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(*S*)-2-(1-Benzyl-5-bromo-1*H*-indol-3-yl)-2-hydroxy-*N*-methylacetamide

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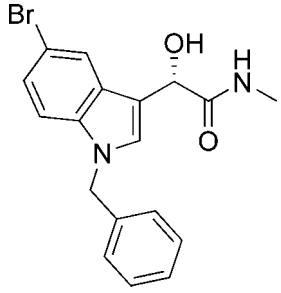
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.063; data-to-parameter ratio = 16.2.

The title compound, $C_{18}H_{17}BrN_2O_2$, is a chiral indole derivative. The crystal structure shows both intra- and intermolecular hydrogen-bonding interactions.

Related literature

For related literature, see: Bandini *et al.* (2005); Ramirez & Garcia-Rubio (2003); Yuan *et al.* (2004).



Experimental

Crystal data

$C_{18}H_{17}BrN_2O_2$
M_r = 373.25
Monoclinic, $P2_1$
a = 4.5759 (6) Å
b = 9.4134 (11) Å
c = 18.540 (2) Å
β = 95.049 (2)°
V = 795.51 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 2.60 mm^{-1}
T = 296 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001*a*)
T_{min} = 0.625, T_{max} = 0.781
5321 measured reflections
3482 independent reflections
2936 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.063
S = 0.95
3482 reflections
215 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Absolute structure: Flack (1983), 1456 Friedel pairs
Flack parameter: 0.015 (7)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.87 (3)	2.32 (3)	2.682 (3)	105 (2)
C18—H18C···O1^{i}	0.96	2.57	3.395 (3)	144
N2—H2A···O2^{ii}	0.87 (3)	2.40 (3)	3.119 (3)	139 (3)
O1—H1···O2^{iii}	0.91 (4)	2.02 (4)	2.839 (2)	149 (3)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2001*b*) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2117).

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supplementary materials

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(*S*)-2-(1-Benzyl-5-bromo-1*H*-indol-3-yl)-2-hydroxy-*N*-methylacetamide

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Comment

Derivatives of indole have received much attention because of their widespread applications in materials science, agri-chemicals, and pharmaceuticals (Ramirez & Garcia-Rubio, 2003). Their preparation and functionalization continues to be a fascinating subject in organic synthesis due to the frequent appearance of indoles in biologically interesting compounds (Bandini *et al.*, 2005). The title compound, an example of a derivatized indole core, is shown in Figure 1. In the crystal, C—H···O hydrogen bonds, (Table 1) link the molecules in rows along the *c* axis (Fig. 2)

Experimental

The title compound was synthesized according to procedure of Yuan *et al.*, 2004. Crystals appropriate for data collection were obtained by slow evaporation of a CH_3COCH_3/C_6H_6 (100:1, *v*/*v*) solution at 283 K.

Refinement

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms bonded to carbon were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å and U_{iso}(H) = 1.2U_{eq}(C). The O—H and N—H H atom coordinates were allowed to refine but the thermal parameters were fixed.

Figures

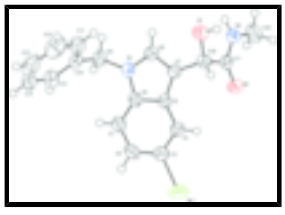


Fig. 1. The structure of (I), showing the atom-labelling scheme and thermal ellipsoids at the 50% probability level.

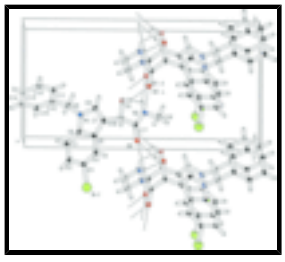


Fig. 2. The molecular packing of (I) viewed along the *c* axis. Hydrogen bonds are drawn as dashed lines.

