

The effect of hydrogen bonding on the conformations of 2-(1*H*-indol-3-yl)-2-oxoacetamide and 2-(1*H*-indol-3-yl)-*N,N*-dimethyl-2-oxoacetamide

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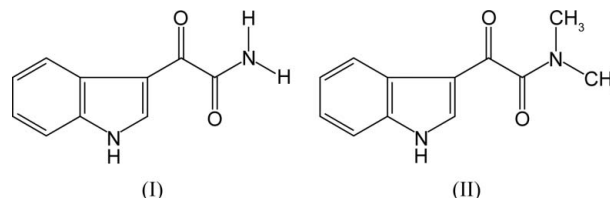
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In the title compounds, C₁₀H₈N₂O₂, (I), and C₁₂H₁₂N₂O₂, (II), the two carbonyl groups are oriented with torsion angles of $-149.3(3)$ and $-88.55(15)^\circ$, respectively. The single-bond distances linking the two carbonyl groups are 1.528(4) and 1.5298(17) Å, respectively. In (I), the molecules are linked by an elaborate system of N–H···O hydrogen bonds, which form adjacent *R*₂²(8) and *R*₂²(8) ring motifs to generate a ladder-like construct. Adjacent ladders are further linked by N–H···O hydrogen bonds to build a three-dimensional network. The hydrogen bonding in (II) is far simpler, consisting of helical chains of N–H···O-linked molecules that follow the 2₁ screw of the *b* axis. It is the presence of an elaborate hydrogen-bonding system in the crystal structure of (I) that leads to the different torsion angle for the orientation of the two adjacent carbonyl groups from that in (II).

Comment

The title compounds, 2-(1*H*-indol-3-yl)-2-oxoacetamide, (I), and 2-(1*H*-indol-3-yl)-*N,N*-dimethyl-2-oxoacetamide, (II), are synthetic intermediates in the preparation of tryptamine and *N,N*-dimethyltryptamine, and are prepared by treating indole with oxalyl chloride followed by quenching with aqueous ammonia or dimethylamine, respectively. The products were characterized by spectroscopic analysis and their X-ray crystal structure determinations were carried out to ascertain the conformations of these molecules, especially the orientations of the carbonyl groups, as 1,2-diones have been shown to be potent inhibitors of carboxylesterases (Hyatt *et al.*, 2007). The inhibitory properties of ethane-1,2-diones toward carboxylesterases are such that, when the carbonyl O atoms are *cis*-coplanar, the compounds demonstrate specificity for human liver carboxylesterase. Conversely, when the dione O atoms are not planar (or are *trans*-coplanar), the compounds are

more potent at human intestinal carboxylesterase inhibition (Hyatt *et al.*, 2007). Thus, the title compounds and their analogues were screened for their inhibition of human carboxylesterases and it was planned to determine their conformations to compare with the observation reported for ethane-1,2-diones.



X-ray crystallography confirmed the molecular structures and atom connectivities of (I) and (II) (Figs. 1 and 3), and selected geometric parameters are listed in Tables 1 and 3. The two carbonyl groups are in a *trans* orientation, with torsion angles O1–C9–C10–O2 = $-149.3(3)^\circ$ in (I) and $-88.55(15)^\circ$ in (II). The deviation from 180° may be due to repulsive interactions between nonbonded electron pairs on atoms O1 and O2. However, in (I) the extensive hydrogen bonding introduces rigidity into the molecule. Since in both compounds the C9=O1 groups are coplanar with the indole nucleus, there is extended conjugation from atom O1 through to the indole ring. This is evident from the shortening of the

