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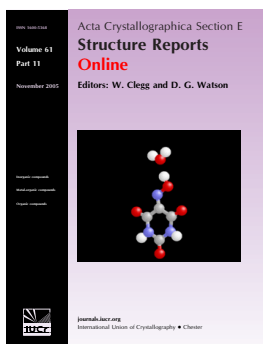
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Racemic 9,10-dimethoxy-3-methyl-6-phenyl-7,7a-dihydro-benzo[*b*]benzo[4,5]isothiazolo[2,3-*d*][1,4]diazepine 12,12-dioxide

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Racemic 9,10-dimethoxy-3-methyl-6-phenyl-7,7a-dihydrobenzo[*b*]benzo[4,5]-isothiazolo[2,3-*d*][1,4]diazepine 12,12-dioxide

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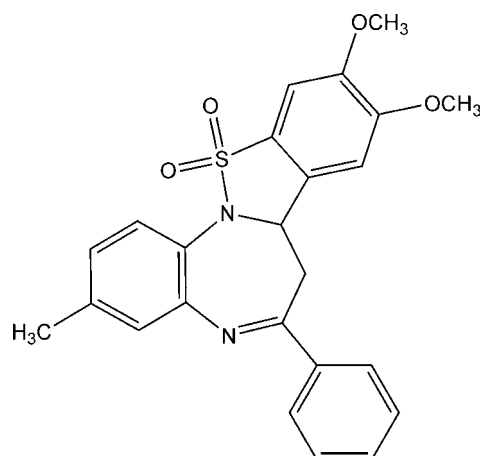
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.041; wR factor = 0.106; data-to-parameter ratio = 13.1.

There are two molecules in the asymmetric unit of the title compound, $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_4\text{S}$. The conformation of the seven-membered ring is twisted boat for both molecules. The molecule is chiral, but crystal symmetry generates a racemate. The crystal packing is stabilized by weak intermolecular C—H \cdots O hydrogen bonds.

Related literature

For related structures, see: Zia-ul-Haq *et al.* (2007); Boudina *et al.* (2007); Doubia *et al.* (2007); Sanudo *et al.* (2009); Spencer *et al.* (2009); Swamy *et al.* (2008). For the psychotropic properties of aptazapine [systematic name: 2-methyl-1,3,4,14b-tetrahydro-2*H*,10*H*-pyrazino[1,2-*a*]pyrrolo[2,1-*c*][1,4]benzodiazepine] and bretazenil [systematic name: *t*-butyl-8-bromo-11,12,13,13a-tetrahydro-9-oxo-9*H*-imidazo(1,5-*a*)pyrrolo(2,1-*c*)(1,4)benzodiazepine-1-carboxylate], see: Silvestri *et al.* (1994); Landquist (1984); Insuasty *et al.* (2008); Bennamane *et al.* (2008); Schutz (1982). For the bioactivity of benzodiazepines, see: Constanzo *et al.* (1990); Kelly *et al.* (1997). For the effect on the bioactivity of fusing different heterocyclic rings to the 1,4- and 1,5-benzodiazepine system, see: Chimirri *et al.* (1993*a,b*). For the synthesis of the title compound, see: Bassin *et al.* (2000).



Experimental

Crystal data

$\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_4\text{S}$
 $M_r = 434.51$
 Triclinic, $P\bar{1}$
 $a = 11.9007$ (4) Å
 $b = 12.8521$ (4) Å
 $c = 14.6202$ (5) Å
 $\alpha = 109.852$ (2)°
 $\beta = 90.743$ (2)°
 $\gamma = 92.775$ (2)°
 $V = 2099.76$ (12) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.19$ mm⁻¹
 $T = 120$ K
 $0.50 \times 0.30 \times 0.10$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer
 Absorption correction: multi-scan (SORTAV; Blessing, 1997)
 $T_{\min} = 0.912$, $T_{\max} = 0.981$
 31054 measured reflections
 7393 independent reflections
 5463 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.106$
 $S = 1.01$
 7393 reflections
 566 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots Cl1	0.89	2.44	3.303 (3)	162
N1—H1B \cdots Cl1 ⁱ	0.89	2.36	3.236 (2)	170
N1—H1C \cdots O1 ⁱⁱ	0.89	2.05	2.901 (4)	159
O1—H1H \cdots Cl1	0.85	2.45	3.290 (3)	170
O1—H1I \cdots Cl1 ⁱⁱⁱ	0.85	2.39	3.228 (2)	170

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

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997); data reduction: DENZO, COLLECT and maXus (Mackay *et al.*, 1999); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: CAMERON (Watkin, *et al.*, 1993); 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: RK2256).

