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Key indicators

Single-crystal X-ray study

T = 90 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.065

wR factor = 0.176

Data-to-parameter ratio = 13.3

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

1-(Azepan-1-yl)-2-(1H-indol-3-yl)ethane-1,2-dione

In the crystal structure of the title compound, $\text{C}_{16}\text{H}_{18}\text{N}_2\text{O}_2$, the indole moiety is nearly planar. The two carbonyl groups are almost perpendicular, with a torsion angle of $-105.7(4)^\circ$, and the single C—C bond linking the two carbonyl groups is $1.515(5) \text{ \AA}$ in length. The molecules are linked together by $\text{N}-\text{H}\cdots\text{O}$ intermolecular hydrogen bonds.

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Comment

The title compound, (I), is a synthetic intermediate in the preparation of rigid analogues of tryptamine, and is prepared by treating indole with oxalyl chloride, followed by reaction with azepane. The product was characterized by spectroscopic analysis. Its X-ray structure determination was carried out in order to study the conformation of the molecule, and the results are presented here.

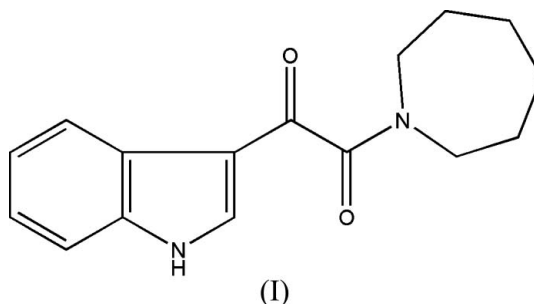


Fig. 1 illustrates the molecular structure of (I) with the atom-numbering scheme. Selected geometric parameters are presented in Table 1. The indole moiety is nearly planar, with a standard deviation of 0.0141 \AA . The $\text{C}1-\text{C}2-\text{C}9-\text{O}1$ torsion angle of $177.8(3)^\circ$ suggests that the indole moiety and the $\text{C}9=\text{O}1$ bond are nearly coplanar, which facilitates extended conjugation starting from atom O1 through the indole moiety. This is further supported by the fact that the $\text{C}2-\text{C}9$ bond distance of $1.427(5) \text{ \AA}$ is somewhat shorter than the standard value for a $\text{C}_{\text{ar}}-\text{C}_{\text{sp}^2}$ single bond [$1.470(15) \text{ \AA}$; Allen *et al.*, 1987].

The two carbonyl groups are in a *trans* orientation, with a $\text{O}1-\text{C}9-\text{C}10-\text{O}2$ torsion angle of $-105.7(4)^\circ$. It is important to note that, in the structurally related compound 1-(1H-indol-3-ylcarbonyl)-N-(4-methoxybenzyl)formamide (Sonar *et al.*, 2006), the $\text{O}1-\text{C}9-\text{C}10-\text{O}2$ torsion angle is $144.8(3)^\circ$. This difference is mainly because of the presence of the secondary glyoxylamide group, which can undergo hydrogen bonding, leading to restricted rotation of the $\text{C}9-\text{C}10$ bond.

From the entries in the Cambridge Structural Database (Allen *et al.*, 1991) of many previously reported X-ray crystal

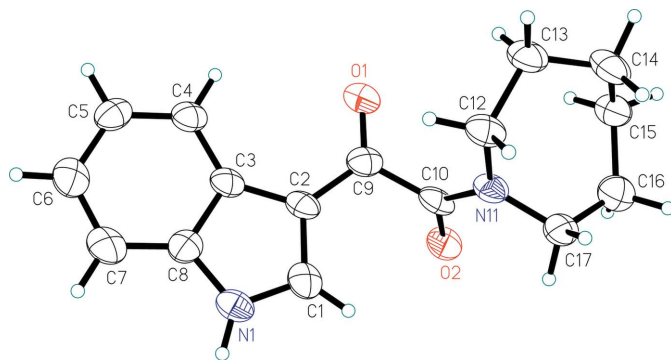


Figure 1

A view of the asymmetric unit of (I); displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

