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## (3*aR*,8*bR*)-3*a*,8*b*-Dihydroxy-2-methyl-sulfanyl-3-nitro-1-phenyl-1,8*b*-dihydroindeno[1,2-*b*]pyrrol-4(3*aH*)-one

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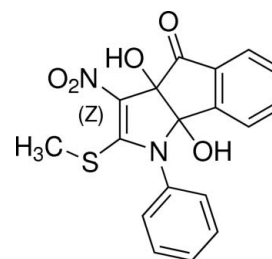
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.092; data-to-parameter ratio = 17.5.

In the title compound,  $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_5$ , the pyrrolidine ring adopts a shallow envelope conformation, with the C atom bearing the OH group (and remote from the N atom) displaced by 0.257 (2) Å from the other atoms. The cyclopentane ring has a twisted conformation about the C—C bond bearing one =O and one —OH grouping. The dihedral angle between the five-membered rings (all atoms) is 65.54 (9)° and the OH groups lie to the same side of the ring-junction. The molecular structure features a weak intramolecular O—H...O bond and a possible C—H... $\pi$  interaction. In the crystal, the molecules are linked into [010] chains by O—H...O hydrogen bonds. Weak C—H...O bonds connect the chains into (100) sheets.

### Related literature

For background to pyrrolidine derivatives, see: Grigg (1995); Kravchenko *et al.* (2005). For ring conformation analysis, see: Cremer & Pople (1975). For a related structure, see: Ghorbani (2012).



### Experimental

#### Crystal data

$\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_5$   
 $M_r = 370.37$   
Orthorhombic,  $P2_12_12_1$   
 $a = 9.6625$  (3) Å  
 $b = 10.8994$  (2) Å  
 $c = 15.8154$  (4) Å

$V = 1665.61$  (7) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.23$  mm<sup>-1</sup>  
 $T = 293$  K  
0.21 × 0.19 × 0.18 mm

#### Data collection

Bruker Kappa APEXII  
diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.967$ ,  $T_{\max} = 0.974$

9249 measured reflections  
4140 independent reflections  
3800 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.018$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.092$   
 $S = 1.00$   
4140 reflections  
236 parameters  
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983)  
Absolute structure parameter:  
0.00 (6)

**Table 1**

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C13–C18 benzene ring.

| $D-H\cdots A$              | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------|-------|-------------|-------------|---------------|
| O3—H3...O5                 | 0.82  | 2.34        | 2.895 (2)   | 126           |
| O2—H2...O5 <sup>i</sup>    | 0.82  | 2.00        | 2.8155 (18) | 171           |
| C11—H11...O4 <sup>ii</sup> | 0.93  | 2.51        | 3.423 (3)   | 166           |
| C9—H9...Cg1                | 0.93  | 2.89        | 3.545 (2)   | 129           |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); 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: HB7154).

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## supplementary materials

*Acta Cryst.* (2013). E69, o1770–o1771 [doi:10.1107/S1600536813029577]

**(3a*R*,8b*R*)-3a,8b-Dihydroxy-2-methylsulfanyl-3-nitro-1-phenyl-1,8b-dihydro-indeno[1,2-*b*]pyrrol-4(3a*H*)-one**

**R. A. Nagalakshmi, J. Suresh, V. Jeyachandran, R. Ranjith Kumar and P. L. Nilantha Lakshman**

**1. Comment**

Pyrrolidine-containing compounds are of significant importance because of their biological activities and widespread employment in catalysis (Grigg, 1995; Kravchenko *et al.*, 2005). As part of our own studies in this area, we have undertaken the crystal structure determination of the title compound, a pyrrolidine derivative, and the results are presented here.

