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3-[(5-Ethyl-1,3,4-thiadiazol-2-yl)diazenyl]-1,2-dimethyl-1*H*-indole

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Key indicators

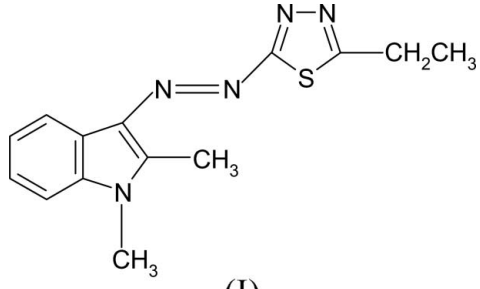
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.061
wR factor = 0.153
Data-to-parameter ratio = 23.1

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

In the title compound, $C_{14}H_{15}N_5S$, the indole ring system is nearly planar, with a dihedral angle of 2.74 (7)° between the two rings. The thiadiazole ring forms a dihedral angle of 5.44 (5)° with the indole ring system.

Comment

Thiadiazole and its derivatives are used for their biological activities, such as antiviral, antibacterial, antifungal and antitubercular properties. Thus, they have been exploited by the pharmaceutical industry in the development of drugs or drug candidates (Ward *et al.*, 1998; Hanasaki *et al.*, 1995). On the other hand, substituted thiadiazole derivatives have found useful applications in many different technological areas (Weinstock & Shinkai, 1984). 1,3,4-Thiadiazole and its derivates 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, 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). The indole ring system is nearly planar, with a dihedral angle of 2.74 (7)° between rings *A* (C4–C9) and *B* (N1/C3/C4/C9/C10). In the closely related compounds (Seferoğlu *et al.*, 2006, 2006*a*,*b*,*c*, 2007*a*,*b*,*c*), the observed *A*/*B* and/or *A*′/*B*′dihedral

angles are in the range 0.59 (7)–4.26 (7)°. The orientation of the ring *C* (S1/N4/N5/C1/C2) with respect to the indole ring system may be described by the dihedral angle of 5.44 (5)°.

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, 8 ml). The solution was rapidly cooled in an ice–salt bath and then added dropwise with stirring to a cold solution of nitrosulfuric acid (95%, 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 and then added dropwise with stirring to 1,2-dimethylindole (290 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, washed with cold water and crystallized from ethanol (yield; 440 mg, 83%; m.p. 473-475 K).

Crystal data

$C_{14}H_{15}N_5S$
$M_r = 285.38$
Triclinic, $P\overline{1}$
$a = 8.1879$ (1) Å
$b = 8.2359$ (2) Å
$c = 10.5343$ (3) Å
$\alpha = 92.56$ (1)°
$\beta = 96.66$ (2)°
$\gamma = 92.31$ (3)°
$V = 704.14$ (4) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.23$ mm^{-1}
$T = 294$ (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.925$, $T_{max} = 0.957$
21347 measured reflections
4281 independent reflections
2750 reflections with $I > 2\sigma(I)$
$R_{int} = 0.083$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.153$
$S = 1.05$
4281 reflections
185 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

H atoms 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, and $x = 1.2$ for all other 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).

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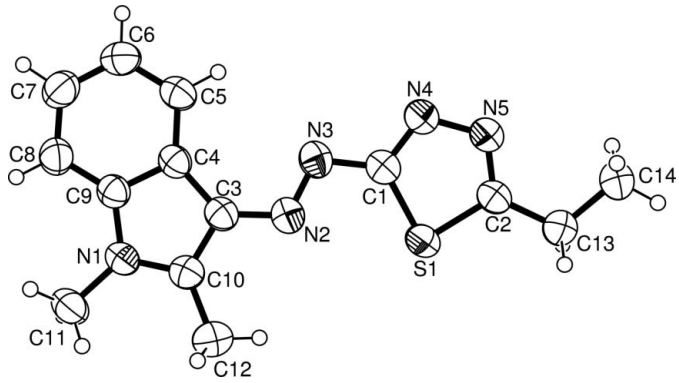

Figure 1
The title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for H atoms).

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