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

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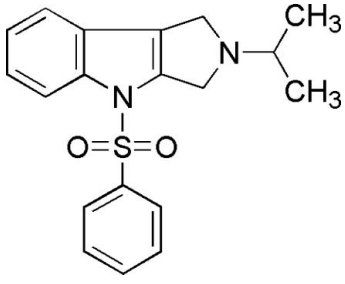
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.006 Å; disorder in main residue; R factor = 0.054; wR factor = 0.185; data-to-parameter ratio = 17.5.

The indole ring and the pyrrolidine C atoms of the title compound, $C_{19}H_{19}N_2O_2S$, are essentially coplanar. The angle between the planes of the phenylsulfonyl ring and the indole ring system is 88.0 (1)°. The pyrrolidine N atom is disordered over two positions, with a site-occupancy ratio of 3:2, and has bond angles totaling 357.0°, indicating significant flattening from a purely pyramidal N atom, which in ammonia has angles totaling 324°.

Related literature

For synthesis and chemistry see: Gribble (2003); Gribble *et al.* (2005); Roy, Kishbaugh *et al.* (2007); Roy, Pelkey *et al.* (2007); Kishbaugh & Gribble (2002); Mohanakrishnan & Srinivasan (1995).



Experimental

Crystal data

$C_{19}H_{19}N_2O_2S$
M_r = 339.42
Triclinic, $P\overline{1}$
a = 8.196 (2) Å
b = 9.559 (3) Å
c = 12.359 (2) Å
α = 70.801 (19)°
β = 98.54 (2)°
γ = 108.88 (2)°
V = 864.2 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 296 K
0.40 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-6S diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.924, T_{max} = 0.961
3977 measured reflections
3977 independent reflections
1720 reflections with $I > 2\sigma(I)$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.185
S = 1.01
3977 reflections
227 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 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: FL2136).

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supplementary materials

Acta Cryst. (2007). E**63**, o3408 [**doi:10.1107/S1600536807032400**]

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

T. L. S. Kishbaugh, G. W. Gribble and J. P. Jasinski

Comment

The title compound, $C_{19}H_{19}N_2O_2S$, was synthesized as a precursor to the corresponding pyrrolo[3,4-*b*]indole as a stable synthetic analogue of indole-2,3-quinodimethane. The X-ray crystallographic analysis, Fig. 1, confirms the molecular structure and atom connectivity that we had proposed for this compound based on NMR spectroscopy and chemical reactions (Kishbaugh & Gribble, 2002).

The three isopropyl carbons lie nearly in the molecular plane with torsion angles of 19.4 (7)° (C1—N2—C17—C18) and −18.1 (7)° (C2—N2—C17—C19), respectively.

Experimental

To a 35°C solution of 2,3-dibromomethyl-1-(phenylsulfonyl)indole (470 mg, 1.06 mmol) (Mohanakrishnan & Srinivasan, 1995) and K_2CO_3 (404 mg, 2.90 mmol) in tetrahydrofuran (20 ml) was added a solution of isopropyl amine (100 µ*L*, 1.17 mmol) in tetrahydrofuran (20 ml) slowly *via* an addition funnel. After 2 h, the opaque solution was filtered through a Celite pad with ethyl acetate rinses. The combined yellow organic solution was concentrated in vacuo to yield (I) as a pale yellow solid. After column chromatography (1:1; ethyl acetate: hexanes), 197 mg of (I) (61%) was isolated as a white solid: m.p. 419–420 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 = 0.93–0.98 Å, and with U_{iso}(H) = 1.19–1.20U_{eq}(C).

Figures

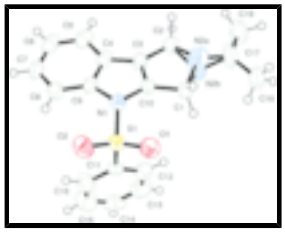


Fig. 1. Molecular structure of (I) showing atom labeling and 50% probability displacement ellipsoids. N2 is disordered with N2a (0.6) and N2b (0.4) at partial occupancy.

