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2-*tert*-Butyl-4-(phenylsulfonyl)-1,2,3,4-tetrahydropyrrolo[3,4-*b*]indole

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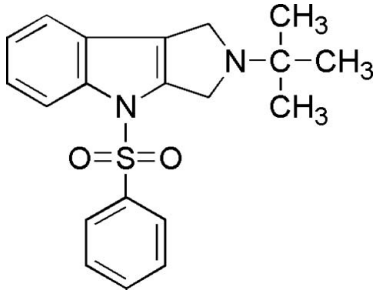
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; R factor = 0.062; wR factor = 0.283; data-to-parameter ratio = 17.8.

The indole and pyrrolidine ring systems of the title compound, $C_{20}H_{22}N_2O_2S$, are essentially coplanar. The angle between the planes of the phenylsulfonyl group and the indole ring system is 80.2 (2)°. The pyrrolidine N atom has bond angles totalling 343.1°, indicating some flattening from a purely pyramidal N atom, compared with 324° for ammonia.

Related literature

For the synthesis of the title compound and related chemistry, see: Gribble (2003); Gribble *et al.* (2005); Roy, Kishbaugh *et al.* (2007); Kishbaugh & Gribble (2002); Mohanakrishnan & Srinivasan (1995). For a related structure, see: Kishbaugh *et al.* (2007).



Experimental

Crystal data

$C_{20}H_{22}N_2O_2S$
M_r = 354.46
Orthorhombic, *Pbca*
a = 18.113 (5) Å
b = 16.140 (9) Å
c = 12.146 (8) Å
V = 3551 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 296 K
0.50 × 0.50 × 0.40 mm

Data collection

Rigaku AFC-6*S* diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.907, T_{max} = 0.925
4059 measured reflections
4059 independent reflections
1736 reflections with $I > 2\sigma(I)$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.283
S = 1.06
4059 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2137).

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supplementary materials

Acta Cryst. (2007). E**63**, o3409 [**doi:10.1107/S1600536807032412**]

2-*tert*-Butyl-4-(phenylsulfonyl)-1,2,3,4-tetrahydropyrrolo[3,4-*b*]indole

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Comment

The title compound, $C_{20}H_{22}N_2O_2S$, was synthesized as a precursor to the corresponding pyrrolo[3,4-*b*]indole as a stable synthetic analogue of indole-2,3-quinodimethane. The *tert*-butyl group was chosen to block oxidation of the pyrrolidine ring by adventitious oxygen, a side-reaction that is more pronounced with the isopropyl analogue discussed in the preceding paper (Kishbaugh *et al.*, 2007). The X-ray crystallographic analysis confirms the molecular structure and atom connectivity for (I) that we had proposed for this compound based on NMR spectroscopy and chemical reactions (Kishbaugh & Gribble, 2002).

The tertiary butyl group has one methyl group (C18) bisecting the molecular plane with torsion angles of 65.2 (5)° for C1—N2—C17—C18 and −67.5 (5)° for C2—N2—C17—C18. The other two methyl groups, C19 and C20, adopt a *gauche* butane arrangement with the pyrrolidine methylene groups C3 and C1, respectively.

Experimental

To a refluxing solution of 2,3-dibromomethyl-1-(phenylsulfonyl)indole (294 mg, 0.664 mmol) (Mohanakrishnan & Srinivasan, 1995) and K_2CO_3 (312 mg, 2.25 mmol) in tetrahydrofuran (10 ml) was added a solution of *tert*-butylamine (90 µ*L*, 0.80 mmol) in tetrahydrofuran (15 ml) slowly *via* addition funnel. After 10 h, the reaction mixture was cooled to rt, and the opaque solution was filtered through a Celite pad with ethyl acetate rinses. The resulting combined yellow solution was concentrated *in vacuo* to yield a pale yellow solid which was purified by column chromatography (2:1 hexanes: ethyl acetate) to yield (I) as a white solid (161 mg, 68%): m.p. 457–458 K. As (I) was unstable toward oxidation, an elemental analysis was not attempted. Recrystallization from hexane–dichloromethane (3:1) yielded crystals that were suitable for X-ray crystallography.

Refinement

The H atoms were included in the riding model approximation with C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.19–1.20U_{eq}(C).

Figures

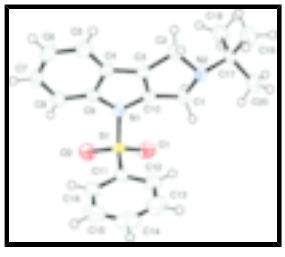


Fig. 1. Molecular structure of (I) showing atom labelling and 50% probability displacement ellipsoids.