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supplementary materials

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Racemic 9,10-dimethoxy-3-methyl-6-phenyl-7,7a-dihydrobenzo[*b*]benzo[4,5]isothiazolo[2,3-*d*][1,4]diazepine 12,12-dioxide

J. P. Bassin, V. P. Shah, L. Martin and P. N. Horton

Comment

In recent years tetracyclic benzodiazepines have received a great deal of attention due to the psychotropic properties of such compounds as aptazapine and bretazenil (Silvestri *et al.*, 1994; Landquist, 1984; Insuasty *et al.*, 2008; Bennamane *et al.*, 2008; Schutz, 1982). A number of structures of benzodiazepines have been reported: Zia-ul-Haq *et al.*, (2007); Boudina *et al.*, (2007); Doubia *et al.*, (2007); Sanudo *et al.*, (2009); Spencer *et al.*, (2009); Swamy *et al.*, (2008). Benzodiazepines containing heterocycles fused to the seven-membered ring have shown important and varied bioactivities (Constanzo *et al.*, 1990; Kelly *et al.*, 1997). It has been demonstrated that by fusing different heterocyclic rings to the 1,4- and 1,5-benzodiazepine system enhances the biological activity of these compounds (Chimirri *et al.*, 1993*a,b*). We previously reported the synthesis of a new heterocyclic ring system dihydrobenzo[*b*]benzo[4,5]isothiazolo[2,3-*d*][1,4]diazepine 12,12-dioxide (Bassin *et al.*, 2000). As, an extension of this work we report the crystal structure of the enantiomeric mixture of 9,10-dimethoxy-3-methyl-6-phenyl-7,7*a*-dihydrobenzo[*b*] benzo[4,5]isothiazolo [2,3-*d*][1,4]diazepine 12,12-dioxide.

Experimental

The title compound was synthesised following a modified procedure (Bassin *et al.*, 2000) by refluxing (*E*)-4,5-dimethoxy-2-(3-oxo-3-phenylprop-1-en-1-yl)benzene-1-sulfonyl chloride with 3,4-diaminotoluene in ethanol for 1 hour. The reaction mixture was allowed to cool to room temperature the resulting precipitated product was filtered under suction and thoroughly washed with aqueous ethanol. The air dried product was re-crystallised from ethanol. A yellow crystalline product (yield: 82%, m.p.: 498 K) was obtained.

Refinement

H atoms were positioned geometrically [C–H = 0.95 Å (for aromatic), 0.98 Å (for methyl groups) and 0.99 Å (for methylene groups)] and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{C})$ for methyl groups.

Figures

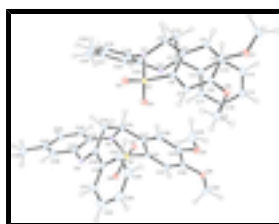


Fig. 1. The molecular structure of the title compound, showing the atom labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

9,10-dimethoxy-3-methyl-6-phenyl-7,7a-dihydrobenzo[*b*]benzo[4,5]isothiazolo[2,3-*d*][1,4]diazepine 12,12-dioxide

Crystal data

$C_{24}H_{22}N_2O_4S$	$Z = 4$
$M_r = 434.51$	$F(000) = 912$
Triclinic, $P\bar{1}$	$D_x = 1.375 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Melting point: 498 K
$a = 11.9007 (4) \text{ \AA}$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$b = 12.8521 (4) \text{ \AA}$	Cell parameters from 25443 reflections
$c = 14.6202 (5) \text{ \AA}$	$\theta = 2.9\text{--}27.5^\circ$
$\alpha = 109.852 (2)^\circ$	$\mu = 0.19 \text{ mm}^{-1}$
$\beta = 90.743 (2)^\circ$	$T = 120 \text{ K}$
$\gamma = 92.775 (2)^\circ$	Prism, yellow
$V = 2099.76 (12) \text{ \AA}^3$	$0.50 \times 0.30 \times 0.10 \text{ mm}$

Data collection

Bruker–Nonius KappaCCD diffractometer	7393 independent reflections
Radiation source: fine-focus sealed tube graphite	5463 reflections with $I > 2\sigma(I)$
Detector resolution: $9.091 \text{ pixels mm}^{-1}$ φ - and ω scans	$R_{\text{int}} = 0.060$
Absorption correction: multi-scan (SORTAV; Blessing, 1997)	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 3.0^\circ$
$T_{\text{min}} = 0.912$, $T_{\text{max}} = 0.981$	$h = -14 \rightarrow 14$
31054 measured reflections	$k = -15 \rightarrow 15$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.041$	H-atom parameters constrained
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0595P)^2 + 0.1891P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
7393 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
566 parameters	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.46 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0053 (7)

