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Ethyl 2-[2,2-bis(ethoxycarbonyl)ethyl]-1-(phenylsulfonyl)indole-3-carboxylate

G. Chakkaravarthi,[a]* V. Dhayalan,[b] A. K. Mohanakrishnan[b] and V. Manivannan[c]

[a]Department of Physics, CPCL Polytechnic College, Chennai 600 068, India, [b]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]Department of Physics, Presidency College, Chennai 600 005, India
Correspondence e-mail: chakkaravarthi_2005@yahoo.com

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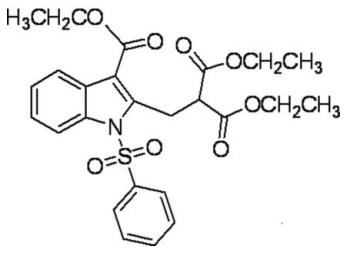
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.060; wR factor = 0.191; data-to-parameter ratio = 17.9.

In the title compound, $C_{25}H_{27}NO_8S$, the phenyl ring forms a dihedral angle of 83.28 (7)° with the indole ring system. The methyl C atoms of the ester groups and one of the carbonyl O atoms are each disordered over two positions; the site occupancies for the C atoms are 0.75/0.25, 0.87/0.13, 0.64/0.36 and for the O atom 0.57/0.43. The molecular structure is stabilized by weak intramolecular C–H···O interactions.

Related literature

For related literature, see: Liu *et al.* (2007); Nieto *et al.* (2005); Pomarnacka & Kozlarska-Kedra (2003); Schultz *et al.* (2001); Yang *et al.* (2002). A similar compound with an ethoxycarbonyl group has been reported (Chakkaravarthi *et al.*, 2007).



Experimental

Crystal data

$C_{25}H_{27}NO_8S$
M_r = 501.54
Monoclinic, $P2_1/c$
a = 7.9066 (2) Å
b = 27.9935 (6) Å
c = 11.8849 (2) Å
β = 100.832 (1)°
V = 2583.66 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 295 (2) K
0.26 × 0.18 × 0.14 mm

Data collection

Bruker Kappa APEX2 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.892, T_{max} = 0.976
29408 measured reflections
6381 independent reflections
4267 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.192
S = 1.09
6381 reflections
356 parameters
10 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1	0.93	2.52	2.895 (3)	104
C10—H10···O4	0.93	2.46	2.944 (3)	113
C13—H13···O1	0.93	2.51	3.059 (3)	118
C18—H18*A*···O3	0.97	2.28	3.004 (15)	131
C18—H18*B*···O2	0.97	2.34	2.827 (3)	110
C19—H19···O2	0.98	2.39	3.025 (3)	122

Data collection: *APEX2* (Bruker, 2004); 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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2566).

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supplementary materials

Acta Cryst. (2007). E**63**, o4724 [**doi:10.1107/S1600536807057078**]

Ethyl 2-[2,2-bis(ethoxycarbonyl)ethyl]-1-(phenylsulfonyl)indole-3-carboxylate

G. Chakkaravarthi, V. Dhayalan, A. K. Mohanakrishnan and V. Manivannan

Comment

Benzenesulfonamide derivatives exhibit significant biological activities, such as antibacterial (Nieto *et al.*, 2005), anticancer and anti - HIV (Pomarnacka & Kozlarska-Kedra, 2003) and antitumor (Yang *et al.*, 2002). Sulfur containing compounds mostly act as simple narcotics (Schultz *et al.*, 2001).

Geometric parameters in the title compound (Fig. 1) agree with the reported values of similar structures (Chakkaravarthi *et al.*, 2007; Liu *et al.*, 2007). The sum of the bond angles around N1 (358.19°) indicates sp^2 hybridization.

The torsion angles C14—N1—S1—O1 and C7—N1—S1—O2 [−54.54 (17)° and 13.62 (2)°, respectively] indicate the *syn* conformation of the sulfonyl moiety with indole ring system.

The molecular structure is stabilized by weak intramolecular C - H···O interactions (Table 1). A similar compound with ethoxycarbonyl group has been reported (Chakkaravarthi *et al.*, 2007).