(*S*)-2-(1-Benzyl-5-bromo-1*H*-indol-3-yl)-2-hydroxy-*N*- methylacetamide

Crystal data

$C_{18}H_{17}BrN_2O_2$	F_{000} = 380
M_r = 373.25	D_x = 1.558 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 2442 reflections
a = 4.5759 (6) Å	θ = 0.00–0.00º
b = 9.4134 (11) Å	μ = 2.60 mm^{-1}
c = 18.540 (2) Å	T = 296 (2) K
β = 95.049 (2)º	Block, colorless
V = 795.51 (17) Å^3	0.20 × 0.20 × 0.10 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	3482 independent reflections
Radiation source: fine-focus sealed tube	2936 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 296(2) K	θ_{max} = 28.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001a)	h = −6→5
T_{min} = 0.625, T_{max} = 0.781	k = −11→12
5321 measured reflections	l = −22→24

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.063	$(\Delta/\sigma)_{max}$ = 0.002
S = 0.95	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
3482 reflections	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
215 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1456 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.015 (7)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.14694 (7)	−0.31988 (3)	0.737099 (16)	0.06370 (11)
C1	0.6534 (6)	0.0039 (3)	0.81575 (12)	0.0391 (6)
H1A	0.5425	0.0180	0.8548	0.047*
C2	0.8019 (6)	−0.1216 (3)	0.80734 (13)	0.0433 (6)
H2	0.7932	−0.1936	0.8414	0.052*
C3	0.9654 (6)	−0.1411 (3)	0.74796 (13)	0.0391 (5)
C4	0.9924 (5)	−0.0383 (2)	0.69649 (12)	0.0341 (5)
H4	1.1062	−0.0534	0.6580	0.041*
C5	0.8429 (5)	0.0900 (2)	0.70371 (11)	0.0298 (5)
C6	0.6757 (5)	0.1082 (3)	0.76376 (12)	0.0318 (5)
C7	0.8163 (5)	0.2179 (2)	0.66256 (11)	0.0317 (5)
C8	0.6355 (5)	0.3059 (3)	0.69713 (12)	0.0357 (5)
H8	0.5795	0.3967	0.6817	0.043*
C9	0.3612 (5)	0.3018 (3)	0.81004 (12)	0.0403 (5)
H9A	0.2210	0.2306	0.8225	0.048*
H9B	0.2518	0.3806	0.7874	0.048*
C10	0.5304 (5)	0.3531 (3)	0.87805 (12)	0.0362 (5)
C11	0.7243 (6)	0.4648 (3)	0.87613 (14)	0.0482 (6)
H11	0.7512	0.5078	0.8320	0.058*
C12	0.8776 (7)	0.5135 (4)	0.93757 (19)	0.0689 (9)
H12	1.0081	0.5887	0.9350	0.083*
C13	0.8399 (8)	0.4520 (5)	1.00341 (18)	0.0757 (11)
H13	0.9437	0.4858	1.0453	0.091*
C14	0.6496 (9)	0.3411 (4)	1.00709 (15)	0.0733 (11)
H14	0.6246	0.2985	1.0514	0.088*
C15	0.4936 (7)	0.2922 (3)	0.94434 (13)	0.0545 (7)
H15	0.3624	0.2173	0.9470	0.065*
C16	0.9457 (5)	0.2509 (2)	0.59302 (12)	0.0352 (5)
H16	1.1449	0.2120	0.5951	0.042*
C17	0.7595 (5)	0.1810 (3)	0.53065 (10)	0.0338 (4)
C18	0.3941 (7)	0.2177 (3)	0.42835 (13)	0.0538 (8)
H18A	0.5083	0.1786	0.3921	0.081*
H18B	0.2787	0.2955	0.4081	0.081*