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3992 (2)	-0.2363 (2)	0.14280 (18)	0.0360 (6)
H1A	0.4094	-0.1782	0.2067	0.054*
H1B	0.4607	-0.2867	0.1329	0.054*
H1C	0.3272	-0.2778	0.1396	0.054*
C2	0.42922 (19)	-0.05068 (19)	-0.13723 (16)	0.0311 (5)
H2A	0.3605	-0.0683	-0.1784	0.047*
H2B	0.4933	-0.0824	-0.1766	0.047*
H2C	0.4423	0.0299	-0.1089	0.047*
C3	0.16980 (16)	0.00163 (15)	0.14164 (14)	0.0188 (4)
C4	0.24041 (17)	-0.08172 (16)	0.14182 (14)	0.0217 (5)
H4	0.2314	-0.1188	0.1877	0.026*
C5	0.32354 (17)	-0.10949 (16)	0.07437 (15)	0.0225 (5)
C6	0.33481 (17)	-0.05692 (16)	0.00370 (14)	0.0215 (5)
C7	0.26602 (17)	0.02657 (16)	0.00462 (14)	0.0222 (5)
H7	0.2738	0.0637	-0.0413	0.027*
C8	0.18439 (16)	0.05466 (15)	0.07553 (14)	0.0197 (4)
C9	0.07022 (16)	0.03569 (16)	0.20530 (14)	0.0195 (4)
H9	0.0141	-0.0289	0.1893	0.023*
C10	0.09793 (17)	0.07586 (16)	0.31449 (14)	0.0211 (4)
H10A	0.0271	0.0850	0.3505	0.025*
H10B	0.1404	0.0195	0.3303	0.025*
C11	0.16693 (16)	0.18492 (16)	0.34651 (13)	0.0194 (4)
C12	0.29050 (17)	0.18973 (16)	0.36576 (14)	0.0210 (5)
C13	0.34603 (18)	0.10086 (17)	0.37451 (15)	0.0265 (5)
H13	0.3046	0.0338	0.3690	0.032*
C14	0.46215 (18)	0.10953 (19)	0.39138 (16)	0.0306 (5)
H14	0.4992	0.0483	0.3976	0.037*
C15	0.52371 (18)	0.20556 (19)	0.39914 (15)	0.0317 (5)
H15	0.6030	0.2107	0.4100	0.038*
C16	0.46915 (18)	0.2947 (2)	0.39095 (17)	0.0334 (6)
H16	0.5111	0.3614	0.3962	0.040*
C17	0.35396 (17)	0.28733 (18)	0.37510 (15)	0.0272 (5)
H17	0.3173	0.3495	0.3705	0.033*

supplementary materials

C18	-0.04226 (16)	0.20694 (16)	0.24328 (14)	0.0207 (5)
C19	-0.15279 (17)	0.21991 (17)	0.22056 (15)	0.0250 (5)
H19	-0.1860	0.1735	0.1601	0.030*
C20	-0.21585 (17)	0.29909 (17)	0.28400 (15)	0.0259 (5)
H20	-0.2914	0.3066	0.2665	0.031*
C21	-0.16983 (17)	0.36800 (16)	0.37326 (15)	0.0235 (5)
C22	-0.05783 (17)	0.35632 (16)	0.39450 (15)	0.0235 (5)
H22	-0.0245	0.4042	0.4543	0.028*
C23	0.00699 (17)	0.27780 (16)	0.33205 (15)	0.0213 (5)
C24	-0.23848 (18)	0.45185 (18)	0.44470 (16)	0.0308 (5)
H24A	-0.2971	0.4754	0.4095	0.046*
H24B	-0.1895	0.5162	0.4820	0.046*
H24C	-0.2737	0.4185	0.4892	0.046*
N1	0.02092 (13)	0.12406 (13)	0.17640 (11)	0.0204 (4)
N2	0.12208 (13)	0.27710 (13)	0.35685 (12)	0.0219 (4)
O1	0.40000 (12)	-0.18679 (11)	0.06850 (10)	0.0290 (4)
O2	0.41702 (12)	-0.09601 (12)	-0.06133 (10)	0.0282 (3)
O3	0.13467 (12)	0.26485 (11)	0.12453 (10)	0.0278 (4)
O4	0.01497 (12)	0.12890 (11)	0.00291 (10)	0.0265 (3)
S1	0.08537 (4)	0.15358 (4)	0.08842 (4)	0.02013 (14)
C25	0.4183 (2)	0.30217 (18)	0.13538 (18)	0.0388 (6)
H25A	0.3708	0.2635	0.1693	0.058*
H25B	0.4746	0.2526	0.0987	0.058*
H25C	0.3714	0.3246	0.0905	0.058*
C26	0.64950 (18)	0.67805 (19)	0.35264 (17)	0.0348 (6)
H26A	0.6307	0.7317	0.3214	0.052*
H26B	0.7296	0.6639	0.3453	0.052*
H26C	0.6332	0.7079	0.4220	0.052*
C27	0.23523 (16)	0.57054 (16)	0.29537 (14)	0.0188 (4)
C28	0.29256 (17)	0.47623 (16)	0.24852 (14)	0.0218 (5)
H28	0.2523	0.4079	0.2147	0.026*
C29	0.40916 (17)	0.48312 (16)	0.25179 (14)	0.0233 (5)
C30	0.46967 (16)	0.58300 (16)	0.30635 (14)	0.0215 (5)
C31	0.41321 (17)	0.67598 (16)	0.35242 (14)	0.0210 (5)
H31	0.4527	0.7439	0.3885	0.025*
C32	0.29588 (16)	0.66768 (16)	0.34460 (14)	0.0192 (4)
C33	0.10869 (17)	0.57821 (16)	0.30258 (14)	0.0205 (4)
H33	0.0811	0.5345	0.3437	0.025*
C34	0.04001 (16)	0.53834 (16)	0.20675 (14)	0.0201 (4)
H34A	0.0568	0.4608	0.1700	0.024*
H34B	-0.0411	0.5395	0.2210	0.024*
C35	0.06480 (17)	0.60900 (16)	0.14439 (14)	0.0213 (5)
C36	0.13310 (17)	0.56838 (16)	0.05648 (14)	0.0224 (5)
C37	0.17889 (19)	0.46524 (18)	0.02645 (16)	0.0309 (5)
H37	0.1614	0.4149	0.0598	0.037*
C38	0.2498 (2)	0.4349 (2)	-0.05163 (17)	0.0401 (6)
H38	0.2808	0.3642	-0.0712	0.048*
C39	0.2754 (2)	0.5066 (2)	-0.10083 (17)	0.0419 (6)
H39	0.3262	0.4868	-0.1528	0.050*

