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2-(Benzylsulfanyl)-4-phenyl-3*H*-1,5-benzodiazepine

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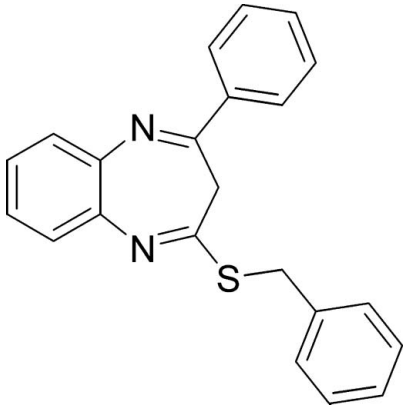
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.121; data-to-parameter ratio = 23.8.

The title compound, $C_{22}H_{18}N_2S$, is a benzodiazepine derivative with interesting pharmacological properties. The molecule is built up from two fused six- and seven-membered rings with benzylsulfanyl and phenyl substituents. The seven-membered ring displays a twist-chair conformation. There is weak slipped π–π stacking between symmetry-related molecules, with an interplanar distance of 3.45 Å and a centroid-to-centroid vector of 3.805 (1) Å (involving the benzyl aromatic rings), which ensures the cohesion of the crystal structure, together with van der Waals forces.

Related literature

For related literature, see: Akkurt *et al.* (2005); Cremer & Pople (1975); El Azzaoui *et al.* (2006); Ghomsi *et al.* (2004); Grossi *et al.* (2002); Kusanur *et al.* (2004); Morimoto *et al.* (2002); Vijay *et al.* (2002); Zellou *et al.* (1999).



Experimental

Crystal data

$C_{22}H_{18}N_2S$
M_r = 342.44
Monoclinic, $P2_1/c$
a = 8.9633 (2) Å
b = 8.4904 (2) Å
c = 23.1997 (5) Å
β = 92.206 (1)°
V = 1764.24 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 293 (2) K
0.36 × 0.20 × 0.11 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: none
20651 measured reflections
5390 independent reflections
3087 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.121
S = 1.01
5390 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2191).

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supplementary materials

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2-(Benzylsulfanyl)-4-phenyl-3*H*-1,5-benzodiazepine

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Comment

Benzodiazepines are very important compounds widely used in the last decades as antipyretic (Grossi *et al.*, 2002), anti-anxiety (Kusanur *et al.*, 2004) and hypnotic agents (Zellou *et al.*, 1999).

In addition to the well known pharmacological profile of 1,4-benzodiazepines, it has been shown that also some 1,5-benzodiazepines exert a biological activity (Vijay *et al.*, 2002, Morimoto *et al.*, 2002), similar to that of 1,4-derivatives. Moreover, 1,5-benzodiazepines are valuable synthons used for the synthesis of new heterocyclic compounds, such as benzimidazole, isoxazole and pyrazole (El Azzaoui *et al.*, 2006, Akkurt *et al.* 2005, Ghomsi *et al.*, 2004).

The 2-(benzylsulfanyl)-4-phenyl-3*H*-1,5-benzodiazepine molecule (I) is built up from two fused six-membered and seven-membered rings linked to benzylsulfanyl and phenyl, but not coplanar, as show in Fig. 1. The seven-membered ring displays a twist-chair conformation in the moleclule, as indicated by the total puckering amplitude Q_T=0.859 (2) Å and spherical polar angle θ_2=74.83 (9)° with φ_2=22.3 (2)° and φ_3=128.4 (4)° (Cremer & Pople, 1975).

The crystal structure is stabilized by weak slipped π-π interaction involving the benzene C11–C16 ring between symmetry related molecules with interplanar distance of 3.45Å and centroid to centroid vector of 3.805 (1)Å and Van der Waals forces.

Experimental

To a solution of 4-phenyl-1,5-benzodiazepine-2-thione (1 g, 3.96 mmol) and benzylbromide (0.70 ml, 4.36 mmol) in DMF (20 ml), 0.5 mmol of tetra-n-butylammonium bromide and 4.36 mmol (0.60 g) of anhydrous potassium carbonate were added. After filtration, the solvent was evaporated under reduced pressure and the crude residue was recrystallized from ethanol giving the compound I in 83% yield.