H18C	0.2667	0.1457	0.4446	0.081*
H1	1.088 (8)	0.418 (4)	0.5528 (18)	0.081*
H2A	0.572 (7)	0.358 (3)	0.4998 (16)	0.065*
N1	0.5491 (5)	0.2407 (2)	0.75786 (10)	0.0357 (5)
N2	0.5881 (5)	0.2677 (2)	0.48896 (11)	0.0400 (5)
O1	0.9600 (4)	0.40069 (17)	0.58655 (9)	0.0473 (4)
O2	0.7673 (4)	0.05083 (18)	0.52216 (9)	0.0470 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0778 (2)	0.03513 (14)	0.0805 (2)	0.01527 (16)	0.02007 (15)	0.01439 (16)
C1	0.0485 (15)	0.0387 (14)	0.0309 (11)	−0.0056 (11)	0.0081 (11)	0.0018 (10)
C2	0.0576 (16)	0.0373 (14)	0.0348 (12)	−0.0030 (12)	0.0021 (11)	0.0105 (10)
C3	0.0440 (14)	0.0296 (12)	0.0428 (13)	0.0029 (10)	−0.0012 (11)	0.0021 (10)
C4	0.0362 (12)	0.0327 (12)	0.0333 (11)	−0.0003 (9)	0.0023 (10)	−0.0031 (9)
C5	0.0320 (12)	0.0310 (12)	0.0260 (11)	−0.0047 (9)	0.0010 (9)	−0.0029 (9)
C6	0.0364 (14)	0.0324 (13)	0.0262 (11)	−0.0044 (10)	0.0001 (10)	−0.0015 (10)
C7	0.0372 (12)	0.0282 (13)	0.0295 (11)	−0.0015 (8)	0.0017 (9)	−0.0013 (8)
C8	0.0437 (14)	0.0301 (12)	0.0329 (12)	0.0022 (10)	0.0010 (10)	−0.0012 (10)
C9	0.0381 (13)	0.0436 (14)	0.0400 (13)	0.0040 (11)	0.0082 (10)	−0.0024 (11)
C10	0.0405 (13)	0.0346 (13)	0.0347 (12)	0.0062 (10)	0.0096 (10)	−0.0053 (10)
C11	0.0534 (17)	0.0466 (16)	0.0447 (14)	−0.0031 (13)	0.0046 (13)	−0.0055 (12)
C12	0.0581 (19)	0.065 (2)	0.081 (2)	−0.0043 (16)	−0.0046 (17)	−0.0260 (18)
C13	0.078 (2)	0.086 (3)	0.058 (2)	0.036 (2)	−0.0207 (18)	−0.0336 (19)
C14	0.113 (3)	0.077 (3)	0.0312 (15)	0.044 (2)	0.0104 (17)	0.0035 (15)
C15	0.075 (2)	0.0504 (17)	0.0411 (15)	0.0064 (16)	0.0212 (14)	0.0033 (13)
C16	0.0404 (13)	0.0287 (12)	0.0371 (12)	−0.0001 (10)	0.0070 (10)	0.0034 (9)
C17	0.0456 (11)	0.0293 (10)	0.0283 (9)	0.0020 (13)	0.0135 (9)	0.0016 (12)
C18	0.0660 (17)	0.058 (2)	0.0360 (12)	−0.0011 (13)	−0.0042 (12)	0.0037 (11)
N1	0.0426 (12)	0.0356 (11)	0.0297 (10)	0.0011 (10)	0.0072 (9)	−0.0021 (8)
N2	0.0551 (13)	0.0321 (11)	0.0327 (10)	0.0015 (10)	0.0028 (9)	0.0017 (9)
O1	0.0682 (12)	0.0299 (10)	0.0453 (9)	−0.0101 (9)	0.0148 (9)	0.0029 (7)
O2	0.0721 (13)	0.0283 (9)	0.0405 (9)	0.0045 (9)	0.0045 (9)	−0.0032 (7)

Geometric parameters (Å, °)

Br1—C3	1.895 (2)	C10—C15	1.380 (3)
C1—C2	1.378 (4)	C11—C12	1.363 (4)
C1—C6	1.386 (3)	C11—H11	0.9300
C1—H1A	0.9300	C12—C13	1.376 (5)
C2—C3	1.397 (4)	C12—H12	0.9300
C2—H2	0.9300	C13—C14	1.365 (5)
C3—C4	1.372 (3)	C13—H13	0.9300
C4—C5	1.400 (3)	C14—C15	1.389 (5)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.416 (3)	C15—H15	0.9300
C5—C7	1.425 (3)	C16—O1	1.418 (3)
C6—N1	1.375 (3)	C16—C17	1.524 (3)