C40	0.2270 (2)	0.6071 (2)	-0.07446 (17)	0.0416 (6)
H40	0.2429	0.6558	-0.1096	0.050*
C41	0.1557 (2)	0.63781 (19)	0.00233 (16)	0.0320 (5)
H41	0.1215	0.7068	0.0187	0.038*
C42	-0.00310 (16)	0.75312 (16)	0.34128 (15)	0.0209 (5)
C43	-0.05867 (18)	0.81701 (18)	0.42232 (16)	0.0274 (5)
H43	-0.0384	0.8152	0.4848	0.033*
C44	-0.14299 (18)	0.88309 (18)	0.41306 (17)	0.0310 (5)
H44	-0.1793	0.9274	0.4694	0.037*
C45	-0.17537 (17)	0.88550 (17)	0.32215 (17)	0.0284 (5)
C46	-0.11840 (17)	0.82177 (17)	0.24161 (16)	0.0263 (5)
H46	-0.1396	0.8231	0.1792	0.032*
C47	-0.03145 (17)	0.75615 (16)	0.24917 (15)	0.0222 (5)
C48	-0.2672 (2)	0.9570 (2)	0.3111 (2)	0.0432 (6)
H48A	-0.2339	1.0214	0.2976	0.065*
H48B	-0.3089	0.9822	0.3714	0.065*
H48C	-0.3186	0.9142	0.2571	0.065*
N3	0.09285 (14)	0.69706 (13)	0.35612 (12)	0.0215 (4)
N4	0.02928 (14)	0.70698 (14)	0.16533 (12)	0.0235 (4)
O5	0.47410 (12)	0.39866 (11)	0.20519 (11)	0.0310 (4)
O6	0.58380 (11)	0.57683 (12)	0.30791 (10)	0.0289 (4)
O7	0.21847 (12)	0.81169 (12)	0.50136 (10)	0.0318 (4)
O8	0.21966 (12)	0.86010 (11)	0.35488 (11)	0.0325 (4)
S2	0.20873 (4)	0.77439 (4)	0.39734 (4)	0.02169 (14)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0384 (14)	0.0364 (13)	0.0432 (15)	0.0115 (11)	0.0032 (11)	0.0250 (12)
C2	0.0318 (13)	0.0387 (13)	0.0250 (13)	0.0040 (10)	0.0060 (10)	0.0130 (10)
C3	0.0219 (11)	0.0144 (10)	0.0171 (11)	-0.0015 (8)	-0.0029 (8)	0.0020 (8)
C4	0.0272 (12)	0.0181 (10)	0.0210 (11)	-0.0001 (9)	-0.0018 (9)	0.0085 (9)
C5	0.0243 (11)	0.0168 (10)	0.0257 (12)	0.0038 (9)	0.0001 (9)	0.0058 (9)
C6	0.0221 (11)	0.0213 (11)	0.0181 (11)	0.0007 (9)	0.0001 (9)	0.0028 (9)
C7	0.0250 (11)	0.0215 (11)	0.0212 (11)	0.0003 (9)	-0.0003 (9)	0.0089 (9)
C8	0.0219 (11)	0.0154 (10)	0.0197 (11)	-0.0015 (8)	-0.0028 (9)	0.0038 (8)
C9	0.0208 (11)	0.0170 (10)	0.0205 (11)	0.0008 (8)	-0.0005 (8)	0.0061 (8)
C10	0.0222 (11)	0.0209 (11)	0.0195 (11)	0.0018 (9)	0.0003 (8)	0.0061 (9)
C11	0.0229 (11)	0.0201 (11)	0.0136 (10)	0.0013 (9)	0.0003 (8)	0.0038 (8)
C12	0.0238 (11)	0.0241 (11)	0.0129 (10)	0.0015 (9)	0.0005 (8)	0.0034 (8)
C13	0.0261 (12)	0.0243 (12)	0.0271 (12)	0.0036 (9)	0.0006 (9)	0.0058 (9)
C14	0.0295 (13)	0.0343 (13)	0.0266 (13)	0.0117 (11)	-0.0012 (10)	0.0074 (10)
C15	0.0224 (12)	0.0484 (15)	0.0234 (13)	0.0033 (11)	-0.0003 (9)	0.0110 (11)
C16	0.0235 (12)	0.0401 (14)	0.0390 (14)	-0.0061 (11)	-0.0050 (10)	0.0180 (11)
C17	0.0249 (12)	0.0279 (12)	0.0306 (13)	0.0016 (10)	-0.0031 (9)	0.0123 (10)
C18	0.0211 (11)	0.0182 (10)	0.0224 (11)	0.0027 (9)	0.0023 (9)	0.0063 (9)
C19	0.0231 (11)	0.0267 (11)	0.0224 (12)	0.0012 (9)	-0.0028 (9)	0.0049 (9)
C20	0.0178 (11)	0.0305 (12)	0.0290 (13)	0.0039 (9)	-0.0006 (9)	0.0095 (10)