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) with U_{iso}(H) = 1.2U_{eq}(C).

Figures

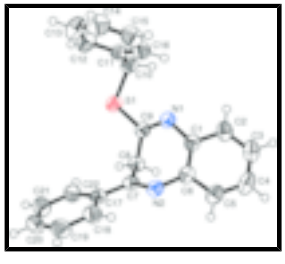


Fig. 1. Molecular structure of (I) with atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

2-(Benzylsulfanyl)-4-phenyl-3*H*-1,5-benzodiazepine

Crystal data

$C_{22}H_{18}N_2S$	F_{000} = 720
M_r = 342.44	D_x = 1.289 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -p 2ybc	Cell parameters from 5390 reflections
a = 8.9633 (2) Å	θ = 2.5–30.6º
b = 8.4904 (2) Å	μ = 0.19 mm^{-1}
c = 23.1997 (5) Å	T = 293 (2) K
β = 92.2060 (10)º	Parallelepiped, pale yellow
V = 1764.24 (7) Å^3	0.36 × 0.20 × 0.11 mm
Z = 4	

Data collection

Bruker X8 APEXII KappaCCD area-detector diffractometer	3087 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.050
Monochromator: graphite	θ_{max} = 30.6º
T = 293(2) K	θ_{min} = 2.6º
φ scans, and ω scans with κ offsets	h = −12→12
Absorption correction: none	k = −11→12
20651 measured reflections	l = −28→33
5390 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.121	$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 0.0842P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
5390 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
226 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.46398 (5)	0.71815 (5)	0.067691 (19)	0.04826 (14)
N1	0.28324 (15)	0.66474 (15)	0.15407 (6)	0.0430 (3)
N2	−0.03087 (15)	0.66376 (15)	0.10001 (6)	0.0441 (3)
C1	0.15618 (18)	0.59743 (18)	0.17860 (7)	0.0418 (4)
C2	0.1790 (2)	0.5380 (2)	0.23459 (7)	0.0537 (4)
H2	0.2727	0.5476	0.2529	0.064*
C3	0.0662 (2)	0.4661 (2)	0.26296 (8)	0.0605 (5)
H3	0.0849	0.4222	0.2992	0.073*
C4	−0.0755 (2)	0.4591 (2)	0.23736 (9)	0.0610 (5)
H4	−0.1520	0.4088	0.2561	0.073*
C5	−0.1033 (2)	0.5264 (2)	0.18437 (8)	0.0535 (4)
H5	−0.2004	0.5278	0.1688	0.064*
C6	0.01218 (18)	0.59340 (18)	0.15305 (7)	0.0421 (4)
C7	0.05337 (17)	0.65608 (18)	0.05623 (7)	0.0402 (4)
C8	0.19674 (17)	0.56213 (18)	0.05994 (7)	0.0423 (4)
H8A	0.1780	0.4572	0.0745	0.051*
H8B	0.2383	0.5529	0.0221	0.051*
C9	0.30306 (17)	0.64850 (17)	0.10023 (7)	0.0389 (3)
C10	0.56719 (19)	0.8065 (2)	0.12818 (8)	0.0480 (4)
H10A	0.5497	0.7450	0.1625	0.058*
H10B	0.6730	0.8005	0.1211	0.058*
C11	0.52765 (17)	0.97528 (19)	0.13977 (7)	0.0430 (4)
C12	0.5712 (2)	1.0944 (2)	0.10325 (9)	0.0605 (5)
H12	0.6242	1.0695	0.0708	0.073*
C13	0.5368 (2)	1.2495 (2)	0.11455 (10)	0.0701 (6)
H13	0.5661	1.3282	0.0895	0.084*
C14	0.4599 (2)	1.2880 (2)	0.16230 (10)	0.0666 (5)
H14	0.4391	1.3929	0.1704	0.080*
C15	0.4135 (2)	1.1711 (2)	0.19825 (8)	0.0627 (5)
H15	0.3592	1.1967	0.2303	0.075*
C16	0.44700 (19)	1.0155 (2)	0.18717 (8)	0.0526 (4)
H16	0.4150	0.9371	0.2118	0.063*
C17	0.00515 (18)	0.74104 (17)	0.00296 (7)	0.0408 (4)
C18	−0.1250 (2)	0.8310 (2)	0.00231 (8)	0.0526 (4)
H18	−0.1791	0.8387	0.0356	0.063*
C19	−0.1745 (2)	0.9085 (2)	−0.04679 (9)	0.0618 (5)
H19	−0.2622	0.9670	−0.0465	0.074*
C20	−0.0956 (2)	0.9002 (2)	−0.09610 (8)	0.0622 (5)

