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N-{[1-(5-Benzylsulfanyl)-1,3,4-oxadiazol-2-yl]-ethyl}-4-chlorobenzenesulfonamide

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.048
wR factor = 0.119
Data-to-parameter ratio = 14.0

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

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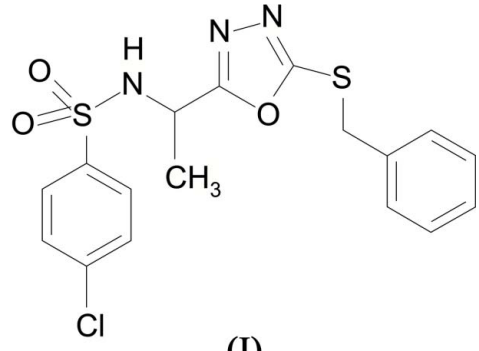
The crystal structure of the title compound, $C_{17}H_{16}ClN_3O_3S_2$, contains hydrogen-bonded chains lying along the *b* axis. In the molecule, the benzene rings lie almost parallel to each other and the heterocyclic oxadiazole ring is oriented nearly perpendicular to the benzene rings.

Comment

Benzenesulfonamides and 1,3,4-oxadiazole derivatives have been reported to possess significant biological activities, such as antimicrobial, anti-HIV, insulin-releasing antidiabetic, carbonic anhydrase inhibitory, high-ceiling diuretic, anti-thyroid, antitumour, *etc*. (Nishimori *et al.*, 2006; Turner, 2002; Supuran & Scozzafava, 2000, 2001, 2003; Masereel *et al.*, 2002; Singh *et al.*, 1997; Khanum *et al.*, 2005). In continuation of our interest in the chemical and pharmacological properties of benzenesulfonamides and 1,3,4-oxadiazole derivatives (Zareef *et al.*, 2006), we have synthesized a series of new compounds. We report the structure of the title compound, (I), in this paper.



(I)

Molecules of (I) (Fig. 1) form hydrogen-bonded chains along the *b* axis *via* strong N—H···N hydrogen bonds (Fig. 2); details of the hydrogen-bonding geometry are provided in Table 2. Unlike the structure of 4-methyl-*N*-[1-(5-mercapto-1,3,4-oxadiazol-2-yl)propyl]benzenesulfonamide (Zareef *et al.*, 2006), which is closely related to (I), the O atoms bonded to atom S2 are not involved in hydrogen bonds. The oxadiazole ring is essentially planar and the benzene rings lie approximately perpendicular to its mean plane: the angle between the mean planes of the oxadiazole and chlorophenyl rings is 71.61 (10)°, while that between the oxadizole and benzyl ring is 84.10 (9)°. The benzene rings lie almost parallel to each other, the angle between the mean planes being 6.25 (18)°. The molecular dimensions in (I) are as expected. A search of the Cambridge Structural Database (2006 release; Allen,

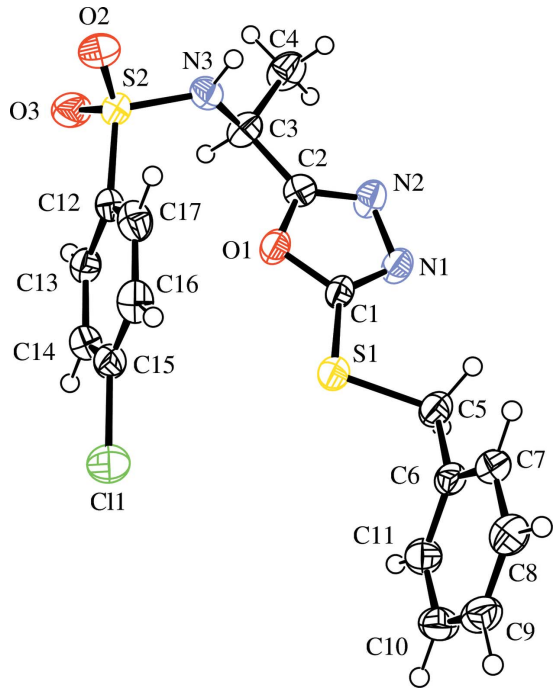


Figure 1
The molecular structure of (I), with displacement ellipsoids plotted at the 50% probability level.

2002) for the 5-thio-1,3,4-oxadiazole skeleton yielded only five entries [refcodes AVULIM (Ozturk *et al.*, 2004), AVULUY (Du *et al.*, 2004), YITMUJ (Ziyaev *et al.*, 1992), IZAJEY (Qiu & Xu, 2004) and UGOBEX (Zhang *et al.*, 2002); the first three compounds are thiones with S=C double bonds, while IZAJEY is a thioglucopyranoside derivative with S—C distances similar to those found in (I). Three-dimensional coordinates for UGOBEX, a benzylsulfanyloxadiazole derivative of a triazole, are not available in the CSD.

