

Zeynel Seferoğlu,<sup>a</sup> Tuncer Hökelek,<sup>b</sup> Ertan Şahin<sup>c</sup> and Nermin Ertan<sup>a\*</sup><sup>a</sup>Gazi University, Department of Chemistry, 06500 Beşevler, Ankara, Turkey, <sup>b</sup>Hacettepe University, Department of Physics, 06800 Beytepe, Ankara, Turkey, and <sup>c</sup>Atatürk University, Department of Chemistry, 22240 Erzurum, Turkey

Correspondence e-mail: merzifon@hacettepe.edu.tr

## Key indicators

Single-crystal X-ray study  
 $T = 294$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.078  
 $wR$  factor = 0.162  
Data-to-parameter ratio = 17.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Ethyl [2-(2-phenyl-1*H*-indol-3-yl)diazenyl]-1,3-thiazol-4-yl]acetate

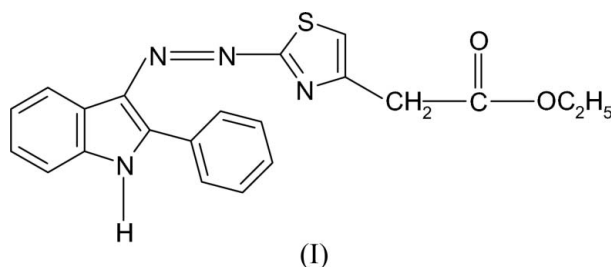
In the molecule of the title compound,  $\text{C}_{21}\text{H}_{18}\text{N}_4\text{O}_2\text{S}$ , all of the rings are individually planar. In the crystal structure, the molecules are linked by intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, forming an infinite chain.

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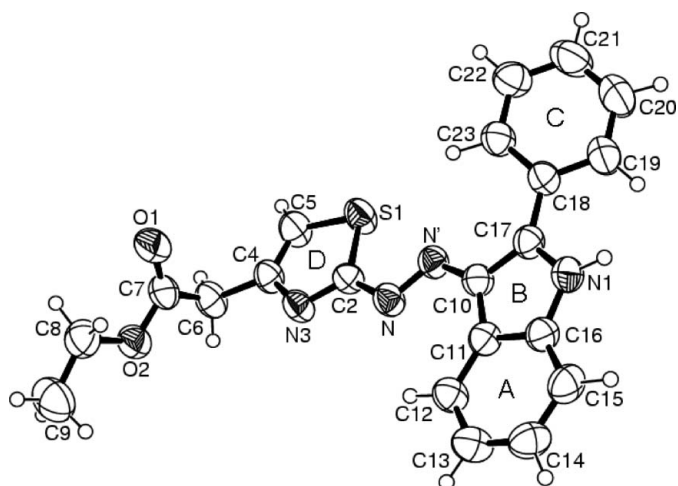
## Comment

Heterocycles containing the 1,3-thiazole ring system exhibit a wide spectrum of biological activities, including acting as antiviral and antifungal agents. 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). Azo dyes have wide applicability as optical materials and their structures have also attracted considerable attention (Biswas & Umapathy, 2000). Indole and its derivatives form a class of toxic recalcitrant N-heterocyclic compounds that are considered as pollutants (Florin *et al.*, 1980) and aryl azoindoles form yellow- or orange-coloured dyes (Aldemir *et al.*, 2003). Many azo-dye breakdown products are carcinogenic, toxic or mutagenic (Ochiai *et al.*, 1986). Although there are many publications on the industrial applications of azo dyes (Tsuda *et al.*, 2000), to the best of our knowledge, few structures of azoindole derivatives have been reported to date (Seferoğlu *et al.*, 2006a,b; Bruni *et al.*, 1995). This study was undertaken in order to ascertain the crystal structure of the title compound, (I).

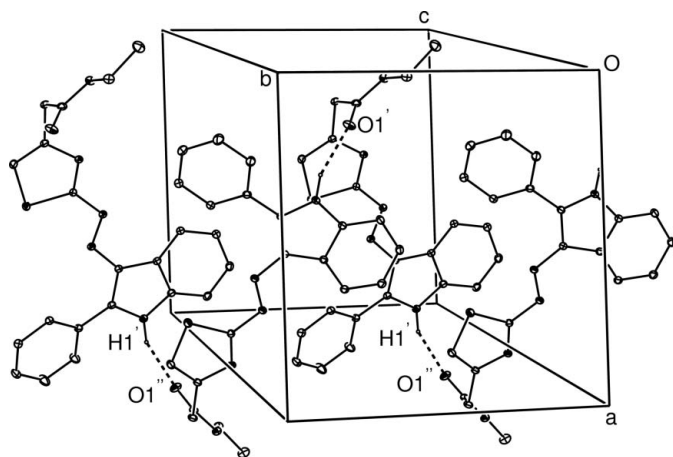


The molecular structure of compound (I) is shown in Fig. 1. The bond lengths and angles (Table 1) are in normal ranges (Allen *et al.*, 1987).