In the title compound (Fig 1) C<sub>18</sub>H<sub>14</sub>N<sub>2</sub>O<sub>5</sub>, the central pyrrolidine ring is enveloped on C2 with the puckering parameters  $q_2 = 0.1576$  (16) Å and  $\varphi_2 = 243.9$  (6) ° (Cremer & Pople, 1975). The cyclopentane ring has a twisted conformation with the puckering parameters  $q_2 = 0.1540$  (18) Å and  $\varphi_2 = 45.5$  (7) °. The benzene ring of the indane ring is in planar with r.m.s. deviation 0.007 (1) Å. The benzene ring attached to the pyrrole ring is also planar with r.m.s. deviation 0.0096 Å. The aryl ring makes the dihedral angle of 57.40 (1) ° with the mean plane of the pyrrolidine ring. The sum of the C—N—C angles around N1 (359.89 (1)°) atom is implying a noticeable flattening of the trigonal pyramidal geometry about N1. The conformation of the methylsulfanyl moiety is in anticlinical conformation as evidenced from the torsion angle C5-S1-C4-C3 = -145.2 °. The nitro group is well ordered and makes a dihedral angle of 20.25 (17) ° with the mean plane of pyrrolidine ring. The twist of the methyl sulfanyl ring attached with the pyrrolidine ring is indicated by the torsion angle C5-S1-C4-N1 = 28.5 °. The bond length C4-S1 = 1.727 (2) Å is shorter than S1-C5 = 1.804 (3) Å as found in similar structure (Ghorbani, 2012). The methyl group of the methyl sulfanyl substituent is tilted towards the plane of the benzene ring as indicated by the angle C4-S1-C5 = 106.52 (11) °. Due to conjugation, the bond length C1-O2 = 1.380 (4) Å is shorter than the bond length C2-O3 = 1.409 (2) Å. The methoxy groups substituted at the phenyl rings are twisted, as it can be seen from the torsion angles C19-O6-C16-C17 = 14.1 (3) °.

The structure features a weak intra-molecular O—H...O interaction. An inter-molecular O2—H2...O5 interaction forms a zig-zag chain along b axis and they are further connected by C11—H11...O4 inter-molecular interaction, forming a zig-zag chain along c axis. A weak C—H...Cg1 interaction is also observed, as in Table 1 (Cg1 is the centroid of the benzene ring C13-C18).

**2. Experimental**

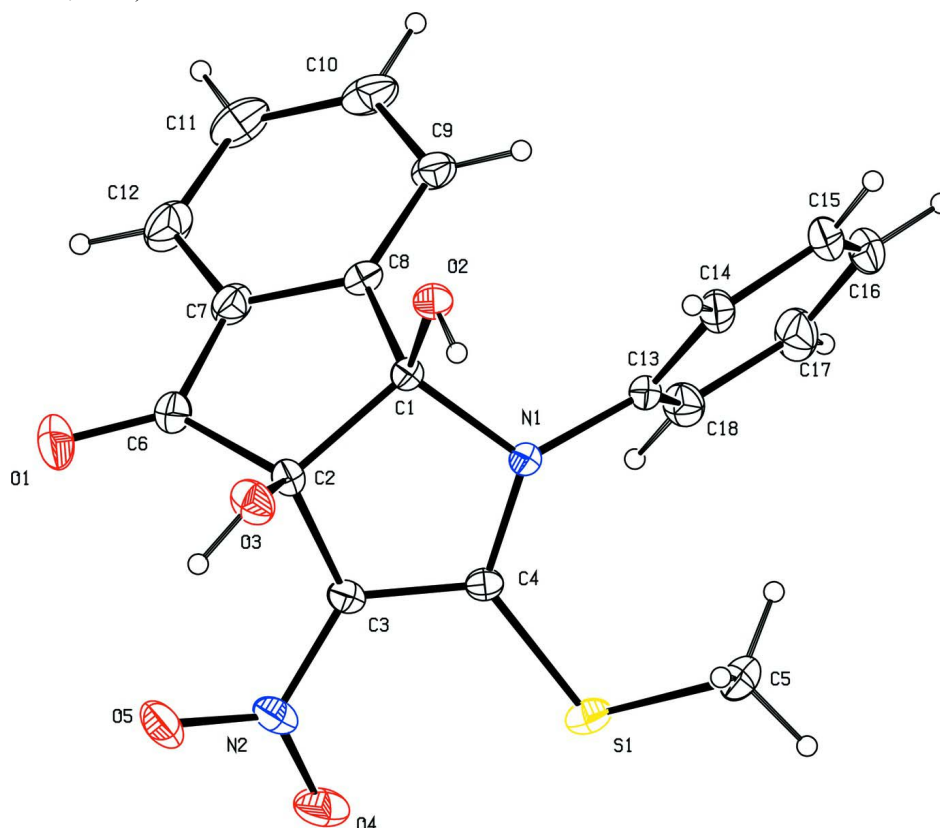
A mixture of (E)-N-(1-(methylthio)-2-nitrovinyl)aniline (1 mmol) with ninhydrin (1 mmol) in presence of glacial AcOH (3-5 drops) was thoroughly ground in a pestle and mortar at room temperature for 2-10 mins. The reaction progress was monitored by thin layer chromatography. After completion of the reaction, the reaction mixture was triturated with crushed ice, the resulting solid filtered off and washed with water to afford the pure product. The compound was further recrystallized from ethanol to obtain colourless blocks. Melting point : 451K - 453K. Yield : 94%.