2-*tert*-Butyl-4-(phenylsulfonyl)-1,2,3,4-tetrahydropyrrolo[3,4-*b*]indole

Crystal data

$C_{20}H_{22}N_2O_2S$	$D_x = 1.326$ Mg m^{-3}
$M_r = 354.46$	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
Orthorhombic, *Pbca*	Cell parameters from 20 reflections
$a = 18.113$ (5) Å	$\theta = 10.4$–$13.2°$
$b = 16.140$ (9) Å	$\mu = 0.20$ mm^{-1}
$c = 12.146$ (8) Å	$T = 296$ K
$V = 3551$ (3) Å^3	Prism, yellow
$Z = 8$	0.50 × 0.50 × 0.40 mm
$F_{000} = 1504$	

Data collection

Rigaku AFC-6S diffractometer	$R_{int} = 0.0000$
Radiation source: normal-focus sealed tube	$\theta_{max} = 27.5°$
Monochromator: graphite	$\theta_{min} = 2.3°$
$T = 296$ K	$h = 0 \rightarrow 23$
$\omega/2\theta$ scans	$k = 0 \rightarrow 20$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -15 \rightarrow 0$
$T_{min} = 0.907$, $T_{max} = 0.925$	3 standard reflections
4059 measured reflections	every 150 reflections
4059 independent reflections	intensity decay: none
1736 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.062$	$w = 1/[\sigma^2(F_o^2) + (0.1437P)^2 + 2.0864P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.283$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
4059 reflections	$\Delta\rho_{min} = -0.41$ e Å^{-3}
228 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0011 (9)
Secondary atom site location: difference Fourier map	

Special details

Experimental. ^{1}H (CD_2Cl_2) δ 7.98 (dd, 1H, 1, 7 Hz), 7.83–7.87 (m, 2H), 7.43–7.58 (m, 3H), 7.19–7.35 (m, 3H), 4.30 (m, 2H), 3.96 (m, 2H), 1.18 (s, 9H); ^{13}C ($CDCl_3$) δ 139.7, 138.5, 134.0, 129.6, 128.9, 126.9, 126.1, 123.9, 123.8, 122.9, 119.4, 114.3, 54.1, 48.8, 46.7, 26.3; IR (film) λ_{max} 3061, 2962, 1447, 1364, 1216, 1171, 1089, 995, 913, 748, 720, 682 cm-1; UV (EtOH) λ_{max} 258 nm.

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	0.25733 (7)	−0.01040 (8)	0.61347 (10)	0.0343 (4)
O1	0.21803 (19)	0.0451 (2)	0.5441 (3)	0.0461 (10)
O2	0.2272 (2)	−0.0358 (3)	0.7161 (3)	0.0517 (11)
N1	0.3376 (2)	0.0366 (3)	0.6408 (3)	0.0347 (10)
N2	0.4291 (2)	0.1561 (2)	0.4210 (3)	0.0325 (10)
C1	0.3561 (3)	0.1342 (3)	0.4654 (4)	0.0386 (13)
H5	0.3264	0.1050	0.4115	0.046*
H6	0.3295	0.1828	0.4907	0.046*
C2	0.4876 (3)	0.1369 (3)	0.5033 (4)	0.0380 (12)
H7	0.5045	0.1866	0.5405	0.046*
H8	0.5295	0.1096	0.4692	0.046*
C3	0.4484 (3)	0.0802 (3)	0.5810 (4)	0.0313 (11)
C4	0.4609 (3)	0.0343 (3)	0.6813 (4)	0.0327 (12)
C5	0.5234 (3)	0.0151 (3)	0.7450 (5)	0.0408 (13)
H1	0.5703	0.0311	0.7222	0.049*
C6	0.5136 (4)	−0.0279 (4)	0.8423 (5)	0.0486 (15)
H3	0.5545	−0.0399	0.8859	0.058*
C7	0.4448 (3)	−0.0533 (4)	0.8758 (4)	0.0453 (14)
H4	0.4400	−0.0823	0.9416	0.054*
C8	0.3825 (3)	−0.0368 (3)	0.8138 (4)	0.0402 (13)
H2	0.3362	−0.0544	0.8371	0.048*
C9	0.3908 (3)	0.0064 (3)	0.7163 (4)	0.0318 (11)
C10	0.3770 (3)	0.0794 (3)	0.5584 (4)	0.0310 (11)
C11	0.2819 (3)	−0.0994 (3)	0.5378 (4)	0.0335 (12)
C12	0.2947 (3)	−0.0918 (4)	0.4257 (5)	0.0465 (14)
H9	0.2884	−0.0412	0.3904	0.056*
C13	0.3169 (4)	−0.1611 (4)	0.3675 (6)	0.0621 (19)
H10	0.3267	−0.1565	0.2925	0.075*
C14	0.3247 (4)	−0.2362 (4)	0.4185 (7)	0.065 (2)