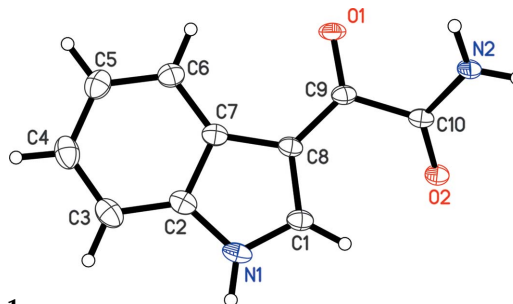


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

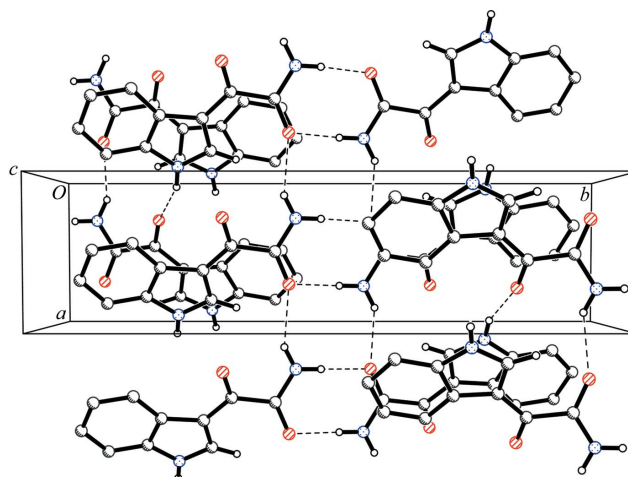


Figure 2
A packing diagram for (I), viewed down the *c* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in these interactions have been omitted for clarity.

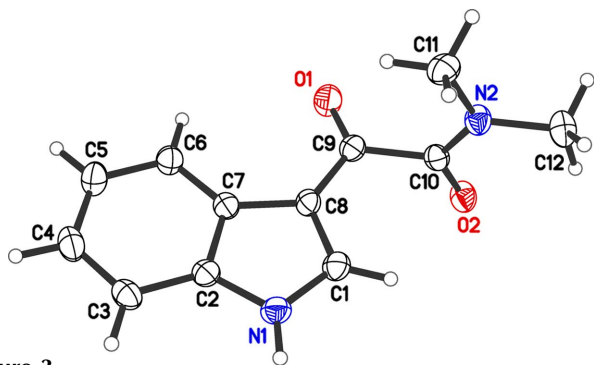


Figure 3
A view of the molecule of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

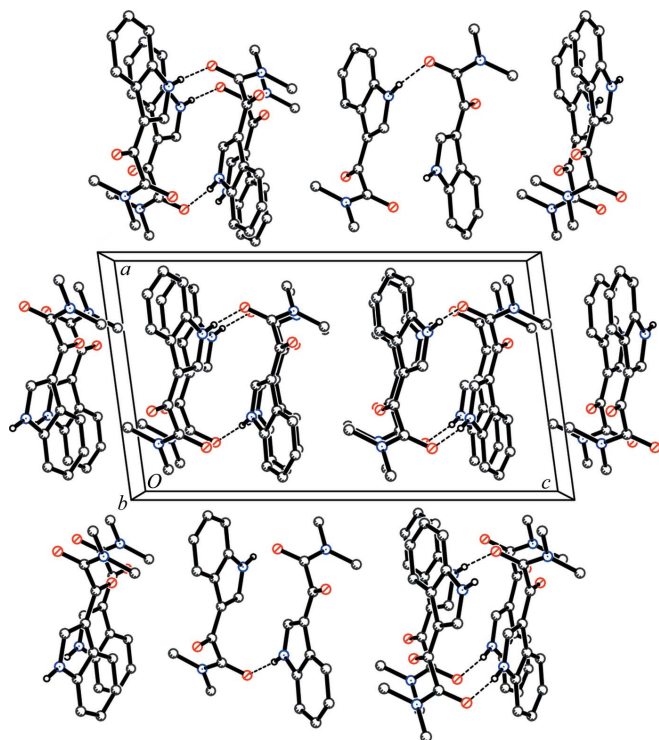


Figure 4
A packing diagram for (II), viewed down the *b* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in these interactions have been omitted for clarity.

C8—C9 bonds [1.421 (4) Å in (I) and 1.4380 (17) Å in (II)] bonds compared with the standard value for a single bond connecting a C_{ar} atom to a Csp^2 atom [1.470 (15) Å; Allen *et al.*, 1987]. Possibly due to repulsive interactions between nonbonded electron pairs, the C9—C10 bonds are stretched, resulting in bond lengths of 1.528 (4) and 1.5298 (17) Å in (I) and (II), respectively. These are longer than expected, the characteristic value for a Csp^2 — Csp^2 bond being 1.50 Å (Zukerman-Schpector *et al.*, 1994). The C10—N2 bond lengths [1.323 (4) Å in (I) and 1.3287 (16) Å in (II)], and the bond angles around the N2 atoms, suggest that the lone pairs of electrons on N2 undergo delocalization, bestowing double-bond character on the C10—N2 bonds.

The packing of compounds (I) and (II), as viewed down the *c* and *b* axes, are illustrated in Figs. 2 and 4, respectively.