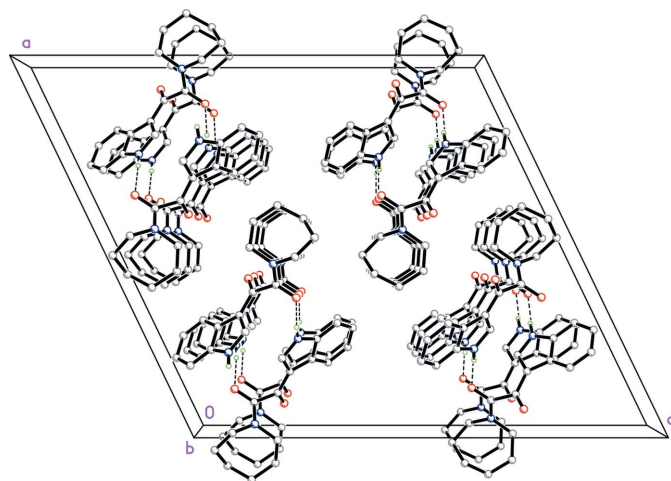


Figure 2

A packing diagram of (I), viewed down the *b* axis, showing the hydrogen-bonding interactions (dashed lines). H atoms, except those involved in hydrogen bonding, have been omitted for clarity.

structures of molecules containing the glyoxylamide function, it is observed that primary and secondary glyoxylamides have torsion angles ranging from 162.4 to 179.4°, while tertiary glyoxylamides, which lack the hydrogen-bonding ability, have torsion angles ranging from 79.0 to 101.1° (Black *et al.*, 1996). In the case of the title compound, absence of a similar kind of hydrogen bonding results in maximum deviation. Because of the above, the C9—C10 bond length of 1.515 (5) Å is longer than expected, the characteristic value for a Csp^2-Csp^2 bond being 1.50 Å (Zukerman-Schpector *et al.*, 1994). The C10—N11 bond length [1.337 (4) Å] and the bond angles around atom N11 suggest that the lone pair of electrons on N11 undergoes delocalization, affording double-bond character to the C10—N11 bond and forcing the atoms O2/C10/N11/C12 into an almost planar conformation.

The packing of compound (I), as viewed down the *b* axis, is illustrated in Fig. 2. The molecules are linked by intermolecular N—H···O hydrogen bonds, details of which are given in Table 2.

Experimental

To a solution of indole (1 g, 8.5 mmol) in dry diethyl ether (10 ml), oxalyl chloride (1.45 ml, 16.9 mmol) in dry diethyl ether (5 ml) was added dropwise with rapid stirring, maintaining the temperature below 283 K. After addition was complete, the mixture was stirred for 45 min. Filtration afforded 3-indoleglyoxalyl chloride as a yellow solid. 3-Indoleglyoxalyl chloride (1.245 g, 6 mmol) was suspended in dry diethyl ether (30 ml) and stirred at 273 K, and azepane (1.19 g, 12 mmol) in dry diethyl ether (15 ml) was added dropwise. After complete addition, the mixture was stirred for 3 h. The resulting solid was washed with water and dried. The product was obtained as colourless crystals by recrystallization from ethanol.

Crystal data

$C_{16}H_{18}N_2O_2$	$Z = 8$
$M_r = 270.32$	$D_x = 1.316 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 22.6214 (5) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 5.2942 (12) \text{ \AA}$	$T = 90.0 (2) \text{ K}$
$c = 25.309 (3) \text{ \AA}$	Rod, colourless
$\beta = 115.768 (4)^\circ$	$0.25 \times 0.12 \times 0.03 \text{ mm}$
$V = 2729.7 (7) \text{ \AA}^3$	

Data collection

Nonius KappaCCD area-detector diffractometer	8245 measured reflections
ω scans	2410 independent reflections
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)	1123 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.978$, $T_{\max} = 0.998$	$R_{\text{int}} = 0.155$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.065$	$w = 1/[\sigma^2(F_o^2) + (0.0789P)^2]$
$wR(F^2) = 0.176$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2410 reflections	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
181 parameters	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N1—C1	1.337 (4)	C9—C10	1.515 (5)
O1—C9	1.230 (4)	C10—N11	1.337 (4)
O2—C10	1.250 (4)	N11—C17	1.449 (4)
C1—C2	1.394 (5)	N11—C12	1.463 (4)
C1—C2—C9	125.2 (4)	C2—C9—C10	116.1 (3)
O1—C9—C2	125.1 (4)	O2—C10—N11	121.9 (3)
O1—C9—C10	118.7 (3)	O2—C10—C9	119.2 (3)
C1—C2—C9—O1	177.8 (3)	O1—C9—C10—O2	−105.7 (4)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1N···O2 ⁱ	0.88	1.93	2.800 (4)	171

Symmetry code: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

All H atoms were located in difference Fourier syntheses and were subsequently positioned geometrically and refined with a riding model. Bond distances for H atoms were set to 0.95 (C_{ar}–H), 0.99 (C_{sec}–H) and 0.88 Å (N–H), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the parent atom.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

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