Experimental

To a well stirred suspension of sodium hydride (0.27 g, 5.62 mmol) in dry tetrahydrofuron (15 ml) at room temperature, a solution of diethyl malonate (1.4 ml, 9.47 mmol) in dry tetrahydrofuron (10 ml) was slowly added over a period of 20 min. After the evolution of hydrogen gas ceased, a solution of ethyl-1-phenyl sulfonyl-2-bromo methyl indole-3-carboxylate (2 g, 4.73 mmol) in dry THF (20 ml) was added in dropwise with vigorous stirring. Then the reaction mixture was stirred for 3 h. The reaction mixture was quenched with ice containing few drop of concentrated HCl, extracted with dichloro methane (3 *X* 5 ml) and dried (Na_2SO_4). The solvent was removed under vacuum. Then the crude product was purified by column chromatography on siliga gel (hexane-ethyl acetate 9:1). Single crystals suitable for X-ray analysis were grown by slow evaporation of ethyl acetate solution at room temperature.

Refinement

The site occupancy factors for disordered C and O atoms were refined as C22 = 0.25 (2), C22A = 0.75 (2), C25 = 0.134 (10), C25A = 0.866 (10), C17 = 0.36 (2), C17A = 0.64 (2), O3 = 0.43 (6) and O3A = 0.57 (6) during anisotropic refinement. The distances C16—C17, C21—C22, C21—C22A, C24—C25 and C24—C25A were restrained to 1.55 (1) Å; the bond distances C24—C25/C24—C25A and C21—C22/C21—C22A were restrained to be equal within 0.02Å and the non-bonding distances O6—C25/O6—C25A and O8—C22/O8—C22A were restrained to be equal within 0.04 Å. H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C) for aromatic C—H, C—H = 0.98 Å and U_{iso}(H) = 1.2Ueq(C) for C—H, C—H = 0.97 Å and U_{iso}(H) = 1.2Ueq(C) for CH_2 and C—H = 0.96 Å and U_{iso}(H) = 1.5Ueq(C) for CH_3.

Figures

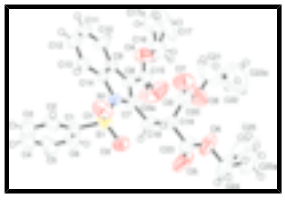


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

Ethyl 2-[2,2-bis(ethoxycarbonyl)ethyl]-1-(phenylsulfonyl)indole-3-carboxylate

Crystal data

$C_{25}H_{27}NO_8S$	F_{000} = 1056
M_r = 501.54	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 7119 reflections
a = 7.9066 (2) Å	θ = 2.7–24.6º
b = 27.9935 (6) Å	μ = 0.17 mm^{-1}
c = 11.8849 (2) Å	T = 295 (2) K
β = 100.832 (1)º	Block, colourless
V = 2583.66 (10) Å^3	0.26 × 0.18 × 0.14 mm
Z = 4	

Data collection

Bruker Kappa APEX2 diffractometer	6381 independent reflections
Radiation source: fine-focus sealed tube	4267 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 295(2) K	θ_{max} = 28.2º
ω and φ scan	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→10
T_{min} = 0.892, T_{max} = 0.976	k = −37→37
29408 measured reflections	l = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained
$wR(F^2)$ = 0.192	$w = 1/[\sigma^2(F_o^2) + (0.1023P)^2 + 0.2706P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.09	$(\Delta/\sigma)_{max} < 0.001$