### 3. Refinement

H atoms were placed at calculated positions and allowed to ride on their carrier atoms with  $C-H = 0.93-0.98\text{\AA}$ .  $U_{iso} = 1.2U_{eq}(C)$  for  $CH_2$  and  $CH$  groups and  $U_{iso} = 1.5U_{eq}(C)$  for  $CH_3$  group.

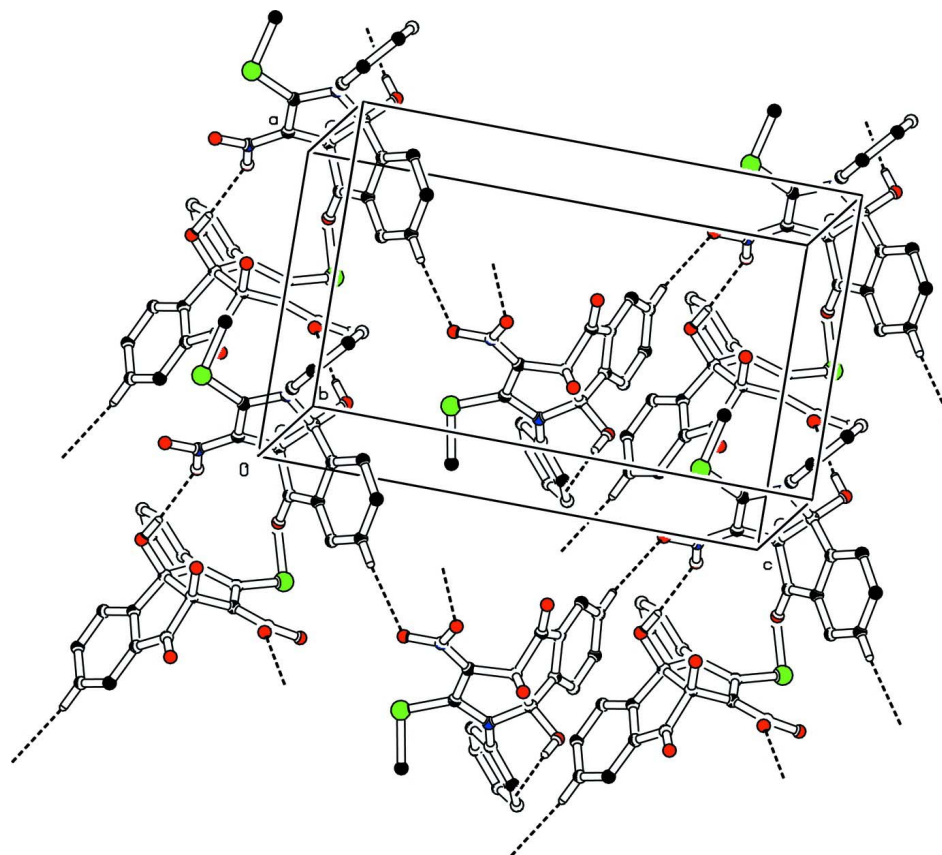
### Computing details

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).



**Figure 1**

The molecular structure of (I), showing 20% probability displacement ellipsoids. H-atoms are omitted for clarity.

**Figure 2**

The partial packing diagram showing O—H···O and C—H···O interactions.