H11	0.3395	−0.2823	0.3783	0.078*
C15	0.3106 (4)	−0.2435 (4)	0.5302 (7)	0.0639 (19)
H12	0.3155	−0.2946	0.5648	0.077*
C16	0.2893 (3)	−0.1747 (4)	0.5906 (5)	0.0474 (15)
H13	0.2800	−0.1792	0.6657	0.057*
C17	0.4340 (3)	0.2364 (3)	0.3643 (4)	0.0362 (12)
C18	0.4207 (4)	0.3109 (3)	0.4420 (5)	0.0521 (15)
H14	0.3690	0.3239	0.4434	0.062*
H15	0.4479	0.3580	0.4158	0.062*
H16	0.4370	0.2970	0.5149	0.062*
C19	0.5108 (3)	0.2429 (4)	0.3128 (6)	0.0565 (17)
H17	0.5442	0.2676	0.3646	0.068*
H18	0.5083	0.2766	0.2478	0.068*
H19	0.5281	0.1886	0.2935	0.068*
C20	0.3769 (3)	0.2372 (4)	0.2715 (5)	0.0551 (17)
H20	0.3990	0.2163	0.2052	0.066*
H21	0.3602	0.2929	0.2595	0.066*
H22	0.3357	0.2029	0.2915	0.066*

Atomic displacement parameters (Å^2)