6381 reflections	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
356 parameters	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O8	0.4654 (3)	0.06412 (9)	0.6271 (2)	0.1218 (9)	
C21	0.3304 (9)	0.0330 (2)	0.6543 (5)	0.183 (2)	
H21A	0.2246	0.0507	0.6533	0.219*	0.25 (2)
H21B	0.3657	0.0183	0.7290	0.219*	0.25 (2)
H21C	0.3080	0.0386	0.7307	0.219*	0.75 (2)
H21D	0.3644	−0.0001	0.6489	0.219*	0.75 (2)
C22	0.308 (4)	−0.0035 (6)	0.5616 (17)	0.131 (11)	0.25 (2)
H22A	0.2206	−0.0258	0.5726	0.197*	0.25 (2)
H22B	0.4149	−0.0202	0.5637	0.197*	0.25 (2)
H22C	0.2749	0.0119	0.4887	0.197*	0.25 (2)
C22A	0.1991 (19)	0.0263 (9)	0.5545 (10)	0.278 (10)	0.75 (2)
H22D	0.1128	0.0050	0.5723	0.418*	0.75 (2)
H22E	0.2493	0.0130	0.4940	0.418*	0.75 (2)
H22F	0.1473	0.0566	0.5305	0.418*	0.75 (2)
O6	0.4575 (3)	0.14642 (9)	0.45463 (15)	0.1016 (7)	
C24	0.4363 (5)	0.1513 (2)	0.3289 (3)	0.1361 (17)	
H24A	0.5340	0.1402	0.2976	0.163*	0.134 (10)
H24B	0.3991	0.1827	0.3000	0.163*	0.134 (10)
H24C	0.5035	0.1781	0.3099	0.163*	0.866 (10)
H24D	0.4758	0.1226	0.2963	0.163*	0.866 (10)
C25	0.292 (4)	0.1152 (11)	0.322 (2)	0.141 (17)	0.134 (10)
H25A	0.2418	0.1094	0.2429	0.211*	0.134 (10)
H25B	0.2052	0.1274	0.3609	0.211*	0.134 (10)
H25C	0.3368	0.0858	0.3569	0.211*	0.134 (10)
C25A	0.2606 (6)	0.1588 (2)	0.2843 (3)	0.119 (2)	0.866 (10)
H25D	0.2435	0.1612	0.2024	0.179*	0.866 (10)
H25E	0.2235	0.1878	0.3152	0.179*	0.866 (10)
H25F	0.1949	0.1325	0.3050	0.179*	0.866 (10)
O4	0.7444 (3)	0.00175 (6)	0.93343 (16)	0.0833 (6)	
C16	0.7172 (5)	−0.04847 (10)	0.9064 (3)	0.1012 (10)	
H16A	0.6205	−0.0525	0.8435	0.121*	0.64 (2)
H16B	0.8188	−0.0621	0.8841	0.121*	0.64 (2)
H16C	0.8276	−0.0636	0.9064	0.121*	0.36 (2)
H16D	0.6486	−0.0516	0.8300	0.121*	0.36 (2)
C17A	0.6831 (15)	−0.0717 (3)	1.0072 (9)	0.103 (3)	0.64 (2)
H17A	0.6643	−0.1052	0.9921	0.155*	0.64 (2)
H17B	0.5823	−0.0580	1.0284	0.155*	0.64 (2)
H17C	0.7797	−0.0675	1.0687	0.155*	0.64 (2)
C17	0.626 (4)	−0.0743 (8)	0.991 (2)	0.200 (15)	0.36 (2)

H17D	0.6391	−0.1082	0.9835	0.300*	0.36 (2)
H17E	0.5064	−0.0662	0.9761	0.300*	0.36 (2)
H17F	0.6769	−0.0649	1.0680	0.300*	0.36 (2)
O3A	0.864 (3)	0.0147 (5)	0.7829 (16)	0.086 (5)	0.43 (6)
O3	0.811 (5)	0.0131 (5)	0.7638 (18)	0.147 (6)	0.57 (6)
C1	1.0656 (3)	0.22467 (7)	0.92948 (18)	0.0498 (5)	
C2	1.0958 (3)	0.25755 (9)	1.0163 (2)	0.0696 (7)	
H2	1.0057	0.2748	1.0367	0.084*	
C3	1.2626 (4)	0.26446 (13)	1.0724 (3)	0.0914 (9)	
H3	1.2856	0.2865	1.1320	0.110*	
C4	1.3943 (4)	0.23944 (13)	1.0417 (3)	0.0927 (9)	
H4	1.5065	0.2447	1.0801	0.111*	
C5	1.3637 (3)	0.20698 (11)	0.9559 (3)	0.0828 (8)	
H5	1.4547	0.1899	0.9362	0.099*	
C6	1.1983 (3)	0.19911 (8)	0.8978 (2)	0.0630 (6)	
H6	1.1765	0.1770	0.8384	0.076*	
C7	0.8124 (3)	0.11715 (8)	0.82435 (16)	0.0495 (5)	
C8	0.8111 (3)	0.07924 (8)	0.89519 (17)	0.0530 (5)	
C9	0.8128 (3)	0.09659 (8)	1.00985 (16)	0.0500 (5)	
C10	0.8153 (3)	0.07444 (9)	1.11520 (19)	0.0665 (6)	
H10	0.8151	0.0413	1.1210	0.080*	
C11	0.8179 (4)	0.10257 (10)	1.2106 (2)	0.0746 (7)	
H11	0.8195	0.0882	1.2813	0.089*	
C12	0.8183 (3)	0.15172 (10)	1.2030 (2)	0.0675 (6)	
H12	0.8189	0.1698	1.2687	0.081*	
C13	0.8178 (3)	0.17465 (8)	1.10046 (17)	0.0552 (5)	
H13	0.8196	0.2078	1.0956	0.066*	
C14	0.8145 (2)	0.14609 (7)	1.00462 (16)	0.0459 (4)	
C15	0.7996 (4)	0.02893 (9)	0.8579 (2)	0.0707 (7)	
C18	0.7955 (3)	0.11677 (9)	0.69716 (17)	0.0602 (6)	
H18A	0.8128	0.0845	0.6720	0.072*	
H18B	0.8846	0.1368	0.6759	0.072*	
C19	0.6184 (3)	0.13479 (9)	0.63587 (17)	0.0592 (6)	
H19	0.6076	0.1680	0.6598	0.071*	
C20	0.4725 (4)	0.10741 (11)	0.6689 (2)	0.0766 (7)	
C23	0.6122 (4)	0.13528 (11)	0.5078 (2)	0.0769 (7)	
N1	0.8127 (2)	0.15922 (6)	0.88973 (13)	0.0476 (4)	
O1	0.7418 (2)	0.24538 (6)	0.90117 (16)	0.0747 (5)	
O2	0.8539 (2)	0.21559 (6)	0.73534 (14)	0.0744 (5)	
O5	0.7302 (3)	0.12815 (12)	0.46237 (18)	0.1306 (11)	
O7	0.3763 (3)	0.12298 (10)	0.7269 (2)	0.1108 (8)	
S1	0.85601 (7)	0.215323 (19)	0.85505 (5)	0.05376 (19)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O8	0.1224 (19)	0.0987 (18)	0.153 (2)	−0.0370 (15)	0.0491 (16)	−0.0315 (16)
C21	0.183 (5)	0.166 (5)	0.208 (6)	−0.085 (4)	0.062 (5)	−0.022 (4)