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

Crystal data

$C_{19}H_{19}N_2O_2S$	$Z = 2$
$M_r = 339.42$	$F_{000} = 358$
Triclinic, $P\overline{1}$	$D_x = 1.304$ Mg m^{-3}
$a = 8.196$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$b = 9.559$ (3) Å	Cell parameters from 20 reflections
$c = 12.359$ (2) Å	$\theta = 6.4$–9.1°
$\alpha = 70.801$ (19)°	$\mu = 0.20$ mm^{-1}
$\beta = 98.54$ (2)°	$T = 296$ K
$\gamma = 108.88$ (2)°	Prism, colorless
$V = 864.2$ (4) Å^3	0.40 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-6S diffractometer	$R_{int} =$
Radiation source: normal-focus sealed tube	$\theta_{max} = 27.5^\circ$
Monochromator: graphite	$\theta_{min} = 1.8^\circ$
$T = 296$ K	$h = -10 \rightarrow 10$
$\omega/2\theta$ scans	$k = 0 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = -15 \rightarrow 16$
$T_{min} = 0.924$, $T_{max} = 0.961$	3 standard reflections
3977 measured reflections	every 150 reflections
3977 independent reflections	intensity decay: none
1720 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H-atom parameters constrained
$wR(F^2) = 0.185$	$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.1181P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3977 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
227 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. ^{1}H (CD_2Cl_2) δ 8.00 (m, 1H), 7.87 (m, 2H), 7.51–7.57 (m, 1H), 7.42–7.47 (m, 2H), 7.33–7.37 (m, 2H), 7.20–7.30 (m, 2H), 4.27–4.30 (m, 2H), 3.91–3.94 (m, 2H), 2.96 (septet, 1H, 6 Hz), 1.20 (d, 6H, 6 Hz); ^{13}C (CD_2Cl_2) δ 140.5, 139.9, 138.5, 134.3, 129.8, 127.0, 126.4, 124.1, 124.0, 123.9, 119.6, 114.5, 54.5, 52.7, 50.7, 21.3; IR (film) ν_{max} 2968, 2889, 2789, 1448, 1371, 1179, 996, 748, 726, 685 cm^{-1}; UV (EtOH) ν_{max} 260 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}	Occ. (<1)
S1	0.81739 (12)	0.75976 (11)	0.83788 (8)	0.0434 (3)	
O1	0.8348 (3)	0.6439 (3)	0.7955 (2)	0.0561 (7)	
O2	0.9307 (3)	0.8042 (3)	0.9280 (2)	0.0596 (7)	
N1	0.8420 (4)	0.9168 (3)	0.7251 (2)	0.0412 (7)	
N2A	0.7365 (11)	0.8952 (11)	0.4282 (8)	0.063 (2)	0.60
N2B	0.6592 (11)	0.8685 (15)	0.4443 (11)	0.033 (2)	0.40
C1	0.7554 (4)	0.7990 (4)	0.5483 (3)	0.0451 (9)	
H1A	0.6776	0.6989	0.5859	0.049*	
H1B	0.8539	0.7684	0.5482	0.055*	
C2	0.7259 (5)	1.0457 (4)	0.4256 (3)	0.0502 (9)	
H2A	0.7037	1.1286	0.3588	0.060*	
H2B	0.8407	1.1291	0.3666	0.060*	
C3	0.7756 (4)	1.0509 (4)	0.5453 (3)	0.0417 (8)	
C4	0.8172 (4)	1.1538 (4)	0.6144 (3)	0.0429 (8)	
C5	0.8286 (5)	1.3103 (4)	0.5912 (4)	0.0578 (10)	
H5	0.8076	1.3702	0.5173	0.069*	
C6	0.8719 (5)	1.3728 (5)	0.6813 (4)	0.0631 (11)	
H6	0.8796	1.4763	0.6677	0.076*	
C7	0.9043 (5)	1.2846 (5)	0.7919 (4)	0.0583 (11)	
H7	0.9314	1.3305	0.8505	0.070*	
C8	0.8975 (5)	1.1326 (5)	0.8172 (3)	0.0512 (9)	
H8	0.9208	1.0748	0.8913	0.061*	
C9	0.8541 (4)	1.0679 (4)	0.7267 (3)	0.0405 (8)	
C10	0.7886 (4)	0.9137 (4)	0.6119 (3)	0.0392 (8)	
C11	0.6027 (4)	0.7119 (4)	0.8750 (3)	0.0393 (8)	
C12	0.4712 (5)	0.6450 (5)	0.8095 (4)	0.0611 (11)	
H12	0.4946	0.6227	0.7467	0.073*	