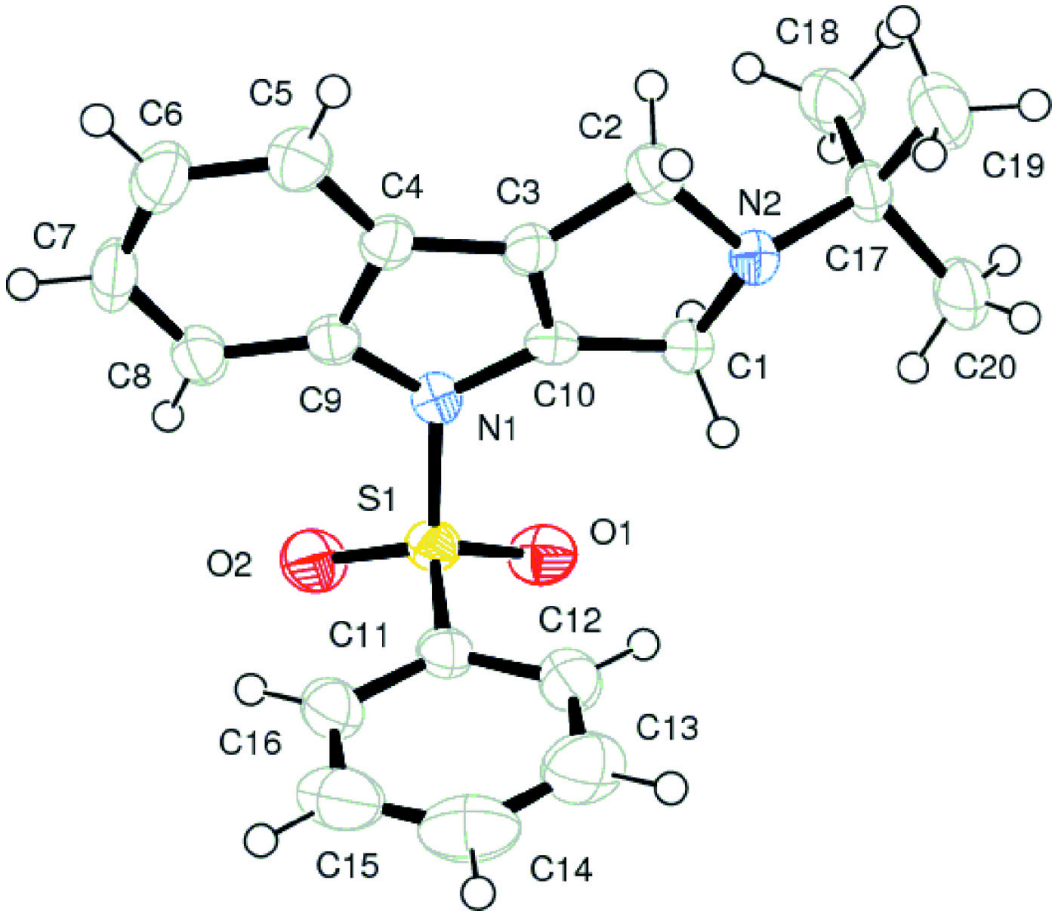
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0316 (7)	0.0367 (7)	0.0347 (7)	−0.0061 (5)	0.0014 (6)	−0.0008 (6)
O1	0.037 (2)	0.044 (2)	0.058 (2)	0.0048 (17)	−0.0071 (19)	−0.0025 (19)
O2	0.048 (2)	0.064 (3)	0.042 (2)	−0.016 (2)	0.0144 (19)	−0.003 (2)
N1	0.039 (2)	0.035 (2)	0.031 (2)	−0.0063 (19)	−0.0016 (19)	0.0039 (18)
N2	0.037 (2)	0.032 (2)	0.028 (2)	−0.0021 (18)	−0.0027 (18)	0.0041 (18)
C1	0.043 (3)	0.037 (3)	0.037 (3)	−0.007 (2)	−0.008 (2)	0.007 (2)
C2	0.038 (3)	0.038 (3)	0.038 (3)	−0.002 (2)	0.004 (2)	0.002 (2)
C3	0.036 (3)	0.028 (3)	0.030 (3)	−0.002 (2)	−0.003 (2)	−0.002 (2)
C4	0.044 (3)	0.025 (2)	0.030 (3)	0.002 (2)	−0.004 (2)	−0.004 (2)
C5	0.035 (3)	0.042 (3)	0.045 (3)	0.004 (2)	−0.002 (3)	0.001 (3)
C6	0.060 (4)	0.045 (4)	0.041 (3)	0.007 (3)	−0.015 (3)	0.004 (3)
C7	0.063 (4)	0.045 (3)	0.029 (3)	0.005 (3)	−0.005 (3)	0.011 (3)
C8	0.050 (3)	0.037 (3)	0.033 (3)	−0.007 (3)	0.004 (3)	0.001 (2)
C9	0.039 (3)	0.026 (2)	0.030 (3)	−0.003 (2)	−0.001 (2)	−0.004 (2)
C10	0.034 (3)	0.026 (2)	0.033 (3)	−0.007 (2)	−0.004 (2)	−0.003 (2)
C11	0.028 (3)	0.035 (3)	0.037 (3)	−0.006 (2)	−0.001 (2)	0.002 (2)
C12	0.054 (4)	0.043 (3)	0.043 (3)	−0.006 (3)	0.005 (3)	0.000 (3)
C13	0.067 (4)	0.067 (5)	0.052 (4)	−0.009 (4)	0.014 (3)	−0.021 (3)
C14	0.054 (4)	0.052 (4)	0.091 (6)	0.002 (3)	−0.006 (4)	−0.025 (4)
C15	0.056 (4)	0.042 (4)	0.094 (6)	−0.006 (3)	−0.025 (4)	0.002 (4)
C16	0.043 (3)	0.046 (3)	0.054 (4)	−0.010 (3)	−0.009 (3)	0.012 (3)
C17	0.045 (3)	0.033 (3)	0.030 (3)	−0.001 (2)	0.004 (2)	0.008 (2)
C18	0.066 (4)	0.036 (3)	0.054 (4)	−0.004 (3)	0.004 (3)	0.001 (3)
C19	0.051 (4)	0.062 (4)	0.056 (4)	−0.006 (3)	0.006 (3)	0.020 (3)
C20	0.061 (4)	0.055 (4)	0.050 (4)	−0.006 (3)	−0.007 (3)	0.023 (3)

Geometric parameters (Å, °)