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C21	0.0239 (11)	0.0202 (11)	0.0273 (12)	0.0037 (9)	0.0021 (9)	0.0090 (9)
C22	0.0248 (11)	0.0200 (11)	0.0227 (12)	0.0008 (9)	-0.0036 (9)	0.0037 (9)
C23	0.0215 (11)	0.0186 (10)	0.0235 (11)	-0.0001 (9)	-0.0010 (9)	0.0069 (9)
C24	0.0274 (12)	0.0310 (12)	0.0320 (13)	0.0075 (10)	0.0024 (10)	0.0074 (10)
N1	0.0225 (9)	0.0190 (9)	0.0200 (9)	0.0036 (7)	0.0003 (7)	0.0067 (7)
N2	0.0194 (9)	0.0219 (9)	0.0214 (10)	0.0016 (7)	-0.0016 (7)	0.0036 (7)
O1	0.0336 (9)	0.0255 (8)	0.0316 (9)	0.0116 (7)	0.0054 (7)	0.0131 (7)
O2	0.0317 (8)	0.0301 (8)	0.0250 (8)	0.0102 (7)	0.0094 (7)	0.0110 (7)
O3	0.0316 (8)	0.0169 (7)	0.0333 (9)	-0.0017 (6)	0.0000 (7)	0.0069 (6)
O4	0.0308 (8)	0.0269 (8)	0.0217 (8)	0.0024 (7)	-0.0058 (6)	0.0084 (6)
S1	0.0222 (3)	0.0166 (3)	0.0209 (3)	0.0006 (2)	-0.0011 (2)	0.0056 (2)
C25	0.0392 (14)	0.0256 (12)	0.0386 (14)	0.0070 (11)	-0.0016 (11)	-0.0064 (11)
C26	0.0240 (12)	0.0359 (13)	0.0395 (14)	-0.0033 (10)	0.0003 (10)	0.0072 (11)
C27	0.0199 (10)	0.0196 (11)	0.0193 (11)	0.0006 (9)	-0.0005 (8)	0.0098 (9)
C28	0.0230 (11)	0.0168 (10)	0.0243 (12)	0.0010 (9)	-0.0004 (9)	0.0053 (9)
C29	0.0280 (12)	0.0202 (11)	0.0213 (12)	0.0077 (9)	0.0028 (9)	0.0057 (9)
C30	0.0188 (11)	0.0258 (11)	0.0201 (11)	0.0022 (9)	0.0005 (8)	0.0080 (9)
C31	0.0238 (11)	0.0178 (10)	0.0203 (11)	-0.0015 (9)	-0.0015 (9)	0.0057 (9)
C32	0.0231 (11)	0.0172 (10)	0.0185 (11)	0.0022 (9)	0.0019 (8)	0.0076 (8)
C33	0.0242 (11)	0.0162 (10)	0.0210 (11)	0.0027 (9)	0.0017 (9)	0.0060 (8)
C34	0.0184 (10)	0.0183 (10)	0.0213 (11)	0.0017 (8)	0.0008 (8)	0.0037 (9)
C35	0.0207 (11)	0.0218 (11)	0.0193 (11)	-0.0007 (9)	-0.0038 (8)	0.0046 (9)
C36	0.0243 (11)	0.0217 (11)	0.0187 (11)	0.0002 (9)	-0.0014 (9)	0.0039 (9)
C37	0.0371 (13)	0.0298 (12)	0.0243 (13)	0.0038 (10)	0.0039 (10)	0.0068 (10)
C38	0.0436 (15)	0.0391 (14)	0.0313 (14)	0.0121 (12)	0.0101 (11)	0.0024 (11)
C39	0.0426 (15)	0.0534 (17)	0.0231 (13)	0.0006 (13)	0.0106 (11)	0.0046 (12)
C40	0.0554 (16)	0.0442 (15)	0.0267 (14)	-0.0024 (13)	0.0079 (12)	0.0145 (12)
C41	0.0407 (14)	0.0310 (12)	0.0237 (13)	0.0012 (11)	0.0012 (10)	0.0086 (10)
C42	0.0183 (10)	0.0191 (10)	0.0247 (12)	0.0022 (9)	0.0019 (9)	0.0065 (9)
C43	0.0256 (12)	0.0298 (12)	0.0247 (12)	0.0041 (10)	0.0021 (9)	0.0060 (10)
C44	0.0263 (12)	0.0283 (12)	0.0329 (14)	0.0087 (10)	0.0076 (10)	0.0020 (10)
C45	0.0227 (11)	0.0209 (11)	0.0386 (14)	0.0036 (9)	-0.0028 (10)	0.0059 (10)
C46	0.0257 (12)	0.0221 (11)	0.0301 (13)	0.0008 (9)	-0.0056 (9)	0.0078 (10)
C47	0.0223 (11)	0.0177 (10)	0.0248 (12)	0.0001 (9)	0.0004 (9)	0.0053 (9)
C48	0.0348 (14)	0.0366 (14)	0.0522 (17)	0.0131 (11)	-0.0065 (12)	0.0059 (12)
N3	0.0223 (9)	0.0180 (9)	0.0219 (9)	0.0034 (7)	-0.0019 (7)	0.0037 (7)
N4	0.0261 (10)	0.0224 (10)	0.0214 (10)	0.0021 (8)	-0.0008 (8)	0.0066 (8)
O5	0.0253 (8)	0.0238 (8)	0.0367 (9)	0.0079 (7)	0.0020 (7)	0.0002 (7)
O6	0.0196 (8)	0.0302 (8)	0.0313 (9)	0.0003 (6)	0.0004 (6)	0.0032 (7)
O7	0.0326 (9)	0.0336 (9)	0.0205 (8)	0.0070 (7)	-0.0052 (6)	-0.0024 (7)
O8	0.0325 (9)	0.0213 (8)	0.0480 (10)	0.0031 (7)	-0.0025 (7)	0.0172 (7)
S2	0.0230 (3)	0.0175 (3)	0.0231 (3)	0.0027 (2)	-0.0020 (2)	0.0048 (2)

