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

**Tara L. S. Kishbaugh,[a] Gordon W. Gribble[a] and Jerry P. Jasinski[b]***

[a]Department of Chemistry, Dartmouth College, Hanover, NH 03755-3564, USA, and [b]Department of Chemistry, Keene State College, Keene, NH 03435-2001, USA
Correspondence e-mail: jjasinski@keene.edu

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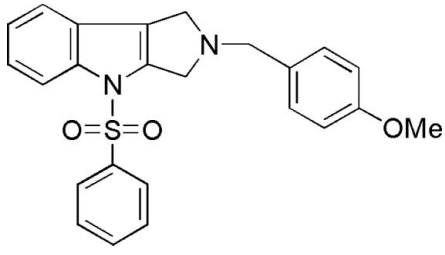
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.061; $wR$ factor = 0.283; data-to-parameter ratio = 17.4.

The indole and pyrrolidine ring systems of the title compound, $C_{24}H_{22}N_2O_3S$, are essentially coplanar. The angle between the planes of the phenylsulfonyl group and the indole ring system is 89.1 (2)°, which is characteristic of 1-(phenylsulfonyl)-indoles. The benzyl ring is nearly perpendicular to the pyrroloindole unit, with an angle between the planes of 82.2 (2)°.

## Related literature

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




## Experimental

### *Crystal data*

$C_{25}H_{24}N_2O_2S$
$M_r$ = 416.52
Monoclinic, $P2_1/c$
$a$ = 11.929 (7) Å
$b$ = 9.397 (7) Å
$c$ = 19.198 (4) Å
$\beta$ = 106.47 (3)°
$V$ = 2063.6 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.18 mm$^{-1}$
$T$ = 296 K
0.50 × 0.40 × 0.20 mm

### *Data collection*

Rigaku AFC-6*S* diffractometer
Absorption correction: $\psi$ scan (North *et al.*, 1968)
$T_{min}$ = 0.915, $T_{max}$ = 0.965
4739 measured reflections
4739 independent reflections
1590 reflections with $I > 2\sigma(I)$
3 standard reflections
every 150 reflections
intensity decay: none

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.284
$S$ = 1.03
4739 reflections
273 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.38 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: FL2139).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3411 [ **doi:10.1107/S1600536807032436** ]

# 2-(4-Methoxybenzyl)-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, (I), was synthesized as a precursor to the corresponding pyrrolo[3,4-*b*]indole as a stable synthetic analogue of indole-2,3-quinodimethane and having a readily removable 4-methoxybenzyl protecting group. 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 pyrrolidine nitrogen N2 has bond angles totaling 337.8 (4)°, indicating slight flattening from a purely pyramidal nitrogen, which is 324° for ammonia with H—N—H bond angles of 108°, and is consistent with the inductive electron-withdrawing properties of a benzyl group.

## Experimental

To a refluxing solution of 2,3-dibromomethyl-1-(phenylsulfonyl)indole (415 mg, 0.937 mmol) (Mohanakrishnan & Srinivasan, 1995) and $K_2CO_3$ (397 mg, 2.80 mmol) in tetrahydrofuran (20 ml) was added a solution of 4-methoxybenzylamine (150 µ*L*, 1.15 mmol) in tetrahydrofuran (20 ml) slowly *via* addition funnel. After 10 h, the opaque solution was filtered through a Celite pad with ethyl acetate rinses. The resulting 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 (290 mg, 74% yield): m.p. 391–391.5 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) = 1.19–1.20$U_{eq}$(C).

## Figures

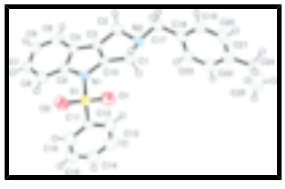


Fig. 1. Molecular structure of (I) showing atom labelling and 50% probability displacement ellipsoids.

