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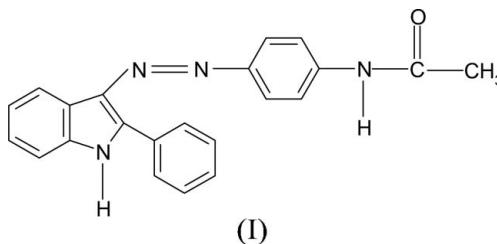
Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(C-C) = 0.006$ Å
 R factor = 0.077
 wR factor = 0.156
Data-to-parameter ratio = 10.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*N*-{4-[(2-Phenyl-1*H*-indol-3-yl)diazenyl]phenyl}-acetamide

In the title molecule, $C_{22}H_{18}N_4O$, all of the rings and the indole system are individually planar. In the crystal structure, molecules are linked by intermolecular $N-H \cdots O$ hydrogen bonds, forming infinite chains running along the [001] direction.

Comment

Azo dyes have wide applicability as optical materials and their structures have also attracted considerable attention (Biswas & Umaphathy, 2000; Willner & Rubin, 1996). Indole and its derivatives form a class of toxic recalcitrant *N*-heterocyclic compounds that are considered as pollutants (Florin *et al.*, 1980; Ishiguro & Sugawara, 1978), and aryl azoindoles give yellow or orange 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 (Bruni *et al.*, 1995).



The molecular structure of the title compound, (I), is shown in Fig. 1. It consists of acetamidophenyldiazenyl and phenylindole units. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987).

An examination of the deviations from the least-squares planes through individual rings shows that all of the rings are planar. The indole ring system is planar, with a puckering amplitude $Q_T = 0.035$ (4) Å (Cremer & Pople, 1975) and the dihedral angle between rings *A* and *B* is 1.63 (14)° (rings are defined in Fig. 1). In the closely related compound 3-(4-chlorophenyldiazenyl)-1-methyl-2-phenyl-1*H*-indole, the observed *A/B* dihedral angles were 1.56 (11) and 0.77 (12)° ($Z' = 2$; Seferoğlu *et al.*, 2006). The orientations of the phenyl and benzene rings *C* and *D* with respect to the indole ring system may be described by the dihedral angles 11.64 (11) and 14.36 (10)°, respectively. The phenyldiazenyl unit is not planar, with a puckering amplitude $Q_T = 0.106$ (3) Å.

As can be seen from the packing diagram (Fig. 2), the $N-H \cdots O$ intermolecular hydrogen bonds [$H1 \cdots O1^i = 2.03$ (4) Å, $N1 \cdots O1^i = 2.889$ (4) Å and $N1-H1 \cdots O1^i =$

