Crystal structure of 2-(2,3-dihydrobenzo[1,4]dioxin-6-yl)-5-(4-nitro benzylsulfanyl)-[1,3,4]oxadiazole, $C_{17}H_{13}N_3O_5S$

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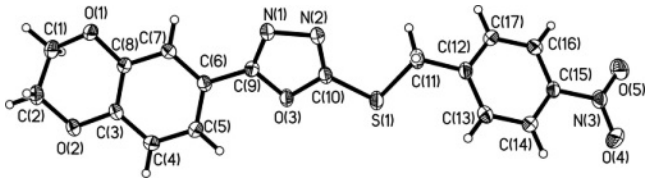

Abstract

$C_{17}H_{13}N_3O_5S$, triclinic, $P\bar{1}$ (no. 2), a = 7.199(1) Å, b = 8.002(2) Å, c = 14.547(3) Å, α = 96.722(2)°, β = 94.865(2)°, γ = 105.649(2)°, V = 795.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.0390, $wR_{ref}(F^2)$ = 0.0955, T = 298 K.

Source of material

To a stirred solution of 5-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-1,3,4-oxadiazo-le-2-thiol (1mmol, 0.268 g) and sodium hydroxide (1 mmol) in acetonitrile (50 mL) was added dropwise acetonitrile (5 mL) *p*-nitrobenzylbromide (1mmol), which of the latter being from the plant Flavescens. The resulting mixture was heated under reflux for 24 h and the reaction was monitored by thin layer chromatography (TLC). Afterwards the solution was cooled to room temperature and the organic solvent was removed in vacuo. The residue was dissolved in ethyl acetate and the organic layer was washed with saturated brine. Then the organic phase was dried over anhydrous Na_2SO_4, filtered, and removed in vacuo. The purification of the residue by recrystallization from acetonitrile yielded the colourless block-shaped single crystals of the title compound.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.18×0.20×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.41 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6427, 3372
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2672
$N(param)_{refined}$:	235
Programs:	SHELX [11]

Experimental details

All H atoms were constrained to ideal geometries, with C–H = 0.93-0.97 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Discussion

Oxadiazole compounds have been extensively studied for their biological activities [1-6]. As a continuation of the work on the structures of such compounds [7-9], in this paper, a new oxadiazole derivative is reported. The molecule of the compound is shown in Figure 1. The C(9)–O(3)–C(10)–N(2)–N(1) ring forms dihedral angles of 20.4(3) and 49.9(3)° with the C(3)–C(8) and C(12)–C(17) benzene rings, respectively. The dihedral angle between the C(3)–C(8) and C(12)–C(17) benzene rings is 34.5(3)°. All the bond lengths in the molecule are within normal values [10].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*i*	0.0297	1.0264	1.1159	0.065
H(1B)	2*i*	0.1920	1.2063	1.1287	0.065
H(2A)	2*i*	0.4036	1.0830	1.2095	0.058
H(2B)	2*i*	0.2189	1.0785	1.2605	0.058
H(4)	2*i*	0.2258	0.5339	1.1550	0.053
H(5)	2*i*	0.2583	0.3893	1.0137	0.051
H(7)	2*i*	0.2903	0.8265	0.8985	0.044
H(11A)	2*i*	0.2692	0.1548	0.5682	0.057
H(11B)	2*i*	0.4660	0.1299	0.6112	0.057
H(13)	2*i*	0.3938	–0.2208	0.6383	0.052
H(14)	2*i*	0.3278	–0.4912	0.5464	0.049
H(16)	2*i*	0.0800	–0.3058	0.3385	0.048
H(17)	2*i*	0.1450	–0.0362	0.4315	0.048

