2-{4-Methyl-N-[(2,3,4,9-tetrahydro-1H-carbazol-3-yl)methyl]benzenesulfon-amido}ethyl 4-methylbenzenesulfonate

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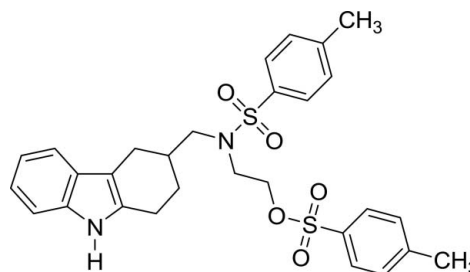Correspondence e-mail: merzifon@hacettepe.edu.tr

In the title compound, $C_{29}H_{32}N_2O_5S_2$, the indole ring system is nearly planar, with a maximum deviation of 0.013 (2) Å, and the cyclohexenone ring has an envelope conformation with the methine C atom as the flap. The two methylbenzene rings are approximately perpendicular to each other, making a dihedral angle of 89.09 (8)°. In the crystal, N—H···O hydrogen bonds link the molecules into a chain running along the a-axis direction, and weak C—H···O hydrogen bonds and C—H···π interactions are observed between the chains.

Related literature

For tetrahydrocarbazole systems present in the framework of a number of indole-type alkaloids of biological interest, see: Saxton (1983). For related structures, see: Hökelek *et al.* (2009); Çaylak *et al.* (2007); Uludağ *et al.* (2009); Gündoğdu *et al.* (2011). For the use of tetrahydrocarbazolone in the synthesis of central-nervous-system-active drugs, see: Romeo *et al.* (2006). For the syntheses of tetrahydrocarbazolone-based antitumor-active compounds from tetrahydro-carbazoles, see: Chen *et al.* (2009). For the syntheses of aminotetrahydrocarbazoles as central nervous system agents, see: Mooradian *et al.* (1977). For bond-length data, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_{29}H_{32}N_2O_5S_2$	$V = 2741.9$ (2) Å^3
$M_r = 552.69$	$Z = 4$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
$a = 10.5719$ (5) Å	$\mu = 2.11$ mm^{-1}
$b = 10.8783$ (6) Å	$T = 150$ K
$c = 23.8868$ (11) Å	$0.21 \times 0.16 \times 0.13$ mm
$\beta = 93.507$ (2)°	

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	168526 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	5101 independent reflections
$T_{min} = 0.701$, $T_{max} = 0.761$	4955 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	
$S = 1.05$	
5101 reflections	$\Delta\rho_{max} = 0.43$ e Å^{-3}
349 parameters	$\Delta\rho_{min} = -0.43$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

$Cg2$ and $Cg5$ are the centroids of the C4A/C5A/C8A/N9/C9A and C20–C25 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N9—H9···O5^i	0.84 (2)	2.18 (2)	2.9561 (19)	154.1 (18)
C21—H21···O4^{ii}	0.95	2.38	3.230 (2)	148
C8—H8···$Cg5^{iii}$	0.95	2.81	3.5127 (18)	131
C10—H10B···$Cg2^{iv}$	0.99	2.58	3.5538 (18)	168

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 2, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

organic compounds

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2005). *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.

Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc. Madison, Wisconsin, USA.

Çaylak, N., Hökelek, T., Uludağ, N. & Patır, S. (2007). *Acta Cryst.* E**63**, o3913–o3914.

Chen, J., Lou, J., Liu, T., Wu, R., Dong, X., He, Q., Yang, B. & Hu, Y. (2009). *Arch. Pharm.* **342**, 165–172.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

Gündoğdu, C., Göçmentürk, M., Ergün, Y., Tercan, B. & Hökelek, T. (2011). *Acta Cryst.* E**67**, o1470–o1471.

Hökelek, T., Dal, H., Tercan, B., Göçmentürk, M. & Ergün, Y. (2009). *Acta Cryst.* E**65**, o1702–o1703.

Mooradian, A., Dupont, P. E., Hlavac, A. G., Aceto, M. D. & Pearl, J. (1977). *J. Med. Chem.* **20**, 487–492.

Romeo, G., Materia, L., Pittalá, V., Modica, M., Salerno, L., Siracusa, M., Russo, F. & Minneman, K. P. (2006). *Bioorg. Med. Chem.* **14**, 5211–5219.

Saxton, J. E. (1983). Editor. *Heterocyclic Compounds*, Vol. 25, *The Monoterpenoid Indole Alkaloids*, ch. 8 and 11. New York: Wiley.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Uludağ, N., Öztürk, A., Hökelek, T. & Erdoğan, Ü. I. (2009). *Acta Cryst.* E**65**, o595–o596.

