2*i*	0.2618(2)	0.5394(1)	0.5213(1)	0.0119(9)	0.014(1)	0.013(1)	−0.0012(7)	−0.0026(7)	−0.0041(8)
C(1A)	2*i*	0.1767(3)	0.6001(2)	0.2453(2)	0.007(1)	0.019(1)	0.010(1)	−0.0001(9)	−0.0010(9)	−0.006(1)
C(2A)	2*i*	0.1654(3)	0.6574(2)	0.1349(2)	0.014(1)	0.019(1)	0.014(1)	−0.0029(9)	−0.0026(9)	−0.001(1)
C(3A)	2*i*	0.1963(3)	0.5714(2)	0.0686(2)	0.015(1)	0.028(1)	0.011(1)	−0.003(1)	−0.0021(9)	−0.006(1)
C(4A)	2*i*	0.0868(3)	0.4820(2)	0.1227(2)	0.015(1)	0.022(1)	0.014(1)	−0.0021(9)	−0.0026(9)	−0.008(1)
C(5A)	2*i*	0.1333(3)	0.4176(2)	0.2273(2)	0.014(1)	0.018(1)	0.017(1)	−0.0016(9)	−0.0032(9)	−0.007(1)
C(6A)	2*i*	0.1623(3)	0.4922(2)	0.2885(2)	0.007(1)	0.016(1)	0.012(1)	0.0000(9)	−0.0002(9)	−0.006(1)
C(7A)	2*i*	0.1889(2)	0.4609(2)	0.3935(2)	0.007(1)	0.014(1)	0.014(1)	−0.0002(8)	0.0009(9)	−0.005(1)
C(8A)	2*i*	0.1838(3)	0.3574(2)	0.4652(2)	0.009(1)	0.016(1)	0.013(1)	0.0007(9)	−0.0022(9)	−0.006(1)
C(9A)	2*i*	0.2243(3)	0.5462(2)	0.4260(2)	0.008(1)	0.014(1)	0.013(1)	0.0005(9)	−0.0005(9)	−0.0049(9)
C(10A)	2*i*	0.2605(3)	0.4448(2)	0.5838(2)	0.008(1)	0.010(1)	0.015(1)	−0.0009(8)	−0.0003(9)	−0.006(1)
C(11A)	2*i*	0.3042(2)	0.4216(2)	0.6875(2)	0.007(1)	0.015(1)	0.013(1)	0.0002(8)	0.0004(9)	−0.0055(9)
C(12A)	2*i*	0.2970(3)	0.3210(2)	0.7571(2)	0.013(1)	0.011(1)	0.019(1)	−0.0024(9)	−0.0008(9)	−0.005(1)
C(13A)	2*i*	0.3375(3)	0.3042(2)	0.8541(2)	0.012(1)	0.013(1)	0.014(1)	0.0004(9)	−0.0018(9)	−0.0005(9)
C(14A)	2*i*	0.3866(3)	0.3872(2)	0.8824(2)	0.010(1)	0.021(1)	0.012(1)	0.0008(9)	−0.0021(9)	−0.004(1)
C(15A)	2*i*	0.3955(3)	0.4883(2)	0.8129(2)	0.011(1)	0.016(1)	0.015(1)	−0.0015(9)	−0.0031(9)	−0.006(1)
C(16A)	2*i*	0.3552(3)	0.5053(2)	0.7161(2)	0.012(1)	0.011(1)	0.015(1)	−0.0010(9)	−0.0018(9)	−0.0023(9)
S(1B)	2*i*	0.85336(7)	−0.08916(5)	0.30267(5)	0.0236(3)	0.0111(3)	0.0224(3)	0.0012(2)	−0.0103(2)	−0.0052(2)
O(1B)	2*i*	0.5437(2)	0.2531(1)	0.1065(1)	0.031(1)	0.0138(9)	0.0199(9)	0.0047(7)	−0.0053(7)	−0.0028(7)
O(2B)	2*i*	0.6602(2)	0.1123(1)	0.0272(1)	0.0210(9)	0.0143(8)	0.0146(9)	−0.0006(7)	−0.0021(7)	−0.0034(7)
N(1B)	2*i*	0.8150(2)	−0.0570(2)	0.1032(1)	0.019(1)	0.014(1)	0.017(1)	−0.0043(8)	−0.0037(8)	−0.0047(9)
C(1B)	2*i*	0.7572(3)	0.0187(2)	0.3661(2)	0.015(1)	0.013(1)	0.020(1)	−0.0045(9)	−0.0046(9)	−0.005(1)
C(2B)	2*i*	0.7770(3)	0.0150(2)	0.4736(2)	0.024(1)	0.018(1)	0.021(1)	−0.003(1)	−0.010(1)	−0.006(1)
C(3B)	2*i*	0.7598(3)	0.1339(2)	0.4849(2)	0.024(1)	0.018(1)	0.018(1)	−0.005(1)	−0.004(1)	−0.007(1)
C(4B)	2*i*	0.5914(3)	0.2030(2)	0.4547(2)	0.025(1)	0.016(1)	0.021(1)	−0.003(1)	0.000(1)	−0.006(1)
C(5B)	2*i*	0.5922(3)	0.2136(2)	0.3393(2)	0.029(1)	0.015(1)	0.021(1)	−0.001(1)	−0.005(1)	−0.005(1)
C(6B)	2*i*	0.6749(3)	0.1072(2)	0.3045(2)	0.013(1)	0.014(1)	0.016(1)	−0.0045(9)	−0.0002(9)	−0.003(1)
C(7B)	2*i*	0.6890(3)	0.0880(2)	0.2029(2)	0.014(1)	0.012(1)	0.016(1)	−0.0050(9)	−0.0022(9)	−0.001(1)
C(8B)	2*i*	0.6224(3)	0.1603(2)	0.1162(2)	0.015(1)	0.016(1)	0.017(1)	−0.004(1)	0.0002(9)	−0.003(1)
C(9B)	2*i*	0.7823(3)	−0.0147(2)	0.1916(2)	0.014(1)	0.013(1)	0.021(1)	−0.0041(9)	−0.0036(9)	−0.003(1)
C(10B)	2*i*	0.7513(3)	0.0080(2)	0.0267(2)	0.013(1)	0.012(1)	0.021(1)	−0.0051(9)	0.001(1)	−0.002(1)
C(11B)	2*i*	0.7690(3)	−0.0260(2)	−0.0721(2)	0.015(1)	0.019(1)	0.014(1)	−0.008(1)	0.0007(9)	−0.004(1)
C(12B)	2*i*	0.6734(3)	0.0340(2)	−0.1469(2)	0.018(1)	0.022(1)	0.019(1)	−0.007(1)	0.001(1)	−0.002(1)
C(13B)	2*i*	0.6865(3)	−0.0044(2)	−0.2355(2)	0.021(1)	0.034(2)	0.017(1)	−0.012(1)	−0.002(1)	−0.000(1)
C(14B)	2*i*	0.7982(3)	−0.1019(2)	−0.2514(2)	0.028(1)	0.033(2)	0.016(1)	−0.017(1)	0.002(1)	−0.008(1)
C(15B)	2*i*	0.8977(3)	−0.1610(2)	−0.1779(2)	0.026(1)	0.022(1)	0.021(1)	−0.009(1)	0.003(1)	−0.009(1)
C(16B)	2*i*	0.8823(3)	−0.1238(2)	−0.0882(2)	0.021(1)	0.018(1)	0.019(1)	−0.008(1)	−0.002(1)	−0.003(1)