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.3806(3)	0.5648(2)	0.7829(1)	0.067(1)	0.0338(8)	0.0326(8)	0.0135(7)	0.0100(7)	0.0003(6)
N(2)	2*i*	0.3731(2)	0.4179(2)	0.7163(1)	0.066(1)	0.0359(8)	0.0311(8)	0.0148(7)	0.0097(7)	–0.0001(6)
N(3)	2*i*	0.1602(2)	–0.5912(2)	0.3756(1)	0.057(1)	0.0339(8)	0.0402(9)	0.0083(7)	0.0100(7)	–0.0015(7)
O(1)	2*i*	0.2432(2)	1.0225(2)	1.03869(8)	0.077(1)	0.0336(7)	0.0381(7)	0.0216(6)	0.0162(7)	0.0050(5)
O(2)	2*i*	0.2204(2)	0.8457(2)	1.19810(8)	0.0707(9)	0.0419(7)	0.0296(6)	0.0180(6)	0.0121(6)	0.0005(5)
O(3)	2*i*	0.2518(2)	0.3244(2)	0.84158(8)	0.0515(8)	0.0323(6)	0.0337(6)	0.0094(5)	0.0117(5)	–0.0019(5)
O(4)	2*i*	0.2048(3)	–0.7095(2)	0.4096(1)	0.106(1)	0.0361(8)	0.066(1)	0.0271(8)	0.0041(9)	–0.0009(7)
O(5)	2*i*	0.0836(3)	–0.6082(2)	0.2960(1)	0.106(1)	0.0533(9)	0.0400(8)	0.0136(9)	–0.0062(8)	–0.0109(7)
S(1)	2*i*	0.23362(8)	0.06172(6)	0.71249(3)	0.0637(3)	0.0330(2)	0.0397(3)	0.0092(2)	0.0151(2)	–0.0031(2)
C(1)	2*i*	0.1689(4)	1.0801(3)	1.1216(1)	0.083(2)	0.042(1)	0.046(1)	0.029(1)	0.018(1)	0.0013(9)
C(2)	2*i*	0.2641(3)	1.0320(2)	1.2047(1)	0.063(1)	0.041(1)	0.039(1)	0.0120(9)	0.0090(9)	–0.0057(8)
C(3)	2*i*	0.2380(3)	0.7658(2)	1.1122(1)	0.040(1)	0.0386(9)	0.0278(8)	0.0116(7)	0.0053(7)	0.0003(7)
C(4)	2*i*	0.2390(3)	0.5928(2)	1.1035(1)	0.064(1)	0.040(1)	0.0338(9)	0.0182(9)	0.0103(9)	0.0098(7)
C(5)	2*i*	0.2593(3)	0.5065(2)	1.0192(1)	0.060(1)	0.0319(9)	0.039(1)	0.0167(8)	0.0087(9)	0.0043(7)
C(6)	2*i*	0.2813(2)	0.5948(2)	0.9423(1)	0.038(1)	0.0354(9)	0.0298(8)	0.0104(7)	0.0035(7)	0.0003(7)
C(7)	2*i*	0.2769(3)	0.7678(2)	0.9501(1)	0.044(1)	0.0354(9)	0.0302(8)	0.0115(7)	0.0072(7)	0.0056(7)
C(8)	2*i*	0.2527(3)	0.8533(2)	1.0347(1)	0.039(1)	0.0294(8)	0.0348(9)	0.0093(7)	0.0046(7)	0.0022(7)
C(9)	2*i*	0.3080(3)	0.5041(2)	0.8538(1)	0.040(1)	0.0305(8)	0.0334(9)	0.0109(7)	0.0023(7)	0.0012(7)
C(10)	2*i*	0.2965(3)	0.2833(2)	0.7548(1)	0.040(1)	0.0373(9)	0.0295(8)	0.0131(8)	0.0036(7)	–0.0027(7)
C(11)	2*i*	0.3262(3)	0.0787(2)	0.6013(1)	0.060(1)	0.037(1)	0.043(1)	0.0074(9)	0.0207(9)	–0.0030(8)
C(12)	2*i*	0.2776(3)	–0.1001(2)	0.5441(1)	0.041(1)	0.0322(9)	0.0348(9)	0.0090(7)	0.0132(7)	0.0003(7)
C(13)	2*i*	0.3309(3)	–0.2377(2)	0.5780(1)	0.052(1)	0.045(1)	0.0305(9)	0.0156(9)	0.0002(8)	–0.0009(7)
C(14)	2*i*	0.2922(3)	–0.3991(2)	0.5235(1)	0.054(1)	0.0368(9)	0.0356(9)	0.0178(8)	0.0043(8)	0.0050(7)
C(15)	2*i*	0.1999(2)	–0.4204(2)	0.4346(1)	0.038(1)	0.0299(8)	0.0329(9)	0.0074(7)	0.0087(7)	0.0000(6)
C(16)	2*i*	0.1433(3)	–0.2878(2)	0.3988(1)	0.047(1)	0.041(1)	0.0313(9)	0.0125(8)	0.0014(8)	0.0042(7)
C(17)	2*i*	0.1828(3)	–0.1272(2)	0.4545(1)	0.051(1)	0.0353(9)	0.041(1)	0.0190(8)	0.0099(8)	0.0081(7)

