Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 8-Chloro-4-[1-(phenylsulfonyl)indol-3-yl]-3a,4,5,9b-tetrahydro-3*H*-cyclopenta[*c*]quinoline

**R. Sankaranarayanan,[a] M. Yogavel,[b] D. Velmurugan,[b]* K. Sekar,[c] G. Babu,[d] P. T. Perumal,[d] S. Shanmuga Sundara Raj[e] and H.-K. Fun[e]**

[a]Molecular Biophysics Unit, Indian Institute of Science, Bangalore 560 012, India, [b]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [c]Bioinformatics Centre, Supercomputer Education and Research Centre, Indian Institute of Science, Bangalore 560 012, India, [d]Organic Chemistry Division, Central Leather Research Institute, Adyar, Chennai 600 020, India, and [e]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: d_velu@yahoo.com

**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.045
*wR* factor = 0.124
Data-to-parameter ratio = 18.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

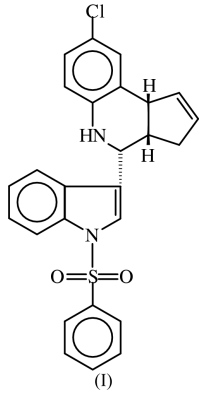


Received 2 December 2002
Accepted 9 December 2002
Online 19 December 2002

In the title compound, $C_{26}H_{21}ClN_2O_2S$, the tetrahydropyridine ring adopts a sofa conformation and the cyclopentene ring adopts an envelope conformation. In the crystal, centrosymmetrically related molecules exist as N—H···O and C—H···O hydrogen-bonded dimers, and the molecular packing is stabilized by C—H···π and van der Waals interactions.

## Comment

A large number of natural products contain the quinoline and indole heterocycles,i and they are found in numerous commercial products, including pharmaceuticals, fragrances and dyes (Padwa *et al.*, 1999). Tetrahydroquinoline derivatives exhibit antitumour activities (Jaton *et al.*, 1997), and also act as potent antipsychotic agents (Norman *et al.*, 1996) and anti-schistosomal drugs (Billings & Heidelberger, 1982). They also possess anti-inflammatory (Ohnishi *et al.*, 1981), antiulcer (Uchida *et al.*, 1989) and analgesic (Shaaban *et al.*, 1977) activities. The X-ray crystal structure analysis of the title compound, (I), was carried out as part of our studies on tetrahydroquinoline compounds containing indole derivatives (Sankaranarayanan *et al.*, 2000).




(I)

Bond lengths and angles in (I) agree with those reported for similar structures (Sivaraman *et al.*, 1994*a*,*b*,*c*; Sivaraman *et al.*, 1996; Sankaranarayanan *et al.*, 2000). The tetrahydropyridine ring, *B*, adopts a sofa conformation with asymmetry parameter (Cremer & Pople, 1975) $\Delta_S$(C6) = 0.006 (1) (Nardelli, 1983); atom C12 deviates from the mean plane through the other atoms in the ring by 0.698 (2) Å. The cyclopentene ring, *A*, is in an envelope conformation, as indicated by the lowest asymmetry parameter $\Delta_S$(C11) of 0.011 (1); atom C11 deviates by 0.289 (2) Å from the mean plane passing through C7, C8, C9 and C10. The indole system is not strictly planar, the dihedral angle between the planes of the fused five- and six-membered rings being 2.1 (1)°. The torsion angles O23—S22—N15—C16 [172.8 (1)°] and O23—S22—C25—C26

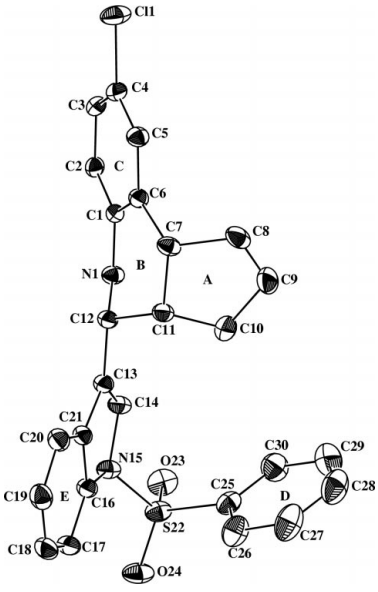



**Figure 1**
View of the title molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 35% probability level. H atoms have been omitted.

