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3-(7-Methoxy-β-carbolin-1-yl)propionic acid monohydrate

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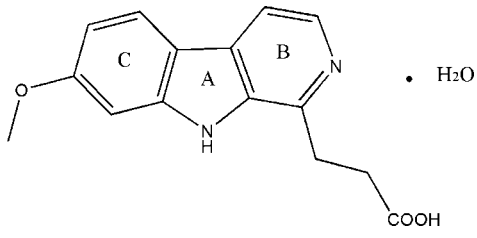
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.116; data-to-parameter ratio = 11.8.

In the title compound, $C_{15}H_{14}N_2O_3\cdot H_2O$ [systematic name: 3-(7-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)propanoic acid monohydrate], the fused rings make dhedral angles of 0.4 (1), 1.1 (2) and 1.4 (2)°. In the crystal, the water molecule is involved in the formation of three independent hydrogen-bonded chains *via* O—H···O and N—H···O hydrogen bonds, while the carboxy group forms an intermolecular O—H···N hydrogen bond.

Related literature

For the isolation of the title compound, see: Kardono *et al.* (1991). For the preparation, see: Kardono *et al.* (1991). For its pharmacological activity, see: Kuo *et al.* (2003). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{15}H_{14}N_2O_3\cdot H_2O$
M_r = 288.30
Monoclinic, $P2_1/n$
a = 4.5114 (1) Å
b = 10.8637 (2) Å
c = 28.0865 (3) Å
β = 92.414 (1)°
V = 1375.31 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 296 K
0.12 × 0.10 × 0.05 mm

Data collection

Bruker APEXII diffractometer
9119 measured reflections
2366 independent reflections
2048 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.05
2366 reflections
201 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N13—H13A···OW^{i}	0.86	1.86	2.7224 (18)	180
O5′—H5′A···N2^{ii}	0.82	1.79	2.5965 (18)	169
OW—HWA···O5′^{iii}	0.85 (3)	1.92 (3)	2.7514 (19)	169 (2)
OW—HWB···O4^{iv}	0.87 (3)	1.89 (3)	2.764 (2)	175 (2)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MW2003).

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supplementary materials

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3-(7-Methoxy-β-carbolin-1-yl)propionic acid monohydrate

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Comment

The title compound, (I), was isolated from the roots of *Eurycoma longifolia*. It is prepared according to the procedure of Kardono *et al.* (Kardono *et al.*, 1991) and recrystallized from methanol.

Bond lengths and angles are in agreement with reported literature values (Allen *et al.*, 1987). The rings (A, B and C) are each essentially planar with r.m.s deviations of 0.0018 (5) Å, 0.0027 (8)Å and 0.0052 (16) Å, respectively. The dihedral angles between the rings are A/B =1.1 (2)°, A/C = 0.4 (1)° and B/C=1.4 (2)°. The lattice water molecule is involved in formation of three independent hydrogen-bonded chains *via* O—H···O and N—H···O hydrogen bonds while the carboxy group forms an intermolecular O—H···N hydrogen bond (Fig.2 and Table 1). The lattice water molecules could be considered to be a hydrogen-bond bridge which provide further stability to the crystal lattice.

Experimental

The title compound was prepared according to the procedure of Kardono *et al.*, 1991.Crystals suitable for data collection were obtained by slow evaporation from methanol solution at 283 K over a period of two weeks.

Refinement

Water H atoms were initially located in a difference Fourier map,and all other H atoms were constrained to an ideal geometry with C—H distances of 0.98 Å and U_{iso}(H) = 1.2U_{eq}(C)for CH; 0.97 Å and U_{iso}(H) = 1.2U_{eq} (C)for CH_2; 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)for CH_3; and 0.82 Å and U_{iso}(H) = 1.5U_{eq}(C)for OH atoms.

Figures

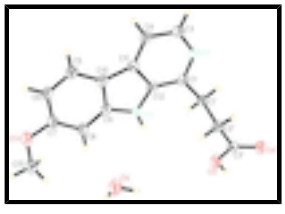

Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

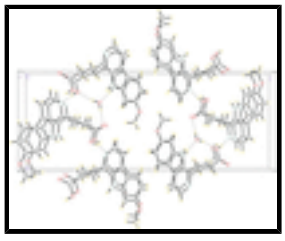

Fig. 2. The packing of the title compound,viewed down the *a* axis. The dashed line indicates the hydrogen bond.

3-(7-methoxy-9*H*-pyrido[3,4-*b*]indol-1-yl)propanoic acid monohydrate

Crystal data

$C_{15}H_{14}N_2O_3 \cdot H_2O$	$F(000) = 608$
$M_r = 288.30$	$D_x = 1.392$ Mg m^{-3}
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Hall symbol: -P 2yn	Cell parameters from 3368 reflections
$a = 4.5114$ (1) Å	$\theta = 3.2$–$67.6°$
$b = 10.8637$ (2) Å	$\mu = 0.85$ mm^{-1}
$c = 28.0865$ (3) Å	$T = 296$ K
$\beta = 92.414$ (1)°	Prism, colourless
$V = 1375.31$ (4) Å^3	0.12 × 0.10 × 0.05 mm
$Z = 4$	