2-{4-Methyl-*N*-[(2,3,4,9-tetrahydro-1*H*-carbazol-3-yl)methyl]benzene-sulfonamido}ethyl 4-methylbenzenesulfonate

Mustafa Göçmentürk, Yavuz Ergün, Berline Mougang-Soume, Nagihan Çaylak Delibaş and Tuncer Hökelek

1. Comment

Tetrahydrocarbazole systems are present in the framework of a number of indole-type alkaloids of biological interest (Saxton, 1983). The structures of tricyclic, tetracyclic and pentacyclic ring systems with dithiolane and other substituents of the tetrahydrocarbazole core, have been reported previously. Tetrahydrocarbazoles have been increasingly important intermediates in the syntheses of indole or carbazole alkaloids and various biologically active heterocyclic compounds because of their unique structures. For instance, tetrahydrocarbazole was used in the syntheses of central nervous system active drugs (Romeo *et al.*, 2006). Tetrahydrocarbazolone based antitumor active compounds were synthesized from tetrahydrocarbazoles (Chen *et al.*, 2009). Aminotetrahydrocarbazoles were also synthesized as central nervous system agents (Mooradian *et al.*, 1977). The present study was undertaken to ascertain the crystal structure of the title compound.

The molecule of the title compound contains a carbazole skeleton with methyl phenylsulfonamide and ethyl methyl benzenesulfonate groups, (Fig. 1), where the bond lengths are close to standard values (Allen *et al.*, 1987) and generally agree with those in the previously reported compounds. In all structures atom N9 is substituted.

An examination of the deviations from the least-squares planes through individual rings shows that rings *B* (C4a/C5a/C8a/N9/C9a) and *C* (C5a/C5—C8/C8a) are nearly coplanar [with a maximum deviation of 0.013 (2) Å for atom C7] with dihedral angle of *B*/*C* = 0.85 (6)°. Ring *A* (C1—C4/C4a/C9a) adopts envelope conformation with atom C3 displaced by -0.605 (2) Å from the plane of the other rings atoms, as in 3a,4,10,10b-tetrahydro-2*H*-furo[2,3-a]carbazol-5(3H)-one (Çaylak *et al.*, 2007), 3,3-ethylenedithio-3,3a,4,5,10,10b-hexahydro-2*H*-furo[2,3-a]carbazole (Uludağ *et al.*, 2009), ethyl 1-oxo-1,2,3,4-tetrahydro-9*H*-carbazole-3-carboxylate (Hökelek *et al.*, 2009) and ethyl 4-oxo-2,3,4,9-tetrahydro-1*H*-carbazole-3-carboxylate (Gündoğdu *et al.*, 2011). Ring *A* has a pseudo twofold axis running through the midpoints of C2–C3 and C4a–C9a bonds. Rings *D* (C13—C18) and *E* (C20—C25) are oriented at a dihedral angle of 89.09 (8)°.

In the crystal, intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into infinite chains along the a-axis (Table 1 and Fig. 2). π···π contacts between the benzene rings, Cg4—Cg4[i] [symmetry code: (i) 1 - *x*, 1 - *y*, 1 - *z*, where Cg4 is the centroid of the ring *D* (C13—C18)] may further stabilize the structure, with centroid-centroid distance of 3.955 (1) Å. There also exist two weak C—H···π interactions (Table 1).

2. Experimental

For the preparation of the title compound, (I), a solution of 2-((2,3,4,9 -tetrahydro-1*H*-carbazole-3-yl)methylamino)-ethanol (1.0 g, 4.1 mmol) in pyridine (5 ml) was cooled to 273 K. Then, p-toluenesulphonyl chloride (1.7 g, 9.0 mmol) was added. The mixture was stirred for 18 h at room temperature, and then washed with hydrochloric acid (10%). The

organic layer was extracted with chloroform and dried over anhydrous magnesium sulfate. The solvent was removed under reduced pressure. The crude product was purified by silica gel column chromatography eluting with ethyl acetate:hexane (1:1). The solvent was evaporated under reduced pressure and the residue was recrystallized from methanol (yield; 1.7 g, 75%, m.p. 425 K).