Amides undergo extensive hydrogen bonding, but in (I), in addition to primary amide functionality, there is a hydrogen-bond-accepting carbonyl group and a variable glyoxylamide torsion angle. In (I), molecules are linked by an elaborate system of N—H...O hydrogen bonds (entries 1 and 2 in Table 2) form adjacent $R_2^2(8)$ and $R_2^2(8)$ ring motifs (Bernstein *et al.*, 1995) to generate a ladder-like construct. Adjacent ladders are further linked by additional N—H...O hydrogen bonds (entry 3 in Table 2) to build a three-dimensional network. The hydrogen bonding in (II) is far simpler, consisting of helical chains of N—H...O-linked molecules that follow the 2_1 screw of the *b* axis (Table 4).

Experimental

The title compounds were prepared according to a previously reported procedure (Da Settimo *et al.*, 1996). Recrystallization from ethyl acetate afforded (I) and (II) as pale-yellow and colourless crystalline products which were suitable for X-ray analysis. Spectroscopic data for (I), 1H NMR (300 MHz, DMSO- d_6): δ 7.22–7.25 (*m*, 2H), 7.49–7.52 (*m*, 1H), 7.70 (*sb*, 1H), 8.06 (*sb*, 1H), 8.19–8.22 (*m*, 1H), 8.67 (*d*, 1H), 12.17 (*sb*, 1H); ^{13}C NMR (75 MHz, DMSO- d_6): δ 111.95, 112.41, 121.10, 122.32, 123.22, 125.98, 136.11, 138.10, 165.80, 182.72. Spectroscopic data for (II), 1H NMR (300 MHz, DMSO- d_6): δ 2.93 (*s*, 3H), 3.01 (*s*, 3H), 7.23–7.32 (*m*, 2H), 7.51–7.57 (*m*, 1H), 8.10 (*dd*, 1H), 8.14 (*s*, 1H), 12.17 (*sb*, 1H); ^{13}C NMR (75 MHz, DMSO- d_6): δ 33.41, 36.81, 112.52, 112.83, 120.71, 122.38, 123.38, 124.75, 136.73, 136.83, 167.11, 186.44.

Compound (I)

Crystal data

$C_{10}H_8N_2O_2$	$V = 844.91 (11) \text{ \AA}^3$
$M_r = 188.18$	$Z = 4$
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation
$a = 5.1253 (4) \text{ \AA}$	$\mu = 0.88 \text{ mm}^{-1}$
$b = 19.4363 (14) \text{ \AA}$	$T = 90 \text{ K}$
$c = 8.4953 (6) \text{ \AA}$	$0.30 \times 0.08 \times 0.06 \text{ mm}$
$\beta = 93.251 (3)^\circ$	

Data collection

Bruker X8 Proteum diffractometer	10887 measured reflections
Absorption correction: multi-scan [SADABS (Bruker, 2006) and XABS2 (Parkin <i>et al.</i> , 1995)]	1518 independent reflections
$T_{min} = 0.744$, $T_{max} = 0.949$	1481 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$	128 parameters
$wR(F^2) = 0.158$	H-atom parameters constrained
$S = 1.24$	$\Delta\rho_{max} = 0.33 \text{ e \AA}^{-3}$
1518 reflections	$\Delta\rho_{min} = -0.33 \text{ e \AA}^{-3}$

Compound (II)

Crystal data

$C_{12}H_{12}N_2O_2$	$V = 1094.27 (4) \text{ \AA}^3$
$M_r = 216.24$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.1813 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 6.0048 (1) \text{ \AA}$	$T = 90 \text{ K}$
$c = 18.0625 (4) \text{ \AA}$	$0.65 \times 0.25 \times 0.15 \text{ mm}$
$\beta = 97.7222 (8)^\circ$	

Table 1

Selected geometric parameters (Å, °) for (I).

N2—C10	1.323 (4)	C8—C9	1.421 (4)
O2—C10	1.232 (3)	C9—C10	1.528 (4)
C1—C8—C9	126.9 (3)	O2—C10—N2	124.1 (3)
O1—C9—C8	124.1 (3)	O2—C10—C9	122.3 (2)
O1—C9—C10	117.1 (3)	N2—C10—C9	113.5 (2)
C1—C8—C9—O1	−178.2 (3)	O1—C9—C10—O2	−149.3 (3)

Table 2

Hydrogen-bond geometry (Å, °) for (I).

