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3-[2-(*N*,*N*-Diethylamino)glyoxyloyl]-1*H*-indol-4-yl acetate monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.128
Data-to-parameter ratio = 16.7

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

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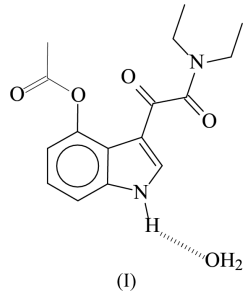
In the crystal structure of the title compound, $C_{16}H_{18}N_2O_4\cdot H_2O$, the substituted indole molecule forms a hydrogen bond to the water molecule. The water molecule in turn interacts with the carbonyl O atom of the acetoxy unit of an adjacent indole molecule and also with the O atom of the acetamide unit of another indole molecule, giving rise to a hydrogen-bonded helical chain structure that runs along the *b* axis of the monoclinic unit cell.

Comment

3-[2-(*N*,*N*-Diethylamino)glyoxyloyl]-1*H*-indol-4-yl acetate is a reagent used in the synthesis of 3-[2-(diethylamino)ethyl]-4-indolol (4-HO-DET), a compound belonging to the 3-(2-aminoethyl)indole structural class of psychoactive drugs (Shulgin & Shulgin, 1997). 4-HO-DET will be converted into other compounds, in an extension of *in vivo* and *in vitro* studies of the structure–activity relationships of such compounds (An *et al.*, 2001; Bu *et al.*, 2002). 3-[2-(*N*,*N*-Diethylamino)glyoxyloyl]-1*H*-indol-4-yl acetate exists as the monohydrate, (I) (Fig. 1), the crystal structure of which is presented here.



(I)

The ketonic fragment of (I) (C9/C11/C12/O3) is approximately coplanar with the indole system [dihedral angle 17.5 (1)°], but almost perpendicular to the amide fragment [C11/C12/N2/O4; dihedral angle 82.8 (1)°]. The large twist is probably necessary to avoid crowding, but it appears to lengthen the C11—C12 bond somewhat.

The indole molecule forms a hydrogen bond to the water molecule, which in turn interacts with the carbonyl O atom of the acetoxy unit of an adjacent indole molecule and the O atom of the acetamide unit of another indole molecule (Table 2). These hydrogen bonds give rise to a helical chain that runs along the *b* axis of the monoclinic unit cell.

Experimental

The title compound was synthesized from 4-acetoxyindole (synthesized from 4-hydroxyindole and acetic anhydride), oxalyl chloride

and diethylamine, using the procedure of Shulgin & Shulgin (1997), and was obtained as colourless crystals by recrystallization from ether. The literature records the compound as being anhydrous.

Crystal data

$C_{16}H_{18}N_2O_4\cdot H_2O$	D_x = 1.254 Mg m^{-3}
M_r = 320.34	Mo $K\alpha$ radiation
Monoclinic, $P2_1/a$	Cell parameters from 931 reflections
a = 12.646 (2) Å	θ = 2.5–26.8°
b = 10.518 (1) Å	μ = 0.09 mm^{-1}
c = 12.904 (2) Å	T = 293 (2) K
β = 98.703 (2)°	Block, colourless
V = 1696.7 (4) Å^3	0.50 × 0.38 × 0.36 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2880 reflections with $I > 2\sigma(I)$
	R_{int} = 0.012
φ and ω scans	θ_{max} = 27.0°
Absorption correction: none	$h = -16 \rightarrow 16$
9454 measured reflections	$k = -11 \rightarrow 13$
3695 independent reflections	$l = -10 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 0.3054P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.128	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
3695 reflections	
221 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.353 (2)	C3—C8	1.399 (2)
O1—C3	1.402 (2)	C4—C5	1.393 (2)
O2—C2	1.199 (2)	C5—C6	1.370 (2)
O3—C11	1.225 (2)	C6—C7	1.392 (2)
O4—C12	1.236 (2)	C7—C8	1.410 (2)
N1—C7	1.379 (2)	C8—C9	1.449 (2)
N1—C10	1.339 (2)	C9—C10	1.383 (2)
N2—C12	1.327 (2)	C9—C11	1.439 (2)
N2—C15	1.467 (2)	C11—C12	1.526 (2)
N2—C13	1.467 (2)	C13—C14	1.503 (3)
C1—C2	1.487 (2)	C15—C16	1.480 (3)
C3—C4	1.372 (2)		
C2—O1—C3	117.0 (1)	C6—C7—C8	123.7 (1)
C7—N1—C10	109.4 (1)	C3—C8—C7	116.5 (1)
C12—N2—C13	122.9 (1)	C3—C8—C9	137.4 (1)
C12—N2—C15	119.5 (1)	C7—C8—C9	106.1 (1)
C13—N2—C15	117.6 (1)	C8—C9—C10	105.9 (1)
O1—C2—O2	122.8 (1)	C10—C9—C11	123.4 (1)
O1—C2—C1	111.6 (1)	C11—C9—C8	130.7 (1)
O2—C2—C1	125.6 (2)	N1—C10—C9	110.7 (1)
O1—C3—C4	117.7 (1)	O3—C11—C9	125.8 (1)
O1—C3—C8	121.7 (1)	O3—C11—C12	116.9 (1)
C4—C3—C8	120.5 (1)	C9—C11—C12	117.3 (1)
C3—C4—C5	121.1 (2)	O4—C12—N2	124.0 (1)
C4—C5—C6	121.0 (2)	O4—C12—C11	118.9 (1)
C5—C6—C7	117.2 (1)	N2—C12—C11	117.0 (1)
N1—C7—C6	128.4 (1)	N2—C13—C14	113.2 (2)
N1—C7—C8	108.0 (1)	N2—C15—C16	112.4 (2)

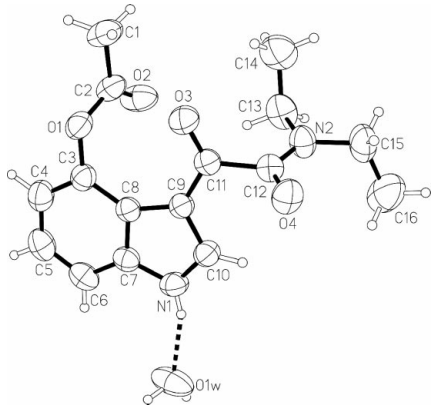


Figure 1
A view of the molecular structure of (I), showing the atom-numbering scheme and with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii. The dashed line indicates a hydrogen bond.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1W	0.86 (1)	1.91 (1)	2.743 (2)	161 (2)
O1W—H1W1···O2^{i}	0.85 (1)	1.99 (1)	2.841 (2)	175 (2)
O1W—H1W2···O4^{ii}	0.85 (1)	1.98 (1)	2.810 (2)	165 (2)

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

H atoms on C atoms were placed at calculated positions in a riding model approximation, with C—H distances of 0.93 for aromatic H atoms, 0.96 for methyl H atoms and 0.97 Å for methylene H atoms, and with U_{iso}(H) = 1.2U_{eq}(C). The amine and water H atoms were located and refined with N—H and O—H distance restraints of 0.85 (1) Å.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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