

## 2-Hydroxy-(2-methyl-1*H*-indol-3-yl-methylidene)benzohydrazide ethanol solvate

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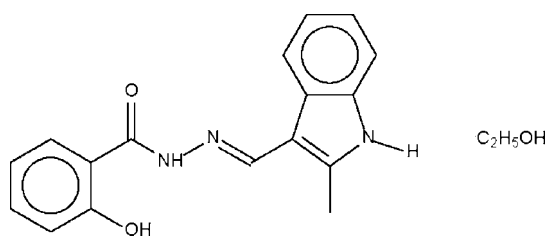
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 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.048;  $wR$  factor = 0.162; data-to-parameter ratio = 17.2.

In the title compound,  $\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2 \cdot \text{C}_2\text{H}_6\text{O}$ , Schiff base molecules are linked by a hydroxy-amido hydrogen bond into a helical chain running along the  $b$  axis. This chain is consolidated by two other hydrogen bonds; the ethanol solvent molecule is a hydrogen-bond donor to the amide group and a hydrogen-bond acceptor for the indolyl NH group of an adjacent Schiff base molecule.

### Related literature

For reports on the medicinal properties of the unsubstituted compound, indol-3-ylmethylidene-2-hydroxybenzohydrazide, see: Alemany *et al.* (1967); Fujikawa *et al.* (1966); Nakata *et al.* (1966); Singh *et al.* (1984).



### Experimental

#### Crystal data

 $\text{C}_{17}\text{H}_{15}\text{N}_3\text{O}_2 \cdot \text{C}_2\text{H}_6\text{O}$   
 $M_r = 339.39$   
 Monoclinic,  $P2_1/c$ 
 $a = 7.5907$  (1) Å  
 $b = 11.2269$  (2) Å  
 $c = 20.3014$  (3) Å

 $\beta = 91.924$  (1)°  
 $V = 1729.11$  (5) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation

 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 $0.34 \times 0.27 \times 0.12$  mm

#### Data collection

 Bruker SMART APEX  
 diffractometer  
 Absorption correction: none  
 20827 measured reflections

 3967 independent reflections  
 2869 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.056$ 

#### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.162$   
 $S = 1.13$   
 3967 reflections

 230 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.30$  e Å<sup>-3</sup>

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1o} \cdots \text{O2}^i$	0.84	1.76	2.594 (2)	176
$\text{O3}-\text{H3o} \cdots \text{O2}$	0.84	2.02	2.843 (2)	165
$\text{N1}-\text{H1n} \cdots \text{O1}$	0.88	1.91	2.618 (2)	136
$\text{N3}-\text{H3n} \cdots \text{O3}^{ii}$	0.88	1.96	2.824 (2)	168

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2238).

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**supplementary materials**

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## 2-Hydroxy-(2-methyl-1*H*-indol-3-ylmethylidene)benzohydrazide ethanol solvate

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### Comment

There are no reports of the title Schiff base but there are several reports on the medicinal properties of the unsubstituted compound, indol-3-ylmethylidene-2-hydroxybenzohydrazide (Alemany *et al.*, 1967; Fujikawa *et al.*, 1966; Nakata *et al.*, 1966; Singh *et al.*, 1984). The methyl-substituted title compound (Scheme I, Fig. 1) is a planar molecule that is linked by a hydroxy...amido hydrogen bond into a helical chain that runs along the *b*-axis of the monoclinic unit cell (Fig. 2). The chain is consolidated by two other hydrogen bonds: the ethanol molecule is hydrogen-bond donor to the amido unit well as hydrogen-bond acceptor to the amino<sub>indolyl</sub> unit of adjacent Schiff bases.

### Experimental

2-Hydroxybenzohydrazide (0.60 g, 4 mmol) and 2-methyl-1*H*-indole-3-carboxaldehyde (0.63 g, 4 mmol) were heated in ethanol (30 ml) for 3 h. The solvent was removed by evaporation and the light brown product recrystallized from ethanol.

### Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with  $U(\text{H})$  set to 1.2–1.5  $U(\text{C})$ .

The oxygen- and nitrogen-bound H-atoms were also generated geometrically (O—H 0.84, N—H 0.88 Å).

### Figures

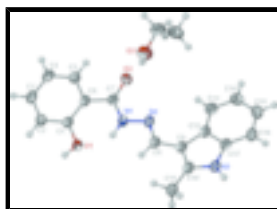


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C<sub>17</sub>H<sub>15</sub>N<sub>3</sub>O<sub>2</sub>·C<sub>2</sub>H<sub>6</sub>O at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radii.