H20	−0.1291	0.9535	−0.1291	0.075*
C21	0.0325 (2)	0.8133 (2)	−0.09660 (8)	0.0631 (5)
H21	0.0861	0.8073	−0.1301	0.076*
C22	0.0831 (2)	0.7341 (2)	−0.04748 (7)	0.0507 (4)
H22	0.1705	0.6753	−0.0483	0.061*

Atomic displacement parameters (Å^2)

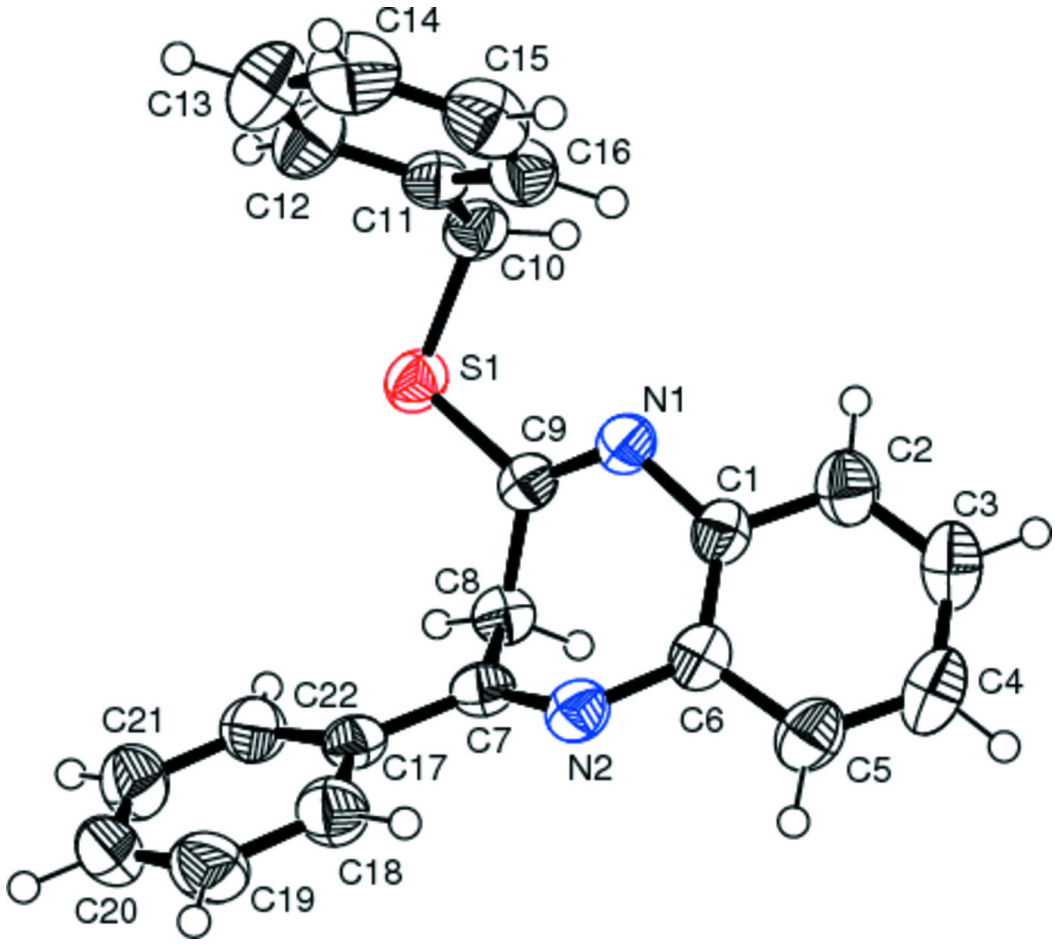
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0448 (2)	0.0518 (3)	0.0487 (3)	−0.00462 (19)	0.00941 (19)	−0.00117 (19)
N1	0.0419 (8)	0.0437 (7)	0.0435 (8)	−0.0033 (6)	0.0037 (6)	0.0006 (6)
N2	0.0388 (8)	0.0449 (7)	0.0488 (8)	−0.0008 (6)	0.0043 (6)	0.0011 (6)
C1	0.0467 (10)	0.0380 (8)	0.0413 (8)	0.0005 (7)	0.0081 (7)	−0.0007 (7)
C2	0.0562 (11)	0.0586 (11)	0.0465 (10)	0.0071 (9)	0.0074 (8)	0.0031 (8)
C3	0.0700 (14)	0.0624 (12)	0.0503 (11)	0.0128 (10)	0.0176 (10)	0.0136 (9)
C4	0.0641 (13)	0.0535 (11)	0.0674 (12)	0.0023 (9)	0.0274 (10)	0.0117 (9)
C5	0.0453 (10)	0.0537 (10)	0.0624 (11)	0.0029 (8)	0.0150 (8)	0.0028 (9)
C6	0.0436 (9)	0.0367 (8)	0.0465 (9)	0.0023 (7)	0.0089 (7)	0.0004 (7)
C7	0.0379 (9)	0.0361 (8)	0.0465 (9)	−0.0041 (6)	−0.0009 (7)	−0.0047 (7)
C8	0.0424 (9)	0.0394 (8)	0.0454 (9)	0.0008 (7)	0.0033 (7)	−0.0051 (7)
C9	0.0365 (8)	0.0339 (8)	0.0463 (9)	0.0018 (6)	0.0027 (7)	0.0013 (7)
C10	0.0337 (8)	0.0529 (10)	0.0573 (10)	−0.0011 (7)	0.0009 (7)	0.0047 (8)