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

*Crystal data*

| | |
|---|---|
| $C_{25}H_{24}N_2O_2S$ | $F_{000}$ = 880 |
| $M_r$ = 416.52 | $D_x$ = 1.341 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71069 Å |
| $a$ = 11.929 (7) Å | Cell parameters from 20 reflections |
| $b$ = 9.397 (7) Å | θ = 6.2–7.8º |
| $c$ = 19.198 (4) Å | μ = 0.18 mm$^{-1}$ |
| β = 106.47 (3)º | $T$ = 296 K |
| $V$ = 2063.6 (19) Å$^3$ | Prism, colourless |
| $Z$ = 4 | 0.50 × 0.40 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Rigaku AFC-6S diffractometer | $R_{int}$ = 0.0000 |
| Radiation source: normal-focus sealed tube | $\theta_{max}$ = 27.5º |
| Monochromator: graphite | $\theta_{min}$ = 1.8º |
| $T$ = 296 K | $h$ = 0→15 |
| ω/2θ scans | $k$ = 0→12 |
| Absorption correction: ψ scan (North *et al.*, 1968) | $l$ = −24→23 |
| $T_{min}$ = 0.915, $T_{max}$ = 0.965 | 3 standard reflections |
| 4739 measured reflections | every 150 reflections |
| 4739 independent reflections | intensity decay: none |
| 1590 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 |
| $R[F^2 > 2\sigma(F^2)]$ = 0.061 | $w = 1/[\sigma^2(F_o^2) + (0.1351P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.284 | $(\Delta/\sigma)_{max} < 0.001$ |
| $S$ = 1.03 | $\Delta\rho_{max}$ = 0.48 e Å$^{-3}$ |
| 4739 reflections | $\Delta\rho_{min}$ = −0.38 e Å$^{-3}$ |
| 273 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: 0.0052 (19) |
| Secondary atom site location: difference Fourier map | |

*Special details*

**Experimental**. $^1$H ($CD_2Cl_2$) δ 8.04 (d, 1H, 8 Hz), 7.88 (m, 2H), 7.24–7.56 (m, 8H), 6.95 (m, 2H), 4.32 (m, 2H), 4.00 (s, 2H), 3.96 (m, 2H), 3.86 (s, 3H); $^{13}$C ($CD_2Cl_2$) δ 159.2, 140.3, 139.8, 138.4, 134.3, 131.4, 130.2, 129.7, 129.3, 129.0, 128.8, 126.9, 126.7, 126.3, 124.0, 123.7, 119.6, 114.5, 114.4, 114.1, 60.1, 55.5, 54.9, 53.1; IR (film) $\lambda_{max}$ 3056, 2933, 2832, 1611, 1511, 1447, 1369, 1247, 1177, 996, 750, 723, 685 cm-1; UV (EtOH) $\lambda_{max}$ 224, 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.29345 (13) | 0.16620 (16) | 0.82158 (8) | 0.0408 (4) |
| O1 | 0.3017 (3) | 0.0241 (4) | 0.7988 (2) | 0.0510 (11) |
| O2 | 0.3618 (3) | 0.2133 (5) | 0.8910 (2) | 0.0532 (12) |
| N1 | 0.3285 (4) | 0.2660 (5) | 0.7598 (2) | 0.0390 (11) |
| N2 | 0.3015 (4) | 0.1362 (5) | 0.5746 (2) | 0.0440 (13) |
| C1 | 0.3125 (6) | 0.0860 (6) | 0.6496 (3) | 0.0481 (15) |
| H1 | 0.2440 | 0.0332 | 0.6521 | 0.058* |
| H2 | 0.3816 | 0.0274 | 0.6681 | 0.058* |
| C2 | 0.3454 (6) | 0.2842 (7) | 0.5768 (3) | 0.0505 (16) |
| H3 | 0.2956 | 0.3415 | 0.5383 | 0.061* |
| H4 | 0.4247 | 0.2866 | 0.5729 | 0.061* |
| C3 | 0.3404 (5) | 0.3338 (6) | 0.6499 (3) | 0.0403 (14) |
| C4 | 0.3571 (5) | 0.4598 (6) | 0.6937 (3) | 0.0410 (14) |
| C5 | 0.3785 (5) | 0.6024 (7) | 0.6835 (4) | 0.0495 (16) |
| H5 | 0.3885 | 0.6333 | 0.6396 | 0.059* |
| C6 | 0.3850 (5) | 0.6982 (7) | 0.7393 (4) | 0.0539 (17) |
| H6 | 0.3998 | 0.7938 | 0.7331 | 0.065* |
| C7 | 0.3693 (5) | 0.6521 (7) | 0.8044 (3) | 0.0482 (15) |
| H7 | 0.3722 | 0.7188 | 0.8407 | 0.058* |
| C8 | 0.3498 (5) | 0.5120 (7) | 0.8177 (3) | 0.0481 (16) |
| H8 | 0.3398 | 0.4831 | 0.8619 | 0.058* |
| C9 | 0.3456 (4) | 0.4154 (6) | 0.7622 (3) | 0.0369 (13) |
| C10 | 0.3227 (4) | 0.2224 (6) | 0.6887 (3) | 0.0342 (12) |
| C11 | 0.1462 (5) | 0.2100 (6) | 0.8114 (3) | 0.0393 (13) |
| C12 | 0.0626 (6) | 0.1670 (8) | 0.7498 (4) | 0.0645 (19) |
| H9 | 0.0837 | 0.1128 | 0.7150 | 0.077* |
| C13 | −0.0525 (6) | 0.2044 (9) | 0.7397 (4) | 0.078 (2) |
| H10 | −0.1095 | 0.1763 | 0.6979 | 0.093* |