C13	0.3034 (6)	0.6111 (5)	0.8375 (4)	0.0744 (13)
H13	0.2128	0.5676	0.7923	0.089*
C14	0.2692 (6)	0.6406 (5)	0.9308 (4)	0.0683 (13)
H14	0.1555	0.6172	0.9493	0.082*
C15	0.4016 (6)	0.7045 (6)	0.9973 (4)	0.0770 (13)
H15	0.3780	0.7225	1.0621	0.092*
C16	0.5692 (5)	0.7421 (5)	0.9689 (3)	0.0603 (11)
H16	0.6594	0.7879	1.0131	0.072*
C17	0.6808 (8)	0.8350 (5)	0.3439 (4)	0.0870 (17)
C18	0.6535 (6)	0.6699 (5)	0.3577 (4)	0.0730 (13)
H18A	0.7534	0.6579	0.3342	0.088*
H18B	0.5543	0.6335	0.3113	0.088*
H18C	0.6351	0.6108	0.4368	0.088*
C19	0.6286 (7)	0.9265 (6)	0.2316 (4)	0.0794 (14)
H19A	0.5112	0.9251	0.2324	0.095*
H19B	0.6383	0.8830	0.1739	0.095*
H19C	0.7022	1.0314	0.2149	0.095*

Atomic displacement parameters (Å^2)