**(3a*R*,8b*R*)-3a,8b-Dihydroxy-2-methylsulfonyl-3-nitro-1-phenyl-1,8b-dihydroindeno [1,2-*b*]pyrrol-4(3a*H*)-one**

*Crystal data*

$C_{18}H_{14}N_2O_5S$

$M_r = 370.37$

Orthorhombic,  $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 9.6625 (3) \text{ \AA}$

$b = 10.8994 (2) \text{ \AA}$

$c = 15.8154 (4) \text{ \AA}$

$V = 1665.61 (7) \text{ \AA}^3$

$Z = 4$

$F(000) = 768$

$D_x = 1.477 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2000 reflections

$\theta = 2\text{--}31^\circ$

$\mu = 0.23 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colourless

$0.21 \times 0.19 \times 0.18 \text{ mm}$

*Data collection*

Bruker Kappa APEXII

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 0 pixels  $\text{mm}^{-1}$

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.967$ ,  $T_{\max} = 0.974$

9249 measured reflections

4140 independent reflections

3800 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 28.3^\circ$ ,  $\theta_{\min} = 2.3^\circ$

$h = -12 \rightarrow 12$

$k = -12 \rightarrow 14$

$l = -18 \rightarrow 21$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.092$   
 $S = 1.00$   
 4140 reflections  
 236 parameters  
 0 restraints  
 Primary atom site location: structure-invariant  
 direct methods  
 Secondary atom site location: difference Fourier  
 map

Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0557P)^2 + 0.2428P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$   
 Absolute structure: Flack (1983), 000 Friedel  
 pairs  
 Absolute structure parameter: 0.00 (6)

Special details

**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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

|     | $x$           | $y$           | $z$           | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|---------------|---------------|----------------------------------|
| C1  | 0.14622 (15)  | -0.06791 (13) | 0.10358 (9)   | 0.0281 (3)                       |
| C2  | 0.07556 (17)  | -0.17588 (14) | 0.05420 (10)  | 0.0322 (3)                       |
| C3  | 0.07626 (16)  | -0.12630 (14) | -0.03423 (9)  | 0.0315 (3)                       |
| C4  | 0.16436 (15)  | -0.02676 (14) | -0.04222 (9)  | 0.0287 (3)                       |
| C5  | 0.3928 (2)    | 0.0803 (2)    | -0.11431 (14) | 0.0551 (5)                       |
| H5A | 0.4342        | 0.1155        | -0.1639       | 0.083*                           |
| H5B | 0.4461        | 0.0106        | -0.0964       | 0.083*                           |
| H5C | 0.3910        | 0.1403        | -0.0699       | 0.083*                           |
| C6  | -0.07130 (19) | -0.18225 (19) | 0.09064 (12)  | 0.0447 (4)                       |
| C7  | -0.09638 (18) | -0.06680 (17) | 0.13669 (12)  | 0.0426 (4)                       |
| C8  | 0.02672 (16)  | -0.00269 (14) | 0.14699 (9)   | 0.0326 (3)                       |
| C9  | 0.0300 (2)    | 0.10338 (17)  | 0.19568 (11)  | 0.0438 (4)                       |
| H9  | 0.1121        | 0.1461        | 0.2044        | 0.053*                           |
| C10 | -0.0939 (3)   | 0.1434 (2)    | 0.23091 (13)  | 0.0583 (6)                       |
| H10 | -0.0941       | 0.2146        | 0.2633        | 0.070*                           |
| C11 | -0.2169 (3)   | 0.0807 (2)    | 0.21922 (15)  | 0.0663 (6)                       |
| H11 | -0.2982       | 0.1110        | 0.2428        | 0.080*                           |
| C12 | -0.2199 (2)   | -0.0259 (2)   | 0.17303 (15)  | 0.0612 (5)                       |
| H12 | -0.3018       | -0.0697       | 0.1661        | 0.073*                           |
| C13 | 0.25948 (16)  | 0.12942 (13)  | 0.05639 (10)  | 0.0318 (3)                       |
| C14 | 0.3684 (2)    | 0.14182 (18)  | 0.11299 (11)  | 0.0446 (4)                       |
| H14 | 0.4143        | 0.0729        | 0.1335        | 0.054*                           |
| C15 | 0.4078 (2)    | 0.2584 (2)    | 0.13854 (14)  | 0.0559 (5)                       |
| H15 | 0.4781        | 0.2674        | 0.1782        | 0.067*                           |