C7—C8	1.369 (3)	C16—H16	0.9800
C7—C16	1.498 (3)	C17—O2	1.236 (4)
C8—N1	1.371 (3)	C17—N2	1.331 (3)
C8—H8	0.9300	C18—N2	1.448 (3)
C9—N1	1.467 (3)	C18—H18A	0.9600
C9—C10	1.500 (3)	C18—H18B	0.9600
C9—H9A	0.9700	C18—H18C	0.9600
C9—H9B	0.9700	N2—H2A	0.87 (3)
C10—C11	1.378 (4)	O1—H1	0.91 (4)
C2—C1—C6	117.5 (2)	C10—C11—H11	119.3
C2—C1—H1A	121.3	C11—C12—C13	120.3 (3)
C6—C1—H1A	121.3	C11—C12—H12	119.9
C1—C2—C3	120.4 (2)	C13—C12—H12	119.9
C1—C2—H2	119.8	C14—C13—C12	119.8 (3)
C3—C2—H2	119.8	C14—C13—H13	120.1
C4—C3—C2	122.9 (2)	C12—C13—H13	120.1
C4—C3—Br1	119.02 (18)	C13—C14—C15	119.6 (3)
C2—C3—Br1	118.10 (18)	C13—C14—H14	120.2
C3—C4—C5	117.9 (2)	C15—C14—H14	120.2
C3—C4—H4	121.1	C10—C15—C14	121.0 (3)
C5—C4—H4	121.1	C10—C15—H15	119.5
C4—C5—C6	118.8 (2)	C14—C15—H15	119.5
C4—C5—C7	134.2 (2)	O1—C16—C7	107.67 (18)
C6—C5—C7	107.0 (2)	O1—C16—C17	113.1 (2)
N1—C6—C1	129.9 (2)	C7—C16—C17	109.08 (18)
N1—C6—C5	107.5 (2)	O1—C16—H16	109.0
C1—C6—C5	122.6 (2)	C7—C16—H16	109.0
C8—C7—C5	106.69 (19)	C17—C16—H16	109.0
C8—C7—C16	125.3 (2)	O2—C17—N2	123.8 (2)
C5—C7—C16	127.91 (19)	O2—C17—C16	120.3 (2)
C7—C8—N1	110.1 (2)	N2—C17—C16	116.0 (2)
C7—C8—H8	125.0	N2—C18—H18A	109.5
N1—C8—H8	125.0	N2—C18—H18B	109.5
N1—C9—C10	113.0 (2)	H18A—C18—H18B	109.5
N1—C9—H9A	109.0	N2—C18—H18C	109.5
C10—C9—H9A	109.0	H18A—C18—H18C	109.5
N1—C9—H9B	109.0	H18B—C18—H18C	109.5
C10—C9—H9B	109.0	C8—N1—C6	108.7 (2)
H9A—C9—H9B	107.8	C8—N1—C9	126.5 (2)
C11—C10—C15	118.0 (2)	C6—N1—C9	124.8 (2)
C11—C10—C9	120.7 (2)	C17—N2—C18	122.7 (2)
C15—C10—C9	121.3 (2)	C17—N2—H2A	121 (2)
C12—C11—C10	121.3 (3)	C18—N2—H2A	116 (2)
C12—C11—H11	119.3	C16—O1—H1	106 (2)
C6—C1—C2—C3	−0.5 (4)	C11—C12—C13—C14	0.4 (5)
C1—C2—C3—C4	1.3 (4)	C12—C13—C14—C15	−0.6 (5)
C1—C2—C3—Br1	−176.7 (2)	C11—C10—C15—C14	−0.7 (4)
C2—C3—C4—C5	−1.4 (4)	C9—C10—C15—C14	−179.5 (2)