| | | | | |
|---|---|---|---|---|
| C14 | −0.0823 (6) | 0.2839 (9) | 0.7920 (4) | 0.074 (2) |
| H11 | −0.1601 | 0.3087 | 0.7856 | 0.088* |
| C15 | 0.0002 (6) | 0.3265 (8) | 0.8529 (4) | 0.074 (2) |
| H12 | −0.0209 | 0.3801 | 0.8879 | 0.089* |
| C16 | 0.1157 (6) | 0.2897 (7) | 0.8625 (4) | 0.0613 (18) |
| H13 | 0.1727 | 0.3194 | 0.9039 | 0.074* |
| C17 | 0.3508 (6) | 0.0386 (7) | 0.5328 (3) | 0.0546 (17) |
| H14 | 0.4272 | 0.0083 | 0.5624 | 0.066* |
| H15 | 0.3614 | 0.0883 | 0.4909 | 0.066* |
| C18 | 0.2758 (5) | −0.0919 (7) | 0.5071 (3) | 0.0451 (15) |
| C19 | 0.3262 (6) | −0.2176 (7) | 0.4941 (3) | 0.0519 (16) |
| H16 | 0.4069 | −0.2241 | 0.5039 | 0.062* |
| C20 | 0.2568 (7) | −0.3341 (7) | 0.4664 (4) | 0.0613 (18) |
| H17 | 0.2922 | −0.4179 | 0.4577 | 0.074* |
| C21 | 0.1367 (6) | −0.3297 (7) | 0.4512 (3) | 0.0574 (17) |
| C22 | 0.0872 (6) | −0.2050 (7) | 0.4657 (4) | 0.0569 (18) |
| H18 | 0.0066 | −0.1990 | 0.4572 | 0.068* |
| C23 | 0.1563 (6) | −0.0897 (7) | 0.4924 (4) | 0.0565 (17) |
| H19 | 0.1206 | −0.0062 | 0.5010 | 0.068* |
| C24 | 0.0782 (5) | −0.4476 (5) | 0.4251 (3) | 0.0391 (14) |
| H20 | 0.1015 | −0.5207 | 0.4621 | 0.047* |
| H21 | 0.1039 | −0.4784 | 0.3839 | 0.047* |
| C25 | −0.0396 (10) | −0.4433 (9) | 0.4036 (5) | 0.109 (3) |
| H22 | −0.0689 | −0.4663 | 0.4439 | 0.131* |
| H23 | −0.0687 | −0.5110 | 0.3653 | 0.131* |
| H24 | −0.0650 | −0.3495 | 0.3863 | 0.131* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0453 (9) | 0.0433 (9) | 0.0367 (8) | 0.0029 (7) | 0.0163 (6) | 0.0038 (7) |
| O1 | 0.062 (3) | 0.039 (3) | 0.057 (3) | 0.007 (2) | 0.025 (2) | 0.007 (2) |
| O2 | 0.051 (3) | 0.070 (3) | 0.036 (2) | 0.008 (2) | 0.0096 (19) | 0.002 (2) |
| N1 | 0.045 (3) | 0.035 (3) | 0.040 (3) | −0.001 (2) | 0.018 (2) | −0.001 (2) |
| N2 | 0.056 (3) | 0.045 (3) | 0.035 (3) | −0.006 (2) | 0.020 (2) | −0.004 (2) |
| C1 | 0.058 (4) | 0.047 (4) | 0.044 (4) | −0.007 (3) | 0.022 (3) | −0.005 (3) |
| C2 | 0.065 (4) | 0.047 (4) | 0.044 (3) | 0.003 (3) | 0.022 (3) | 0.005 (3) |
| C3 | 0.045 (3) | 0.043 (3) | 0.036 (3) | −0.003 (3) | 0.017 (3) | −0.004 (3) |
| C4 | 0.033 (3) | 0.043 (4) | 0.050 (4) | −0.002 (3) | 0.015 (3) | −0.002 (3) |
| C5 | 0.042 (3) | 0.047 (4) | 0.060 (4) | 0.000 (3) | 0.016 (3) | 0.010 (3) |
| C6 | 0.046 (4) | 0.039 (4) | 0.074 (5) | −0.006 (3) | 0.011 (3) | −0.005 (3) |
| C7 | 0.047 (4) | 0.043 (4) | 0.053 (4) | 0.002 (3) | 0.011 (3) | −0.012 (3) |
| C8 | 0.051 (4) | 0.056 (4) | 0.038 (3) | 0.000 (3) | 0.012 (3) | −0.005 (3) |
| C9 | 0.029 (3) | 0.046 (4) | 0.033 (3) | 0.002 (3) | 0.006 (2) | 0.001 (3) |
| C10 | 0.036 (3) | 0.039 (3) | 0.030 (3) | −0.002 (2) | 0.012 (2) | 0.000 (3) |
| C11 | 0.041 (3) | 0.041 (3) | 0.039 (3) | −0.001 (3) | 0.016 (3) | 0.001 (3) |
| C12 | 0.050 (4) | 0.088 (5) | 0.056 (4) | −0.006 (4) | 0.016 (3) | −0.017 (4) |
| C13 | 0.048 (5) | 0.108 (7) | 0.071 (5) | −0.010 (4) | 0.006 (4) | −0.009 (5) |