|     |               |               |               |              |
|-----|---------------|---------------|---------------|--------------|
| C16 | 0.3443 (3)    | 0.36057 (19)  | 0.10607 (15)  | 0.0605 (6)   |
| H16 | 0.3729        | 0.4383        | 0.1229        | 0.073*       |
| C17 | 0.2376 (2)    | 0.34801 (17)  | 0.04819 (16)  | 0.0566 (5)   |
| H17 | 0.1954        | 0.4173        | 0.0255        | 0.068*       |
| C18 | 0.1936 (2)    | 0.23165 (15)  | 0.02401 (12)  | 0.0430 (4)   |
| H18 | 0.1203        | 0.2227        | -0.0136       | 0.052*       |
| N1  | 0.21036 (13)  | 0.00884 (11)  | 0.03503 (7)   | 0.0280 (2)   |
| N2  | 0.02304 (15)  | -0.19222 (14) | -0.10135 (10) | 0.0405 (3)   |
| O1  | -0.14751 (19) | -0.26911 (18) | 0.08517 (13)  | 0.0783 (6)   |
| O2  | 0.24185 (12)  | -0.10349 (10) | 0.16359 (7)   | 0.0339 (2)   |
| H2  | 0.3065        | -0.1386       | 0.1404        | 0.051*       |
| O3  | 0.14813 (16)  | -0.28697 (11) | 0.06474 (9)   | 0.0464 (3)   |
| H3  | 0.1156        | -0.3392       | 0.0331        | 0.070*       |
| O4  | 0.01453 (17)  | -0.14460 (16) | -0.17197 (9)  | 0.0568 (4)   |
| O5  | -0.01833 (16) | -0.30050 (13) | -0.08714 (10) | 0.0559 (4)   |
| S1  | 0.21819 (5)   | 0.03237 (4)   | -0.13829 (2)  | 0.04134 (11) |

Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|--------------|--------------|--------------|--------------|
| C1  | 0.0308 (7)  | 0.0277 (6)  | 0.0257 (6)   | -0.0027 (5)  | 0.0015 (5)   | 0.0009 (5)   |
| C2  | 0.0339 (7)  | 0.0293 (7)  | 0.0336 (7)   | -0.0048 (6)  | -0.0008 (6)  | -0.0018 (6)  |
| C3  | 0.0317 (7)  | 0.0334 (7)  | 0.0295 (7)   | 0.0016 (6)   | -0.0019 (6)  | -0.0056 (6)  |
| C4  | 0.0297 (6)  | 0.0302 (7)  | 0.0262 (6)   | 0.0051 (6)   | 0.0003 (5)   | -0.0009 (5)  |
| C5  | 0.0551 (11) | 0.0604 (12) | 0.0497 (10)  | -0.0134 (10) | 0.0204 (9)   | -0.0003 (9)  |
| C6  | 0.0372 (8)  | 0.0548 (11) | 0.0421 (9)   | -0.0125 (8)  | 0.0044 (7)   | -0.0029 (8)  |
