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3-(5-Ethyl-1,3,4-thiadiazol-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) = 0.006 Å
R factor = 0.102
wR factor = 0.212
Data-to-parameter ratio = 20.9

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

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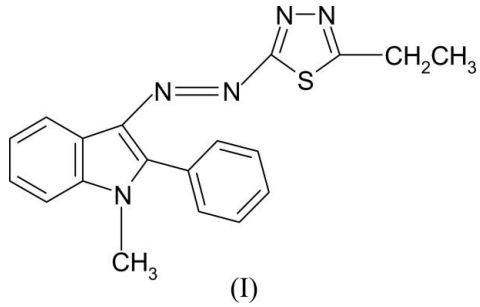
In the molecule of the title compound, $C_{19}H_{17}N_5S$, all the rings are individually planar. Within the indole ring system, the dihedral angle between the five- and six-membered rings is 0.59 (12)°. The thiadiazole and phenyl rings form dihedral angles of 6.32 (10) and 44.36 (13)°, respectively, with the indole ring system.

Comment

The thiadiazole nucleus has been exploited by the pharmaceutical industry in the development of drugs or drug candidates, due to its exceptional pharmacological properties (Ward *et al.*, 1998; Hanasaki *et al.*, 1995), and substituted thiadiazole derivatives have found useful applications in many different technological areas (Weinstock & Shinkai, 1984). Thiadiazole and its derivatives are also used for their biological activities, such as antiviral, antibacterial, antifungal and antitubercular properties. 1,3,4-Thiadiazole and its derivatives are of great interest in chemistry, owing to their bioactivity with regard to certain plant-growth regulating effects, as well as their antimicrobial activity (Seaborg, 1984). Their special structures and properties have been widely reported in the fields of synthesis and spectroscopic analysis, and in traditional applications such as medicines and pesticides (Al-Mulaikel & El-Emary, 2003; Shouji *et al.*, 1996). Indole and its derivatives form a class of toxic recalcitrant N-heterocyclic compounds that are considered as pollutants (Florin *et al.*, 1980). Azo dyes have wide applicability as optical materials and their structures have also 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*a*,*b*,*c*; Seferoğlu, Hökelek, Şahin *et al.*, 2006; Seferoğlu *et al.*, 2007*a*,*b*). 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).

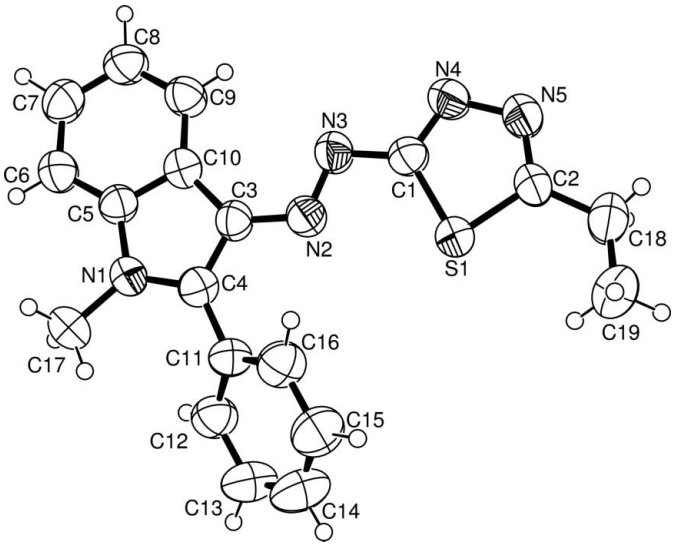


Figure 1
The title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for H atoms).

