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(3*E*)-3-[(4-Hexylphenyl)imino]1*H*-indol-2(3*H*)-one

Sema Öztürk,[a] Mehmet Akkurt,[a]* Mahmure Üstün Özgür,[b] Ayşe Erçağ[c] and Frank W. Heinemann[d]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, Yıldız Technical University, 34210 Davutpaşa Campus, İstanbul, Turkey, [c]Chemistry Department, Engineering Faculty, İstanbul University, 34850 İstanbul, Turkey, and [d]Institut für Anorganische Chemie, Universität Erlangen-Nürnberg, Egerlandstrasse 1, D-91058 Erlangen, Germany

Correspondence e-mail: akkurt@erciyes.edu.tr

Key indicators

Single-crystal X-ray study
T = 294 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.132
Data-to-parameter ratio = 12.1

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

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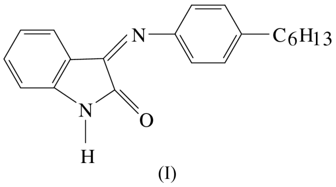
In the title compound, $C_{20}H_{22}N_2O$, the indole and hexylphenyl groups are connected by a C—N bond of 1.431 (2) Å. The molecule is not planar.

Comment

The synthetic versatility of isatin has lead to the extensive use of this compound in organic synthesis. It has stemmed from the interest in the biological and pharmacological properties of the derivatives (Silva *et al.*, 2001; Pardasani *et al.*, 1999). Isatin and derivatives of isatin have been used as reagents in the dye industry also. Although, it was firstl synthesized nearly a hundred years ago, recently it was discovered in mamalian tissues and in body fluids. It was observed that monoamine oxidase was inhibited by isatin and derivatives in *in vitro* studies.



(I)

Schiff bases of isatin have been reported to posses antibacterial (Sarangapani & Reddy, 1994; Varma & Nobles, 1975), antifungal (Pandeya *et al.*, 1999), antiviral (Singh *et al.*, 1983), anti-HIV (Pandeya *et al.*, 2000), antiprotozoal (Varma & Khan, 1977) and antihelminthic (Sarciron *et al.*, 1993) activities.

Our interest in this category of ligands is justified by their already proven medical and biological implications (Popp & Pajouhesh, 1982).

In this study, our purpose was to synthesize and characterize of a new Schiff base ligand, shown in Fig. 1. We report analytical and X-ray crystallographic studies of the title compound, (I).

The C1—C2 bond length of 1.529 (3) Å is longer than the standard value for this type of bond (Allen *et al.*, 1987). The C2—N2—C9 angle is 119.2 (2)°. In the hexyl group, the average value of the C—C—C bond angle is 115.2 (3)° and this group shows a chair conformation. The indol group is planar [maximum displacements are −0.025 (2) Å for C3 and 0.021 (2) Å for C7] and forms a dihedral angle of 88.01 (5)° with the benzene ring plane.

To determine the structural and electronic parameters of this isatin derivative, conformational analyses of the compound and its quantum mechanic optimizations was performed.

The quantum-chemical calculation, using the *PM*3 method, showed that the charges at atoms N1, N2 and O1 are 0.0374, 0.0547 and −0.2933 e, respectively. The final heat of formation

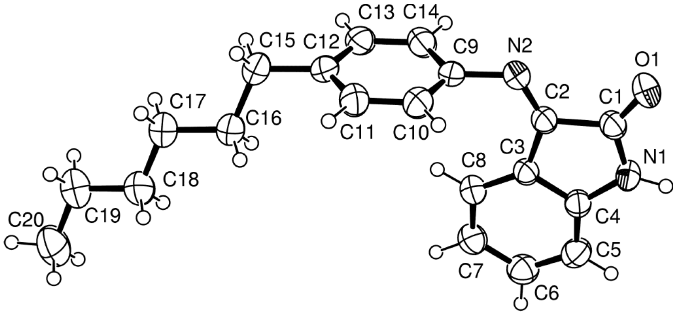


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

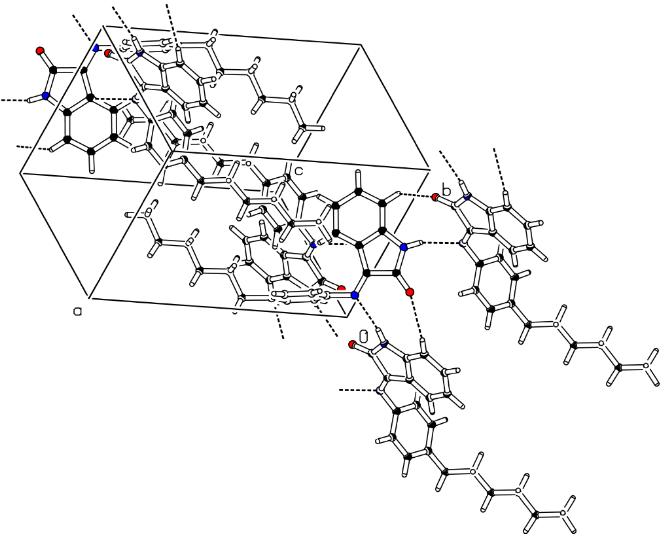


Figure 2
A view of the crystal packing and the short hydrogen-bond contacts.

of the title compound is 5.53 kcal and its total energy is −3326.84 eV. The values of the HOMO and LUMO energies are −9.02078 and −1.05314 eV, respectively. The calculated molecule dipole moment is 4.467 Debye (1 D = 3.33564 × 10^{-30} Cm).