| C7  | 0.0355 (8)  | 0.0529 (10) | 0.0395 (8)   | -0.0020 (7)  | 0.0064 (7)   | 0.0023 (8)   |
| C8  | 0.0355 (7)  | 0.0361 (7)  | 0.0262 (6)   | 0.0027 (6)   | 0.0047 (6)   | 0.0031 (6)   |
| C9  | 0.0557 (11) | 0.0398 (8)  | 0.0359 (8)   | 0.0028 (8)   | 0.0116 (8)   | -0.0025 (7)  |
| C10 | 0.0778 (16) | 0.0532 (11) | 0.0441 (10)  | 0.0179 (11)  | 0.0222 (10)  | -0.0019 (9)  |
| C11 | 0.0537 (13) | 0.0828 (15) | 0.0625 (13)  | 0.0234 (12)  | 0.0275 (11)  | 0.0038 (12)  |
| C12 | 0.0374 (9)  | 0.0844 (15) | 0.0616 (12)  | 0.0002 (11)  | 0.0152 (9)   | 0.0022 (11)  |
| C13 | 0.0357 (8)  | 0.0300 (7)  | 0.0297 (7)   | -0.0073 (6)  | 0.0046 (6)   | -0.0022 (5)  |
| C14 | 0.0445 (9)  | 0.0487 (10) | 0.0408 (9)   | -0.0166 (8)  | -0.0042 (7)  | 0.0046 (7)   |
| C15 | 0.0618 (12) | 0.0596 (11) | 0.0462 (10)  | -0.0313 (10) | -0.0019 (10) | -0.0048 (10) |
| C16 | 0.0737 (14) | 0.0452 (11) | 0.0626 (12)  | -0.0282 (10) | 0.0175 (11)  | -0.0168 (9)  |
| C17 | 0.0615 (13) | 0.0334 (8)  | 0.0749 (14)  | -0.0024 (9)  | 0.0114 (11)  | -0.0033 (9)  |
| C18 | 0.0438 (10) | 0.0353 (8)  | 0.0498 (10)  | 0.0015 (7)   | 0.0015 (8)   | -0.0031 (7)  |
| N1  | 0.0304 (6)  | 0.0273 (5)  | 0.0262 (5)   | -0.0031 (5)  | 0.0021 (5)   | -0.0003 (4)  |
| N2  | 0.0346 (7)  | 0.0460 (8)  | 0.0409 (8)   | 0.0039 (6)   | -0.0072 (6)  | -0.0161 (6)  |
| O1  | 0.0633 (10) | 0.0836 (12) | 0.0880 (12)  | -0.0434 (10) | 0.0184 (9)   | -0.0231 (10) |
| O2  | 0.0361 (6)  | 0.0368 (5)  | 0.0288 (5)   | 0.0014 (5)   | -0.0028 (4)  | 0.0014 (4)   |
| O3  | 0.0615 (8)  | 0.0269 (5)  | 0.0507 (7)   | 0.0006 (5)   | -0.0068 (6)  | -0.0009 (5)  |
| O4  | 0.0583 (9)  | 0.0755 (10) | 0.0365 (7)   | 0.0049 (8)   | -0.0143 (6)  | -0.0108 (6)  |
| O5  | 0.0563 (8)  | 0.0461 (7)  | 0.0652 (9)   | -0.0123 (6)  | -0.0069 (7)  | -0.0198 (7)  |
| S1  | 0.0506 (2)  | 0.0458 (2)  | 0.02760 (17) | 0.00591 (19) | 0.00479 (17) | 0.00507 (16) |

