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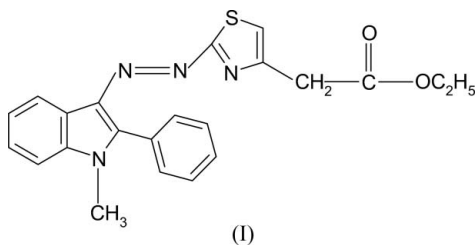
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Key indicators

Single-crystal X-ray study
T = 294 K
Mean $\sigma(\text{C}-\text{C})$ = 0.003 Å
R factor = 0.064
wR factor = 0.153
Data-to-parameter ratio = 23.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Ethyl 2-{2-[(1-methyl-2-phenyl-1*H*-indol-3-yl)-diazenyl]thiazol-4-yl}acetateIn the molecule of the title compound, C₂₂H₂₀N₄O₂S, all of the rings are individually planar. The thiazole and phenyl rings form dihedral angles of 13.33 (5) and 50.60 (6)°, respectively, with the indole ring system.Received 20 October 2006
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Comment

The 1,3-thiazole ring 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); heterocycles containing it exhibit a wide spectrum of biological activities, including antiviral and antifungal agents. 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, only a few structures of azoindole derivatives have been reported to date (Bruni *et al.*, 1995; Seferoğlu *et al.*, 2006*a,b,c*). This study was undertaken in order to ascertain the crystal structure of the title compound, (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 all of the rings are planar. The indole ring system is planar, with a dihedral angle of 0.59 (7)° between rings *A* (C9–C14) and *B* (N1/C8/C9/C14/C15). In the closely related compounds 3-(4-chlorophenyldiazanyl)-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*), and ethyl[2-(2-phenyl-1*H*-indol-3-yl)diazenyl]-1,3-thiazol-4-yl]acetate, (IV) (Seferoğlu *et al.*, 2006*c*), the observed *A/B* dihedral angles are 1.56 (11) and 0.77 (12)° in (II), 1.63 (14)° in (III) and 0.99 (10)° in (IV). The orientations of rings *C* (C16–C21) and *D* (S1/N4/C1–C3) with respect to the indole ring system may be described by dihedral angles of 50.60 (6) and 13.33 (5)°, respectively.

Experimental

For the preparation of the title compound, 2-amino-4-ethylthiazoleacetate (0.37 g, 2.0 mmol) was dissolved in a hot glacial acetic acid–propionic acid mixture (2:1, 6 ml). The solution was rapidly cooled in an ice-salt bath and then added dropwise with stirring to a cold solution of nitrosylsulfuric acid (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 also cooled in an ice-salt bath and then added dropwise with stirring to 1-methyl-2-phenylindole (0.414 g, 2.0 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 4–5 by the simultaneous addition of a saturated sodium carbonate solution (30 ml). The mixture was stirred for a further 1 d. The resulting solid was filtered off, washed with cold water and crystallized from toluene (yield 0.69 g, 86%; m.p 450–452 K).

Crystal data

| | |
|--------------------------------|---|
| $C_{22}H_{20}N_4O_2S$ | $Z = 4$ |
| $M_r = 404.48$ | $D_x = 1.318 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation |
| $a = 9.5545 (3) \text{ \AA}$ | $\mu = 0.19 \text{ mm}^{-1}$ |
| $b = 16.5418 (2) \text{ \AA}$ | $T = 294 (2) \text{ K}$ |
| $c = 13.1399 (3) \text{ \AA}$ | Prism, red |
| $\beta = 101.07 (11)^\circ$ | $0.35 \times 0.25 \times 0.15 \text{ mm}$ |
| $V = 2038.1 (8) \text{ \AA}^3$ | |

Data collection

| | |
|--|--|
| Rigaku R-AXIS RAPID-S diffractometer | 46983 measured reflections |
| ω scans | 6248 independent reflections |
| Absorption correction: multi-scan (Blessing, 1995) | 3841 reflections with $I > 2\sigma(I)$ |
| $T_{\min} = 0.938$, $T_{\max} = 0.973$ | $R_{\text{int}} = 0.075$ |
| | $\theta_{\text{max}} = 30.6^\circ$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | $w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.5149P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.153$ | $(\Delta/\sigma)_{\text{max}} = 0.001$ |
| $S = 1.05$ | $\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$ |
| 6248 reflections | $\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$ |
| 264 parameters | |
| H-atom parameters constrained | |

H atoms were positioned geometrically with C–H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms.

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

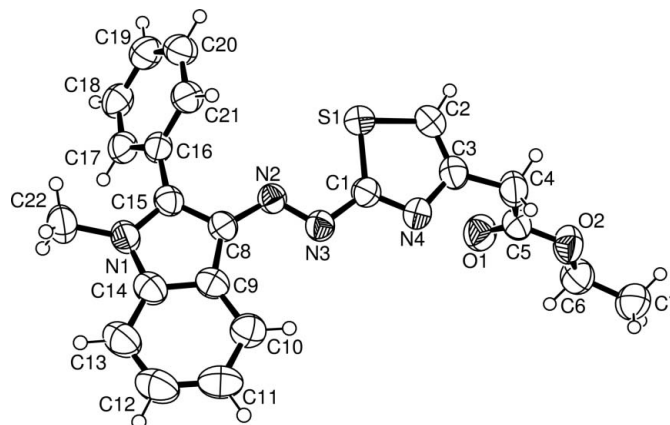


Figure 1

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

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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References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Biswas, N. & Umopathy, S. (2000). *J. Phys. Chem. A*, **104**, 2734–2745.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruni, P., Bocelli, G., Cantoni, A., Giorgini, E., Iacussi, M., Maurelli, E. & Tosi, G. (1995). *J. Chem. Crystallogr.* **25**, 683–691.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Florin, I., Rutburge, L., Curvall, M. & Enzell, C. R. (1980). *Toxicology*, **18**, 219–232.
- Metzger, J. V. (1984). *Thiazoles and their Benzo Derivatives*, edited by K. T. Potts, Vol. 6. New York: Pergamon Press.
- Rigaku/MS (2005). *CrystalClear*. Rigaku/MS, The Woodlands, Texas, USA.
- Seferoğlu, Z., Hökelek, T., Şahin, E. & Ertan, N. (2006a). *Acta Cryst.* **E62**, o2108–o2110.
- Seferoğlu, Z., Hökelek, T., Şahin, E. & Ertan, N. (2006b). *Acta Cryst.* **E62**, o3492–o3494.
- Seferoğlu, Z., Hökelek, T., Şahin, E. & Ertan, N. (2006c). *Acta Cryst.* **E62**, o3835–o3837.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Zabriskie, T. M., Mayne, C. L. & Ireland, C. M. (1988). *J. Am. Chem. Soc.* **110**, 7919–7920.