Geometric parameters (Å, °)

C1—O1	1.433 (2)	C25—O5	1.437 (3)
C1—H1A	0.9800	C25—H25A	0.9800
C1—H1B	0.9800	C25—H25B	0.9800
C1—H1C	0.9800	C25—H25C	0.9800

C2—O2	1.424 (2)	C26—O6	1.429 (3)
C2—H2A	0.9800	C26—H26A	0.9800
C2—H2B	0.9800	C26—H26B	0.9800
C2—H2C	0.9800	C26—H26C	0.9800
C3—C8	1.367 (3)	C27—C32	1.373 (3)
C3—C4	1.394 (3)	C27—C28	1.390 (3)
C3—C9	1.505 (3)	C27—C33	1.517 (3)
C4—C5	1.378 (3)	C28—C29	1.385 (3)
C4—H4	0.9500	C28—H28	0.9500
C5—O1	1.362 (2)	C29—O5	1.354 (2)
C5—C6	1.418 (3)	C29—C30	1.415 (3)
C6—O2	1.364 (2)	C30—O6	1.365 (2)
C6—C7	1.377 (3)	C30—C31	1.370 (3)
C7—C8	1.398 (3)	C31—C32	1.395 (3)
C7—H7	0.9500	C31—H31	0.9500
C8—S1	1.740 (2)	C32—S2	1.730 (2)
C9—N1	1.482 (2)	C33—N3	1.484 (3)
C9—C10	1.528 (3)	C33—C34	1.528 (3)
C9—H9	1.0000	C33—H33	1.0000
C10—C11	1.514 (3)	C34—C35	1.512 (3)
C10—H10A	0.9900	C34—H34A	0.9900
C10—H10B	0.9900	C34—H34B	0.9900
C11—N2	1.286 (2)	C35—N4	1.285 (3)
C11—C12	1.488 (3)	C35—C36	1.482 (3)
C12—C13	1.388 (3)	C36—C37	1.388 (3)
C12—C17	1.397 (3)	C36—C41	1.399 (3)
C13—C14	1.393 (3)	C37—C38	1.386 (3)
C13—H13	0.9500	C37—H37	0.9500
C14—C15	1.374 (3)	C38—C39	1.374 (3)
C14—H14	0.9500	C38—H38	0.9500
C15—C16	1.383 (3)	C39—C40	1.374 (4)
C15—H15	0.9500	C39—H39	0.9500
C16—C17	1.381 (3)	C40—C41	1.376 (3)
C16—H16	0.9500	C40—H40	0.9500
C17—H17	0.9500	C41—H41	0.9500
C18—C19	1.384 (3)	C42—C43	1.388 (3)
C18—C23	1.408 (3)	C42—C47	1.398 (3)
C18—N1	1.430 (3)	C42—N3	1.432 (3)
C19—C20	1.382 (3)	C43—C44	1.380 (3)
C19—H19	0.9500	C43—H43	0.9500
C20—C21	1.392 (3)	C44—C45	1.390 (3)
C20—H20	0.9500	C44—H44	0.9500
C21—C22	1.391 (3)	C45—C46	1.392 (3)
C21—C24	1.504 (3)	C45—C48	1.503 (3)
C22—C23	1.384 (3)	C46—C47	1.393 (3)
C22—H22	0.9500	C46—H46	0.9500
C23—N2	1.413 (3)	C47—N4	1.402 (3)
C24—H24A	0.9800	C48—H48A	0.9800
C24—H24B	0.9800	C48—H48B	0.9800

