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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.074
wR factor = 0.198
Data-to-parameter ratio = 23.4

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

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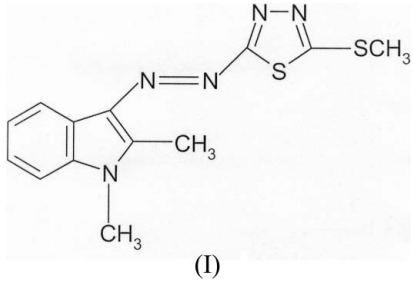
The asymmetric unit of the title compound, $C_{13}H_{13}N_5S_2$, contains two molecules. The thiadiazole rings form dihedral angles of 4.19 (7) and 1.41 (7)° with the indole ring systems in the two molecules.

Comment

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 unusual structures and properties have been widely reported in the fields of synthesis and spectroscopic analysis, and in applications such as medicines and pesticides (Al-Muaikel & El-Emary, 2003; Shouji *et al.*, 1996). 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 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 *et al.*, 2006; Seferoğlu *et al.*, 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 asymmetric unit contains two molecules. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

The orientations of the rings *C* (S1/N4/N5/C11/C12) and *C*′ (S1′/N4′/N5′/C11′/C12′) with respect to the indole ring systems may be described by dihedral angles of 4.19 (7) and 1.41 (7)°, respectively.

Experimental

For the preparation of the title compound, 2-amino-5-methylthio-1,3,4-thiadiazole (290 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 off, washed with cold water and crystallized from ethanol (yield 510 mg, 84%; m.p. 519–520 K).

Crystal data

$C_{13}H_{13}N_5S_2$
M_r = 303.42
Triclinic, $P\overline{1}$
a = 7.2739 (2) Å
b = 13.4818 (2) Å
c = 14.7373 (4) Å
α = 78.488 (4)°
β = 87.707 (5)°
γ = 85.551 (5)°
V = 1411.43 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 294 (2) K
0.35 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer
Absorption correction: none
40820 measured reflections
8574 independent reflections
4852 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.198
S = 1.04
8574 reflections
367 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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 H 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).

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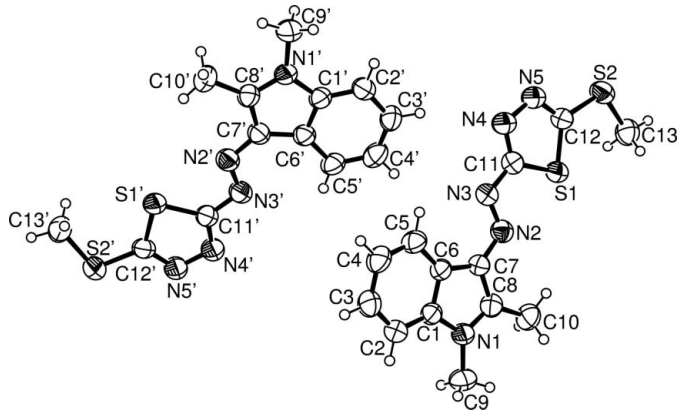

Figure 1
The asymmetric unit of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level (arbitrary spheres for H atoms).

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