3. Refinement

The H9 atom is located in a difference Fourier synthesis and refined isotropically. The remaining C-bound H-atoms were positioned geometrically with C—H = 0.95, 1.00, 0.99 and 0.98 Å, for aromatic, methine, methylene and methyl H-atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.5 for methyl H-atoms and k = 1.2 for all other H-atoms.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).
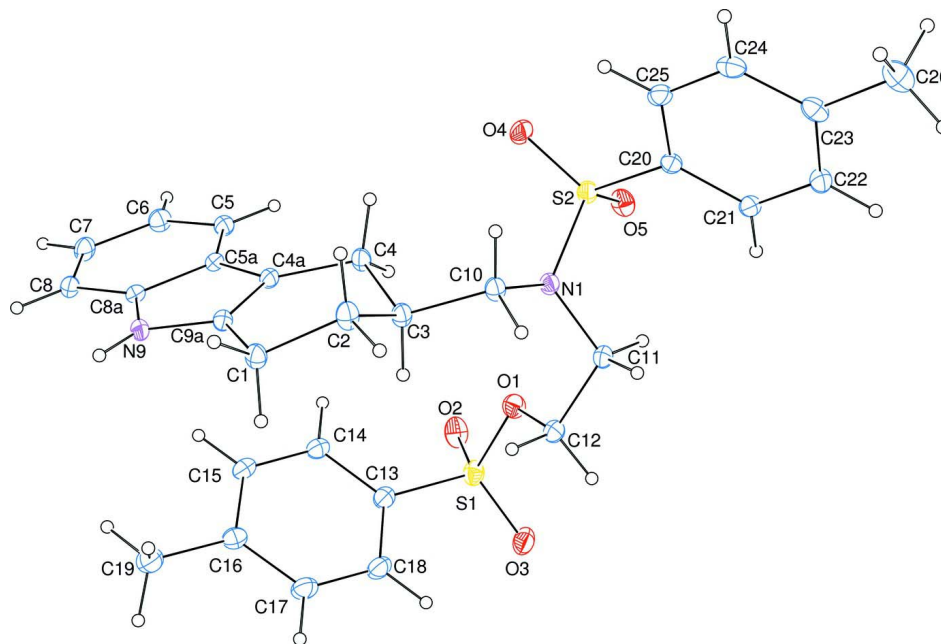
Figure 1

The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.
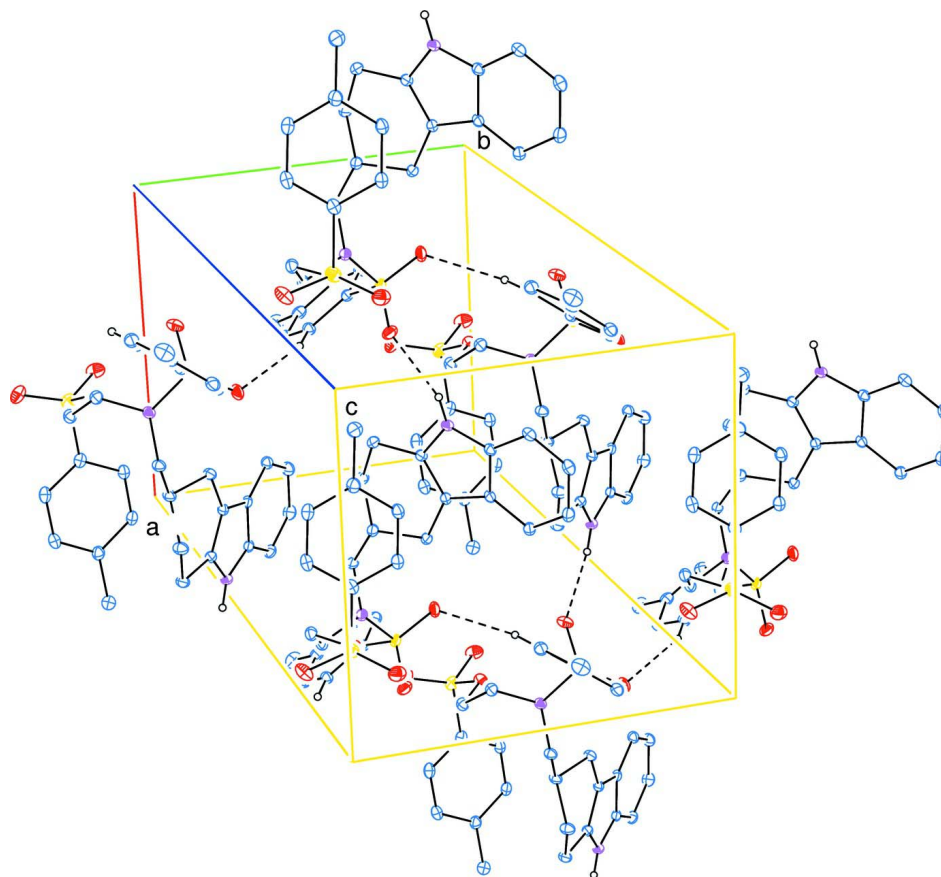
Figure 2

A view of the crystal packing of the title compound. The N—H···O and C—H···O hydrogen bonds are shown as dashed lines [H-atoms not involved in hydrogen bonding have been omitted for clarity].

2-{4-Methyl-*N*-[(2,3,4,9-tetrahydro-1*H*-carbazol-3-yl)methyl]benzenesulfonamido}ethyl 4-methylbenzenesulfonate

Crystal data

$C_{29}H_{32}N_2O_5S_2$	$F(000) = 1168$
$M_r = 552.69$	$D_x = 1.339$ Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Hall symbol: -P 2ybc	Cell parameters from 9835 reflections
$a = 10.5719$ (5) Å	$\theta = 3.7$–69.1°
$b = 10.8783$ (6) Å	$\mu = 2.11$ mm^{-1}
$c = 23.8868$ (11) Å	$T = 150$ K
$\beta = 93.507$ (2)°	Block, pink
$V = 2741.9$ (2) Å^3	$0.21 \times 0.16 \times 0.13$ mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
Radiation source: fine-focus sealed tube	$T_{min} = 0.701$, $T_{max} = 0.761$
Graphite monochromator	168526 measured reflections
φ and ω scans	5101 independent reflections