S1—O1	1.421 (4)	C8—C9	1.383 (7)
S1—O2	1.422 (4)	C8—H2	0.9300
S1—N1	1.673 (4)	C11—C16	1.380 (7)
S1—C11	1.762 (5)	C11—C12	1.387 (7)
N1—C10	1.411 (6)	C12—C13	1.382 (8)
N1—C9	1.417 (6)	C12—H9	0.9300
N2—C17	1.470 (6)	C13—C14	1.370 (10)
N2—C1	1.472 (6)	C13—H10	0.9300
N2—C2	1.490 (7)	C14—C15	1.386 (10)
C1—C10	1.483 (7)	C14—H11	0.9300
C1—H5	0.9700	C15—C16	1.386 (9)
C1—H6	0.9700	C15—H12	0.9300
C2—C3	1.494 (7)	C16—H13	0.9300
C2—H7	0.9700	C17—C19	1.528 (8)
C2—H8	0.9700	C17—C20	1.530 (7)
C3—C10	1.322 (6)	C17—C18	1.548 (7)
C3—C4	1.443 (7)	C18—H14	0.9600
C4—C5	1.406 (7)	C18—H15	0.9600
C4—C9	1.412 (7)	C18—H16	0.9600
C5—C6	1.382 (8)	C19—H17	0.9600
C5—H1	0.9300	C19—H18	0.9600
C6—C7	1.374 (8)	C19—H19	0.9600
C6—H3	0.9300	C20—H20	0.9600
C7—C8	1.382 (8)	C20—H21	0.9600
C7—H4	0.9300	C20—H22	0.9600
O1—S1—O2	120.7 (3)	C3—C10—N1	110.6 (4)
O1—S1—N1	105.5 (2)	C3—C10—C1	113.7 (4)
O2—S1—N1	106.9 (2)	N1—C10—C1	134.7 (4)
O1—S1—C11	109.3 (2)	C16—C11—C12	121.2 (5)
O2—S1—C11	108.6 (3)	C16—C11—S1	120.0 (4)
N1—S1—C11	104.7 (2)	C12—C11—S1	118.8 (4)
C10—N1—C9	106.4 (4)	C13—C12—C11	118.7 (6)
C10—N1—S1	121.4 (3)	C13—C12—H9	120.7
C9—N1—S1	124.4 (3)	C11—C12—H9	120.7
C17—N2—C1	116.0 (4)	C14—C13—C12	120.9 (6)
C17—N2—C2	117.1 (4)	C14—C13—H10	119.5
C1—N2—C2	110.0 (4)	C12—C13—H10	119.5
N2—C1—C10	101.1 (4)	C13—C14—C15	120.0 (7)
N2—C1—H5	111.5	C13—C14—H11	120.0
C10—C1—H5	111.5	C15—C14—H11	120.0
N2—C1—H6	111.5	C14—C15—C16	120.1 (6)
C10—C1—H6	111.5	C14—C15—H12	120.0
H5—C1—H6	109.4	C16—C15—H12	120.0
N2—C2—C3	102.3 (4)	C11—C16—C15	119.1 (6)
N2—C2—H7	111.3	C11—C16—H13	120.4
C3—C2—H7	111.3	C15—C16—H13	120.4

N2—C2—H8	111.3	N2—C17—C19	107.9 (4)
C3—C2—H8	111.3	N2—C17—C20	108.2 (4)
H7—C2—H8	109.2	C19—C17—C20	108.3 (5)
C10—C3—C4	108.9 (4)	N2—C17—C18	112.9 (4)
C10—C3—C2	109.9 (4)	C19—C17—C18	109.7 (5)
C4—C3—C2	140.6 (5)	C20—C17—C18	109.7 (5)
C5—C4—C9	119.1 (5)	C17—C18—H14	109.5
C5—C4—C3	134.7 (5)	C17—C18—H15	109.5
C9—C4—C3	106.1 (4)	H14—C18—H15	109.5
C6—C5—C4	118.6 (5)	C17—C18—H16	109.5
C6—C5—H1	120.7	H14—C18—H16	109.5
C4—C5—H1	120.7	H15—C18—H16	109.5
C7—C6—C5	121.3 (5)	C17—C19—H17	109.5
C7—C6—H3	119.4	C17—C19—H18	109.5
C5—C6—H3	119.4	H17—C19—H18	109.5
C6—C7—C8	121.5 (5)	C17—C19—H19	109.5
C6—C7—H4	119.3	H17—C19—H19	109.5
C8—C7—H4	119.3	H18—C19—H19	109.5
C7—C8—C9	118.3 (5)	C17—C20—H20	109.5
C7—C8—H2	120.8	C17—C20—H21	109.5
C9—C8—H2	120.8	H20—C20—H21	109.5
C8—C9—C4	121.2 (5)	C17—C20—H22	109.5
C8—C9—N1	130.8 (5)	H20—C20—H22	109.5
C4—C9—N1	107.9 (4)	H21—C20—H22	109.5

Fig. 1


C18
C5
C6
C2
C19
C4
C3
N2
C7
C17
C8
C9
C10
C1
C20
N1
S1
O1
O2
C11
C12
C16
C13
C15
C14