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# 2-(2-Acetamido-5-methylbenzoyl)-1*H*-indole

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.005 Å
*R* factor = 0.077
*wR* factor = 0.216
Data-to-parameter ratio = 15.5

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



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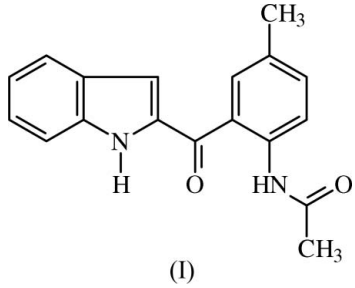
In the title compound, $C_{18}H_{16}N_2O_2$, the dihedral angle between the indole ring system and the benzoyl benzene ring is 31.5 (1)°. In the crystalline state, symmetry-related molecules are linked *via* N—H···O and C—H···O hydrogen bonds, and π–π interactions to form a layer structure parallel to the *bc* plane.

## Comment

Many indole derivatives, both natural and synthetic, show important biological activities such as antibacterial, antifungal (Wang & Ng, 2002; Singh *et al.*, 2000; Tsotinis *et al.*, 1997; Quetin-Leclercq *et al.*, 1995), antitumour (Andreani *et al.*, 2001; Bradlow *et al.*, 1999; Cirrincione *et al.*, 1999; Tiwari *et al.*, 1994; Dashwood *et al.*, 1994) and antimicrobial activities (Piscopo, Diurno, Mazzoni & Ciaccio, 1990; Piscopo, Diurno, Mazzoni, Ciaccio & Veneruso, 1990). Some indole derivatives are used as neuroprotectants (Stolc, 1999). Recently, we have reported the crystal structures of some phenylsulfonylindole derivatives (Ravishankar *et al.*, 2003*a*,*b*, 2005*a*,*b*). We report here the crystal structure of the title compound, (I).




(I)

The indole ring system in (I) (Fig. 1) is planar, with a maximum deviation of 0.017 (3) Å for atom C2. The N—C$sp^2$ bond lengths, *viz*. N1—C1 [1.377 (4) Å] and N1—C8 [1.369 (4) Å], are comparable with the mean value of 1.355 (14) Å reported for N atoms with planar configurations (Allen *et al.*, 1987). These values are significantly longer in phenylsulfonyl indole derivatives (Ravishankar *et al.*, 2003*a*,*b*, 2005*a*,*b*). The conformation of the attachment of the benzoyl substituent to the indole ring system is described by the C2—C1—C9—C10 torsion angle of 7.2 (5)°; the C1—C9—C10—C11 torsion angle of 28.2 (4)° shows how the C10–C15 ring is oriented. The mean plane through the acetylamide group makes a dihedral angle of 15.1 (2)° with the C10–C15 ring. This orientation is influenced by the intramolecular N2—H2*N*···O1 and C14—H14···O2 hydrogen bonds involving carbonyl atoms O1 and O2 (Table 1). Each of these interactions generates rings of graph-set motif *S*(6) (Bernstein *et al.*, 1995; Etter, 1990).

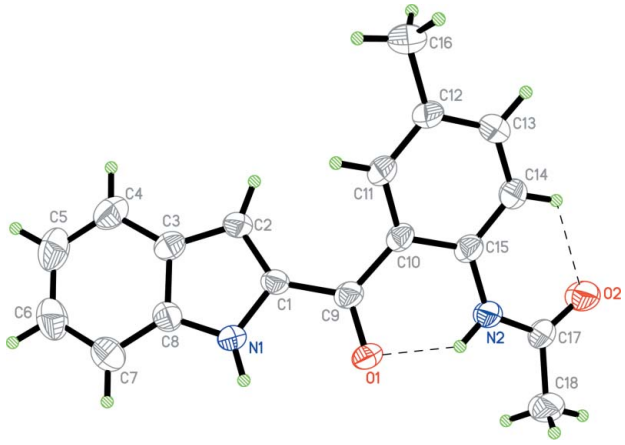



**Figure 1**
The structure of (I), showing the atom-numbering scheme and intramolecular hydrogen bonds (as dashed lines). Displacement ellipsoids are drawn at the 50% probability level.

The packing of the molecules, shown in Fig. 2, reveals that symmetry-related molecules are linked *via* N—H···O and C—H···O hydrogen bonds to form a layer structure parallel to the *bc* plane. Within a layer, the C10–C15 benzene rings at the symmetry positions ($x$, $y$, $z$) and ($\frac{1}{2} - x$, $\frac{1}{2} - y$, $1 - z$) are stacked with their centroids 3.688 (2) Å apart, indicating the presence of $\pi$–$\pi$ interactions.