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 puckering amplitude  $Q_T = 0.029$  (3) Å (Cremer & Pople, 1975), and the dihedral angle between rings *A* and *B* is  $0.99$  (10)° (rings are as defined in Fig. 1). In the closely related compounds 3-(4-chlorophenyldiazenyl)-1-methyl-2-phenyl-1*H*-indole, (II) (Seferoğlu *et al.*, 2006a), and *N*-{4-[(2-phenyl-1*H*-indol-3-



**Figure 1**  
The molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**  
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

yl)diazonyl]phenyl]acetamide, (III) (Seferoğlu *et al.*, 2006b), the observed *A/B* dihedral angles are 1.56 (11) and 0.77 (12)° in (II) and 1.63 (14)° in (III).

The orientations of rings *C* and *D* with respect to the indole ring system may be described by dihedral angles of 22.39 (7) and 11.21 (6)°, respectively. The thiazole-diazonyl unit is not planar, with a puckering amplitude  $Q_T = 0.149$  (3) Å. Rings *B* and *D* have pseudo-twofold axes, running through atom C16 and the mid-point of the C10–C17 bond (ring *B*), and through atom S1 and the mid-point of the N3–C4 bond (ring *D*) (Table 1).

As can be seen from the packing diagram (Fig. 2), N–H...O intermolecular hydrogen bonds [H1...O1<sup>i</sup> 2.03 (3) Å, N1...O1<sup>i</sup> 2.885 (3) Å and N1–H1...O1<sup>i</sup> 153.4(2.3)°; symmetry code: (i)  $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ] link the molecules to form an infinite chain. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

## 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 nitrosyl sulfuric 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 2-phenylindole (0.386 g, 2.0 mmol) in an acetic acid–propionic acid mixture (2:1; 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 0.68 g, 87%, m.p. 461 K).

## Crystal data

$C_{21}H_{18}N_4O_2S$	$Z = 4$
$M_r = 390.45$	$D_x = 1.364 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 10.6111$ (1) Å	$\mu = 0.20 \text{ mm}^{-1}$
$b = 10.5040$ (2) Å	$T = 294$ (2) K
$c = 17.3345$ (2) Å	Block, brown
$\beta = 100.248$ (1)°	$0.35 \times 0.25 \times 0.15 \text{ mm}$
$V = 1901.26$ (5) Å <sup>3</sup>	

## Data collection

Rigaku R-AXIS RAPID-S diffractometer	36040 measured reflections
$\omega$ scans	5786 independent reflections
Absorption correction: multi-scan (Blessing, 1995)	3501 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.935$ , $T_{\max} = 0.971$	$R_{\text{int}} = 0.077$
	$\theta_{\max} = 30.5^\circ$

## Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.4823P]$
$R[F^2 > 2\sigma(F^2)] = 0.078$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.162$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.11$	$\Delta\rho_{\max} = 0.19 \text{ e } \text{Å}^{-3}$
5786 reflections	$\Delta\rho_{\min} = -0.19 \text{ e } \text{Å}^{-3}$
325 parameters	
All H-atom parameters refined	

**Table 1**

Selected geometric parameters (Å, °).

S1–C5	1.714 (3)	N3–C2	1.305 (3)
S1–C2	1.733 (2)	N3–C4	1.374 (3)
N–C2	1.394 (3)	C4–C5	1.341 (4)
N'–N	1.289 (2)	C10–C17	1.402 (3)
N'–C10	1.367 (3)	C10–C11	1.444 (3)
N1–C17	1.360 (3)	C11–C16	1.403 (3)
C5–S1–C2	88.19 (13)	C5–C4–N3	115.6 (2)
N'–N–C2	111.23 (19)	C4–C5–S1	111.0 (2)
N–N'–C10	114.4 (2)	N'–C10–C17	122.1 (2)
C17–N1–C16	110.93 (19)	N'–C10–C11	130.0 (2)
C2–N3–C4	109.96 (19)	C17–C10–C11	107.9 (2)
N3–C2–N	121.4 (2)	N1–C16–C11	107.7 (2)
N3–C2–S1	115.24 (17)	N1–C17–C10	107.4 (2)
N–C2–S1	123.32 (16)		
C5–S1–C2–N3	0.2 (2)	N3–C4–C5–S1	−0.5 (3)
C2–S1–C5–C4	0.2 (2)	C11–C10–C17–N1	1.1 (3)
C16–N1–C17–C10	−0.8 (3)	C17–C10–C11–C16	−0.9 (3)
C2–N3–C4–C5	0.7 (3)	C10–C11–C16–N1	0.4 (3)
C4–N3–C2–S1	−0.5 (3)	C11–C16–N1–C17	0.2 (3)

H atoms were located in a difference synthesis and refined isotropically. Refined values of bond lengths and  $U_{\text{iso}}$  parameters are:

N—H = 0.92 (2) Å and  $U_{\text{iso}}(\text{H}) = 0.067$  (8) Å<sup>2</sup>, and C—H = 0.90 (3)–1.07 (5) Å and  $U_{\text{iso}}(\text{H}) = 0.068$  (7)–0.151 (18) Å<sup>2</sup>.

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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