C22	0.145 (19)	0.073 (11)	0.176 (19)	−0.059 (11)	0.030 (14)	−0.044 (10)
C22A	0.154 (11)	0.36 (2)	0.300 (15)	−0.119 (15)	−0.001 (9)	0.009 (14)
O6	0.0976 (14)	0.156 (2)	0.0446 (9)	0.0306 (14)	−0.0028 (9)	−0.0103 (11)
C24	0.121 (3)	0.228 (6)	0.0530 (17)	0.026 (3)	−0.0003 (18)	−0.009 (2)
C25	0.23 (5)	0.13 (3)	0.081 (18)	0.04 (3)	0.07 (2)	0.027 (18)
C25A	0.123 (4)	0.161 (5)	0.061 (2)	0.031 (3)	−0.016 (2)	−0.007 (2)
O4	0.1192 (16)	0.0486 (10)	0.0908 (13)	−0.0107 (9)	0.0415 (11)	−0.0058 (8)
C16	0.131 (3)	0.0452 (15)	0.128 (3)	−0.0072 (16)	0.025 (2)	−0.0089 (16)
C17A	0.112 (6)	0.045 (3)	0.154 (6)	−0.013 (3)	0.030 (4)	0.032 (4)
C17	0.18 (2)	0.145 (17)	0.32 (3)	−0.077 (14)	0.16 (2)	−0.086 (19)
O3A	0.142 (11)	0.055 (5)	0.074 (6)	0.033 (5)	0.052 (5)	−0.004 (4)
O3	0.294 (16)	0.067 (4)	0.078 (5)	−0.004 (7)	0.032 (9)	−0.017 (3)
C1	0.0519 (11)	0.0445 (11)	0.0572 (11)	−0.0058 (8)	0.0207 (9)	0.0034 (8)
C2	0.0670 (15)	0.0653 (15)	0.0847 (16)	−0.0141 (12)	0.0351 (12)	−0.0182 (12)
C3	0.0797 (19)	0.104 (2)	0.093 (2)	−0.0374 (17)	0.0241 (15)	−0.0354 (17)
C4	0.0608 (16)	0.104 (2)	0.111 (2)	−0.0208 (16)	0.0101 (15)	−0.0108 (19)
C5	0.0522 (14)	0.0758 (18)	0.124 (3)	−0.0012 (12)	0.0265 (14)	−0.0021 (16)
C6	0.0608 (14)	0.0551 (13)	0.0788 (15)	−0.0043 (10)	0.0277 (11)	−0.0037 (11)
C7	0.0520 (11)	0.0522 (12)	0.0454 (10)	0.0024 (9)	0.0118 (8)	0.0022 (8)
C8	0.0620 (12)	0.0482 (12)	0.0501 (11)	0.0037 (9)	0.0138 (9)	0.0014 (9)
C9	0.0546 (11)	0.0490 (12)	0.0472 (10)	−0.0020 (9)	0.0120 (8)	0.0046 (8)
C10	0.0927 (17)	0.0560 (13)	0.0531 (12)	−0.0010 (12)	0.0198 (11)	0.0119 (10)
C11	0.0991 (19)	0.0809 (19)	0.0462 (12)	−0.0054 (15)	0.0203 (12)	0.0097 (11)
C12	0.0818 (16)	0.0740 (16)	0.0485 (12)	−0.0100 (13)	0.0166 (11)	−0.0077 (11)
C13	0.0605 (13)	0.0527 (12)	0.0530 (11)	−0.0096 (10)	0.0124 (9)	−0.0042 (9)
C14	0.0444 (10)	0.0499 (11)	0.0439 (10)	−0.0039 (8)	0.0093 (7)	0.0035 (8)
C15	0.1019 (19)	0.0535 (14)	0.0579 (13)	0.0065 (13)	0.0182 (14)	0.0002 (11)
C18	0.0746 (15)	0.0650 (14)	0.0438 (11)	0.0060 (11)	0.0184 (10)	0.0018 (9)
C19	0.0708 (14)	0.0671 (14)	0.0395 (10)	0.0045 (11)	0.0102 (9)	−0.0031 (9)
C20	0.0836 (18)	0.089 (2)	0.0559 (14)	−0.0028 (15)	0.0091 (12)	−0.0058 (13)
C23	0.0880 (19)	0.095 (2)	0.0470 (13)	0.0132 (15)	0.0102 (13)	−0.0012 (12)
N1	0.0553 (10)	0.0449 (9)	0.0426 (8)	−0.0067 (7)	0.0091 (7)	0.0054 (7)
O1	0.0578 (9)	0.0539 (10)	0.1148 (14)	0.0109 (8)	0.0222 (9)	0.0140 (9)
O2	0.0931 (12)	0.0708 (11)	0.0568 (9)	−0.0131 (9)	0.0077 (8)	0.0229 (8)
O5	0.1146 (18)	0.231 (3)	0.0512 (11)	0.0421 (19)	0.0288 (11)	0.0124 (14)
O7	0.1057 (17)	0.141 (2)	0.0982 (16)	−0.0020 (14)	0.0508 (13)	−0.0124 (14)
S1	0.0543 (3)	0.0467 (3)	0.0604 (3)	−0.0007 (2)	0.0111 (2)	0.0133 (2)