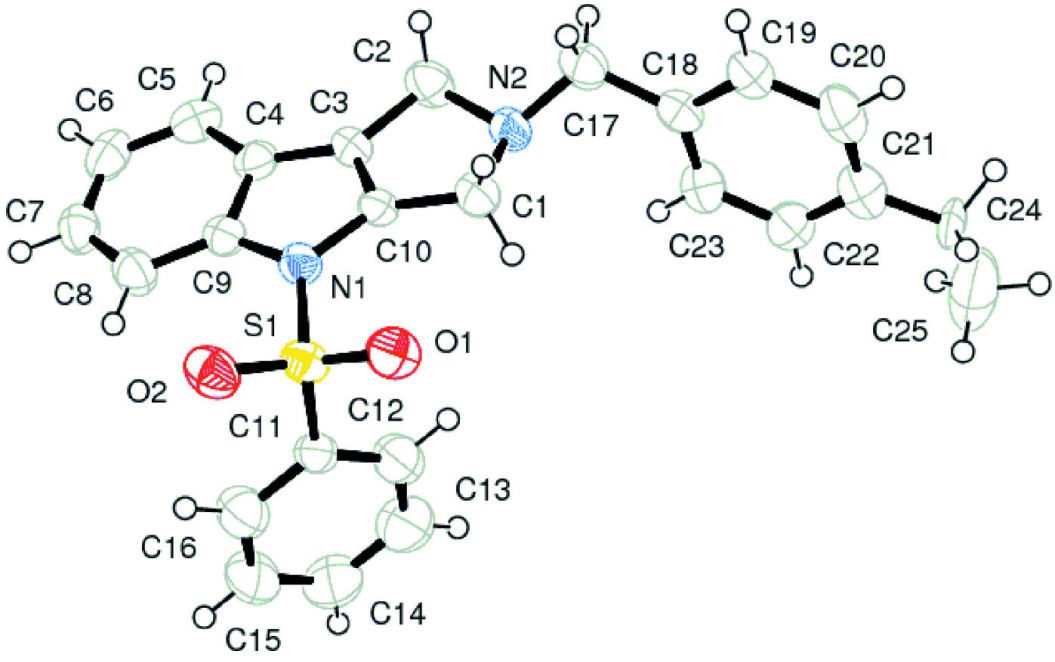
| | | | | | | |
|---|---|---|---|---|---|---|
| C14 | 0.040 (4) | 0.091 (6) | 0.090 (6) | 0.000 (4) | 0.018 (4) | −0.005 (5) |
| C15 | 0.055 (4) | 0.087 (6) | 0.090 (5) | 0.006 (4) | 0.036 (4) | −0.028 (5) |
| C16 | 0.055 (4) | 0.073 (5) | 0.057 (4) | 0.003 (4) | 0.017 (3) | −0.008 (4) |
| C17 | 0.068 (4) | 0.062 (4) | 0.038 (3) | −0.004 (4) | 0.022 (3) | −0.003 (3) |
| C18 | 0.052 (4) | 0.052 (4) | 0.035 (3) | 0.001 (3) | 0.018 (3) | −0.002 (3) |
| C19 | 0.054 (4) | 0.055 (4) | 0.051 (4) | 0.000 (3) | 0.021 (3) | −0.010 (3) |
| C20 | 0.081 (5) | 0.048 (4) | 0.058 (4) | 0.010 (4) | 0.025 (4) | −0.010 (4) |
| C21 | 0.069 (5) | 0.052 (4) | 0.051 (4) | 0.009 (4) | 0.017 (3) | −0.003 (3) |
| C22 | 0.049 (4) | 0.063 (5) | 0.060 (4) | −0.007 (3) | 0.017 (3) | −0.012 (4) |
| C23 | 0.064 (5) | 0.046 (4) | 0.062 (4) | −0.001 (3) | 0.022 (3) | −0.011 (3) |
| C24 | 0.042 (3) | 0.019 (3) | 0.052 (4) | −0.006 (3) | 0.006 (3) | −0.012 (3) |
| C25 | 0.168 (11) | 0.062 (6) | 0.087 (7) | −0.036 (6) | 0.018 (6) | −0.014 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—O1 | 1.416 (4) | C12—C13 | 1.376 (9) |
| S1—O2 | 1.421 (4) | C12—H9 | 0.9300 |
| S1—N1 | 1.656 (5) | C13—C14 | 1.377 (10) |
| S1—C11 | 1.760 (6) | C13—H10 | 0.9300 |