Br1—C3—C4—C5	176.59 (17)	C13—C14—C15—C10	0.7 (4)
C3—C4—C5—C6	0.8 (3)	C8—C7—C16—O1	25.1 (3)
C3—C4—C5—C7	−179.7 (2)	C5—C7—C16—O1	−158.0 (2)
C2—C1—C6—N1	178.8 (2)	C8—C7—C16—C17	−97.9 (3)
C2—C1—C6—C5	0.0 (4)	C5—C7—C16—C17	78.9 (3)
C4—C5—C6—N1	−179.2 (2)	O1—C16—C17—O2	167.5 (2)
C7—C5—C6—N1	1.2 (2)	C7—C16—C17—O2	−72.7 (3)
C4—C5—C6—C1	−0.1 (3)	O1—C16—C17—N2	−14.3 (3)
C7—C5—C6—C1	−179.7 (2)	C7—C16—C17—N2	105.5 (2)
C4—C5—C7—C8	179.5 (2)	C7—C8—N1—C6	0.4 (3)
C6—C5—C7—C8	−1.0 (3)	C7—C8—N1—C9	178.4 (2)
C4—C5—C7—C16	2.1 (4)	C1—C6—N1—C8	−180.0 (2)
C6—C5—C7—C16	−178.3 (2)	C5—C6—N1—C8	−1.0 (2)
C5—C7—C8—N1	0.4 (3)	C1—C6—N1—C9	1.9 (4)
C16—C7—C8—N1	177.8 (2)	C5—C6—N1—C9	−179.1 (2)
N1—C9—C10—C11	65.1 (3)	C10—C9—N1—C8	−101.6 (3)
N1—C9—C10—C15	−116.1 (3)	C10—C9—N1—C6	76.2 (3)
C15—C10—C11—C12	0.5 (4)	O2—C17—N2—C18	−0.7 (3)
C9—C10—C11—C12	179.4 (3)	C16—C17—N2—C18	−178.8 (2)
C10—C11—C12—C13	−0.3 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.87 (3)	2.32 (3)	2.682 (3)	105 (2)
C18—H18C···O1^{i}	0.96	2.57	3.395 (3)	144
N2—H2A···O2^{ii}	0.87 (3)	2.40 (3)	3.119 (3)	139 (3)
O1—H1···O2^{iii}	0.91 (4)	2.02 (4)	2.839 (2)	149 (3)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+1; (ii) −*x*+1, *y*+1/2, −*z*+1; (iii) −*x*+2, *y*+1/2, −*z*+1.