Experimental

Compound (I) was synthesized in four steps. D-2-(4-Chlorophenylsulfonamido)propanoic acid was esterified with ethanol in an acidic medium using the standard method (Furniss *et al.*, 1978). A mixture of the resulting ethyl-2-(4-chlorophenylsulfonamido)propanoate (10 mmol) and hydrazine monohydrate (80%) in absolute ethanol (60 ml) was refluxed for 9 h. The excess solvent was distilled off and the residue was filtered off, washed with water and recrystallized from 60% aqueous ethanol to yield 2-(4-chlorophenylsulfonamido)propane hydrazide. 4-Chloro-*N*-[1-(5-mercapto-1,3,4-oxadiazol-2-yl)ethyl]benzenesulfonamide was obtained by the reaction of this hydrazide (5.5 mmol) in absolute ethanol (80 ml) with carbon disulfide (6.6 mmol) and aqueous potassium hydroxide (5.5 mmol) at reflux temperature for 17 h. Finally, 4-chloro-*N*-[1-(5-mercapto-1,3,4-oxadiazol-2-yl)ethyl]benzenesulfonamide (0.75 mmol), Et_3N (0.22 mmol) and a catalytic amount of 4-(dimethylamino)pyridine (DMAP) (25 mg) were stirred in dry CH_3Cl (25 ml) for 15 min. Benzyl bromide (0.8 mmol) was added and the mixture was stirred for 5 h at 323–343 K. The reaction mixture was washed with dilute HCl, brine and water, and dried over Na_2SO_4 (anhydrous). The excess solvent was distilled off and the product was recrystallized from aqueous ethanol to obtain the pure title compound, (I). Crystals suitable for crystallographic study were grown from a solution of (I) in ethanol by slow evaporation at room temperature.

Crystal data

$C_{17}H_{16}ClN_3O_3S_2$
M_r = 409.90
Monoclinic, $P2_1/c$
a = 10.353 (3) Å
b = 7.592 (3) Å
c = 23.655 (10) Å
β = 100.329 (19)°
V = 1829.2 (12) Å^3
Z = 4
D_x = 1.488 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.46 mm^{-1}
T = 173 (2) K
Needle, colourless
0.25 × 0.06 × 0.03 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.894, T_{max} = 0.986
11365 measured reflections
3314 independent reflections
2422 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.119
S = 1.05
3314 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 1.95P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C15	1.736 (3)	O1—C1	1.360 (3)
S1—C1	1.723 (3)	O1—C2	1.365 (4)
S1—C5	1.821 (3)	N1—C1	1.296 (4)
S2—O3	1.425 (2)	N1—N2	1.418 (4)
S2—O2	1.437 (3)	N2—C2	1.283 (4)
S2—N3	1.614 (3)	N3—C3	1.449 (4)
S2—C12	1.760 (3)		
C1—S1—C5	98.76 (15)	N3—S2—C12	108.00 (14)
O3—S2—O2	120.08 (15)	C1—O1—C2	103.0 (2)
O3—S2—N3	109.84 (16)	C1—N1—N2	106.2 (2)
O2—S2—N3	103.25 (15)	C2—N2—N1	106.0 (3)
O3—S2—C12	106.85 (14)	C3—N3—S2	124.2 (2)
O2—S2—C12	108.33 (16)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N1^{i}	0.88	2.19	2.936 (4)	143

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

H atoms were included in the refinement in geometrically idealized positions, with N—H = 0.88 and C—H = 0.95–1.00 Å, and $U_{iso} = 1.2U_{eq}$(C,N).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure:

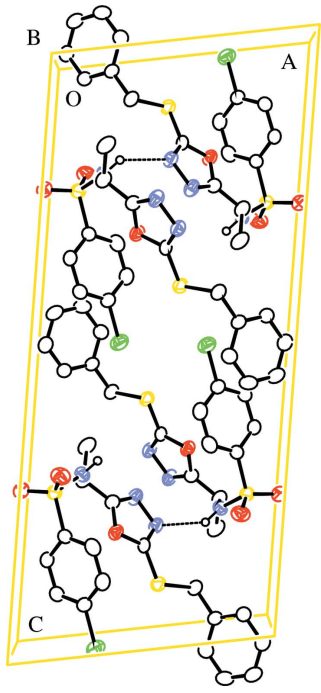


Figure 2
The packing of (I), showing the N—H···N hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

SHELXL97 (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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