| N1—C10 | 1.407 (7) | C14—C15 | 1.357 (10) |
| N1—C9 | 1.417 (7) | C14—H11 | 0.9300 |
| N2—C17 | 1.448 (7) | C15—C16 | 1.380 (9) |
| N2—C2 | 1.482 (7) | C15—H12 | 0.9300 |
| N2—C1 | 1.485 (7) | C16—H13 | 0.9300 |
| C1—C10 | 1.474 (8) | C17—C18 | 1.516 (8) |
| C1—H1 | 0.9700 | C17—H14 | 0.9700 |
| C1—H2 | 0.9700 | C17—H15 | 0.9700 |
| C2—C3 | 1.495 (8) | C18—C23 | 1.373 (8) |
| C2—H3 | 0.9700 | C18—C19 | 1.380 (8) |
| C2—H4 | 0.9700 | C19—C20 | 1.384 (9) |
| C3—C10 | 1.336 (7) | C19—H16 | 0.9300 |
| C3—C4 | 1.433 (8) | C20—C21 | 1.379 (9) |
| C4—C5 | 1.389 (8) | C20—H17 | 0.9300 |
| C4—C9 | 1.423 (7) | C21—C24 | 1.330 (8) |
| C5—C6 | 1.385 (9) | C21—C22 | 1.375 (9) |
| C5—H5 | 0.9300 | C22—C23 | 1.370 (8) |
| C6—C7 | 1.384 (9) | C22—H18 | 0.9300 |
| C6—H6 | 0.9300 | C23—H19 | 0.9300 |
| C7—C8 | 1.373 (9) | C24—C25 | 1.348 (11) |
| C7—H7 | 0.9300 | C24—H20 | 0.9700 |
| C8—C9 | 1.390 (8) | C24—H21 | 0.9700 |
| C8—H8 | 0.9300 | C25—H22 | 0.9600 |
| C11—C16 | 1.365 (8) | C25—H23 | 0.9600 |
| C11—C12 | 1.374 (8) | C25—H24 | 0.9600 |
| O1—S1—O2 | 120.7 (3) | C11—C12—C13 | 119.9 (6) |
| O1—S1—N1 | 105.0 (2) | C11—C12—H9 | 120.1 |
| O2—S1—N1 | 107.5 (3) | C13—C12—H9 | 120.1 |
| O1—S1—C11 | 109.9 (3) | C12—C13—C14 | 119.3 (7) |
| O2—S1—C11 | 108.7 (3) | C12—C13—H10 | 120.4 |