Geometric parameters (Å, °)

O8—C20	1.307 (4)	C17—H17F	0.9600
O8—C21	1.459 (5)	O3A—C15	1.176 (11)
C21—C22A	1.434 (7)	O3—C15	1.222 (14)
C21—C22	1.489 (8)	C1—C2	1.369 (3)
C21—H21A	0.9700	C1—C6	1.380 (3)
C21—H21B	0.9700	C1—S1	1.745 (2)
C21—H21C	0.9700	C2—C3	1.375 (4)
C21—H21D	0.9700	C2—H2	0.9300
C22—H21D	1.0530	C3—C4	1.361 (4)

C22—H22A	0.9600	C3—H3	0.9300
C22—H22B	0.9600	C4—C5	1.353 (4)
C22—H22C	0.9600	C4—H4	0.9300
C22A—H22D	0.9600	C5—C6	1.377 (4)
C22A—H22E	0.9600	C5—H5	0.9300
C22A—H22F	0.9600	C6—H6	0.9300
O6—C23	1.305 (3)	C7—C8	1.356 (3)
O6—C24	1.479 (4)	C7—N1	1.411 (3)
C24—C25A	1.406 (5)	C7—C18	1.492 (3)
C24—C25	1.516 (9)	C8—C9	1.444 (3)
C24—H24A	0.9700	C8—C15	1.474 (3)
C24—H24B	0.9700	C9—C14	1.387 (3)
C24—H24C	0.9700	C9—C10	1.394 (3)
C24—H24D	0.9700	C10—C11	1.378 (3)
C25—H25A	0.9600	C10—H10	0.9300
C25—H25B	0.9600	C11—C12	1.379 (4)
C25—H25C	0.9600	C11—H11	0.9300
C25A—H25D	0.9600	C12—C13	1.377 (3)
C25A—H25E	0.9600	C12—H12	0.9300
C25A—H25F	0.9600	C13—C14	1.388 (3)
O4—C15	1.312 (3)	C13—H13	0.9300
O4—C16	1.449 (3)	C14—N1	1.411 (2)
C16—C17A	1.433 (8)	C18—C19	1.537 (3)
C16—C17	1.527 (10)	C18—H18A	0.9700
C16—H16A	0.9700	C18—H18B	0.9700
C16—H16B	0.9700	C19—C20	1.498 (4)
C16—H16C	0.9700	C19—C23	1.514 (3)
C16—H16D	0.9700	C19—H19	0.9800
C17A—H17A	0.9600	C20—O7	1.200 (3)
C17A—H17B	0.9600	C23—O5	1.179 (3)
C17A—H17C	0.9600	N1—S1	1.6754 (17)
C17—H17D	0.9600	O1—S1	1.4178 (18)
C17—H17E	0.9600	O2—S1	1.4197 (17)
C20—O8—C21	116.7 (3)	H17A—C17A—H17C	109.5
C22A—C21—O8	109.9 (6)	H17B—C17A—H17C	109.5
O8—C21—C22	103.6 (9)	C16—C17—H17D	109.5
O8—C21—H21A	111.0	C16—C17—H17E	109.5
C22—C21—H21A	111.0	H17D—C17—H17E	109.5
C22A—C21—H21B	137.7	C16—C17—H17F	109.5
O8—C21—H21B	111.0	H17D—C17—H17F	109.5
C22—C21—H21B	111.0	H17E—C17—H17F	109.5
H21A—C21—H21B	109.0	C2—C1—C6	121.3 (2)
C22A—C21—H21C	124.3	C2—C1—S1	119.81 (17)
O8—C21—H21C	112.4	C6—C1—S1	118.83 (17)
C22—C21—H21C	141.9	C1—C2—C3	118.4 (2)
O8—C21—H21D	109.4	C1—C2—H2	120.8
H21A—C21—H21D	137.2	C3—C2—H2	120.8
H21C—C21—H21D	108.5	C4—C3—C2	120.7 (3)