Geometric parameters (Å, °)

|            |             |             |             |
|------------|-------------|-------------|-------------|
| C1—O2      | 1.3802 (18) | C10—C11     | 1.383 (4)   |
| C1—N1      | 1.5030 (18) | C10—H10     | 0.9300      |
| C1—C8      | 1.520 (2)   | C11—C12     | 1.373 (4)   |
| C1—C2      | 1.569 (2)   | C11—H11     | 0.9300      |
| C2—O3      | 1.409 (2)   | C12—H12     | 0.9300      |
| C2—C3      | 1.499 (2)   | C13—C18     | 1.382 (2)   |
| C2—C6      | 1.533 (2)   | C13—C14     | 1.388 (2)   |
| C3—N2      | 1.3811 (19) | C13—N1      | 1.4377 (18) |
| C3—C4      | 1.385 (2)   | C14—C15     | 1.387 (3)   |
| C4—N1      | 1.3567 (18) | C14—H14     | 0.9300      |
| C4—S1      | 1.7304 (15) | C15—C16     | 1.371 (3)   |
| C5—S1      | 1.807 (2)   | C15—H15     | 0.9300      |
| C5—H5A     | 0.9600      | C16—C17     | 1.386 (4)   |
| C5—H5B     | 0.9600      | C16—H16     | 0.9300      |
| C5—H5C     | 0.9600      | C17—C18     | 1.391 (3)   |
| C6—O1      | 1.202 (2)   | C17—H17     | 0.9300      |
| C6—C7      | 1.474 (3)   | C18—H18     | 0.9300      |
| C7—C8      | 1.389 (2)   | N2—O4       | 1.234 (2)   |
| C7—C12     | 1.398 (3)   | N2—O5       | 1.266 (2)   |
| C8—C9      | 1.389 (2)   | O2—H2       | 0.8200      |
| C9—C10     | 1.391 (3)   | O3—H3       | 0.8200      |
| C9—H9      | 0.9300      |             |             |
| O2—C1—N1   | 112.11 (12) | C11—C10—C9  | 122.07 (19) |
| O2—C1—C8   | 109.23 (12) | C11—C10—H10 | 119.0       |
| N1—C1—C8   | 112.26 (11) | C9—C10—H10  | 119.0       |
| O2—C1—C2   | 115.02 (12) | C12—C11—C10 | 120.5 (2)   |
| N1—C1—C2   | 103.76 (11) | C12—C11—H11 | 119.7       |
| C8—C1—C2   | 104.18 (12) | C10—C11—H11 | 119.7       |
| O3—C2—C3   | 114.69 (13) | C11—C12—C7  | 118.1 (2)   |
| O3—C2—C6   | 112.16 (14) | C11—C12—H12 | 121.0       |
| C3—C2—C6   | 111.79 (14) | C7—C12—H12  | 121.0       |
| O3—C2—C1   | 111.66 (13) | C18—C13—C14 | 120.66 (15) |
| C3—C2—C1   | 101.07 (12) | C18—C13—N1  | 119.86 (15) |
| C6—C2—C1   | 104.46 (13) | C14—C13—N1  | 119.39 (15) |
| N2—C3—C4   | 124.50 (14) | C15—C14—C13 | 119.01 (19) |
| N2—C3—C2   | 121.85 (14) | C15—C14—H14 | 120.5       |
| C4—C3—C2   | 111.73 (13) | C13—C14—H14 | 120.5       |
| N1—C4—C3   | 110.08 (13) | C16—C15—C14 | 120.83 (19) |
| N1—C4—S1   | 125.85 (11) | C16—C15—H15 | 119.6       |
| C3—C4—S1   | 123.82 (11) | C14—C15—H15 | 119.6       |
| S1—C5—H5A  | 109.5       | C15—C16—C17 | 120.00 (17) |
| S1—C5—H5B  | 109.5       | C15—C16—H16 | 120.0       |
| H5A—C5—H5B | 109.5       | C17—C16—H16 | 120.0       |
| S1—C5—H5C  | 109.5       | C16—C17—C18 | 120.0 (2)   |
| H5A—C5—H5C | 109.5       | C16—C17—H17 | 120.0       |
| H5B—C5—H5C | 109.5       | C18—C17—H17 | 120.0       |
| O1—C6—C7   | 127.37 (19) | C13—C18—C17 | 119.48 (18) |