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C24—H24C	0.9800	C48—H48C	0.9800
N1—S1	1.6479 (16)	N3—S2	1.6406 (17)
O3—S1	1.4374 (14)	O7—S2	1.4321 (15)
O4—S1	1.4295 (14)	O8—S2	1.4383 (15)
O1—C1—H1A	109.5	O5—C25—H25A	109.5
O1—C1—H1B	109.5	O5—C25—H25B	109.5
H1A—C1—H1B	109.5	H25A—C25—H25B	109.5
O1—C1—H1C	109.5	O5—C25—H25C	109.5
H1A—C1—H1C	109.5	H25A—C25—H25C	109.5
H1B—C1—H1C	109.5	H25B—C25—H25C	109.5
O2—C2—H2A	109.5	O6—C26—H26A	109.5
O2—C2—H2B	109.5	O6—C26—H26B	109.5
H2A—C2—H2B	109.5	H26A—C26—H26B	109.5
O2—C2—H2C	109.5	O6—C26—H26C	109.5
H2A—C2—H2C	109.5	H26A—C26—H26C	109.5
H2B—C2—H2C	109.5	H26B—C26—H26C	109.5
C8—C3—C4	119.80 (19)	C32—C27—C28	119.02 (18)
C8—C3—C9	114.71 (17)	C32—C27—C33	114.27 (17)
C4—C3—C9	125.35 (17)	C28—C27—C33	126.65 (17)
C5—C4—C3	118.94 (18)	C29—C28—C27	119.12 (18)
C5—C4—H4	120.5	C29—C28—H28	120.4
C3—C4—H4	120.5	C27—C28—H28	120.4
O1—C5—C4	124.86 (18)	O5—C29—C28	124.54 (18)
O1—C5—C6	114.49 (17)	O5—C29—C30	114.72 (17)
C4—C5—C6	120.65 (18)	C28—C29—C30	120.74 (18)
O2—C6—C7	125.27 (18)	O6—C30—C31	125.14 (18)
O2—C6—C5	114.49 (17)	O6—C30—C29	114.80 (17)
C7—C6—C5	120.23 (18)	C31—C30—C29	120.06 (18)
C6—C7—C8	117.64 (18)	C30—C31—C32	117.85 (18)
C6—C7—H7	121.2	C30—C31—H31	121.1
C8—C7—H7	121.2	C32—C31—H31	121.1
C3—C8—C7	122.65 (18)	C27—C32—C31	123.09 (18)
C3—C8—S1	111.35 (15)	C27—C32—S2	111.58 (15)
C7—C8—S1	125.92 (15)	C31—C32—S2	125.30 (15)
N1—C9—C3	105.11 (15)	N3—C33—C27	104.51 (15)
N1—C9—C10	111.04 (15)	N3—C33—C34	110.71 (15)
C3—C9—C10	114.78 (16)	C27—C33—C34	116.61 (16)
N1—C9—H9	108.6	N3—C33—H33	108.2
C3—C9—H9	108.6	C27—C33—H33	108.2
C10—C9—H9	108.6	C34—C33—H33	108.2
C11—C10—C9	111.25 (15)	C35—C34—C33	112.47 (16)
C11—C10—H10A	109.4	C35—C34—H34A	109.1
C9—C10—H10A	109.4	C33—C34—H34A	109.1
C11—C10—H10B	109.4	C35—C34—H34B	109.1
C9—C10—H10B	109.4	C33—C34—H34B	109.1
H10A—C10—H10B	108.0	H34A—C34—H34B	107.8
N2—C11—C12	117.02 (17)	N4—C35—C36	116.87 (18)
N2—C11—C10	121.73 (17)	N4—C35—C34	122.00 (18)
C12—C11—C10	121.24 (17)	C36—C35—C34	121.12 (17)