Fig. 1

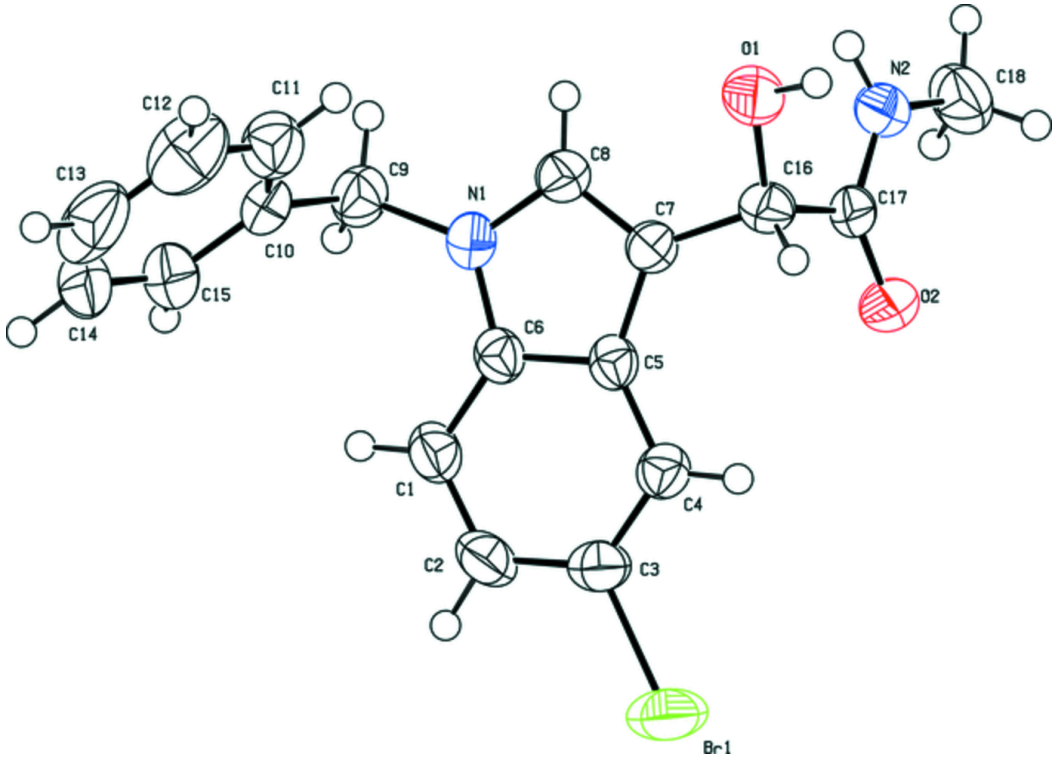
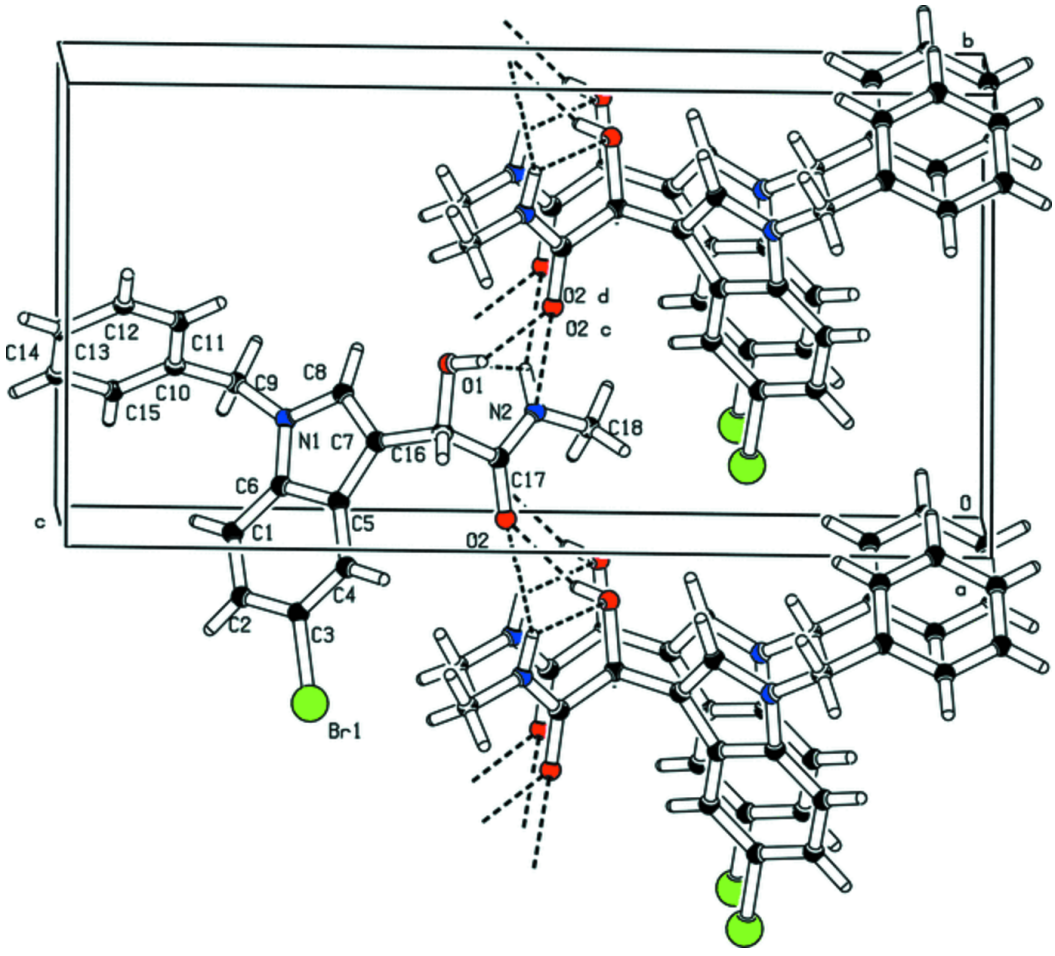

O1
N2
C18
C12
C11
C13
C9
C8
N1
C16
C17
C7
C10
O2
C15
C14
C6
C5
C1
C4
C2
C3
Br1

Fig. 2


b
O2 d
O2 c
C12
C11
C14
C13
C10
C9
C8
C15
O1
N2
C18
N1
C7
C16
C17
C6
C1
C5
O2
c
O
a
C4
C2
C3
Br1