The crystal structure is stabilized by N—H···N and C—H···O hydrogen bonds (Fig. 2), details of which are given in Table 2.

Experimental

All chemicals used were of analytical purity, namely isatin, 4-butylaniline and phosphorus pentoxide. Ethanol and methanol were distilled and dried prior to use, according to methods reported in the literature. The carbon, hydrogen, and nitrogen microanalyses were carried out on a Carlo Erba 1106 elemental analyser. The Fourier transform IR spectra (FT–IR) were recorded with a Unicam Mattson 1000 FT–IR spectrophotometer (in the range 400–4000 cm^{-1}), using the KBr pellet (1 mg per 100 mg) technique. The electronic spectra were recorded on a Philips PU 8700 spectrophotometer (in the region 190–700 nm) using 10^{-3} mol dm^{-3} solutions in DMF and ethanol. ^{1}H NMR data were recorded on a Bruker AC 200 L. The free ligand, Schiff base, was synthesized by the usual condensation reaction. The preparation of the Schiff base was carried out as follows: an ethanol solution of isatin (0.001 mol per 25 ml) was added to an ethanol solution of 4-hexylaniline (0.001 mol per 25 ml) and refluxed for 4 h on a water bath. After solution concentration, the precipitate was separated by suction filtration, washed with ethanol and dried over P_2O_5 *in vacuo*. The orange product was recrystallized from methanol: yield 75–80%, m.p. 456 K. Analysis calculated: C 78.30, H 6.98, N 9.20%; found: C 78.43, H 7.19, N 9.15%. λ_{max} (DMF, nm): 328 440 The IR spectral data of the title compound [frequency (cm^{-1}), KBr]: 3182 (NH), 2927–2851 (Ar-H3), 1753 (C=O), 1651 (C=N), 1600 (C=C). ^{1}H NMR (D_2O, *d*, p.p.m.) spectral data of the title compound: 0.85 (*m*, 3H, CH_3), 1.23 (*s*, 6H, $-CH_2-$), 1.62 (*m*, 2H, $-CH_2-$), 2.6 (*m*, 2H, $-CH_2$), 6.42–6.65 (two symmetric *m*, 2H, Ar-H), 7.00–7.40 (two *m*, 4H, Ar-H), 10.98 (*s*, 1H, NH).

Crystal data

$C_{20}H_{22}N_2O$
$M_r = 306.40$
Monoclinic, $P2_1/c$
$a = 17.1506\ (2)$ Å
$b = 9.6464\ (2)$ Å
$c = 10.4167\ (2)$ Å
$\beta = 99.693\ (2)^\circ$
$V = 1698.75\ (5)$ Å^3
$Z = 4$

$D_x = 1.198$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 155 reflections
$\theta = 6.0$–20.0°
$\mu = 0.07\ \text{mm}^{-1}$
$T = 294\ (2)$ K
Plate, orange
0.52 × 0.31 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
30666 measured reflections
3326 independent reflections
2121 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.089$
$\theta_{\text{max}} = 26.0^\circ$
$h = -21 \rightarrow 21$
$k = -11 \rightarrow 11$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.132$
$S = 1.05$
3326 reflections
274 parameters
Only coordinates of H atoms refined

$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.3745P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}} = 0.14$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.213 (3)	N2—C2	1.280 (2)
N1—C1	1.357 (3)	N2—C9	1.431 (2)
N1—C4	1.408 (3)		
C1—N1—C4	111.63 (16)	N1—C4—C5	128.40 (17)
C2—N2—C9	119.23 (16)	N2—C9—C14	122.24 (17)
N1—C1—C2	105.88 (16)	N2—C9—C10	118.26 (16)
O1—C1—C2	125.99 (18)	C15—C16—C17	114.86 (19)
O1—C1—N1	128.10 (19)	C16—C17—C18	114.6 (2)
N2—C2—C3	135.16 (17)	C17—C18—C19	115.9 (2)
N2—C2—C1	118.78 (17)	C18—C19—C20	115.4 (3)
N1—C4—C3	109.96 (16)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N2$^{\text{i}}$	0.88 (3)	2.22 (3)	3.069 (2)	163 (2)
C5—H5···O1$^{\text{i}}$	0.96 (3)	2.55 (2)	3.265 (3)	131.4 (16)

Symmetry code: (i) $-x, \frac{1}{2}+y, -\frac{1}{2}-z$.

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All H atoms were positioned geometrically and their coordinates refined.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg, 1998); data reduction: *EVALCCD*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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