<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—H1...O1 ⁱ	0.88	1.92	2.768 (3)	160
N2—H2A...O2 ⁱⁱ	0.88	2.08	2.926 (3)	161
N2—H2B...O2 ⁱⁱⁱ	0.88	2.14	2.898 (3)	144

Symmetry codes: (i) $x - 1, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $x + 1, y, z$.**Data collection**

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)
 $T_{\min} = 0.943, T_{\max} = 0.986$

4780 measured reflections
2515 independent reflections
2031 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.112$
 $S = 1.06$
2515 reflections

147 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.20 \text{ e } \text{Å}^{-3}$

H atoms were found in difference Fourier maps and subsequently placed in idealized positions, with C—H = 0.95 (aromatic), 0.98 (methyl) or 0.88 Å (NH₂), and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms or $1.2U_{\text{eq}}(\text{C}, \text{N})$ otherwise.

Data collection: APEX2 (Bruker, 2006) for (I); COLLECT (Nonius, 1999) for (II). Cell refinement: APEX2 for (I); SCALEPACK (Otwinowski & Minor, 1997) for (II). Data reduction: APEX2 for (I); DENZO-SMN (Otwinowski & Minor, 1997) for (II). For both compounds, program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: XP in SHELXTL (Sheldrick,

Table 3

Selected geometric parameters (Å, °) for (II).

N2—C10	1.3287 (16)	C8—C9	1.4380 (17)
O2—C10	1.2431 (15)	C9—C10	1.5298 (17)
C1—C8—C9	125.50 (11)	O2—C10—N2	124.03 (11)
O1—C9—C8	124.97 (12)	O2—C10—C9	118.05 (11)
O1—C9—C10	118.72 (11)	N2—C10—C9	117.91 (10)
C1—C8—C9—O1	175.49 (12)	O1—C9—C10—O2	−88.55 (15)

Table 4

Hydrogen-bond geometry (Å, °) for (II).

<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—H1A...O2 ⁱ	0.88	1.92	2.7885 (14)	170

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

2008); software used to prepare material for publication: SHELXL97 and local procedures.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: YP3017). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2012). C68, o405–o407 [doi:10.1107/S0108270112038322]

The effect of hydrogen bonding on the conformations of 2-(1*H*-indol-3-yl)-2-oxoacetamide and 2-(1*H*-indol-3-yl)-*N,N*-dimethyl-2-oxoacetamide

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(I) 2-(1*H*-indol-3-yl)-2-oxoacetamide

Crystal data

C₁₀H₈N₂O₂

M_r = 188.18

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 5.1253 (4) Å

b = 19.4363 (14) Å

c = 8.4953 (6) Å

β = 93.251 (3)°

V = 844.91 (11) Å³

Z = 4

F(000) = 392

D_x = 1.479 Mg m⁻³

Cu *K*α radiation, λ = 1.54178 Å

Cell parameters from 8721 reflections

θ = 4.6–69.0°

μ = 0.88 mm⁻¹

T = 90 K

Needle, colourless

0.30 × 0.08 × 0.06 mm

Data collection

Bruker X8 Proteum
diffractometer

Radiation source: fine-focus rotating anode

Graded multilayer optics monochromator

Detector resolution: 18 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

[*SADABS* (Bruker, 2006) and *XABS2* (Parkin *et al.*, 1995)]

T_{min} = 0.744, *T_{max}* = 0.949

10887 measured reflections

1518 independent reflections

1481 reflections with *I* > 2σ(*I*)

R_{int} = 0.042

θ_{max} = 68.9°, θ_{min} = 4.6°

h = -4→6

k = -23→23

l = -10→10

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.058

wR(*F*²) = 0.158

S = 1.24

1518 reflections

128 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.037*P*)² + 1.8838*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.33 e Å⁻³