| | | | |
|---|---|---|---|
| N1—S1—C11 | 103.5 (2) | C14—C13—H10 | 120.4 |
| C10—N1—C9 | 106.7 (4) | C15—C14—C13 | 120.9 (7) |
| C10—N1—S1 | 125.2 (4) | C15—C14—H11 | 119.5 |
| C9—N1—S1 | 126.9 (4) | C13—C14—H11 | 119.5 |
| C17—N2—C2 | 114.5 (5) | C14—C15—C16 | 119.7 (7) |
| C17—N2—C1 | 113.4 (5) | C14—C15—H12 | 120.2 |
| C2—N2—C1 | 109.7 (4) | C16—C15—H12 | 120.2 |
| C10—C1—N2 | 100.9 (4) | C11—C16—C15 | 120.0 (6) |
| C10—C1—H1 | 111.6 | C11—C16—H13 | 120.0 |
| N2—C1—H1 | 111.6 | C15—C16—H13 | 120.0 |
| C10—C1—H2 | 111.6 | N2—C17—C18 | 113.5 (5) |
| N2—C1—H2 | 111.6 | N2—C17—H14 | 108.9 |
| H1—C1—H2 | 109.4 | C18—C17—H14 | 108.9 |
| N2—C2—C3 | 102.2 (5) | N2—C17—H15 | 108.9 |
| N2—C2—H3 | 111.3 | C18—C17—H15 | 108.9 |
| C3—C2—H3 | 111.3 | H14—C17—H15 | 107.7 |
| N2—C2—H4 | 111.3 | C23—C18—C19 | 117.1 (6) |
| C3—C2—H4 | 111.3 | C23—C18—C17 | 122.5 (6) |
| H3—C2—H4 | 109.2 | C19—C18—C17 | 120.3 (5) |
| C10—C3—C4 | 109.7 (5) | C18—C19—C20 | 120.2 (6) |
| C10—C3—C2 | 109.5 (5) | C18—C19—H16 | 119.9 |
| C4—C3—C2 | 140.6 (5) | C20—C19—H16 | 119.9 |
| C5—C4—C9 | 118.9 (5) | C21—C20—C19 | 121.9 (6) |
| C5—C4—C3 | 135.6 (6) | C21—C20—H17 | 119.0 |
| C9—C4—C3 | 105.5 (5) | C19—C20—H17 | 119.0 |
| C6—C5—C4 | 119.3 (6) | C24—C21—C22 | 125.2 (6) |
| C6—C5—H5 | 120.3 | C24—C21—C20 | 117.2 (6) |
| C4—C5—H5 | 120.3 | C22—C21—C20 | 117.6 (6) |
| C7—C6—C5 | 120.2 (6) | C23—C22—C21 | 120.1 (6) |
| C7—C6—H6 | 119.9 | C23—C22—H18 | 119.9 |
| C5—C6—H6 | 119.9 | C21—C22—H18 | 119.9 |
| C8—C7—C6 | 122.7 (6) | C22—C23—C18 | 123.0 (6) |
| C8—C7—H7 | 118.6 | C22—C23—H19 | 118.5 |
| C6—C7—H7 | 118.6 | C18—C23—H19 | 118.5 |
| C7—C8—C9 | 117.0 (6) | C21—C24—C25 | 118.7 (6) |
| C7—C8—H8 | 121.5 | C21—C24—H20 | 107.6 |
| C9—C8—H8 | 121.5 | C25—C24—H20 | 107.6 |
| C8—C9—N1 | 130.2 (5) | C21—C24—H21 | 107.6 |
| C8—C9—C4 | 121.7 (6) | C25—C24—H21 | 107.6 |
| N1—C9—C4 | 108.1 (5) | H20—C24—H21 | 107.1 |
| C3—C10—N1 | 110.0 (5) | C24—C25—H22 | 109.5 |
| C3—C10—C1 | 113.6 (5) | C24—C25—H23 | 109.5 |
| N1—C10—C1 | 136.2 (5) | H22—C25—H23 | 109.5 |
| C16—C11—C12 | 120.3 (6) | C24—C25—H24 | 109.5 |
| C16—C11—S1 | 120.6 (5) | H22—C25—H24 | 109.5 |
| C12—C11—S1 | 119.0 (5) | H23—C25—H24 | 109.5 |

**Fig. 1**


C2
N2
C18
C19
C20
C5
C6
C4
C3
C17
C21
C7
C1
C24
C10
C23
C22
C8
C9
N1
S1
O1
C25
O2
C11
C12
C13
C16
C14
C15