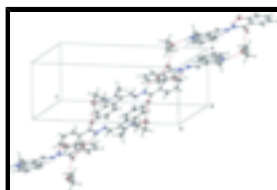


Fig. 2. Hydrogen-bonded chain motif.

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### Crystal data

$C_{17}H_{15}N_3O_2 \cdot C_2H_6O$	$F_{000} = 720$
$M_r = 339.39$	$D_x = 1.304 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
Hall symbol: -P 2ybc	$\lambda = 0.71073 \text{ \AA}$
$a = 7.5907 (1) \text{ \AA}$	Cell parameters from 6771 reflections
$b = 11.2269 (2) \text{ \AA}$	$\theta = 2.7\text{--}28.2^\circ$
$c = 20.3014 (3) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\beta = 91.924 (1)^\circ$	$T = 100 (2) \text{ K}$
$V = 1729.11 (5) \text{ \AA}^3$	Prism, yellow
$Z = 4$	$0.34 \times 0.27 \times 0.12 \text{ mm}$

### Data collection

Bruker SMART APEX diffractometer	2869 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.056$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 100(2) \text{ K}$	$\theta_{\text{min}} = 2.0^\circ$
$\omega$ scans	$h = -9 \rightarrow 9$
Absorption correction: None	$k = -14 \rightarrow 14$
20827 measured reflections	$l = -26 \rightarrow 22$
3967 independent reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.162$	$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.9553P]$
$S = 1.13$	where $P = (F_o^2 + 2F_c^2)/3$
3967 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
230 parameters	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
	Extinction correction: none

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.60372 (18)	0.39960 (12)	0.26124 (6)	0.0287 (3)

H1O	0.6488	0.3342	0.2507	0.043*
O2	0.26721 (17)	0.69310 (12)	0.26809 (7)	0.0272 (3)
O3	-0.0963 (2)	0.65666 (15)	0.29094 (8)	0.0409 (4)
H3O	0.0126	0.6698	0.2916	0.061*
N1	0.3897 (2)	0.53879 (15)	0.32419 (7)	0.0255 (4)
H1N	0.4563	0.4746	0.3236	0.031*
N2	0.3081 (2)	0.57121 (15)	0.38183 (8)	0.0263 (4)
N3	0.1852 (2)	0.47451 (15)	0.59623 (8)	0.0265 (4)
H3N	0.1730	0.4372	0.6339	0.032*
C1	0.5678 (2)	0.46480 (17)	0.20605 (9)	0.0243 (4)
C2	0.6339 (3)	0.43017 (18)	0.14536 (10)	0.0283 (4)
H2	0.7025	0.3597	0.1425	0.034*
C3	0.5997 (3)	0.4978 (2)	0.08976 (10)	0.0313 (5)
H3	0.6462	0.4742	0.0489	0.038*
C4	0.4981 (3)	0.60009 (19)	0.09301 (10)	0.0311 (4)
H4	0.4760	0.6471	0.0547	0.037*
C5	0.4291 (2)	0.63320 (18)	0.15254 (9)	0.0270 (4)
H5	0.3583	0.7028	0.1545	0.032*
C6	0.4616 (2)	0.56639 (17)	0.20990 (9)	0.0233 (4)
C7	0.3674 (2)	0.60467 (17)	0.26980 (9)	0.0233 (4)
C8	0.3263 (2)	0.49282 (18)	0.42813 (9)	0.0258 (4)
H8	0.3859	0.4204	0.4194	0.031*
C9	0.2589 (2)	0.51224 (18)	0.49235 (9)	0.0258 (4)
C10	0.2617 (2)	0.42767 (18)	0.54211 (9)	0.0266 (4)
C11	0.3331 (3)	0.30424 (19)	0.54307 (10)	0.0324 (5)
H11A	0.2672	0.2556	0.5739	0.049*
H11B	0.4578	0.3060	0.5572	0.049*
H11C	0.3212	0.2698	0.4988	0.049*
C12	0.1752 (2)	0.61732 (18)	0.51780 (9)	0.0262 (4)
C13	0.1327 (3)	0.73007 (19)	0.49295 (10)	0.0304 (4)
H13	0.1621	0.7517	0.4494	0.036*
C14	0.0472 (3)	0.8094 (2)	0.53269 (11)	0.0343 (5)
H14	0.0185	0.8864	0.5161	0.041*
C15	0.0014 (3)	0.7792 (2)	0.59706 (11)	0.0359 (5)
H15	-0.0586	0.8356	0.6231	0.043*
C16	0.0425 (3)	0.66914 (19)	0.62286 (10)	0.0314 (5)
H16	0.0123	0.6483	0.6664	0.038*
C17	0.1302 (2)	0.58892 (18)	0.58288 (9)	0.0262 (4)
C18	-0.1888 (3)	0.7654 (2)	0.28171 (11)	0.0384 (5)
H18A	-0.3059	0.7584	0.3014	0.046*
H18B	-0.2077	0.7799	0.2339	0.046*
C19	-0.0926 (3)	0.8698 (2)	0.31203 (13)	0.0453 (6)
H19A	-0.1676	0.9407	0.3088	0.068*
H19B	0.0163	0.8839	0.2886	0.068*
H19C	-0.0637	0.8529	0.3585	0.068*