4955 reflections with $I > 2\sigma(I)$

$R_{int} = 0.048$

$\theta_{max} = 69.3°$, $\theta_{min} = 3.7°$

$h = -12 \rightarrow 12$

$k = -12 \rightarrow 9$

$l = -28 \rightarrow 28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.042$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.116$	
$S = 1.05$	H atoms treated by a mixture of independent and constrained refinement
5101 reflections	
349 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0702P)^2 + 1.3803P]$
0 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} = 0.001$
	$\Delta\rho_{max} = 0.43$ e Å⁻³
	$\Delta\rho_{min} = -0.43$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F² against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F², conventional R-factors R are based on F, with F set to zero for negative F². The threshold expression of F² > 2sigma(F²) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F² 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 (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.74839 (4)	0.45706 (4)	0.440644 (18)	0.03720 (14)
S2	0.91673 (4)	0.22863 (4)	0.250089 (17)	0.03013 (13)
O1	0.79745 (12)	0.41437 (12)	0.38233 (5)	0.0376 (3)
O2	0.79715 (13)	0.36599 (15)	0.47877 (6)	0.0483 (4)
O3	0.77959 (14)	0.58320 (14)	0.44931 (6)	0.0477 (3)
O4	0.85386 (12)	0.11380 (11)	0.23933 (6)	0.0414 (3)
O5	1.01947 (11)	0.23485 (13)	0.29196 (6)	0.0401 (3)
N1	0.81003 (12)	0.32611 (12)	0.26881 (6)	0.0279 (3)
N9	0.26066 (13)	0.12277 (12)	0.33533 (6)	0.0274 (3)
H9	0.183 (2)	0.1403 (18)	0.3313 (8)	0.029 (5)*
C1	0.33412 (15)	0.27199 (16)	0.26254 (7)	0.0323 (4)
H1A	0.2622	0.2488	0.2362	0.039*
H1B	0.3134	0.3508	0.2806	0.039*
C2	0.45468 (16)	0.28622 (18)	0.23078 (8)	0.0364 (4)
H2A	0.4602	0.2173	0.2040	0.044*
H2B	0.4497	0.3636	0.2090	0.044*
C3	0.57387 (15)	0.28803 (15)	0.26999 (7)	0.0309 (4)
H3	0.5629	0.3536	0.2986	0.037*
C4	0.59305 (14)	0.16623 (15)	0.30117 (7)	0.0289 (3)
H4A	0.6225	0.1027	0.2753	0.035*
H4B	0.6585	0.1761	0.3323	0.035*
C4A	0.47025 (14)	0.12658 (14)	0.32400 (6)	0.0256 (3)