C21—C22—H22A	109.5	C4—C3—H3	119.7

H21D—C22—H22A	96.1	C2—C3—H3	119.7
C21—C22—H22B	109.5	C5—C4—C3	120.7 (3)
H21D—C22—H22B	79.2	C5—C4—H4	119.7
H22A—C22—H22B	109.5	C3—C4—H4	119.7
C21—C22—H22C	109.5	C4—C5—C6	120.3 (3)
H21D—C22—H22C	147.3	C4—C5—H5	119.9
H22A—C22—H22C	109.5	C6—C5—H5	119.9
H22B—C22—H22C	109.5	C5—C6—C1	118.6 (2)
C21—C22A—H22D	109.5	C5—C6—H6	120.7
C21—C22A—H22E	109.5	C1—C6—H6	120.7
H22D—C22A—H22E	109.5	C8—C7—N1	108.11 (17)
C21—C22A—H22F	109.5	C8—C7—C18	127.9 (2)
H22D—C22A—H22F	109.5	N1—C7—C18	123.68 (18)
H22E—C22A—H22F	109.5	C7—C8—C9	108.83 (19)
C23—O6—C24	115.4 (2)	C7—C8—C15	124.7 (2)
C25A—C24—O6	108.1 (3)	C9—C8—C15	126.4 (2)
O6—C24—C25	86.3 (10)	C14—C9—C10	119.08 (19)
C25A—C24—H24A	134.8	C14—C9—C8	106.99 (17)
O6—C24—H24A	114.3	C10—C9—C8	133.9 (2)
C25—C24—H24A	114.3	C11—C10—C9	118.7 (2)
C25A—C24—H24B	61.3	C11—C10—H10	120.6
O6—C24—H24B	114.3	C9—C10—H10	120.6
C25—C24—H24B	114.3	C10—C11—C12	121.1 (2)
H24A—C24—H24B	111.4	C10—C11—H11	119.5
C25A—C24—H24C	110.1	C12—C11—H11	119.5
O6—C24—H24C	110.1	C13—C12—C11	121.6 (2)
C25—C24—H24C	160.6	C13—C12—H12	119.2
C25A—C24—H24D	110.1	C11—C12—H12	119.2
O6—C24—H24D	110.1	C12—C13—C14	117.0 (2)
C25—C24—H24D	73.8	C12—C13—H13	121.5
H24B—C24—H24D	135.2	C14—C13—H13	121.5
H24C—C24—H24D	108.4	C9—C14—C13	122.51 (18)
C24—C25—H25A	109.5	C9—C14—N1	107.75 (16)
C24—C25—H25B	109.5	C13—C14—N1	129.74 (19)
H25A—C25—H25B	109.5	O3A—C15—O4	124.6 (8)
C24—C25—H25C	109.5	O3—C15—O4	120.5 (9)
H25A—C25—H25C	109.5	O3A—C15—C8	122.8 (9)
H25B—C25—H25C	109.5	O3—C15—C8	127.6 (8)
C24—C25A—H25D	109.5	O4—C15—C8	111.0 (2)
C24—C25A—H25E	109.5	C7—C18—C19	112.18 (18)
H25D—C25A—H25E	109.5	C7—C18—H18A	109.2
C24—C25A—H25F	109.5	C19—C18—H18A	109.2
H25D—C25A—H25F	109.5	C7—C18—H18B	109.2
H25E—C25A—H25F	109.5	C19—C18—H18B	109.2
C15—O4—C16	117.8 (2)	H18A—C18—H18B	107.9
C17A—C16—O4	107.4 (5)	C20—C19—C23	112.8 (2)
O4—C16—C17	112.5 (10)	C20—C19—C18	112.7 (2)
C17A—C16—H16A	110.2	C23—C19—C18	109.50 (19)
O4—C16—H16A	110.2	C20—C19—H19	107.2

