Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(6-Methoxybenzothiazol-2-yldiazenyl)-1-methyl-2-phenyl-1*H*-indole

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.158
Data-to-parameter ratio = 22.8

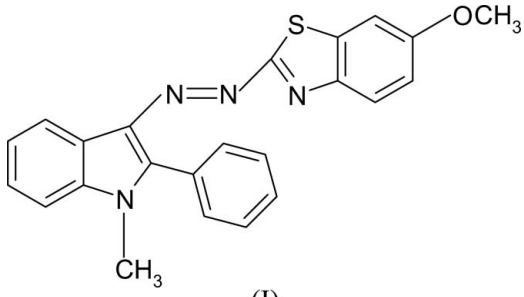
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 28 March 2007
Accepted 2 May 2007

In the title compound, $C_{23}H_{18}N_4OS$, the indole and benzothiazole ring systems are both approximately planar, with dihedral angles between the fused five- and six-membered rings of 1.16 (7) and 8.71 (5)°, respectively. The phenyl ring forms a dihedral angle of 43.43 (6)° with the indole ring system.

Comment

Heterocycles containing the 1,3-thiazole ring system exhibit a wide spectrum of biological activities. In addition to acting as antiviral and antifungal agents, this system has been identified as a central structural element of a number of biologically active natural products (Zabriskie *et al.*, 1988) and of pharmacologically active compounds (Metzger, 1984). Indole and its derivatives form a class of toxic recalcitrant *N*-heterocyclic compounds that are considered pollutants (Florin *et al.*, 1980). Azo dyes have wide applicability as optical materials and their structures have attracted considerable attention (Biswas & Umapathy, 2000). To the best of our knowledge, few structures of azoindole derivatives have been reported to date (Bruni *et al.*, 1995; Seferoğlu *et al.*, 2006, 2006*a*,*b*,*c*, 2007*a*,*b*,*c*). The present study was undertaken in order to ascertain the crystal structure of the title compound, (I).



(I)

The molecular structure of (I), is shown in Fig. 1. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987). An examination of the deviations from the least-squares planes through the individual rings shows that the indole and benzothiazole ring systems are both virtually planar, with dihedral angles between the fused five- and six-membered rings of 1.16 (7) and 8.71 (5)°, respectively. Table 1 demonstrates how these angles compare with those in closely related compounds. The phenyl ring forms a dihedral angle of 43.43 (6)° with the indole ring system.

As can be seen from the packing diagram (Fig. 2), the molecules of (I) are stacked along the *a* axis. Dipole–dipole and van der Waals interactions are effective in the molecular packing.