C5	0.52340 (16)	−0.03680 (15)	0.40220 (7)	0.0297 (3)
H5	0.6127	−0.0374	0.3996	0.036*
C5A	0.44685 (15)	0.04014 (13)	0.36752 (6)	0.0252 (3)
C6	0.46753 (17)	−0.11183 (16)	0.44023 (7)	0.0358 (4)
H6	0.5192	−0.1639	0.4640	0.043*
C7	0.33587 (17)	−0.11239 (17)	0.44440 (8)	0.0365 (4)
H7	0.2996	−0.1660	0.4704	0.044*
C8	0.25776 (16)	−0.03621 (15)	0.41124 (7)	0.0314 (4)
H8	0.1686	−0.0359	0.4144	0.038*
C8A	0.31414 (15)	0.03974 (14)	0.37326 (6)	0.0258 (3)
C9A	0.35577 (14)	0.17487 (14)	0.30590 (7)	0.0268 (3)
C10	0.68646 (14)	0.32474 (15)	0.23591 (7)	0.0292 (3)
H10A	0.6918	0.2666	0.2043	0.035*
H10B	0.6703	0.4076	0.2199	0.035*
C11	0.85600 (16)	0.44698 (15)	0.28894 (7)	0.0308 (3)
H11A	0.8517	0.5055	0.2571	0.037*
H11B	0.9460	0.4394	0.3026	0.037*
C12	0.78106 (16)	0.49826 (16)	0.33539 (7)	0.0324 (4)
H12A	0.8126	0.5811	0.3463	0.039*
H12B	0.6903	0.5048	0.3229	0.039*
C13	0.58318 (17)	0.44288 (16)	0.43154 (7)	0.0321 (4)
C14	0.52784 (17)	0.32846 (16)	0.43769 (7)	0.0328 (4)
H14	0.5789	0.2582	0.4462	0.039*
C15	0.39759 (17)	0.31795 (16)	0.43132 (7)	0.0332 (4)
H15	0.3594	0.2399	0.4361	0.040*
C16	0.32127 (17)	0.41895 (16)	0.41813 (7)	0.0344 (4)
C17	0.37962 (19)	0.53315 (17)	0.41211 (8)	0.0411 (4)
H17	0.3288	0.6033	0.4032	0.049*
C18	0.50931 (19)	0.54588 (16)	0.41887 (8)	0.0393 (4)
H18	0.5477	0.6241	0.4149	0.047*
C19	0.17940 (19)	0.40483 (19)	0.41047 (9)	0.0445 (4)
H19A	0.1532	0.4090	0.3704	0.067*
H19B	0.1545	0.3252	0.4256	0.067*
H19C	0.1383	0.4711	0.4304	0.067*
C20	0.97518 (15)	0.27802 (14)	0.18609 (7)	0.0283 (3)
C21	1.05368 (15)	0.38076 (15)	0.18504 (7)	0.0305 (3)
H21	1.0743	0.4261	0.2183	0.037*
C22	1.10105 (16)	0.41571 (17)	0.13478 (8)	0.0346 (4)
H22	1.1539	0.4862	0.1338	0.041*
C23	1.07314 (16)	0.34982 (18)	0.08539 (7)	0.0363 (4)
C24	0.99322 (17)	0.24845 (17)	0.08763 (8)	0.0374 (4)
H24	0.9725	0.2028	0.0544	0.045*
C25	0.94355 (16)	0.21302 (16)	0.13735 (8)	0.0338 (4)
H25	0.8880	0.1445	0.1381	0.041*
C26	1.1295 (2)	0.3854 (2)	0.03154 (9)	0.0509 (5)
H26A	1.0676	0.3703	0.0000	0.076*
H26B	1.2058	0.3363	0.0267	0.076*
H26C	1.1518	0.4728	0.0327	0.076*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0384 (2)	0.0419 (3)	0.0316 (2)	−0.00848 (18)	0.00467 (17)	0.00134 (17)
S2	0.0248 (2)	0.0244 (2)	0.0415 (2)	0.00333 (14)	0.00396 (16)	0.00732 (15)
O1	0.0419 (7)	0.0361 (7)	0.0354 (6)	0.0008 (5)	0.0079 (5)	0.0068 (5)
O2	0.0413 (7)	0.0644 (9)	0.0388 (7)	−0.0054 (6)	−0.0007 (6)	0.0135 (6)
O3	0.0521 (8)	0.0474 (8)	0.0444 (7)	−0.0204 (7)	0.0113 (6)	−0.0087 (6)
O4	0.0390 (7)	0.0218 (6)	0.0642 (8)	0.0015 (5)	0.0102 (6)	0.0080 (5)
O5	0.0288 (6)	0.0466 (8)	0.0446 (7)	0.0086 (5)	0.0008 (5)	0.0120 (6)
N1	0.0234 (6)	0.0258 (7)	0.0348 (7)	0.0009 (5)	0.0039 (5)	0.0044 (5)
N9	0.0202 (6)	0.0273 (7)	0.0347 (7)	−0.0013 (5)	0.0028 (5)	0.0003 (5)
C1	0.0244 (8)	0.0316 (9)	0.0406 (9)	0.0017 (6)	0.0001 (7)	0.0076 (7)
C2	0.0299 (9)	0.0392 (10)	0.0399 (9)	0.0004 (7)	0.0012 (7)	0.0130 (7)
C3	0.0254 (8)	0.0277 (8)	0.0397 (9)	0.0013 (6)	0.0035 (6)	0.0087 (7)
C4	0.0231 (7)	0.0272 (8)	0.0367 (8)	0.0014 (6)	0.0032 (6)	0.0078 (6)
C4A	0.0251 (7)	0.0213 (7)	0.0305 (8)	−0.0009 (6)	0.0022 (6)	0.0014 (6)
C5	0.0291 (8)	0.0261 (8)	0.0342 (8)	0.0000 (6)	0.0032 (6)	0.0017 (6)
C5A	0.0263 (8)	0.0203 (7)	0.0292 (8)	−0.0031 (6)	0.0041 (6)	−0.0023 (6)
C6	0.0398 (9)	0.0306 (9)	0.0370 (9)	0.0007 (7)	0.0024 (7)	0.0077 (7)
C7	0.0417 (10)	0.0322 (9)	0.0365 (9)	−0.0075 (7)	0.0092 (7)	0.0056 (7)
C8	0.0294 (8)	0.0302 (8)	0.0351 (9)	−0.0067 (6)	0.0070 (7)	−0.0034 (6)
C8A	0.0266 (8)	0.0217 (7)	0.0294 (8)	−0.0024 (6)	0.0030 (6)	−0.0049 (6)
C9A	0.0237 (7)	0.0252 (8)	0.0317 (8)	−0.0020 (6)	0.0020 (6)	−0.0002 (6)
C10	0.0250 (8)	0.0292 (8)	0.0336 (8)	0.0012 (6)	0.0025 (6)	0.0076 (6)
C11	0.0299 (8)	0.0283 (8)	0.0347 (8)	−0.0031 (6)	0.0063 (6)	0.0035 (6)
C12	0.0338 (8)	0.0286 (8)	0.0350 (8)	−0.0012 (7)	0.0046 (7)	0.0037 (7)
C13	0.0396 (9)	0.0304 (8)	0.0267 (8)	−0.0045 (7)	0.0053 (7)	−0.0013 (6)
C14	0.0405 (9)	0.0257 (8)	0.0323 (8)	0.0002 (7)	0.0024 (7)	0.0004 (6)
C15	0.0422 (9)	0.0247 (8)	0.0325 (8)	−0.0039 (7)	0.0008 (7)	−0.0021 (6)
C16	0.0408 (9)	0.0308 (9)	0.0314 (8)	0.0003 (7)	0.0006 (7)	−0.0035 (7)
C17	0.0469 (11)	0.0262 (9)	0.0500 (11)	0.0044 (7)	0.0014 (8)	0.0023 (7)
C18	0.0503 (11)	0.0253 (9)	0.0428 (10)	−0.0047 (7)	0.0074 (8)	0.0013 (7)
C19	0.0429 (10)	0.0385 (10)	0.0513 (11)	0.0006 (8)	−0.0037 (8)	−0.0032 (8)
C20	0.0227 (7)	0.0241 (8)	0.0382 (9)	0.0043 (6)	0.0032 (6)	0.0000 (6)
C21	0.0270 (8)	0.0272 (8)	0.0375 (9)	−0.0006 (6)	0.0031 (6)	−0.0041 (6)
C22	0.0288 (8)	0.0327 (9)	0.0428 (9)	−0.0009 (7)	0.0070 (7)	0.0011 (7)
C23	0.0312 (8)	0.0413 (10)	0.0367 (9)	0.0114 (7)	0.0052 (7)	0.0007 (7)
C24	0.0337 (9)	0.0375 (9)	0.0404 (9)	0.0098 (7)	−0.0010 (7)	−0.0104 (8)
C25	0.0274 (8)	0.0263 (8)	0.0475 (10)	0.0029 (6)	0.0007 (7)	−0.0063 (7)
C26	0.0508 (12)	0.0624 (14)	0.0406 (10)	0.0102 (10)	0.0119 (9)	0.0045 (9)