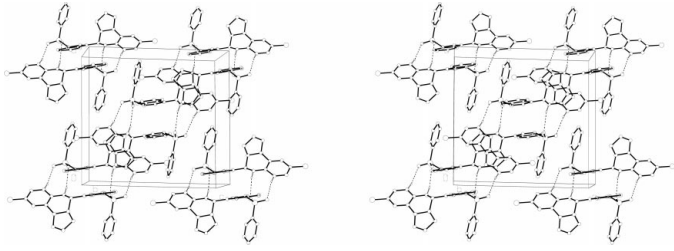


**Figure 2**
Stereoview of the packing of the molecules, viewed down the *b* axis.

[−176.3 (2)°] describe the conformation of the phenylsulfonyl group with respect to the indole system; the mean planes of the indole system and the phenyl ring (*D*) form a dihedral angle of 69.8 (1)°. The mean plane of the cyclopentene ring makes a dihedral angle of 69.4 (1)° with the mean plane passing through the plane of the tetrahydroquinoline moiety excluding atoms C11 and C12. Atom N15 deviates by 0.325 (2) Å from the plane through C14, C16 and S22, and the sum of the angles around N15 is 346.3 (1)°. This pyramidalization behaviour is also observed in related indoles (Yokum & Fronczek, 1997; Sankaranarayanan *et al.*, 2000).

The orientation of the phenyl ring, *D*, is influenced by the weak C30—H30···O23 interaction, while the orientation of the indole substituent is influenced by the weak interaction C17—H17···O24 (Table 2). The torsion angles O23—S22—C25—C30 = 6.1 (2)°, N15—S22—C25—C30 = −106.5 (2)°, O24—S22—N15—C16 = 43.9 (2)° and S22—N15—C16—C17 = −42.2 (3)° quantitatively define these orientations. In the solid state, centrosymmetrically related molecules form dimeric pairs through N—H···O and C—H···O hydrogen bonds involving the sulfonyl O atoms (Table 2 and Fig. 2). In addition to van der Waals interactions, the molecular packing in the crystal is stabilized by C—H...$\pi$ interactions [C11—H11 = 0.93, H11···*Cg* = 2.81, C11···*Cg* = 3.668 (2) Å and C11—H11···*Cg* = 147°; *Cg* denotes the centroid of ring *C* of a symmetry-related molecule at $(\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z)$].

## Experimental

To a solution of 4-phenylsulfonyl-3-[*N*-(*p*-chlorophenyl)formimidoyl]indole (0.648 g, 0.018 mol) and cyclopentadiene (0.237 g, 0.036 mol) in acetonitrile (10 ml), protected by a guard tube, was added indium trichloride (0.081 g, 20 mol%), and the mixture was stirred at room temperature for 30 min. Water (20 ml) was added to the reaction mixture, which was then extracted with chloroform (3 × 10 ml), washed with brine (10 ml), dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude product was purified by column chromatography using silica gel (60–120 mesh) and eluted with petroleum ether–ethyl acetate (90:10) to afford the title compound. The compound was recrystallized by slow evaporation of a methanol–chloroform (1:1) solution.