Received 11 July 2006
Accepted 20 July 2006

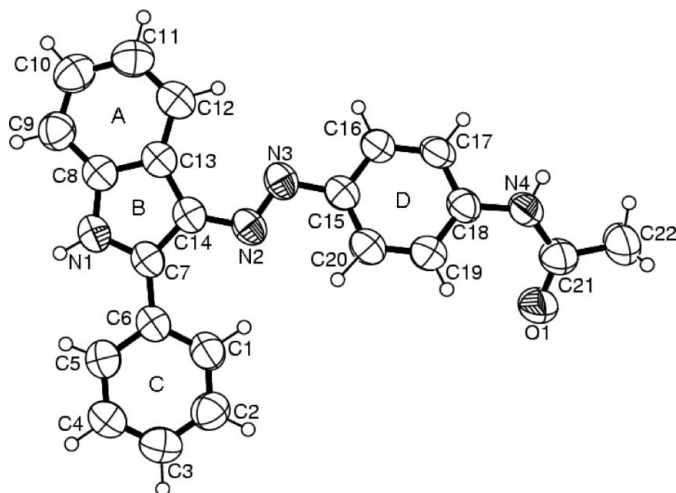


Figure 1
The molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

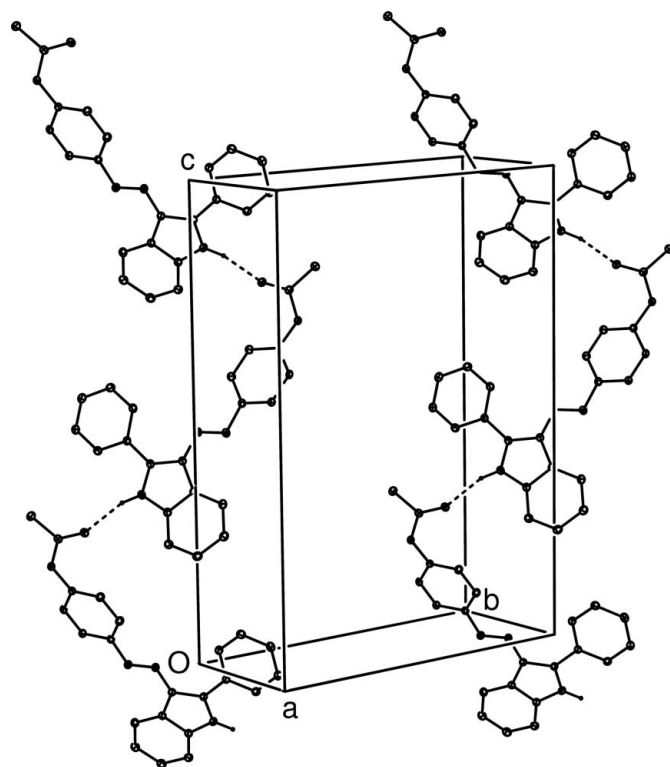


Figure 2
Partial packing diagram of (I), showing hydrogen bonds as dashed lines. Two parallel chains are displayed, related by a translation of (I) along the [010] axis. H atoms not involved in hydrogen bonds have been omitted.

160 (4)°; symmetry code: (i) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$ link the molecules, to form infinite chains along the [001] direction. Dipole–dipole and van der Waals interactions are effective in the molecular packing.

Experimental

4-Aminoacetanilide (0.30 g, 2.0 mmol) was dissolved in HCl (1.5 ml) and water (4.0 ml). The solution was cooled in an ice–salt bath and a

cold solution of NaNO₂ (0.15 g, 2.0 mmol) in water (3.0 ml) was added dropwise with stirring. The resulting diazonium salt was also cooled in an ice–salt bath and then added dropwise with stirring to 2-phenylindole (0.41 g, 2.0 mmol) in an acetic acid/proprionic acid mixture (8 ml, 2:1). The solution was stirred at 273–278 K for 1 h and the pH of the reaction mixture was maintained at 4–6 by the simultaneous addition of a saturated sodium acetate solution (15–20 ml). The mixture was stirred for a further 1 h. The resulting solid was filtered off, washed with cold water and crystallized from ethanol (yield 0.64 g, 91%; m.p. 452–455 K).

Crystal data

C ₂₂ H ₁₈ N ₄ O	Z = 4
<i>M_r</i> = 354.41	<i>D_x</i> = 1.293 Mg m ^{−3}
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Mo <i>K</i> α radiation
<i>a</i> = 5.996 (5) Å	<i>μ</i> = 0.08 mm ^{−1}
<i>b</i> = 14.786 (5) Å	<i>T</i> = 296 (2) K
<i>c</i> = 20.531 (5) Å	Block, red
<i>V</i> = 1820.2 (17) Å ³	0.35 × 0.25 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID-S diffractometer	3181 independent reflections
<i>ω</i> scans	1973 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: none	<i>R</i> _{int} = 0.094
52939 measured reflections	<i>θ</i> _{max} = 30.6°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.2413P]$
$R[F^2 > 2\sigma(F^2)] = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.156$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.17	$\Delta\rho_{max} = 0.11 \text{ e } \text{Å}^{-3}$
3181 reflections	$\Delta\rho_{min} = -0.17 \text{ e } \text{Å}^{-3}$
305 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Methyl H atoms were positioned geometrically with C–H = 0.96 Å, and constrained to ride on their parent atoms with *U*_{iso}(H) = 1.5*U*_{eq}(C). The remaining H atoms were located in a difference map and refined isotropically. Ranges for bond lengths and *U*_{iso} parameters: N–H = 0.83 (4) and 0.89 (4) Å and *U*_{iso}(H) = 0.075 (13) and 0.078 (12) Å²; C–H = 0.89 (4)–1.05 (5) Å and *U*_{iso}(H) = 0.058 (10)–0.117 (15) Å². In the absence of significant anomalous scatterers, 2381 measured Friedel pairs were merged.

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).

The authors are indebted to the Department of Chemistry, Atatürk University, Erzurum, Turkey, for the use of X-ray diffractometer purchased under grant No. 2003/219 of the University Research Fund.

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