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0333 (8)	0.0315 (7)	0.0215 (7)	0.0069 (6)	0.0051 (5)	0.0015 (6)
O2	0.0237 (7)	0.0306 (7)	0.0276 (7)	0.0012 (5)	0.0066 (5)	0.0015 (6)
O3	0.0288 (8)	0.0494 (10)	0.0452 (10)	-0.0007 (7)	0.0132 (7)	0.0105 (7)
N1	0.0239 (8)	0.0333 (9)	0.0198 (8)	0.0029 (7)	0.0060 (6)	-0.0004 (7)
N2	0.0214 (8)	0.0376 (9)	0.0202 (8)	-0.0009 (7)	0.0053 (6)	-0.0021 (7)
N3	0.0232 (8)	0.0368 (9)	0.0197 (8)	-0.0011 (7)	0.0043 (6)	0.0004 (7)
C1	0.0210 (9)	0.0310 (10)	0.0209 (9)	-0.0029 (7)	0.0021 (7)	0.0010 (7)
C2	0.0244 (9)	0.0346 (10)	0.0260 (10)	0.0019 (8)	0.0041 (7)	-0.0026 (8)
C3	0.0292 (10)	0.0440 (12)	0.0211 (10)	-0.0011 (9)	0.0062 (8)	-0.0014 (8)
C4	0.0327 (10)	0.0399 (11)	0.0208 (9)	-0.0021 (9)	0.0019 (8)	0.0045 (8)
C5	0.0251 (9)	0.0308 (10)	0.0252 (10)	-0.0008 (8)	0.0021 (7)	0.0016 (8)
C6	0.0194 (8)	0.0302 (10)	0.0205 (9)	-0.0040 (7)	0.0028 (7)	-0.0016 (7)
C7	0.0185 (8)	0.0279 (9)	0.0236 (9)	-0.0039 (7)	0.0016 (7)	-0.0004 (7)
C8	0.0192 (9)	0.0341 (10)	0.0242 (10)	-0.0012 (7)	0.0029 (7)	-0.0017 (8)
C9	0.0173 (8)	0.0379 (11)	0.0224 (9)	-0.0022 (7)	0.0018 (7)	-0.0010 (8)
C10	0.0184 (8)	0.0391 (11)	0.0224 (9)	-0.0029 (8)	0.0034 (7)	-0.0018 (8)
C11	0.0308 (10)	0.0400 (11)	0.0268 (10)	-0.0006 (9)	0.0069 (8)	0.0005 (9)
C12	0.0167 (8)	0.0396 (11)	0.0225 (9)	-0.0030 (8)	0.0022 (7)	-0.0026 (8)
C13	0.0222 (9)	0.0410 (11)	0.0279 (10)	-0.0020 (8)	0.0021 (7)	0.0017 (9)
C14	0.0264 (10)	0.0362 (11)	0.0402 (12)	0.0025 (9)	-0.0012 (9)	0.0009 (9)
C15	0.0280 (10)	0.0450 (12)	0.0349 (11)	0.0038 (9)	0.0026 (8)	-0.0087 (10)
C16	0.0238 (9)	0.0447 (12)	0.0258 (10)	-0.0009 (8)	0.0042 (8)	-0.0055 (9)
C17	0.0177 (8)	0.0382 (11)	0.0226 (9)	-0.0032 (7)	0.0012 (7)	-0.0020 (8)
C18	0.0253 (10)	0.0568 (14)	0.0332 (11)	0.0033 (10)	0.0047 (8)	0.0130 (10)
C19	0.0292 (11)	0.0548 (15)	0.0520 (15)	0.0108 (10)	0.0026 (10)	0.0041 (12)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C1	1.358 (2)	C8—H8	0.9500
O1—H1O	0.8400	C9—C10	1.386 (3)
O2—C7	1.250 (2)	C9—C12	1.444 (3)
O3—C18	1.417 (3)	C10—C11	1.488 (3)
O3—H3O	0.8400	C11—H11A	0.9800
N1—C7	1.335 (2)	C11—H11B	0.9800
N1—N2	1.391 (2)	C11—H11C	0.9800
N1—H1N	0.8800	C12—C13	1.396 (3)
N2—C8	1.292 (3)	C12—C17	1.412 (3)
N3—C10	1.365 (2)	C13—C14	1.379 (3)
N3—C17	1.375 (3)	C13—H13	0.9500
N3—H3N	0.8800	C14—C15	1.405 (3)
C1—C6	1.401 (3)	C14—H14	0.9500
C1—C2	1.401 (3)	C15—C16	1.374 (3)
C2—C3	1.378 (3)	C15—H15	0.9500
C2—H2	0.9500	C16—C17	1.396 (3)
C3—C4	1.386 (3)	C16—H16	0.9500