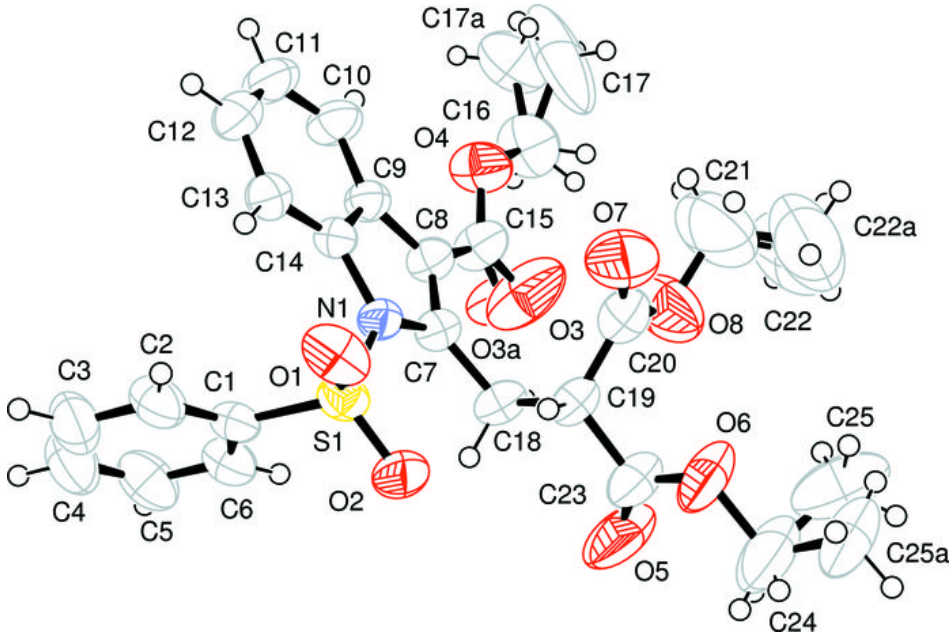
C17—C16—H16A	93.1	C23—C19—H19	107.2
C17A—C16—H16B	110.2	C18—C19—H19	107.2
O4—C16—H16B	110.2	O7—C20—O8	124.6 (3)
C17—C16—H16B	120.7	O7—C20—C19	124.6 (3)
H16A—C16—H16B	108.5	O8—C20—C19	110.8 (3)
C17A—C16—H16C	96.5	O5—C23—O6	124.7 (2)
O4—C16—H16C	109.1	O5—C23—C19	125.5 (2)
C17—C16—H16C	109.1	O6—C23—C19	109.8 (2)
H16A—C16—H16C	121.9	C7—N1—C14	108.31 (15)
C17A—C16—H16D	125.6	C7—N1—S1	128.78 (14)
O4—C16—H16D	109.1	C14—N1—S1	121.10 (13)
C17—C16—H16D	109.1	O1—S1—O2	119.72 (11)
H16B—C16—H16D	93.2	O1—S1—N1	106.66 (9)
H16C—C16—H16D	107.8	O2—S1—N1	106.67 (9)
C16—C17A—H17A	109.5	O1—S1—C1	109.16 (11)
C16—C17A—H17B	109.5	O2—S1—C1	109.79 (10)
H17A—C17A—H17B	109.5	N1—S1—C1	103.56 (9)
C16—C17A—H17C	109.5		
C20—O8—C21—C22A	107.7 (14)	C9—C8—C15—O3	−174 (2)
C20—O8—C21—C22	157.7 (14)	C7—C8—C15—O4	−159.0 (2)
C23—O6—C24—C25A	174.5 (4)	C9—C8—C15—O4	17.2 (4)
C23—O6—C24—C25	125.5 (15)	C8—C7—C18—C19	106.4 (3)
C15—O4—C16—C17A	171.4 (5)	N1—C7—C18—C19	−65.8 (3)
C15—O4—C16—C17	−170.9 (16)	C7—C18—C19—C20	−55.7 (3)
C6—C1—C2—C3	−0.4 (4)	C7—C18—C19—C23	177.9 (2)
S1—C1—C2—C3	−179.3 (2)	C21—O8—C20—O7	−0.3 (5)
C1—C2—C3—C4	0.5 (5)	C21—O8—C20—C19	179.0 (4)
C2—C3—C4—C5	−0.5 (5)	C23—C19—C20—O7	−126.3 (3)
C3—C4—C5—C6	0.5 (5)	C18—C19—C20—O7	109.0 (3)
C4—C5—C6—C1	−0.5 (4)	C23—C19—C20—O8	54.3 (3)
C2—C1—C6—C5	0.4 (4)	C18—C19—C20—O8	−70.3 (3)
S1—C1—C6—C5	179.3 (2)	C24—O6—C23—O5	−1.7 (5)
N1—C7—C8—C9	−0.7 (2)	C24—O6—C23—C19	176.1 (3)
C18—C7—C8—C9	−174.0 (2)	C20—C19—C23—O5	−134.2 (4)
N1—C7—C8—C15	176.0 (2)	C18—C19—C23—O5	−7.8 (4)
C18—C7—C8—C15	2.8 (4)	C20—C19—C23—O6	48.0 (3)
C7—C8—C9—C14	0.2 (2)	C18—C19—C23—O6	174.3 (2)
C15—C8—C9—C14	−176.5 (2)	C8—C7—N1—C14	1.0 (2)
C7—C8—C9—C10	−178.8 (2)	C18—C7—N1—C14	174.60 (18)
C15—C8—C9—C10	4.5 (4)	C8—C7—N1—S1	165.55 (15)
C14—C9—C10—C11	0.5 (4)	C18—C7—N1—S1	−20.9 (3)
C8—C9—C10—C11	179.4 (2)	C9—C14—N1—C7	−0.9 (2)
C9—C10—C11—C12	−0.1 (4)	C13—C14—N1—C7	179.0 (2)
C10—C11—C12—C13	−0.7 (4)	C9—C14—N1—S1	−166.83 (14)
C11—C12—C13—C14	0.9 (4)	C13—C14—N1—S1	13.1 (3)
C10—C9—C14—C13	−0.3 (3)	C7—N1—S1—O1	142.65 (18)
C8—C9—C14—C13	−179.48 (19)	C14—N1—S1—O1	−54.54 (17)
C10—C9—C14—N1	179.60 (19)	C7—N1—S1—O2	13.6 (2)
C8—C9—C14—N1	0.4 (2)	C14—N1—S1—O2	176.42 (15)