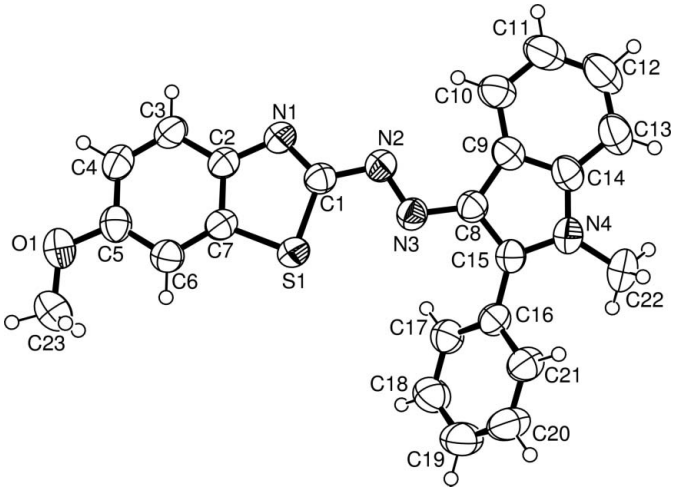


Figure 1
The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

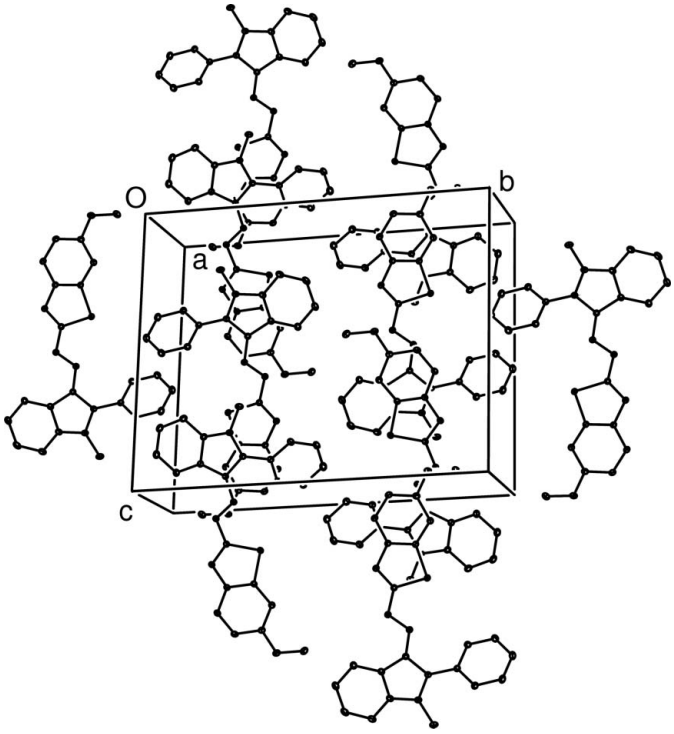


Figure 2
A packing diagram of (I). H atoms have been omitted.

Experimental

2-Amino-6-methoxybenzothiazole (360 mg, 2 mmol) was dissolved in a hot glacial acetic acid–propionic acid mixture (2:1, 6 ml). The solution was cooled rapidly in an ice–salt bath. It was then added dropwise, with stirring, to a cold solution of nitrosylsulfuric acid (3 ml) over a period of 30 min. The mixture was stirred for an additional 2 h at 273 K. The resulting diazonium salt was cooled in an ice–salt bath, then added dropwise, with stirring, to 1-methyl-2-phenylindole (414 mg, 2 mmol) in an acetic acid–propionic acid mixture (3:1, 8 ml). The solution was stirred at 273–278 K for 2 h and the pH of the reaction mixture was maintained at 4–6 by the addition of a saturated sodium carbonate solution (40 ml). The mixture was stirred for a further day. The resulting solid was filtered off, washed with cold water and crystallized from ethanol (yield 550 mg, 69%; m.p. 503–505 K).

Crystal data

$C_{23}H_{18}N_4OS$
$M_r = 398.48$
Monoclinic, $P2_1/c$
$a = 7.3672$ (2) Å
$b = 17.9326$ (5) Å
$c = 15.0284$ (3) Å
$\beta = 99.70$ (2)°
$V = 1957.07$ (13) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.19$ mm^{-1}
$T = 294$ (2) K
0.35 × 0.25 × 0.15 mm

Data collection

Rigaku *R*-AXIS RAPID-S diffractometer
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.927$, $T_{max} = 0.962$
58037 measured reflections
6030 independent reflections
4066 reflections with $I > 2\sigma(I)$
$R_{int} = 0.065$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.158$
$S = 1.06$
6030 reflections
264 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Dihedral angles (°) in closely related compounds.

3-(4-Chlorophenyldiazenyl)-1-methyl-2-phenyl-1*H*-indole[a]	1.56 (11), 0.77 (12)
N-{4-[(2-Phenyl-1*H*-indol-3-yl)diazenyl]phenyl}acetamide[b]	1.63 (14)
Ethyl[2-(2-phenyl-1*H*-indol-3-yldiazenyl)-1,3-thiazol-4-yl]-acetate[c]	0.99 (10)
Ethyl-2-{2-[(1-methyl-2-phenyl-1*H*-indol-3-yl)diazenyl]-thiazol-4-yl}acetate[d]	0.59 (7)
1-Methyl-2-phenyl-3-(1,3,4-thiadiazol-2-yldiazenyl)-1*H*-indole[e]	4.26 (7)
1,2-Dimethyl-3-(thiazol-2-yldazenyl)-1*H*-indole[f]	2.07 (9), 2.04 (9)
3-(5-Ethyl-1,3,4-thiadiazol-2-yldiazenyl)-1-methyl-2-phenyl-1*H*-indole[g]	0.59 (12)

Notes: (*a*) Seferoğlu *et al.* (2006*a*); (*b*) Seferoğlu *et al.* (2006*b*); (*c*) Seferoğlu *et al.* (2006*c*); (*d*) Seferoğlu *et al.* (2006); (*e*) Seferoğlu *et al.* (2007*a*); (*f*) Seferoğlu *et al.* (2007*b*); (*g*) Seferoğlu *et al.* (2007*c*).

H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å, respectively, for aromatic and methyl H atoms, and constrained to ride on their parent atoms with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.2$ for aromatic and $x = 1.5$ for methyl H atoms.

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are indebted to the Department of Chemistry, Atatürk University, Erzurum, Turkey, for the use of the X-ray diffractometer purchased under grant No. 2003/219 of the University Research Fund.

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