Geometric parameters (Å, º)

S1—O1	1.5854 (13)	C9A—C1	1.487 (2)
S1—O2	1.4209 (15)	C9A—C4A	1.365 (2)
S1—O3	1.4234 (15)	C10—H10A	0.9900
S1—C13	1.7536 (18)	C10—H10B	0.9900
S2—O4	1.4308 (13)	C11—C12	1.509 (2)
S2—O5	1.4325 (13)	C11—H11A	0.9900

S2—N1	1.6305 (14)	C11—H11B	0.9900
S2—C20	1.7672 (17)	C12—H12A	0.9900
O1—C12	1.448 (2)	C12—H12B	0.9900
N1—C10	1.483 (2)	C13—C14	1.387 (2)
N1—C11	1.472 (2)	C13—C18	1.388 (3)
N9—C8A	1.376 (2)	C14—C15	1.381 (3)
N9—C9A	1.383 (2)	C14—H14	0.9500
N9—H9	0.84 (2)	C15—C16	1.388 (3)
C1—C2	1.530 (2)	C15—H15	0.9500
C1—H1A	0.9900	C16—C17	1.398 (3)
C1—H1B	0.9900	C16—C19	1.508 (3)
C2—C3	1.523 (2)	C17—C18	1.378 (3)
C2—H2A	0.9900	C17—H17	0.9500
C2—H2B	0.9900	C18—H18	0.9500
C3—C4	1.527 (2)	C19—H19A	0.9800
C3—C10	1.536 (2)	C19—H19B	0.9800
C3—H3	1.0000	C19—H19C	0.9800
C4—C4A	1.502 (2)	C20—C21	1.393 (2)
C4—H4A	0.9900	C20—C25	1.385 (2)
C4—H4B	0.9900	C21—C22	1.382 (2)
C5—C6	1.381 (2)	C21—H21	0.9500
C5—H5	0.9500	C22—C23	1.396 (3)
C5A—C4A	1.434 (2)	C22—H22	0.9500
C5A—C5	1.400 (2)	C23—C24	1.392 (3)
C6—C7	1.401 (3)	C23—C26	1.501 (3)
C6—H6	0.9500	C24—C25	1.382 (3)
C7—C8	1.384 (3)	C24—H24	0.9500
C7—H7	0.9500	C25—H25	0.9500
C8—H8	0.9500	C26—H26A	0.9800
C8A—C5A	1.418 (2)	C26—H26B	0.9800
C8A—C8	1.388 (2)	C26—H26C	0.9800
O1—S1—C13	104.01 (7)	N1—C10—C3	114.07 (13)
O2—S1—O1	103.50 (8)	N1—C10—H10A	108.7
O2—S1—O3	120.48 (9)	N1—C10—H10B	108.7
O2—S1—C13	109.74 (8)	C3—C10—H10A	108.7
O3—S1—O1	108.95 (8)	C3—C10—H10B	108.7
O3—S1—C13	108.84 (9)	H10A—C10—H10B	107.6
O4—S2—O5	119.39 (8)	N1—C11—C12	113.12 (13)
O4—S2—N1	107.19 (7)	N1—C11—H11A	109.0
O4—S2—C20	107.05 (8)	N1—C11—H11B	109.0
O5—S2—N1	106.59 (8)	C12—C11—H11A	109.0
O5—S2—C20	107.42 (8)	C12—C11—H11B	109.0
N1—S2—C20	108.90 (7)	H11A—C11—H11B	107.8
C12—O1—S1	117.76 (11)	O1—C12—C11	107.04 (14)
C10—N1—S2	116.74 (11)	O1—C12—H12A	110.3
C11—N1—S2	116.77 (10)	O1—C12—H12B	110.3
C11—N1—C10	116.39 (13)	C11—C12—H12A	110.3
C8A—N9—C9A	108.75 (13)	C11—C12—H12B	110.3