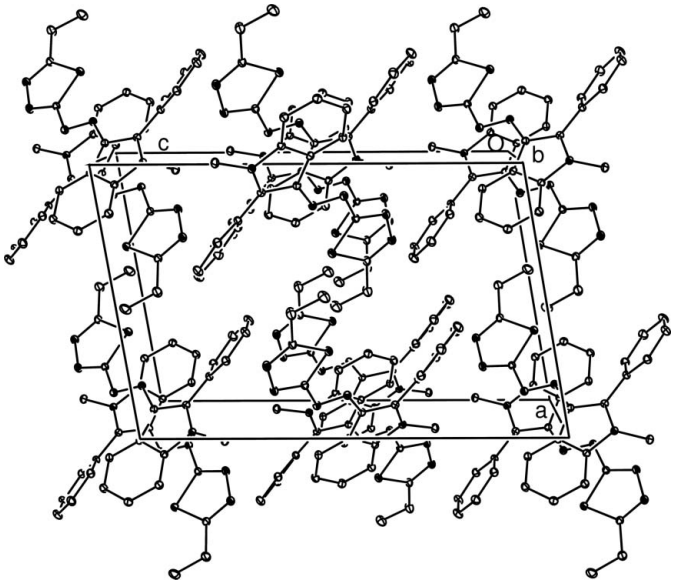


Figure 2
A packing diagram for (I). H atoms have been omitted.

An examination of the deviations from the least-squares planes through the individual rings shows that all the rings are planar. The indole ring system is planar, with a dihedral angle of 0.59 (12)° between rings *A* (C5–C10) and *B* (N1/C3–C5/C10). In the closely related compounds 3-(4-chlorophenyldiazenyl)-1-methyl-2-phenyl-1*H*-indole, (II) (Seferoğlu *et al.*, 2006*a*), *N*-{4-[(2-phenyl-1*H*-indol-3-yl)diazenyl]phenyl}acetamide, (III) (Seferoğlu *et al.*, 2006*b*), ethyl [2-(2-phenyl-1*H*-indol-3-yldiazenyl)-1,3-thiazol-4-yl]acetate, (IV) (Seferoğlu *et al.*, 2006*c*), ethyl 2-{2-[(1-methyl-2-phenyl-1*H*-indol-3-yl)-diazenyl]thiazol-4-yl}acetate, (V) (Seferoğlu, Hökelek, Şahin *et al.*, 2006), 1-methyl-2-phenyl-3-(1,3,4-thiadiazol-2-yldiazenyl)-1*H*-indole, (VI) (Seferoğlu *et al.*, 2007*a*) and 1,2-dimethyl-3-(thiazol-2-yldazenyl)-1*H*-indole, (VII) (Seferoğlu *et al.*, 2007*b*), the observed *A*/*B* and/or *A*′/*B*′ dihedral angles are 1.56 (11) and 0.77 (12)° in (II), 1.63 (14)° in (III), 0.99 (10)° in (IV), 0.59 (7)° in (V), 4.26 (7)° in (VI), and 2.07 (9) and 2.04 (9)° in (VII). The orientations of rings *C* (C11–C16) and *D* (S1/N4/N5/C1/C2) with respect to the indole ring system may be described by the dihedral angles of 44.36 (13) and 6.32 (10)°, respectively.

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

Experimental

For the preparation of the title compound, 2-amino-5-ethyl-1,3,4-thiadiazole (260 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 and then added dropwise with stirring to a cold solution of nitrosylsulfuric acid (95%, 4 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 and 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 simultaneous addition of a saturated sodium carbonate solution (40 ml). The mixture was stirred for a further 1 d. The resulting solid was filtered off, washed with cold water and crystallized from ethanol (yield 610 mg, 88%).

Crystal data

$C_{19}H_{17}N_5S$
M_r = 347.45
Monoclinic, $P2_1/c$
a = 10.7629 (14) Å
b = 10.0115 (8) Å
c = 16.568 (2) Å
β = 99.664 (6)°
V = 1759.9 (3) Å^3
Z = 4
D_x = 1.311 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 294 (2) K
Plate, red
0.35 × 0.25 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
ω scans
Absorption correction: none
51782 measured reflections
5423 independent reflections
2212 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 30.8°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.102
$wR(F^2)$ = 0.212
S = 1.07
5423 reflections
260 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.8413P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

The H atoms bound to atoms C6 and C17–C19 were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å, respectively, for aromatic, methylene and methyl H atoms, and constrained to ride on

their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H or $x = 1.2$ for all other H atoms. The remaining H atoms were located in a difference synthesis and refined isotropically [C—H = 0.87 (4)–1.05 (5) Å and $U_{iso}(H)$ = 0.069 (13)–0.14 (2) Å^2].

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).

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