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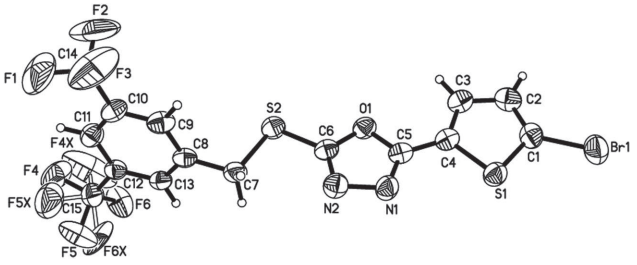
Crystal structure of 2-[3,5-bis(trifluoromethyl) benzylsulfanyl]-5-(5-bromothiophen-2-yl)-1,3,4-oxadiazole, $C_{15}H_7BrF_6N_2OS_2$



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Abstract

$C_{15}H_7BrF_6N_2OS_2$, monoclinic, $P2_1/c$ (no. 14), $a = 18.8292(14)$ Å, $b = 11.4568(9)$ Å, $c = 8.3400(6)$ Å, $\beta = 90.791(3)°$, $V = 1799.0(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.068$, $wR_{ref}(F^2) = 0.199$, $T = 296(2)$.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.57 × 0.35 × 0.07 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	25.8 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21302, 3161, 0.100
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1970
$N(param)_{refined}$:	272
Programs:	SHELX [23], Bruker programs [24]

Source of material

3,5-Bis(trifluoromethyl)benzyl bromide (614 mg, 2.0 mmol) and anhydrous potassium carbonate (276 mg, 2.0 mmol) were added to a solution of 5-(5-bromothiophen-2-yl)-1,3,4-oxadiazole-2-thiol (526 mg, 2.0 mmol), in *N*,*N*-dimethylformamide (5 mL), and the mixture was stirred for 6 h at room temperature. Water (15 mL) was added to the reaction mixture and stirring was continued for 10 min. The precipitated crude product was filtered, washed with water, dried and crystallized from ethanol to yield 646 mg (66%) of the title compound ($C_{15}H_7BrF_6N_2OS_2$) as shiny transparent plates. M.P.: 373–375 K. Single crystals were obtained by slow evaporation from EtOH/$CHCl_3$ (1:2) at room temperature. **^{1}H NMR** (DMSO-d_6, 500.13 MHz): δ 4.73 (s, 2H, CH_2), 7.43 (d, 1H, thiophene-H, $J = 4.0$ Hz), 7.60 (d, 1H, thiophene-H, $J = 4.0$ Hz), 8.03 (s, 1H, Ar—H), 8.24 (s, 2H, Ar—H). **^{13}C NMR** (DMSO-d_6, 125.76 MHz): δ 35.06 (CH_2), 117.87, 121.92, 124.76, 125.75, 130.52, 130.87, 131.51, 132.60, 141.38 (Ar—C, thiophene-C & CF_3), 161.04, 163.01 (oxadiazole-C). **ESI-MS**, m/z: 489.1 $(M + H, 100\%)^+$, 491.1 $(M + 2 + H, 98\%)^+$.

Experimental details

H atoms were placed in calculated positions and were included in the refinement in the riding model approximation. The fluorine atoms of the disorderd CF_3-group were located on a difference Fourier map and refined freely.

Discussion

1,3,4-Oxadiazole derivatives were early identified as a structural motif of particular interest in medicinal chemistry, material sciences and agrochemistry. Some 1,3,4-oxadiazoles have occupied a unique situation in the field of medicinal

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.72439(5)	0.71399(9)	−0.13559(11)	0.0903(4)
F1	−0.0040(4)	0.3361(8)	0.6335(10)	0.177(4)
F2	0.0787(4)	0.2435(6)	0.5630(13)	0.190(4)
F3	0.0346(5)	0.3718(7)	0.4127(8)	0.169(3)
F4[a]	0.0955(12)	0.5157(15)	1.1082(13)	0.120(6)
F5[a]	0.1041(11)	0.6846(12)	1.0352(14)	0.119(6)
F6[a]	0.1969(4)	0.5984(13)	1.0983(9)	0.088(4)
F5X[b]	0.0676(8)	0.588(6)	1.056(6)	0.148(17)
F6X[b]	0.137(2)	0.6986(9)	1.035(5)	0.19(4)
F4X[b]	0.156(5)	0.502(4)	1.117(3)	0.21(4)
S1	0.57631(10)	0.73792(16)	0.0189(2)	0.0639(5)
S2	0.33239(10)	0.51981(15)	0.4713(2)	0.0647(5)
O1	0.4458(2)	0.5598(4)	0.2987(5)	0.0542(11)
N1	0.4281(3)	0.7312(5)	0.1833(7)	0.0625(15)
N2	0.3680(3)	0.7040(5)	0.2801(7)	0.0626(15)
C1	0.6481(4)	0.6519(7)	−0.0195(8)	0.0615(18)
C2	0.6428(4)	0.5444(7)	0.0450(9)	0.0643(19)
H2A	0.6779	0.4853	0.0342	0.077*
C3	0.5797(4)	0.5298(6)	0.1297(8)	0.0584(17)
H3A	0.5673	0.4598	0.1836	0.070*
C4	0.5381(4)	0.6272(5)	0.1264(7)	0.0506(15)
C5	0.4706(4)	0.6447(5)	0.1977(7)	0.0499(15)
C6	0.3828(4)	0.6037(5)	0.3432(8)	0.0528(16)
C7	0.2608(4)	0.6235(5)	0.5014(8)	0.0573(17)
H7A	0.2791	0.6936	0.5576	0.069*
H7B	0.2404	0.6480	0.3967	0.069*
C8	0.2044(3)	0.5650(5)	0.6009(7)	0.0494(15)
C9	0.1578(4)	0.4840(6)	0.5370(8)	0.0605(18)
H9A	0.1619	0.4621	0.4276	0.073*
C10	0.1052(4)	0.4339(6)	0.6279(8)	0.0589(17)
C11	0.0978(4)	0.4661(6)	0.7855(8)	0.0589(17)
H11A	0.0614	0.4326	0.8484	0.071*
C12	0.1435(3)	0.5474(5)	0.8519(7)	0.0489(15)
C13	0.1965(3)	0.5955(5)	0.7604(7)	0.0484(15)
H13A	0.2282	0.6506	0.8076	0.058*
C14	0.0560(6)	0.3450(9)	0.5561(11)	0.088(3)
C15	0.1348(5)	0.5857(7)	1.0223(9)	0.0676(19)

[a]Occupancy: 0.74(3); [b]Occupancy: 0.26(3).

chemistry as pharmacophores or auxophores possessing diverse pharmacological activities, including antibacterial [1–5], anticancer [6–10], antiviral [11–13], antihypertensive [14, 15], anti-inflammatory [16, 17] and antioxidant [18] activities. In addition, some 1,3,4-oxadiazoles are currently used as safe herbicides for crop protection [19–21]. Moreover, 1,3,4-oxadiazole derivatives are widely employed electron-transporting and hole-blocking materials in the development of organic light-emitting diodes (OLEDs) [22].

The asymmetric unit cell of the title compound contains one independent molecule. The oxadiazole ring (O1/C5/N1/N2/C6)) makes dihedral angles 7.74° and 81.39° with the bromothiophene moiety (S1/C1—C4) and phenyl moiety (C8—C13), respectively. The trifluromethyl group is disordered over two sets of sites in a 0.736 : 0.379 ratio. The molecules packed in the crystal structure without any intermolecular hydrogen bonds.

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