|              |              |                 |              |
|--------------|--------------|-----------------|--------------|
| O1—C6—C2     | 125.13 (19)  | C13—C18—H18     | 120.3        |
| C7—C6—C2     | 107.41 (14)  | C17—C18—H18     | 120.3        |
| C8—C7—C12    | 121.52 (18)  | C4—N1—C13       | 125.54 (12)  |
| C8—C7—C6     | 110.28 (15)  | C4—N1—C1        | 110.81 (11)  |
| C12—C7—C6    | 128.00 (18)  | C13—N1—C1       | 118.38 (11)  |
| C7—C8—C9     | 120.20 (16)  | O4—N2—O5        | 122.11 (15)  |
| C7—C8—C1     | 111.24 (14)  | O4—N2—C3        | 120.10 (15)  |
| C9—C8—C1     | 128.48 (15)  | O5—N2—C3        | 117.78 (16)  |
| C8—C9—C10    | 117.6 (2)    | C1—O2—H2        | 109.5        |
| C8—C9—H9     | 121.2        | C2—O3—H3        | 109.5        |
| C10—C9—H9    | 121.2        | C4—S1—C5        | 101.78 (8)   |
| O2—C1—C2—O3  | -15.37 (18)  | N1—C1—C8—C9     | -64.8 (2)    |
| N1—C1—C2—O3  | 107.44 (14)  | C2—C1—C8—C9     | -176.43 (15) |
| C8—C1—C2—O3  | -134.90 (13) | C7—C8—C9—C10    | -1.7 (3)     |
| O2—C1—C2—C3  | -137.77 (13) | C1—C8—C9—C10    | -178.21 (16) |
| N1—C1—C2—C3  | -14.95 (14)  | C8—C9—C10—C11   | 0.5 (3)      |
| C8—C1—C2—C3  | 102.70 (13)  | C9—C10—C11—C12  | 1.2 (4)      |
| O2—C1—C2—C6  | 106.06 (15)  | C10—C11—C12—C7  | -1.7 (4)     |
| N1—C1—C2—C6  | -131.12 (13) | C8—C7—C12—C11   | 0.4 (3)      |
| C8—C1—C2—C6  | -13.47 (16)  | C6—C7—C12—C11   | 174.7 (2)    |
| O3—C2—C3—N2  | 59.5 (2)     | C18—C13—C14—C15 | 1.8 (3)      |
| C6—C2—C3—N2  | -69.58 (19)  | N1—C13—C14—C15  | -174.80 (16) |
| C1—C2—C3—N2  | 179.80 (14)  | C13—C14—C15—C16 | -2.6 (3)     |
| O3—C2—C3—C4  | -105.35 (15) | C14—C15—C16—C17 | 1.3 (3)      |
| C6—C2—C3—C4  | 125.53 (15)  | C15—C16—C17—C18 | 0.9 (3)      |
| C1—C2—C3—C4  | 14.91 (16)   | C14—C13—C18—C17 | 0.3 (3)      |
| N2—C3—C4—N1  | -173.08 (14) | N1—C13—C18—C17  | 176.95 (17)  |
| C2—C3—C4—N1  | -8.67 (18)   | C16—C17—C18—C13 | -1.7 (3)     |
| N2—C3—C4—S1  | 1.5 (2)      | C3—C4—N1—C13    | -156.00 (14) |
| C2—C3—C4—S1  | 165.92 (11)  | S1—C4—N1—C13    | 29.5 (2)     |
| O3—C2—C6—O1  | -40.0 (3)    | C3—C4—N1—C1     | -2.30 (16)   |
| C3—C2—C6—O1  | 90.4 (2)     | S1—C4—N1—C1     | -176.76 (11) |
| C1—C2—C6—O1  | -161.1 (2)   | C18—C13—N1—C4   | 39.6 (2)     |
| O3—C2—C6—C7  | 136.79 (15)  | C14—C13—N1—C4   | -143.72 (16) |
| C3—C2—C6—C7  | -92.78 (16)  | C18—C13—N1—C1   | -112.28 (17) |
| C1—C2—C6—C7  | 15.68 (18)   | C14—C13—N1—C1   | 64.36 (19)   |
| O1—C6—C7—C8  | 164.5 (2)    | O2—C1—N1—C4     | 136.14 (13)  |
| C2—C6—C7—C8  | -12.2 (2)    | C8—C1—N1—C4     | -100.46 (14) |
| O1—C6—C7—C12 | -10.3 (4)    | C2—C1—N1—C4     | 11.43 (15)   |
| C2—C6—C7—C12 | 173.0 (2)    | O2—C1—N1—C13    | -68.05 (16)  |
| C12—C7—C8—C9 | 1.3 (3)      | C8—C1—N1—C13    | 55.35 (17)   |
| C6—C7—C8—C9  | -173.89 (16) | C2—C1—N1—C13    | 167.24 (13)  |
| C12—C7—C8—C1 | 178.35 (18)  | C4—C3—N2—O4     | -23.3 (2)    |
| C6—C7—C8—C1  | 3.1 (2)      | C2—C3—N2—O4     | 173.83 (16)  |
| O2—C1—C8—C7  | -116.54 (15) | C4—C3—N2—O5     | 158.10 (16)  |
| N1—C1—C8—C7  | 118.47 (14)  | C2—C3—N2—O5     | -4.8 (2)     |
| C2—C1—C8—C7  | 6.84 (17)    | N1—C4—S1—C5     | 28.50 (16)   |
| O2—C1—C8—C9  | 60.2 (2)     | C3—C4—S1—C5     | -145.23 (14) |

*Hydrogen-bond geometry (Å, °)*

Cg1 is the centroid of the C13–C18 benzene ring.

| <i>D</i> —H··· <i>A</i>    | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O3—H3···O5                 | 0.82        | 2.34          | 2.895 (2)             | 126                     |
| O2—H2···O5 <sup>i</sup>    | 0.82        | 2.00          | 2.8155 (18)           | 171                     |
| C11—H11···O4 <sup>ii</sup> | 0.93        | 2.51          | 3.423 (3)             | 166                     |
| C9—H9···Cg1                | 0.93        | 2.89          | 3.545 (2)             | 129                     |

Symmetry codes: (i)  $x+1/2, -y-1/2, -z$ ; (ii)  $-x-1/2, -y, z+1/2$ .