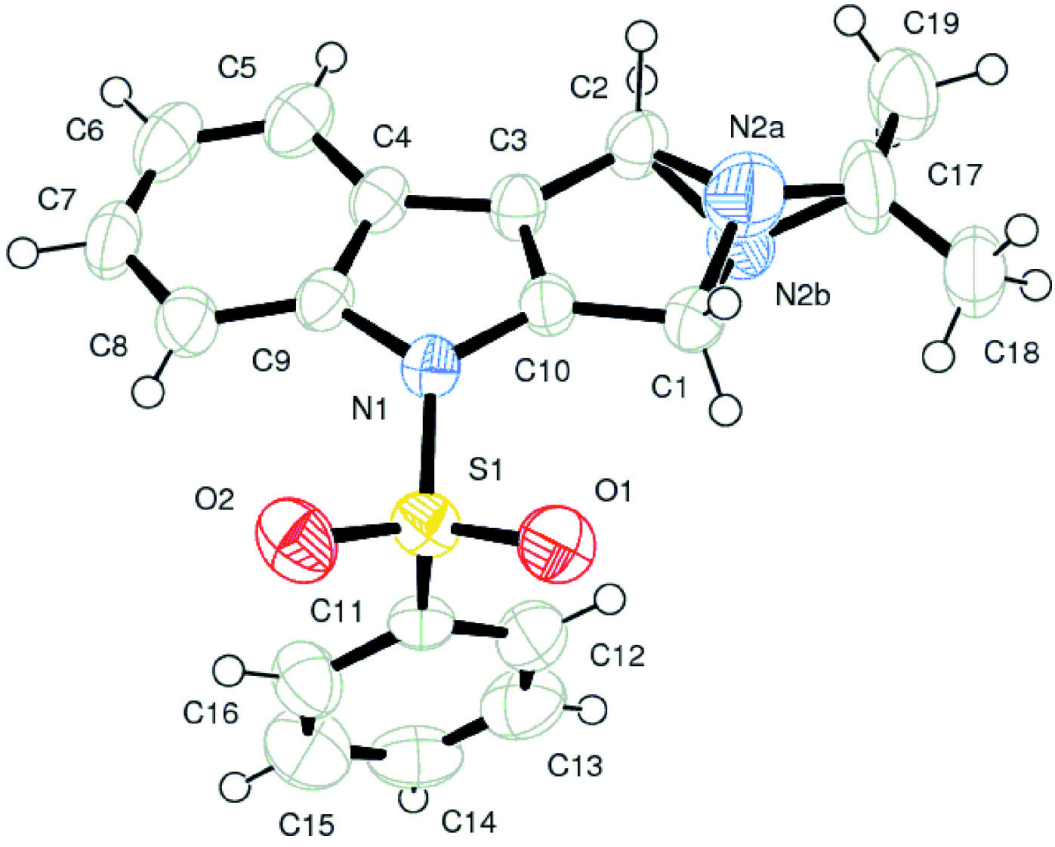
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0455 (5)	0.0454 (5)	0.0412 (5)	0.0190 (4)	0.0019 (4)	−0.0101 (4)
O1	0.0730 (18)	0.0496 (15)	0.0576 (16)	0.0339 (13)	0.0099 (13)	−0.0127 (13)
O2	0.0509 (16)	0.0796 (19)	0.0472 (15)	0.0239 (14)	−0.0116 (12)	−0.0188 (14)
N1	0.0462 (17)	0.0377 (16)	0.0416 (16)	0.0109 (13)	0.0040 (13)	−0.0151 (13)
N2A	0.099 (7)	0.047 (5)	0.045 (5)	0.030 (6)	−0.017 (5)	−0.019 (4)
N2B	0.020 (4)	0.039 (5)	0.039 (5)	0.005 (4)	0.011 (4)	−0.012 (4)
C1	0.052 (2)	0.040 (2)	0.047 (2)	0.0133 (17)	0.0038 (17)	−0.0165 (17)
C2	0.063 (2)	0.040 (2)	0.046 (2)	0.0191 (18)	−0.0077 (18)	−0.0122 (17)
C3	0.045 (2)	0.040 (2)	0.044 (2)	0.0174 (16)	−0.0030 (15)	−0.0160 (16)
C4	0.0395 (19)	0.040 (2)	0.055 (2)	0.0141 (15)	0.0028 (16)	−0.0195 (17)
C5	0.056 (2)	0.045 (2)	0.077 (3)	0.0178 (19)	0.002 (2)	−0.022 (2)
C6	0.062 (3)	0.045 (2)	0.093 (3)	0.016 (2)	0.008 (2)	−0.034 (2)
C7	0.056 (2)	0.056 (3)	0.073 (3)	0.003 (2)	0.010 (2)	−0.042 (2)
C8	0.045 (2)	0.057 (3)	0.055 (2)	0.0053 (17)	0.0106 (17)	−0.0275 (19)
C9	0.0345 (18)	0.038 (2)	0.050 (2)	0.0071 (15)	0.0051 (15)	−0.0177 (16)
C10	0.0411 (19)	0.0396 (19)	0.0396 (19)	0.0116 (15)	0.0025 (15)	−0.0152 (16)
C11	0.0395 (19)	0.0364 (18)	0.0380 (18)	0.0127 (15)	0.0036 (15)	−0.0038 (15)
C12	0.053 (2)	0.059 (3)	0.064 (3)	0.001 (2)	0.007 (2)	−0.023 (2)
C13	0.051 (3)	0.069 (3)	0.081 (3)	0.000 (2)	−0.003 (2)	−0.015 (3)
C14	0.049 (3)	0.065 (3)	0.079 (3)	0.023 (2)	0.022 (2)	0.008 (2)
C15	0.064 (3)	0.094 (4)	0.079 (3)	0.026 (3)	0.019 (3)	−0.023 (3)
C16	0.051 (2)	0.076 (3)	0.055 (2)	0.012 (2)	0.0058 (19)	−0.026 (2)
C17	0.157 (5)	0.065 (3)	0.049 (3)	0.040 (3)	−0.008 (3)	−0.028 (2)
C18	0.094 (3)	0.070 (3)	0.069 (3)	0.028 (3)	−0.002 (3)	−0.040 (2)
C19	0.096 (4)	0.089 (3)	0.057 (3)	0.030 (3)	−0.020 (3)	−0.034 (3)

Geometric parameters (Å, °)