*Crystal data*

$C_{26}H_{21}ClN_2O_2S$
$M_r$ = 460.96
Monoclinic, $P2_1/n$
$a$ = 15.0064 (2) Å
$b$ = 9.4052 (2) Å
$c$ = 15.7077 (2) Å
$\beta$ = 91.093 (1)°
$V$ = 2216.55 (6) Å$^3$
$Z$ = 4
$D_x$ = 1.381 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8685 reflections
$\theta$ = 1.9–28.3°
$\mu$ = 0.29 mm$^{-1}$
$T$ = 293 (2) K
Block, colourless
0.40 × 0.18 × 0.14 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
14 665 measured reflections
5441 independent reflections
4073 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031
$\theta_{max}$ = 28.3°
$h = -19 \rightarrow 19$
$k = -8 \rightarrow 12$
$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.124
$S$ = 1.03
5441 reflections
293 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.7240P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.38 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.7462 (18) | N15—S22 | 1.6665 (17) |
| N1—C1 | 1.408 (2) | S22—O24 | 1.4223 (15) |
| N1—C12 | 1.464 (2) | S22—O23 | 1.4303 (16) |
| C8—C9 | 1.310 (3) | S22—C25 | 1.763 (2) |
| N15—C16 | 1.424 (3) | | |
| C1—N1—C12 | 114.44 (14) | O24—S22—N15 | 106.68 (10) |
| C16—N15—C14 | 106.67 (14) | O23—S22—N15 | 105.62 (9) |
| C16—N15—S22 | 121.99 (12) | O24—S22—C25 | 109.50 (10) |
| C14—N15—S22 | 117.61 (14) | O23—S22—C25 | 109.22 (10) |
| O24—S22—O23 | 120.09 (9) | N15—S22—C25 | 104.55 (8) |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O23$^{i}$ | 0.87 (2) | 2.49 (2) | 3.339 (2) | 167 |
| C2—H2···O24$^{i}$ | 0.93 | 2.59 | 3.503 (2) | 168 |
| C17—H17···O24 | 0.93 | 2.46 | 3.003 (3) | 117 |
| C30—H30···O23 | 0.93 | 2.56 | 2.930 (3) | 104 |

Symmetry code: (i) $1-x, 1-y, 1-z$.

The H atom on N1 was located in a difference Fourier map and refined, while all other H atoms were positioned geometrically and were allowed to ride on their attached atom.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek,1990)'; software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

DV thanks the Department of Science and Technology (DST), India, for financial support.

## References

Billings, P. C. & Heidelberger, C. (1982). *Cancer Res.* **42**, 2692–2696.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Jaton, J. C., Roulin, K., Rose, K., Sirotnak, F. M., Lewenstein, A., Brunner, G., Fankhauser, C. P. & Burger, U. (1997). *J. Nat. Prod*. **60**, 356–360.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Norman, M. H., Navas, F., Thompson, J. B. & Rigdon, G. C. (1996). *J. Med. Chem.* **39**, 4692–4703.
Ohnishi, H., Kosuzume, H., Yamaguchi, K., Ohkura, M., Satoh, M., Uohama, M., Toyonaka, Y. & Suzuki, Y. (1981). *Jpn J. Pharmacol.* **31**, 747–756.
Padwa, A., Brodney, M. A., Liu, B., Satake, K. & Wu, T. (1999). *J. Org. Chem.* **64**, 3595–3607.
Sankaranarayanan, R., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K., Babu, G. & Perumal, P. T. (2000). *Acta Cryst.* C**56**, 475–476.
Shaaban, M. A., Ghoneim, K. M. & Khalifa, M. (1977). *Pharmazie*, **32**, 90–92.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Sadanandam, E. V. (1994*a*). *Acta Cryst.* C**50**, 784–787.
Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Sadanandam, E. V. (1994*b*). *Acta Cryst.* C**50**, 787–789.
Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Sadanandam, E. V. (1994*c*). *Acta Cryst.* C**50**, 789–791.
Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Seetharaman, J. (1996). *J. Mol. Struct.* **385**, 123–128.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Uchida, M., Chihiro, M., Morita, S., Kanbe, T., Yamashita, H., Yamasaki, K., Yabuuchi, Y. & Nakagawa, K. (1989). *Chem. Pharm. Bull.* (*Tokyo*), **37**, 2109–2116.
Yokum, S. T. & Fronczek, F. R. (1997). *Acta Cryst.* C**53**, 362–363.
Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.