C8A—N9—H9	125.7 (13)	H12A—C12—H12B	108.6
C9A—N9—H9	125.5 (13)	C14—C13—S1	119.29 (14)
C2—C1—H1A	110.0	C14—C13—C18	120.80 (17)
C2—C1—H1B	110.0	C18—C13—S1	119.91 (14)
C9A—C1—C2	108.65 (13)	C13—C14—H14	120.4
C9A—C1—H1A	110.0	C15—C14—C13	119.13 (16)
C9A—C1—H1B	110.0	C15—C14—H14	120.4
H1B—C1—H1A	108.3	C14—C15—C16	121.43 (16)
C1—C2—H2A	109.1	C14—C15—H15	119.3
C1—C2—H2B	109.1	C16—C15—H15	119.3
C3—C2—C1	112.28 (14)	C15—C16—C17	118.20 (17)
C3—C2—H2A	109.1	C15—C16—C19	120.47 (16)
C3—C2—H2B	109.1	C17—C16—C19	121.33 (17)
H2A—C2—H2B	107.9	C16—C17—H17	119.4
C2—C3—C4	111.63 (14)	C18—C17—C16	121.29 (17)
C2—C3—C10	108.43 (14)	C18—C17—H17	119.4
C2—C3—H3	107.7	C13—C18—H18	120.4
C4—C3—C10	113.59 (13)	C17—C18—C13	119.14 (17)
C4—C3—H3	107.7	C17—C18—H18	120.4
C10—C3—H3	107.7	C16—C19—H19C	109.5
C3—C4—H4A	109.8	C16—C19—H19B	109.5
C3—C4—H4B	109.8	C16—C19—H19A	109.5
C4A—C4—C3	109.53 (13)	H19C—C19—H19B	109.5
C4A—C4—H4A	109.8	H19C—C19—H19A	109.5
C4A—C4—H4B	109.8	H19B—C19—H19A	109.5
H4A—C4—H4B	108.2	C21—C20—S2	119.98 (13)
C5A—C4A—C4	129.99 (14)	C25—C20—S2	119.51 (13)
C9A—C4A—C4	122.95 (14)	C25—C20—C21	120.51 (16)
C9A—C4A—C5A	107.03 (13)	C20—C21—H21	120.5
C5A—C5—H5	120.4	C22—C21—C20	118.96 (16)
C6—C5—C5A	119.20 (15)	C22—C21—H21	120.5
C6—C5—H5	120.4	C21—C22—C23	121.57 (17)
C5—C5A—C4A	134.57 (14)	C21—C22—H22	119.2
C5—C5A—C8A	118.71 (14)	C23—C22—H22	119.2
C8A—C5A—C4A	106.72 (13)	C22—C23—C26	121.15 (18)
C5—C6—C7	121.07 (16)	C24—C23—C22	118.13 (16)
C5—C6—H6	119.5	C24—C23—C26	120.71 (18)
C7—C6—H6	119.5	C23—C24—H24	119.4
C6—C7—H7	119.4	C25—C24—C23	121.14 (16)
C8—C7—C6	121.13 (15)	C25—C24—H24	119.4
C8—C7—H7	119.4	C20—C25—H25	120.2
C7—C8—C8A	117.73 (15)	C24—C25—C20	119.66 (16)
C7—C8—H8	121.1	C24—C25—H25	120.2
C8A—C8—H8	121.1	C23—C26—H26B	109.5
N9—C8A—C5A	107.66 (13)	C23—C26—H26C	109.5
N9—C8A—C8	130.19 (15)	C23—C26—H26A	109.5
C8—C8A—C5A	122.14 (15)	H26B—C26—H26A	109.5
N9—C9A—C1	124.01 (14)	H26B—C26—H26C	109.5
C4A—C9A—N9	109.84 (14)	H26C—C26—H26A	109.5