## Experimental

A solution of 1-phenylsulfonyl-2-(2-acetamido-5-methylbenzoyl)indole (5 mmol) in methanol (100 mmol) was refluxed with 10% sodium hydroxide solution (10 ml) for 30 min. The solution was poured over ice and the precipitated solid was filtered off, washed with water, dried over anhydrous calcium chloride and purified by crystallization. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

*Crystal data*

$C_{18}H_{16}N_2O_2$
$M_r$ = 292.33
Monoclinic, $C2/c$
$a$ = 21.8765 (12) Å
$b$ = 14.3774 (8) Å
$c$ = 11.7603 (7) Å
$\beta$ = 107.050 (1)°
$V$ = 3536.4 (3) Å$^3$
$Z$ = 8
$D_x$ = 1.098 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 2509 reflections
$\theta$ = 2.3–28.3°
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 (2) K
Block, colourless
0.36 × 0.20 × 0.10 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
8691 measured reflections
3108 independent reflections
2067 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.041
$\theta_{max}$ = 25.0°
$h = -26 \rightarrow 26$
$k = -17 \rightarrow 9$
$l = -13 \rightarrow 13$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.216
$S$ = 1.09
3108 reflections
201 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1234P)^2 + 0.2753P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

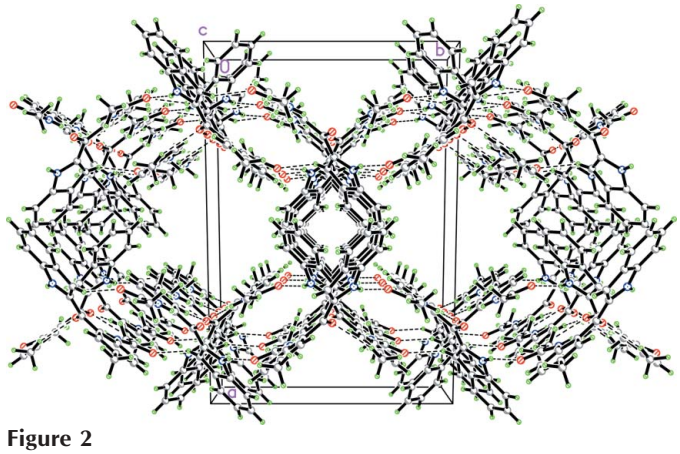



**Figure 2**
A view of the N—H···O and C—H···O hydrogen-bonded layers in (I). Hydrogen bonds are shown as dashed lines.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C9 | 1.235 (3) | N2—C17 | 1.355 (4) |
| O2—C17 | 1.225 (4) | N2—C15 | 1.408 (4) |
| C17—N2—C15 | 129.9 (3) | C11—C12—C16 | 122.2 (3) |
| O1—C9—C1 | 116.9 (3) | O2—C17—N2 | 123.8 (3) |
| C1—C9—C10 | 122.0 (3) | N2—C17—C18 | 114.1 (3) |
| C11—C12—C13 | 116.8 (3) | | |
| C17—N2—C15—C14 | −11.9 (5) | C15—N2—C17—C18 | 175.7 (3) |
| C15—N2—C17—O2 | −3.5 (6) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$N$···O2$^{i}$ | 0.86 | 2.14 | 2.954 (3) | 159 |
| N2—H2$N$···O1 | 0.86 | 1.94 | 2.641 (4) | 138 |
| C2—H2···O2$^{ii}$ | 0.93 | 2.49 | 3.363 (4) | 156 |
| C11—H11···O1$^{iii}$ | 0.93 | 2.53 | 3.397 (4) | 154 |
| C14—H14···O2 | 0.93 | 2.29 | 2.901 (4) | 122 |
| C16—H16$A$···O1$^{iii}$ | 0.96 | 2.53 | 3.442 (5) | 160 |

Symmetry codes: (i) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $x, -y, z + \frac{1}{2}$.