Δρ_{min} = -0.33 e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick,
2008), *F_c** = *kF_c*[1 + 0.001 × *F_c*²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.0033 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.0782 (5)	0.77152 (13)	0.5558 (3)	0.0227 (6)
H1	-0.0529	0.7766	0.6173	0.027*
N2	0.7272 (5)	0.56011 (12)	0.4103 (3)	0.0219 (6)
H2A	0.7242	0.5163	0.4362	0.026*
H2B	0.8761	0.5800	0.3904	0.026*
O1	0.7253 (4)	0.68365 (10)	0.2684 (2)	0.0221 (5)
O2	0.2916 (4)	0.57240 (10)	0.4272 (3)	0.0231 (5)
C1	0.1826 (5)	0.71168 (15)	0.5181 (3)	0.0195 (6)
H1A	0.1282	0.6681	0.5546	0.023*
C2	0.2054 (6)	0.82452 (15)	0.4845 (3)	0.0207 (7)
C3	0.1592 (6)	0.89462 (16)	0.4918 (4)	0.0274 (7)
H3	0.0254	0.9129	0.5524	0.033*
C4	0.3164 (6)	0.93666 (16)	0.4069 (4)	0.0297 (8)
H4	0.2880	0.9850	0.4068	0.036*
C5	0.5163 (6)	0.90938 (16)	0.3211 (4)	0.0270 (7)
H5	0.6249	0.9396	0.2663	0.032*
C6	0.5597 (6)	0.83963 (15)	0.3143 (4)	0.0225 (7)
H6	0.6958	0.8218	0.2548	0.027*
C7	0.4006 (5)	0.79562 (15)	0.3960 (3)	0.0186 (6)
C8	0.3834 (5)	0.72223 (15)	0.4173 (3)	0.0183 (6)
C9	0.5462 (5)	0.67120 (15)	0.3542 (3)	0.0185 (6)
C10	0.5078 (5)	0.59616 (15)	0.4013 (3)	0.0192 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0145 (12)	0.0325 (14)	0.0213 (13)	0.0038 (10)	0.0036 (10)	-0.0025 (10)
N2	0.0094 (11)	0.0211 (13)	0.0356 (15)	-0.0008 (9)	0.0044 (10)	0.0028 (10)
O1	0.0138 (10)	0.0286 (11)	0.0247 (11)	-0.0003 (8)	0.0072 (8)	0.0019 (8)
O2	0.0120 (10)	0.0239 (11)	0.0335 (12)	-0.0002 (8)	0.0037 (8)	0.0031 (9)
C1	0.0142 (14)	0.0269 (15)	0.0175 (14)	0.0009 (11)	0.0007 (11)	0.0001 (11)
C2	0.0155 (14)	0.0276 (16)	0.0186 (15)	0.0018 (11)	-0.0028 (11)	-0.0035 (11)
C3	0.0221 (16)	0.0295 (17)	0.0301 (18)	0.0043 (13)	-0.0037 (13)	-0.0079 (13)
C4	0.0274 (17)	0.0231 (16)	0.0375 (19)	0.0039 (13)	-0.0071 (14)	-0.0030 (13)
C5	0.0239 (16)	0.0261 (16)	0.0302 (18)	-0.0039 (12)	-0.0049 (13)	0.0023 (13)
C6	0.0167 (15)	0.0268 (16)	0.0237 (16)	-0.0012 (12)	-0.0016 (12)	0.0005 (12)
C7	0.0130 (13)	0.0253 (15)	0.0171 (14)	0.0009 (11)	-0.0024 (11)	-0.0001 (11)

C8	0.0124 (13)	0.0229 (14)	0.0195 (14)	0.0015 (11)	0.0011 (11)	-0.0003 (11)
C9	0.0118 (13)	0.0238 (15)	0.0200 (15)	-0.0011 (11)	0.0009 (11)	0.0007 (11)
C10	0.0128 (14)	0.0245 (15)	0.0206 (15)	-0.0003 (11)	0.0028 (11)	-0.0010 (11)

Geometric parameters (Å, °)