C3—H3	0.9500	C18—C19	1.502 (4)
C4—C5	1.384 (3)	C18—H18A	0.9900
C4—H4	0.9500	C18—H18B	0.9900
C5—C6	1.400 (3)	C19—H19A	0.9800
C5—H5	0.9500	C19—H19B	0.9800
C6—C7	1.494 (3)	C19—H19C	0.9800
C8—C9	1.433 (3)		
C1—O1—H1O	109.5	C9—C10—C11	130.10 (18)
C18—O3—H3O	109.5	C10—C11—H11A	109.5
C7—N1—N2	120.20 (16)	C10—C11—H11B	109.5
C7—N1—H1N	119.9	H11A—C11—H11B	109.5
N2—N1—H1N	119.9	C10—C11—H11C	109.5
C8—N2—N1	113.21 (16)	H11A—C11—H11C	109.5
C10—N3—C17	109.62 (16)	H11B—C11—H11C	109.5
C10—N3—H3N	125.2	C13—C12—C17	118.84 (18)
C17—N3—H3N	125.2	C13—C12—C9	135.28 (18)
O1—C1—C6	119.52 (16)	C17—C12—C9	105.88 (17)
O1—C1—C2	120.54 (17)	C14—C13—C12	118.82 (19)
C6—C1—C2	119.93 (17)	C14—C13—H13	120.6
C3—C2—C1	120.29 (19)	C12—C13—H13	120.6
C3—C2—H2	119.9	C13—C14—C15	121.6 (2)
C1—C2—H2	119.9	C13—C14—H14	119.2
C2—C3—C4	120.49 (19)	C15—C14—H14	119.2
C2—C3—H3	119.8	C16—C15—C14	120.8 (2)
C4—C3—H3	119.8	C16—C15—H15	119.6
C5—C4—C3	119.45 (19)	C14—C15—H15	119.6
C5—C4—H4	120.3	C15—C16—C17	117.72 (19)
C3—C4—H4	120.3	C15—C16—H16	121.1
C4—C5—C6	121.42 (19)	C17—C16—H16	121.1
C4—C5—H5	119.3	N3—C17—C16	129.46 (18)
C6—C5—H5	119.3	N3—C17—C12	108.30 (17)
C5—C6—C1	118.38 (17)	C16—C17—C12	122.23 (19)
C5—C6—C7	116.57 (17)	O3—C18—C19	112.73 (18)
C1—C6—C7	124.88 (17)	O3—C18—H18A	109.0
O2—C7—N1	121.54 (17)	C19—C18—H18A	109.0
O2—C7—C6	120.86 (17)	O3—C18—H18B	109.0
N1—C7—C6	117.57 (16)	C19—C18—H18B	109.0
N2—C8—C9	121.71 (18)	H18A—C18—H18B	107.8
N2—C8—H8	119.1	C18—C19—H19A	109.5
C9—C8—H8	119.1	C18—C19—H19B	109.5
C10—C9—C8	124.18 (18)	H19A—C19—H19B	109.5
C10—C9—C12	107.16 (17)	C18—C19—H19C	109.5
C8—C9—C12	128.66 (18)	H19A—C19—H19C	109.5
N3—C10—C9	109.04 (18)	H19B—C19—H19C	109.5
N3—C10—C11	120.86 (17)		
C7—N1—N2—C8	173.12 (16)	C17—N3—C10—C11	-179.95 (17)
O1—C1—C2—C3	-178.81 (17)	C8—C9—C10—N3	-179.13 (17)
C6—C1—C2—C3	2.2 (3)	C12—C9—C10—N3	-0.1 (2)