C12—C13—C14—C9	−0.4 (3)	C7—N1—S1—C1	−102.22 (18)
C12—C13—C14—N1	179.7 (2)	C14—N1—S1—C1	60.59 (17)
C16—O4—C15—O3A	−17.5 (15)	C2—C1—S1—O1	−0.7 (2)
C16—O4—C15—O3	7(2)	C6—C1—S1—O1	−179.55 (17)
C16—O4—C15—C8	176.8 (2)	C2—C1—S1—O2	132.41 (19)
C7—C8—C15—O3A	34.9 (14)	C6—C1—S1—O2	−46.5 (2)
C9—C8—C15—O3A	−148.9 (13)	C2—C1—S1—N1	−114.0 (2)
C7—C8—C15—O3	10 (2)	C6—C1—S1—N1	67.12 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.93	2.52	2.895 (3)	104
C10—H10···O4	0.93	2.46	2.944 (3)	113
C13—H13···O1	0.93	2.51	3.059 (3)	118
C18—H18A···O3	0.97	2.28	3.004 (15)	131
C18—H18B···O2	0.97	2.34	2.827 (3)	110
C19—H19···O2	0.98	2.39	3.025 (3)	122

Fig. 1


C17a
C11
C10
C17
C16
C12
O4
C9
C21
C13
C8
C15
O7
C22a
C14
N1
O3
O8
C22
C2
O3a
C20
C3
C1
O1
C7
C19
S1
C18
O6
C25
C23
O2
C4
C5
C6
O5
C25a
C24