C11	0.0311 (8)	0.0497 (9)	0.0480 (9)	−0.0019 (7)	−0.0017 (7)	0.0007 (7)
C12	0.0589 (12)	0.0553 (11)	0.0687 (12)	−0.0033 (9)	0.0208 (10)	0.0007 (9)
C13	0.0736 (14)	0.0525 (12)	0.0852 (15)	−0.0033 (10)	0.0160 (12)	0.0089 (10)
C14	0.0633 (13)	0.0542 (11)	0.0823 (15)	0.0076 (10)	0.0014 (11)	−0.0104 (11)
C15	0.0589 (12)	0.0746 (14)	0.0547 (11)	0.0090 (10)	0.0036 (9)	−0.0144 (10)
C16	0.0462 (10)	0.0633 (12)	0.0481 (10)	−0.0022 (8)	0.0012 (8)	0.0009 (8)
C17	0.0397 (8)	0.0384 (8)	0.0442 (9)	−0.0073 (6)	−0.0015 (7)	−0.0049 (7)
C18	0.0449 (10)	0.0575 (11)	0.0553 (11)	0.0043 (8)	0.0007 (8)	0.0017 (9)
C19	0.0490 (11)	0.0657 (12)	0.0698 (13)	0.0063 (9)	−0.0098 (10)	0.0060 (10)
C20	0.0692 (14)	0.0648 (12)	0.0511 (11)	−0.0076 (10)	−0.0153 (10)	0.0086 (9)
C21	0.0725 (14)	0.0702 (13)	0.0467 (11)	−0.0054 (11)	0.0038 (10)	0.0010 (9)
C22	0.0494 (10)	0.0539 (10)	0.0488 (10)	−0.0009 (8)	0.0028 (8)	−0.0010 (8)

Geometric parameters (Å, °)

S1—C9	1.7561 (15)	C10—H10B	0.9700
S1—C10	1.8132 (18)	C11—C16	1.382 (2)
N1—C9	1.2759 (19)	C11—C12	1.385 (2)
N1—C1	1.4133 (19)	C12—C13	1.380 (3)
N2—C7	1.2905 (19)	C12—H12	0.9300
N2—C6	1.408 (2)	C13—C14	1.367 (3)
C1—C6	1.400 (2)	C13—H13	0.9300
C1—C2	1.401 (2)	C14—C15	1.371 (3)
C2—C3	1.371 (2)	C14—H14	0.9300
C2—H2	0.9300	C15—C16	1.381 (3)
C3—C4	1.383 (3)	C15—H15	0.9300
C3—H3	0.9300	C16—H16	0.9300