N1—C1	1.327 (4)	C3—C4	1.379 (5)
N1—C2	1.378 (4)	C3—H3	0.9500
N1—H1	0.8800	C4—C5	1.395 (5)
N2—C10	1.323 (4)	C4—H4	0.9500
N2—H2A	0.8800	C5—C6	1.376 (4)
N2—H2B	0.8800	C5—H5	0.9500
O1—C9	1.228 (3)	C6—C7	1.394 (4)
O2—C10	1.232 (3)	C6—H6	0.9500
C1—C8	1.391 (4)	C7—C8	1.441 (4)
C1—H1A	0.9500	C8—C9	1.421 (4)
C2—C3	1.385 (4)	C9—C10	1.528 (4)
C2—C7	1.403 (4)		
C1—N1—C2	110.0 (2)	C6—C5—C4	121.5 (3)
C1—N1—H1	125.0	C6—C5—H5	119.3
C2—N1—H1	125.0	C4—C5—H5	119.3
C10—N2—H2A	120.0	C5—C6—C7	118.9 (3)
C10—N2—H2B	120.0	C5—C6—H6	120.6
H2A—N2—H2B	120.0	C7—C6—H6	120.6
N1—C1—C8	110.0 (3)	C6—C7—C2	118.5 (3)
N1—C1—H1A	125.0	C6—C7—C8	135.4 (3)
C8—C1—H1A	125.0	C2—C7—C8	106.2 (2)
N1—C2—C3	129.1 (3)	C1—C8—C9	126.9 (3)
N1—C2—C7	107.8 (3)	C1—C8—C7	106.0 (2)
C3—C2—C7	123.1 (3)	C9—C8—C7	127.0 (3)
C4—C3—C2	117.0 (3)	O1—C9—C8	124.1 (3)
C4—C3—H3	121.5	O1—C9—C10	117.1 (3)
C2—C3—H3	121.5	C8—C9—C10	118.7 (2)
C3—C4—C5	121.1 (3)	O2—C10—N2	124.1 (3)
C3—C4—H4	119.5	O2—C10—C9	122.3 (2)
C5—C4—H4	119.5	N2—C10—C9	113.5 (2)
C2—N1—C1—C8	-0.9 (3)	N1—C1—C8—C9	178.3 (3)
C1—N1—C2—C3	179.8 (3)	N1—C1—C8—C7	1.0 (3)
C1—N1—C2—C7	0.4 (3)	C6—C7—C8—C1	178.4 (3)
N1—C2—C3—C4	-179.5 (3)	C2—C7—C8—C1	-0.7 (3)
C7—C2—C3—C4	-0.2 (4)	C6—C7—C8—C9	1.0 (5)
C2—C3—C4—C5	-1.5 (5)	C2—C7—C8—C9	-178.1 (3)
C3—C4—C5—C6	1.9 (5)	C1—C8—C9—O1	-178.2 (3)
C4—C5—C6—C7	-0.4 (5)	C7—C8—C9—O1	-1.3 (5)
C5—C6—C7—C2	-1.2 (4)	C1—C8—C9—C10	-1.4 (4)
C5—C6—C7—C8	179.8 (3)	C7—C8—C9—C10	175.5 (3)
N1—C2—C7—C6	-179.0 (3)	O1—C9—C10—O2	-149.3 (3)
C3—C2—C7—C6	1.5 (4)	C8—C9—C10—O2	33.7 (4)

N1—C2—C7—C8	0.2 (3)	O1—C9—C10—N2	30.4 (4)
C3—C2—C7—C8	-179.2 (3)	C8—C9—C10—N2	-146.6 (3)

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—H1...O1 ⁱ	0.88	1.92	2.768 (3)	160
N2—H2A...O2 ⁱⁱ	0.88	2.08	2.926 (3)	161
N2—H2B...O2 ⁱⁱⁱ	0.88	2.14	2.898 (3)	144

Symmetry codes: (i) $x-1, -y+3/2, z+1/2$; (ii) $-x+1, -y+1, -z+1$; (iii) $x+1, y, z$.