References

1. Akhter, M.; Akhter, N.; Alam, M. M.; Zaman, M. S.; Saha, R.; Kumar, A.: Synthesis and biological evaluation of 2,5-disubstituted 1,3,4-oxadiazole derivatives with both COX and LOX inhibitory activity. J. Enzyme Inhib. Med. Chem. **26** (2011) 767-776.
2. Kaplancikli, Z. A.; Altintop, M. D.; Turan-Zitouni, G.; Ozdemir, A.; Ozic, R.; Akalin, G.: Synthesis, antimicrobial activity and cytotoxicity of novel oxadiazole derivatives. J. Enzyme Inhib. Med. Chem. **27** (2012) 51-57.
3. Bondock, S.; Adel, S.; Etman, H. A.; Badria, F. A.: Synthesis and antitumor evaluation of some new 1,3,4-oxadiazole-based heterocycles. Eur. J. Med. Chem. **48** (2012) 192-199.
4. Bakal, R. L.; Gattani, S. G.: Identification and development of 2,5-disubstituted oxadiazole as potential candidate for treatment of XDR and MDR tuberculosis. Eur. J. Med. Chem. **47** (2012) 278-282.
5. Ispikoudi, M.; Amvrazis, M.; Kontogiorgis, C.; Koumbis, A. E.; Litinas, K. E.; Hadjipavlou-Litina, D.; Fylaktakidou, K. C.: Convenient synthesis and biological profile of 5-amino-substituted 1,2,4-oxadiazole derivatives. Eur. J. Med. Chem. **45** (2010) 5635-5645.
6. Zhang, X.-M.; Qiu, M.; Sun, J.; Zhang, Y.-B.; Yang, Y.-S.; Wang, X.-L.; Tang, J.-F.; Zhu, H.-L.: Synthesis, biological evaluation, and molecular docking studies of 1,3,4-oxadiazole derivatives possessing 1,4-benzodioxan moiety as potential anticancer agents. Bioorg. Med. Chem. **19** (2011) 6518-6524.
7. Jiang, Z.-J.; Liu, J.-T.; Lv, H.-S.; Zhao, B.-X.: Synthesis, X-ray crystal structure and optical properties of novel 5-(3-aryl-1*H*-pyrazol-5-yl)-2-(6-methoxy-3-methylbenzofuran-2-yl)-1,3,4-oxadiazole. Spectrochim. Acta **A 86** (2012) 181-186.
8. Fun, H.-K.; Hemamalini, M.; Rai, S.; Isloor, A. M.; Shetty, P.: 2-(5-Bromopyridin-3-yl)-5-[3-(4,5,6,7-tetrahydrothieno[3,2-c]pyridine-5-ylsulfonyl)thiophen-2-yl]-1,3,4-oxadiazole. Acta Crystallogr. **E67** (2011) o2743.
9. Wang, H.-T.; Jia, X.-S.; Qu, S.-N.; Bai, B.-L.; Li, M.: 5,5'-*Bis*(naphthalen-2-yl)-2,2'-*bi*(1,3,4-oxadiazole). Acta Crystallogr. **E67** (2011) o3360.
10. Allen, F. H.; Kennard, O.; Watson, D. G.; Brammer, L.; Orpen, A. G.; Taylor, R.: Tables of bond lengths determined by X-ray and neutron-diffraction.1. Bond lengths in organic-compounds. J. Chem. Soc. Perkin Trans. **2** (1987) S1-S19.
11. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.