C4—C5	1.369 (3)	C17—C22	1.387 (2)
C4—H4	0.9300	C17—C18	1.394 (2)
C5—C6	1.407 (2)	C18—C19	1.374 (2)
C5—H5	0.9300	C18—H18	0.9300
C7—C17	1.481 (2)	C19—C20	1.370 (3)
C7—C8	1.512 (2)	C19—H19	0.9300
C8—C9	1.500 (2)	C20—C21	1.365 (3)
C8—H8A	0.9700	C20—H20	0.9300
C8—H8B	0.9700	C21—C22	1.385 (3)
C10—C11	1.503 (2)	C21—H21	0.9300
C10—H10A	0.9700	C22—H22	0.9300
C9—S1—C10	102.10 (8)	H10A—C10—H10B	107.6
C9—N1—C1	119.68 (14)	C16—C11—C12	118.37 (16)
C7—N2—C6	121.16 (14)	C16—C11—C10	120.79 (15)
C6—C1—C2	118.88 (15)	C12—C11—C10	120.84 (15)
C6—C1—N1	125.70 (14)	C13—C12—C11	120.67 (18)
C2—C1—N1	115.33 (15)	C13—C12—H12	119.7
C3—C2—C1	121.43 (18)	C11—C12—H12	119.7
C3—C2—H2	119.3	C14—C13—C12	120.39 (19)
C1—C2—H2	119.3	C14—C13—H13	119.8
C2—C3—C4	119.69 (18)	C12—C13—H13	119.8
C2—C3—H3	120.2	C13—C14—C15	119.58 (18)
C4—C3—H3	120.2	C13—C14—H14	120.2
C5—C4—C3	119.98 (17)	C15—C14—H14	120.2
C5—C4—H4	120.0	C14—C15—C16	120.43 (18)
C3—C4—H4	120.0	C14—C15—H15	119.8
C4—C5—C6	121.35 (18)	C16—C15—H15	119.8
C4—C5—H5	119.3	C15—C16—C11	120.55 (17)
C6—C5—H5	119.3	C15—C16—H16	119.7
C1—C6—C5	118.40 (15)	C11—C16—H16	119.7
C1—C6—N2	125.23 (14)	C22—C17—C18	117.57 (16)
C5—C6—N2	116.13 (15)	C22—C17—C7	122.88 (15)
N2—C7—C17	118.13 (14)	C18—C17—C7	119.55 (15)
N2—C7—C8	120.23 (14)	C19—C18—C17	120.97 (17)
C17—C7—C8	121.64 (14)	C19—C18—H18	119.5
C9—C8—C7	107.20 (12)	C17—C18—H18	119.5
C9—C8—H8A	110.3	C20—C19—C18	120.47 (18)
C7—C8—H8A	110.3	C20—C19—H19	119.8
C9—C8—H8B	110.3	C18—C19—H19	119.8
C7—C8—H8B	110.3	C21—C20—C19	119.77 (18)
H8A—C8—H8B	108.5	C21—C20—H20	120.1
N1—C9—C8	123.62 (14)	C19—C20—H20	120.1
N1—C9—S1	122.14 (13)	C20—C21—C22	120.28 (18)
C8—C9—S1	114.16 (11)	C20—C21—H21	119.9
C11—C10—S1	114.59 (12)	C22—C21—H21	119.9
C11—C10—H10A	108.6	C21—C22—C17	120.94 (18)
S1—C10—H10A	108.6	C21—C22—H22	119.5
C11—C10—H10B	108.6	C17—C22—H22	119.5
S1—C10—H10B	108.6		

Fig. 1


C14
C15
C13
C16
C12
C11
C10
S1
N1
C9
C2
C1
C3
C8
C4
C22
C21
C7
C6
C17
N2
C5
C18
C20
C19