C13—C12—C17	118.26 (19)	C37—C36—C41	118.0 (2)
C13—C12—C11	122.76 (18)	C37—C36—C35	123.05 (18)
C17—C12—C11	118.97 (18)	C41—C36—C35	118.94 (19)
C12—C13—C14	120.3 (2)	C38—C37—C36	120.7 (2)
C12—C13—H13	119.8	C38—C37—H37	119.6
C14—C13—H13	119.8	C36—C37—H37	119.6
C15—C14—C13	120.8 (2)	C39—C38—C37	120.3 (2)
C15—C14—H14	119.6	C39—C38—H38	119.8
C13—C14—H14	119.6	C37—C38—H38	119.8
C14—C15—C16	119.4 (2)	C38—C39—C40	119.6 (2)
C14—C15—H15	120.3	C38—C39—H39	120.2
C16—C15—H15	120.3	C40—C39—H39	120.2
C17—C16—C15	120.3 (2)	C39—C40—C41	120.7 (2)
C17—C16—H16	119.8	C39—C40—H40	119.7
C15—C16—H16	119.8	C41—C40—H40	119.7
C16—C17—C12	120.9 (2)	C40—C41—C36	120.6 (2)
C16—C17—H17	119.5	C40—C41—H41	119.7
C12—C17—H17	119.5	C36—C41—H41	119.7
C19—C18—C23	118.93 (18)	C43—C42—C47	120.06 (19)
C19—C18—N1	120.14 (18)	C43—C42—N3	118.37 (18)
C23—C18—N1	120.92 (17)	C47—C42—N3	121.01 (17)
C20—C19—C18	121.32 (19)	C44—C43—C42	120.7 (2)
C20—C19—H19	119.3	C44—C43—H43	119.6
C18—C19—H19	119.3	C42—C43—H43	119.6
C19—C20—C21	120.70 (19)	C43—C44—C45	120.6 (2)
C19—C20—H20	119.6	C43—C44—H44	119.7
C21—C20—H20	119.6	C45—C44—H44	119.7
C22—C21—C20	117.61 (19)	C44—C45—C46	118.09 (19)
C22—C21—C24	120.97 (18)	C44—C45—C48	121.1 (2)
C20—C21—C24	121.42 (18)	C46—C45—C48	120.8 (2)
C23—C22—C21	122.67 (19)	C45—C46—C47	122.4 (2)
C23—C22—H22	118.7	C45—C46—H46	118.8
C21—C22—H22	118.7	C47—C46—H46	118.8
C22—C23—C18	118.72 (18)	C46—C47—C42	118.05 (19)
C22—C23—N2	118.58 (17)	C46—C47—N4	117.27 (18)
C18—C23—N2	122.55 (18)	C42—C47—N4	124.33 (18)
C21—C24—H24A	109.5	C45—C48—H48A	109.5
C21—C24—H24B	109.5	C45—C48—H48B	109.5
H24A—C24—H24B	109.5	H48A—C48—H48B	109.5
C21—C24—H24C	109.5	C45—C48—H48C	109.5
H24A—C24—H24C	109.5	H48A—C48—H48C	109.5
H24B—C24—H24C	109.5	H48B—C48—H48C	109.5
C18—N1—C9	120.90 (15)	C42—N3—C33	123.39 (16)
C18—N1—S1	118.90 (13)	C42—N3—S2	116.39 (12)
C9—N1—S1	115.15 (13)	C33—N3—S2	115.50 (13)
C11—N2—C23	120.09 (16)	C35—N4—C47	121.94 (17)
C5—O1—C1	117.25 (16)	C29—O5—C25	116.94 (16)
C6—O2—C2	117.44 (16)	C30—O6—C26	117.05 (16)
O4—S1—O3	114.70 (8)	O7—S2—O8	115.28 (9)

supplementary materials

O4—S1—N1	112.29 (8)	O7—S2—N3	111.89 (9)
O3—S1—N1	110.02 (8)	O8—S2—N3	110.19 (9)
O4—S1—C8	111.83 (9)	O7—S2—C32	111.60 (9)
O3—S1—C8	112.54 (9)	O8—S2—C32	112.07 (9)
N1—S1—C8	93.60 (9)	N3—S2—C32	93.86 (9)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C2—H2C \cdots O1 ⁱ	0.98	2.59	3.423 (3)	142
C4—H4 \cdots O8 ⁱⁱ	0.95	2.55	3.451 (3)	157
C10—H10B \cdots O8 ⁱⁱ	0.99	2.43	3.408 (3)	171
C13—H13 \cdots O8 ⁱⁱ	0.95	2.35	3.295 (3)	174
C15—H15 \cdots O7 ⁱⁱⁱ	0.95	2.55	3.428 (3)	153
C25—H25B \cdots O2 ⁱ	0.98	2.35	3.262 (3)	154
C28—H28 \cdots O3	0.95	2.27	3.205 (3)	169
C34—H34A \cdots O3	0.99	2.60	3.552 (3)	162
C37—H37 \cdots O3	0.95	2.43	3.371 (3)	170
C39—H39 \cdots O6 ^{iv}	0.95	2.42	3.350 (3)	167

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

Fig. 1