(II) 2-(1*H*-indol-3-yl)-*N,N*-dimethyl-2-oxoacetamide

Crystal data

$C_{12}H_{12}N_2O_2$	$F(000) = 456$
$M_r = 216.24$	$D_x = 1.313 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ybc	Cell parameters from 2759 reflections
$a = 10.1813 (2) \text{ \AA}$	$\theta = 1.0\text{--}27.5^\circ$
$b = 6.0048 (1) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 18.0625 (4) \text{ \AA}$	$T = 90 \text{ K}$
$\beta = 97.7222 (8)^\circ$	Rod, colourless
$V = 1094.27 (4) \text{ \AA}^3$	$0.65 \times 0.25 \times 0.15 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD area-detector	4780 measured reflections
diffraction	2515 independent reflections
Radiation source: fine-focus sealed tube	2031 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.033$
Detector resolution: 18 pixels mm^{-1}	$\theta_{\text{max}} = 27.5^\circ, \theta_{\text{min}} = 2.0^\circ$
ω scans at fixed $\chi = 55^\circ$	$h = -13 \rightarrow 13$
Absorption correction: multi-scan	$k = -7 \rightarrow 7$
SCALEPACK (Otwinowski & Minor, 1997)	$l = -23 \rightarrow 23$
$T_{\text{min}} = 0.943, T_{\text{max}} = 0.986$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H-atom parameters constrained
$wR(F^2) = 0.112$	$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.2642P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2515 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
147 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against all reflections. The weighted R-value wR and goodness of fit S are based on F^2 . Conventional R-values R are based on F, with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-values based on F^2 are statistically about twice as large as those based on F, and R-values based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.46067 (12)	0.5029 (2)	0.30948 (7)	0.0250 (3)
H1	0.5263	0.4004	0.2983	0.030*
N1	0.32928 (10)	0.47893 (19)	0.28893 (6)	0.0262 (3)
H1A	0.2914	0.3676	0.2625	0.031*
N2	0.77326 (10)	0.48231 (18)	0.42133 (6)	0.0253 (3)
O1	0.63689 (9)	0.95880 (17)	0.41126 (6)	0.0341 (3)
O2	0.78438 (8)	0.65588 (16)	0.31061 (5)	0.0295 (2)
C2	0.26301 (12)	0.6570 (2)	0.31595 (6)	0.0229 (3)
C3	0.12758 (12)	0.6955 (2)	0.31215 (7)	0.0268 (3)
H3	0.0643	0.5964	0.2866	0.032*
C4	0.08930 (13)	0.8841 (3)	0.34723 (7)	0.0310 (3)
H4	-0.0024	0.9150	0.3463	0.037*
C5	0.18277 (13)	1.0309 (2)	0.38423 (8)	0.0317 (3)
H5	0.1530	1.1600	0.4074	0.038*
C6	0.31755 (12)	0.9925 (2)	0.38792 (7)	0.0264 (3)
H6	0.3802	1.0936	0.4130	0.032*
C7	0.35877 (11)	0.8014 (2)	0.35382 (6)	0.0217 (3)
C8	0.48596 (11)	0.6990 (2)	0.34928 (6)	0.0218 (3)
C9	0.61495 (11)	0.7791 (2)	0.37998 (7)	0.0229 (3)
C10	0.73240 (11)	0.6301 (2)	0.36848 (7)	0.0224 (3)
C11	0.70814 (13)	0.4489 (2)	0.48773 (7)	0.0294 (3)
H11A	0.6463	0.5716	0.4923	0.044*
H11B	0.7750	0.4449	0.5321	0.044*
H11C	0.6594	0.3077	0.4833	0.044*
C12	0.88222 (13)	0.3315 (2)	0.41201 (8)	0.0316 (3)
H12A	0.9080	0.3512	0.3621	0.047*
H12B	0.8538	0.1774	0.4179	0.047*
H12C	0.9581	0.3648	0.4498	0.047*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0232 (6)	0.0284 (7)	0.0245 (6)	-0.0007 (5)	0.0069 (5)	-0.0014 (5)
N1	0.0243 (5)	0.0296 (6)	0.0250 (5)	-0.0045 (4)	0.0048 (4)	-0.0056 (4)
N2	0.0231 (5)	0.0277 (6)	0.0260 (5)	0.0025 (4)	0.0068 (4)	0.0028 (4)
O1	0.0264 (5)	0.0303 (6)	0.0454 (6)	-0.0025 (4)	0.0043 (4)	-0.0106 (4)
O2	0.0247 (5)	0.0379 (6)	0.0275 (5)	0.0043 (4)	0.0092 (4)	0.0068 (4)
C2	0.0233 (6)	0.0271 (7)	0.0190 (5)	-0.0009 (5)	0.0048 (4)	0.0014 (5)
C3	0.0213 (6)	0.0344 (7)	0.0242 (6)	-0.0032 (5)	0.0019 (5)	0.0042 (5)
C4	0.0210 (6)	0.0391 (8)	0.0332 (7)	0.0037 (6)	0.0048 (5)	0.0050 (6)
C5	0.0270 (7)	0.0311 (8)	0.0377 (7)	0.0064 (6)	0.0073 (5)	-0.0012 (6)
C6	0.0245 (6)	0.0261 (7)	0.0289 (6)	0.0002 (5)	0.0048 (5)	-0.0009 (5)

C7	0.0206 (6)	0.0258 (6)	0.0193 (5)	-0.0001 (5)	0.0044 (4)	0.0033 (5)
C8	0.0200 (6)	0.0251 (6)	0.0211 (5)	0.0004 (5)	0.0058 (4)	0.0009 (5)
C9	0.0233 (6)	0.0244 (7)	0.0219 (5)	-0.0004 (5)	0.0059 (4)	0.0006 (5)
C10	0.0180 (5)	0.0249 (7)	0.0243 (6)	-0.0019 (5)	0.0031 (4)	-0.0005 (5)
C11	0.0345 (7)	0.0311 (7)	0.0241 (6)	0.0005 (6)	0.0088 (5)	0.0016 (5)
C12	0.0255 (7)	0.0323 (8)	0.0378 (7)	0.0062 (5)	0.0074 (5)	0.0059 (6)

Geometric parameters (Å, °)