C4A—C9A—C1	126.12 (14)		
O2—S1—O1—C12	−170.46 (12)	C5—C5A—C4A—C9A	−179.36 (17)
O3—S1—O1—C12	−41.11 (15)	C8A—C5A—C4A—C4	178.54 (16)
C13—S1—O1—C12	74.85 (13)	C8A—C5A—C4A—C9A	0.63 (17)
O1—S1—C13—C14	81.47 (14)	C4A—C5A—C5—C6	−179.06 (17)
O1—S1—C13—C18	−99.17 (15)	C8A—C5A—C5—C6	1.0 (2)
O2—S1—C13—C14	−28.72 (16)	C5—C6—C7—C8	−1.2 (3)
O2—S1—C13—C18	150.64 (15)	C6—C7—C8—C8A	0.7 (3)
O3—S1—C13—C14	−162.50 (13)	N9—C8A—C5A—C4A	−0.49 (16)
O3—S1—C13—C18	16.86 (17)	N9—C8A—C5A—C5	179.50 (14)
O4—S2—N1—C10	−43.26 (13)	C8—C8A—C5A—C4A	178.62 (14)
O4—S2—N1—C11	172.49 (12)	C8—C8A—C5A—C5	−1.4 (2)
O5—S2—N1—C10	−172.17 (11)	N9—C8A—C8—C7	179.43 (16)
O5—S2—N1—C11	43.58 (13)	C5A—C8A—C8—C7	0.5 (2)
C20—S2—N1—C10	72.23 (12)	N9—C9A—C1—C2	−167.88 (15)
C20—S2—N1—C11	−72.03 (13)	C4A—C9A—C1—C2	14.3 (2)
O4—S2—C20—C21	−172.61 (13)	N9—C9A—C4A—C4	−178.63 (14)
O4—S2—C20—C25	6.62 (15)	N9—C9A—C4A—C5A	−0.54 (18)
O5—S2—C20—C21	−43.26 (15)	C1—C9A—C4A—C4	−0.6 (3)
O5—S2—C20—C25	135.98 (13)	C1—C9A—C4A—C5A	177.54 (15)
N1—S2—C20—C21	71.81 (14)	N1—C11—C12—O1	62.68 (17)
N1—S2—C20—C25	−108.95 (13)	S1—C13—C14—C15	179.03 (13)
S1—O1—C12—C11	169.71 (11)	C18—C13—C14—C15	−0.3 (3)
S2—N1—C10—C3	116.55 (14)	S1—C13—C18—C17	−179.70 (14)
C11—N1—C10—C3	−99.06 (16)	C14—C13—C18—C17	−0.4 (3)
S2—N1—C11—C12	−144.48 (12)	C13—C14—C15—C16	1.0 (3)
C10—N1—C11—C12	71.14 (18)	C14—C15—C16—C17	−0.9 (3)
C9A—N9—C8A—C5A	0.17 (17)	C14—C15—C16—C19	179.01 (16)
C9A—N9—C8A—C8	−178.84 (16)	C15—C16—C17—C18	0.2 (3)
C8A—N9—C9A—C1	−177.89 (15)	C19—C16—C17—C18	−179.71 (18)
C8A—N9—C9A—C4A	0.24 (18)	C16—C17—C18—C13	0.4 (3)
C9A—C1—C2—C3	−44.4 (2)	S2—C20—C21—C22	178.25 (13)
C1—C2—C3—C4	63.82 (19)	C25—C20—C21—C22	−1.0 (2)
C1—C2—C3—C10	−170.31 (14)	S2—C20—C25—C24	−177.40 (13)
C2—C3—C4—C4A	−46.36 (19)	C21—C20—C25—C24	1.8 (2)
C10—C3—C4—C4A	−169.34 (14)	C20—C21—C22—C23	−0.7 (3)
C2—C3—C10—N1	−178.08 (14)	C21—C22—C23—C24	1.4 (3)
C4—C3—C10—N1	−53.4 (2)	C21—C22—C23—C26	−177.40 (17)
C3—C4—C4A—C5A	−161.21 (16)	C22—C23—C24—C25	−0.6 (3)
C3—C4—C4A—C9A	16.4 (2)	C26—C23—C24—C25	178.28 (17)
C5A—C5—C6—C7	0.3 (3)	C23—C24—C25—C20	−1.1 (3)
C5—C5A—C4A—C4	−1.4 (3)		

Hydrogen-bond geometry (Å, º)

Cg2 and Cg5 are the centroids of the C4A/C5A/C8A/N9/C9A and C20–C25 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O5[i]	0.84 (2)	2.18 (2)	2.9561 (19)	154.1 (18)
C21—H21···O4[ii]	0.95	2.38	3.230 (2)	148

C8—H8···*Cg*5[iii]	0.95	2.81	3.5127 (18)	131
C10—H10*B*···*Cg*2[iv]	0.99	2.58	3.5538 (18)	168

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+2, y+1/2, -z+1/2$; (iii) $-x+1, y-1/2, -z+1/2$; (iv) $-x+1, y+1/2, -z+1/2$.