## supplementary materials

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C1—C2—C3—C4	-0.7 (3)	C8—C9—C10—C11	1.4 (3)
C2—C3—C4—C5	-0.8 (3)	C12—C9—C10—C11	-179.53 (19)
C3—C4—C5—C6	0.8 (3)	C10—C9—C12—C13	-179.7 (2)
C4—C5—C6—C1	0.6 (3)	C8—C9—C12—C13	-0.7 (4)
C4—C5—C6—C7	-174.79 (17)	C10—C9—C12—C17	-0.4 (2)
O1—C1—C6—C5	178.89 (16)	C8—C9—C12—C17	178.61 (18)
C2—C1—C6—C5	-2.1 (3)	C17—C12—C13—C14	-0.4 (3)
O1—C1—C6—C7	-6.1 (3)	C9—C12—C13—C14	178.8 (2)
C2—C1—C6—C7	172.88 (17)	C12—C13—C14—C15	-0.3 (3)
N2—N1—C7—O2	-3.4 (3)	C13—C14—C15—C16	0.7 (3)
N2—N1—C7—C6	178.81 (15)	C14—C15—C16—C17	-0.2 (3)
C5—C6—C7—O2	-1.0 (3)	C10—N3—C17—C16	178.45 (19)
C1—C6—C7—O2	-176.05 (17)	C10—N3—C17—C12	-0.8 (2)
C5—C6—C7—N1	176.83 (16)	C15—C16—C17—N3	-179.69 (19)
C1—C6—C7—N1	1.8 (3)	C15—C16—C17—C12	-0.5 (3)
N1—N2—C8—C9	176.97 (16)	C13—C12—C17—N3	-179.82 (16)
N2—C8—C9—C10	174.44 (18)	C9—C12—C17—N3	0.7 (2)
N2—C8—C9—C12	-4.4 (3)	C13—C12—C17—C16	0.9 (3)
C17—N3—C10—C9	0.5 (2)	C9—C12—C17—C16	-178.58 (17)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1o $\cdots$ O2 <sup>i</sup>	0.84	1.76	2.594 (2)	176
O3—H3o $\cdots$ O2	0.84	2.02	2.843 (2)	165
N1—H1n $\cdots$ O1	0.88	1.91	2.618 (2)	136
N3—H3n $\cdots$ O3 <sup>ii</sup>	0.88	1.96	2.824 (2)	168

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



Fig. 1

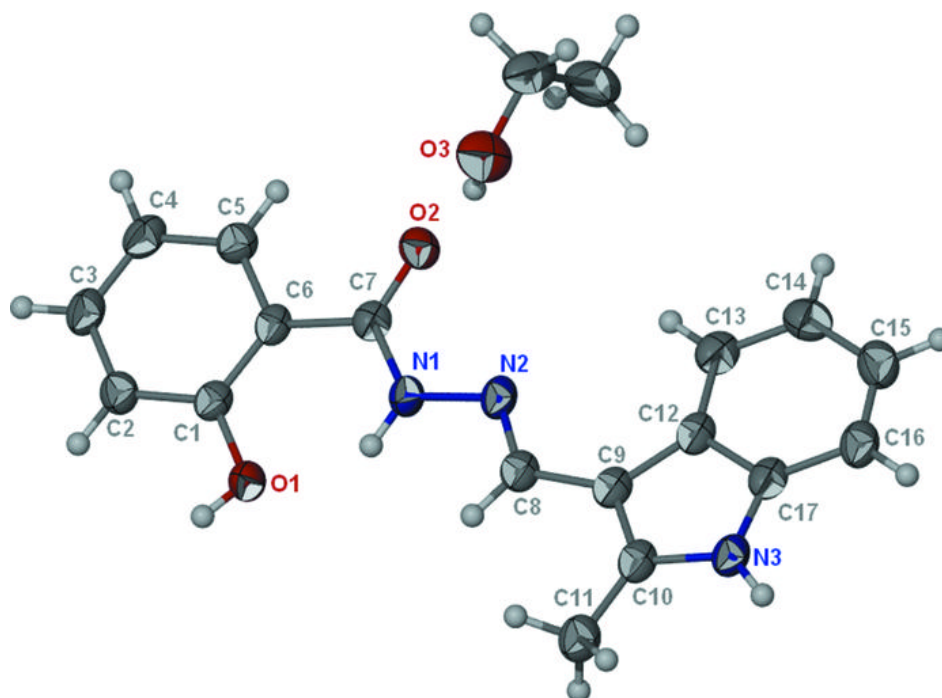


Fig. 2