C1—N1	1.3471 (16)	C4—H4	0.9500
C1—C8	1.3858 (18)	C5—C6	1.3842 (17)
C1—H1	0.9500	C5—H5	0.9500
N1—C2	1.3874 (16)	C6—C7	1.3936 (18)
N1—H1A	0.8800	C6—H6	0.9500
N2—C10	1.3287 (16)	C7—C8	1.4457 (16)
N2—C12	1.4591 (16)	C8—C9	1.4380 (17)
N2—C11	1.4602 (15)	C9—C10	1.5298 (17)
O1—C9	1.2244 (16)	C11—H11A	0.9800
O2—C10	1.2431 (15)	C11—H11B	0.9800
C2—C3	1.3906 (17)	C11—H11C	0.9800
C2—C7	1.4110 (17)	C12—H12A	0.9800
C3—C4	1.380 (2)	C12—H12B	0.9800
C3—H3	0.9500	C12—H12C	0.9800
C4—C5	1.399 (2)		
N1—C1—C8	110.00 (11)	C6—C7—C2	119.39 (11)
N1—C1—H1	125.0	C6—C7—C8	134.46 (12)
C8—C1—H1	125.0	C2—C7—C8	106.09 (11)
C1—N1—C2	109.43 (10)	C1—C8—C9	125.50 (11)
C1—N1—H1A	125.3	C1—C8—C7	106.58 (10)
C2—N1—H1A	125.3	C9—C8—C7	127.92 (12)
C10—N2—C12	120.07 (10)	O1—C9—C8	124.97 (12)
C10—N2—C11	123.31 (11)	O1—C9—C10	118.72 (11)
C12—N2—C11	116.46 (10)	C8—C9—C10	116.23 (11)
N1—C2—C3	129.53 (12)	O2—C10—N2	124.03 (11)
N1—C2—C7	107.88 (10)	O2—C10—C9	118.05 (11)
C3—C2—C7	122.53 (12)	N2—C10—C9	117.91 (10)
C4—C3—C2	116.96 (12)	N2—C11—H11A	109.5
C4—C3—H3	121.5	N2—C11—H11B	109.5
C2—C3—H3	121.5	H11A—C11—H11B	109.5
C3—C4—C5	121.36 (12)	N2—C11—H11C	109.5
C3—C4—H4	119.3	H11A—C11—H11C	109.5
C5—C4—H4	119.3	H11B—C11—H11C	109.5
C6—C5—C4	121.63 (13)	N2—C12—H12A	109.5
C6—C5—H5	119.2	N2—C12—H12B	109.5
C4—C5—H5	119.2	H12A—C12—H12B	109.5
C5—C6—C7	118.12 (12)	N2—C12—H12C	109.5
C5—C6—H6	120.9	H12A—C12—H12C	109.5
C7—C6—H6	120.9	H12B—C12—H12C	109.5

C8—C1—N1—C2	-1.24 (14)	C6—C7—C8—C1	177.34 (13)
C1—N1—C2—C3	-175.89 (12)	C2—C7—C8—C1	0.23 (13)
C1—N1—C2—C7	1.37 (13)	C6—C7—C8—C9	-2.1 (2)
N1—C2—C3—C4	177.12 (12)	C2—C7—C8—C9	-179.18 (11)
C7—C2—C3—C4	0.20 (18)	C1—C8—C9—O1	175.49 (12)
C2—C3—C4—C5	0.64 (19)	C7—C8—C9—O1	-5.2 (2)
C3—C4—C5—C6	-0.6 (2)	C1—C8—C9—C10	-1.37 (17)
C4—C5—C6—C7	-0.3 (2)	C7—C8—C9—C10	177.94 (11)
C5—C6—C7—C2	1.11 (18)	C12—N2—C10—O2	-2.49 (19)
C5—C6—C7—C8	-175.71 (13)	C11—N2—C10—O2	-177.78 (12)
N1—C2—C7—C6	-178.59 (11)	C12—N2—C10—C9	178.11 (11)
C3—C2—C7—C6	-1.10 (18)	C11—N2—C10—C9	2.82 (18)
N1—C2—C7—C8	-0.96 (13)	O1—C9—C10—O2	-88.55 (15)
C3—C2—C7—C8	176.54 (11)	C8—C9—C10—O2	88.51 (14)
N1—C1—C8—C9	-179.96 (11)	O1—C9—C10—N2	90.89 (15)
N1—C1—C8—C7	0.61 (13)	C8—C9—C10—N2	-92.05 (14)

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—H1A...O2 ⁱ	0.88	1.92	2.7885 (14)	170

Symmetry code: (i) $-x+1, y-1/2, -z+1/2$.