H atoms were positioned geometrically and treated as riding on their parent atoms, with an N—H distance of 0.86 Å, C—H distances of 0.93 (aromatic) and 0.96 Å (methyl), and with $U_{iso}$(H) = 1.5$U_{eq}$(methyl C) and 1.2$U_{eq}$(other atoms). A rotating group model was used for the methyl groups. The refinement converged to an $R$ value of 0.095 ($wR$ = 0.248) and the difference map showed three highest peaks of 0.81, 0.51 and 0.48 e Å$^{-3}$. Refinement based on a disordered solvent model led to unstable refinement with very high displacement parameters. A search for solvent-accessible voids in the crystal structure using *PLATON* (Spek, 2003) showed a potential solvent volume of 746.1 Å$^3$ and subsequent application of *SQUEEZE* procedures (Sluis & Spek, 1990) showed only one relevant void with a solvent-accessible volume of 373 Å$^3$. However, this procedure showed no electrons in the void as the weak positive and negative density excursions in the void region sum close to zero. This indicates that the crystal lost nearly all of its solvent of crystallization by the time it was used for data collection, without collapse of the structure.

Further refinement of the structure with data obtained from the above procedure converged to an $R$ value of 0.077 ($wR$ = 0.216). The limited improvement of $R$ values indicates that there are still residual problems associated with the data. As no more crystals were available, we were unable to repeat the experiment at a lower temperature, on a crystal freshly chosen from the mother liquor.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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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.

Andreani, A., Granaiola, M., Leoni, A., Locatelli, A., Morigi, R., Rambaldi, M., Giorgi, G., Salvini, L. & Garaliene, V. (2001). *Anticancer Drug Des.* **16**, 167–174.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bradlow, H. L., Sepkovic, D. W., Telang, N. T. & Osborne, M. P. (1999). *Ann. N. Y. Acad. Sci.* **889**, 204–213.

Cirrincione, G., Almerico, A. M., Barraja, P., Diana, P., Lauria, A., Passannanti, A., Musiu, C., Pani, A., Murtas, P., Minnei, C., Marongiu, M. E. & La Colla. P. (1999). *J. Med. Chem.* **42**, 2561–2568.

Dashwood, R. H., Fong, A. T., Arbogast, D. N., Bjeldanes, L. F., Hendricks, J. D. & Bailey, G. S. (1994). *Cancer Res.* **54**, 3617–3619.

Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.

Piscopo, E., Diurno, M. V., Mazzoni, O., Ciaccio, A. M. (1990). *Boll. Soc. Ital. Biol. Sper.* **66**, 1181–1186.

Piscopo, E., Diurno, M. V., Mazzoni, O., Ciaccio, A. M. & Veneruso, G. (1990). *Boll. Soc. Ital. Biol. Sper.* **66**, 1187–1191.

Quetin-Leclercq, J., Favel, A., Balansard, G., Regli, P. & Angenot, L. (1995). *Planta Med.* **61**, 475–477.

Ravishankar, T., Chinnakali, K., Arumugam, N. & Srinivasan, P. C., Usman, A. & Fun, H.-K. (2003*a*). *Acta Cryst.* C**59**, o137–o140.

Ravishankar, T., Chinnakali, K., Arumugam, N. & Srinivasan, P. C., Usman, A. & Fun, H.-K. (2003*b*). *Acta Cryst.* E**59**, o1903–o1906.

Ravishankar, T., Chinnakali, K., Arumugam, N. & Srinivasan, P. C., Usman, A. & Fun, H.-K. (2005*a*). *Acta Cryst.* E**61**, o998–o1000.

Ravishankar, T., Chinnakali, K., Arumugam, N. & Srinivasan, P. C., Usman, A. & Fun, H.-K. (2005*b*). *Acta Cryst.* E**61**, o1184–o1186.

Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Versions 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Singh, U. P., Sarma, B. K., Mishra, P. K. & Ray, A. B. (2000). *Folia Microbiol.* **45**, 173–176.

Sluis, P. van der & Spek, A. L. (1990). *Acta Cryst.* A**46**, 194–201.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stolc, S. (1999). *Life Sci.* **65**, 1943–1950.

Tiwari, R. K., Guo, L., Bradlow, H. L., Telang, N. T. & Osborne, M. P. (1994). *J. Natl Cancer Inst.* **86**, 126–131.

Tsotinis, A., Varvaresou, A., Calogeropoulou, T., Siatra-Papastaikoudi, T. & Tiligada, A. (1997). *Arzneim.-Forsch.* **47**, 307–310.

Wang, H. X. & Ng, T. B. (2002). *Comput. Biochem. Physiol. C. Toxicol. Pharmacol.* **132**, 261–268.