acidification was collected and recrystallized to give 4,5,6,7-tetrahydrobenzothiophene-2-benzoylamino-3-carboxylic acid. A mixture of 4,5,6,7-tetrahydrobenzothiophene-2-benzoylamino-3-carboxylic acid (0.01 mol) and acetic anhydride (10 mL) was heated on a water bath for 5 h. The solid formed after removal of excess acetic anhydride was triturated with petroleum ether (40–60°C) and recrystallized from petroleum ether (80–100°C) to give 2-phenyl-5,6,7,8-tetrahydro-*4H*-benzo[4,5]thieno[2,3-*d*][1,3]oxazin-4-one as yellow crystals; mp 137–140°C [1].

Experimental details

Cell refinement and data reduction were carried out by Bruker SAINT and APEX2 [16]. The hydrogen atoms were placed on calculated positions using a riding model (AFIX 23 or 43 option of the SHELX program [17]).

Discussion

Tetrahydrobenzothiophene derivatives have many biological activities, such as antivirus, antibacterial, insecticides, acaricides, and antifungal [2, 3] as well as anticancer, antitumor,

antihistaminic and anti-inflammatory activities [4–14]. Furthermore, tetrahydrobenzothiophene derivatives were reported to inhibit hepatitis C virus NS5B polymerase [15].

The asymmetric unit of the crystal structure contains two independent molecules with little difference in bond lengths and angles. In molecule A, the dihedral angle between the phenyl ring (C12—C16) and the thieno[2,3-d][1,3]oxazin-4-one moiety (S1/C1-C10/O2/N1) is 4.54(2)°, whereas this value in molecule B slightly is larger (12.44°). The molecules in the crystal structure at least interact *via* two non-classical intermolecular hydrogen bonds, of which O1B and S1B work as hydrogen bond acceptors and C14A and C12A work as hydrogen bond donors. The distance of the interactions between C14A—H14A$\cdots$O1B^{ii} and C12A—H12A$\cdots$S1B^{i} are 2.59 and 2.84 Å, respectively and the angles are 153 and 175°, respectively. Symmetry codes: (i) $-x+1, -y, -z+1$; (ii) $x, y, z+1$.

The almost flat molecules are stacked along the *a* direction with distances suggesting attractive interactions.

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