S1—O1	1.424 (3)	C6—C7	1.388 (6)
S1—O2	1.425 (3)	C6—H6	0.9300
S1—N1	1.658 (3)	C7—C8	1.366 (5)
S1—C11	1.760 (3)	C7—H7	0.9300
N1—C10	1.410 (4)	C8—C9	1.399 (5)
N1—C9	1.421 (4)	C8—H8	0.9300
N2A—N2B	0.637 (10)	C11—C12	1.364 (5)
N2A—C17	1.307 (10)	C11—C16	1.369 (5)
N2A—C2	1.459 (10)	C12—C13	1.377 (6)
N2A—C1	1.483 (10)	C12—H12	0.9300
N2B—C17	1.427 (13)	C13—C14	1.360 (6)
N2B—C1	1.480 (14)	C13—H13	0.9300
N2B—C2	1.551 (13)	C14—C15	1.365 (6)
C1—C10	1.485 (5)	C14—H14	0.9300
C1—H1A	0.9687	C15—C16	1.371 (6)
C1—H1B	0.9437	C15—H15	0.9300
C2—C3	1.488 (5)	C16—H16	0.9300
C2—H2A	0.9800	C17—C19	1.463 (6)
C2—H2B	1.1780	C17—C18	1.475 (6)
C3—C10	1.328 (5)	C18—H18A	0.9600
C3—C4	1.436 (5)	C18—H18B	0.9600
C4—C5	1.402 (5)	C18—H18C	0.9600
C4—C9	1.406 (5)	C19—H19A	0.9600
C5—C6	1.381 (6)	C19—H19B	0.9600
C5—H5	0.9300	C19—H19C	0.9600
O1—S1—O2	120.78 (17)	C7—C8—H8	121.5
O1—S1—N1	104.87 (15)	C9—C8—H8	121.5
O2—S1—N1	107.01 (16)	C8—C9—C4	122.0 (3)
O1—S1—C11	109.32 (16)	C8—C9—N1	130.1 (3)
O2—S1—C11	108.96 (16)	C4—C9—N1	107.8 (3)
N1—S1—C11	104.66 (15)	C3—C10—N1	111.1 (3)
C10—N1—C9	105.9 (3)	C3—C10—C1	113.0 (3)
C10—N1—S1	124.0 (2)	N1—C10—C1	135.7 (3)
C9—N1—S1	126.0 (2)	C12—C11—C16	120.7 (4)
C17—N2A—C2	123.9 (7)	C12—C11—S1	119.5 (3)
C17—N2A—C1	122.3 (7)	C16—C11—S1	119.8 (3)
C2—N2A—C1	110.3 (6)	C11—C12—C13	119.2 (4)
C17—N2B—C1	114.5 (8)	C11—C12—H12	120.4
C17—N2B—C2	110.1 (8)	C13—C12—H12	120.4
C1—N2B—C2	105.6 (8)	C14—C13—C12	120.4 (4)
N2B—C1—C10	101.1 (6)	C14—C13—H13	119.8
N2A—C1—C10	101.7 (4)	C12—C13—H13	119.8
N2A—C1—H1A	126.4	C13—C14—C15	120.0 (4)
C10—C1—H1A	113.4	C13—C14—H14	120.0
N2B—C1—H1B	124.9	C15—C14—H14	120.0
C10—C1—H1B	112.4	C14—C15—C16	120.1 (4)

N2A—C2—C3	103.9 (4)	C14—C15—H15	119.9
C3—C2—N2B	101.2 (6)	C16—C15—H15	119.9
N2A—C2—H2A	127.9	C11—C16—C15	119.5 (4)
C3—C2—H2A	127.9	C11—C16—H16	120.2
C3—C2—H2B	108.7	C15—C16—H16	120.2
N2B—C2—H2B	123.2	N2A—C17—C19	121.2 (6)
C10—C3—C4	108.2 (3)	N2B—C17—C19	119.2 (7)
C10—C3—C2	109.8 (3)	N2A—C17—C18	122.7 (6)
C4—C3—C2	142.0 (3)	N2B—C17—C18	115.3 (6)
C5—C4—C9	119.3 (3)	C19—C17—C18	115.9 (4)
C5—C4—C3	133.8 (4)	C17—C18—H18A	109.5
C9—C4—C3	106.8 (3)	C17—C18—H18B	109.5
C6—C5—C4	118.0 (4)	H18A—C18—H18B	109.5
C6—C5—H5	121.0	C17—C18—H18C	109.5
C4—C5—H5	121.0	H18A—C18—H18C	109.5
C5—C6—C7	121.5 (4)	H18B—C18—H18C	109.5
C5—C6—H6	119.2	C17—C19—H19A	109.5
C7—C6—H6	119.2	C17—C19—H19B	109.5
C8—C7—C6	122.0 (4)	H19A—C19—H19B	109.5
C8—C7—H7	119.0	C17—C19—H19C	109.5
C6—C7—H7	119.0	H19A—C19—H19C	109.5
C7—C8—C9	117.0 (4)	H19B—C19—H19C	109.5

Fig. 1


C19
C5
C2
C6
C4
C3
N2a
C17
C7
N2b
C8
C9
C10
C1
C18
N1
S1
O2
O1
C11
C12
C16
C13
C15
C14