Data collection

Bruker APEXII diffractometer	2048 reflections with $I > 2\sigma(I)$
Radiation source: sealed tube	$R_{int} = 0.022$
graphite	$\theta_{max} = 67.6°$, $\theta_{min} = 3.2°$
Detector resolution: 0 pixels mm^{-1}	$h = -4 \rightarrow 5$
φ and ω scans	$k = -12 \rightarrow 12$
9119 measured reflections	$l = -33 \rightarrow 33$
2366 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
$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.3134P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.006$
2366 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
201 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0014 (3)

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 $F^2 > \sigma(F^2)$ 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}
C1	0.0343 (3)	0.45008 (14)	0.16867 (5)	0.0351 (3)
N2	0.0495 (3)	0.32718 (11)	0.16361 (5)	0.0402 (3)
C3	0.2158 (4)	0.27148 (15)	0.13080 (6)	0.0458 (4)
H3A	0.2177	0.1860	0.1291	0.055*
C4	0.3807 (4)	0.33780 (15)	0.10014 (6)	0.0438 (4)
H4A	0.4925	0.2985	0.0776	0.053*
C5	0.7128 (4)	0.57535 (16)	0.04262 (6)	0.0443 (4)
H5A	0.7804	0.5050	0.0276	0.053*
C6	0.8037 (4)	0.68936 (17)	0.02872 (6)	0.0487 (4)
H6A	0.9324	0.6962	0.0039	0.058*
C7	0.7055 (4)	0.79694 (16)	0.05145 (6)	0.0460 (4)
C8	0.5138 (4)	0.79159 (15)	0.08833 (6)	0.0427 (4)
H8A	0.4502	0.8624	0.1035	0.051*
C9	0.4200 (3)	0.67511 (14)	0.10178 (5)	0.0374 (4)
C10	0.5163 (3)	0.56590 (14)	0.07978 (5)	0.0381 (4)
C11	0.3771 (3)	0.46622 (14)	0.10355 (5)	0.0370 (3)
C12	0.2008 (3)	0.52071 (14)	0.13862 (5)	0.0350 (3)
N13	0.2299 (3)	0.64599 (12)	0.13699 (4)	0.0381 (3)
H13A	0.1437	0.6978	0.1550	0.046*
O14	0.8192 (3)	0.90335 (12)	0.03401 (5)	0.0602 (4)
C15	0.7305 (6)	1.01607 (19)	0.05520 (8)	0.0734 (7)
H15A	0.8250	1.0837	0.0399	0.110*
H15B	0.7877	1.0156	0.0885	0.110*
H15C	0.5191	1.0248	0.0514	0.110*
C1'	−0.1536 (3)	0.50276 (13)	0.20627 (5)	0.0364 (4)
H1'A	−0.3104	0.4449	0.2129	0.044*
H1'B	−0.2458	0.5780	0.1944	0.044*
C2'	0.0251 (4)	0.53087 (15)	0.25274 (6)	0.0437 (4)
H2'A	0.0966	0.4545	0.2670	0.052*
H2'B	0.1957	0.5811	0.2458	0.052*
C3'	−0.1657 (4)	0.59823 (14)	0.28756 (5)	0.0416 (4)
O4'	−0.2512 (4)	0.54636 (12)	0.32330 (4)	0.0655 (4)
O5'	−0.2315 (4)	0.70783 (11)	0.27682 (5)	0.0618 (4)

H5'A	−0.3268	0.7384	0.2981	0.25 (3)*
OW	0.9592 (4)	0.81052 (13)	0.19395 (6)	0.0690 (5)
HWA	0.894 (5)	0.789 (2)	0.2205 (10)	0.081 (8)*
HWB	0.882 (5)	0.883 (3)	0.1889 (9)	0.076 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0370 (8)	0.0361 (8)	0.0322 (7)	−0.0001 (6)	0.0022 (6)	−0.0003 (6)
N2	0.0474 (8)	0.0356 (7)	0.0381 (7)	0.0008 (5)	0.0062 (6)	−0.0012 (5)
C3	0.0572 (10)	0.0358 (8)	0.0450 (9)	0.0031 (7)	0.0082 (7)	−0.0047 (6)
C4	0.0489 (9)	0.0433 (9)	0.0397 (8)	0.0070 (7)	0.0089 (7)	−0.0061 (6)
C5	0.0431 (9)	0.0531 (10)	0.0374 (8)	0.0014 (7)	0.0097 (7)	−0.0048 (7)
C6	0.0482 (10)	0.0627 (11)	0.0363 (8)	−0.0041 (8)	0.0146 (7)	0.0008 (7)
C7	0.0487 (9)	0.0521 (10)	0.0375 (8)	−0.0068 (7)	0.0064 (7)	0.0072 (7)
C8	0.0483 (9)	0.0419 (9)	0.0383 (8)	−0.0002 (7)	0.0084 (7)	0.0012 (6)
C9	0.0384 (8)	0.0422 (8)	0.0318 (7)	0.0006 (6)	0.0049 (6)	0.0006 (6)
C10	0.0376 (8)	0.0443 (9)	0.0326 (7)	0.0012 (6)	0.0039 (6)	−0.0012 (6)
C11	0.0371 (8)	0.0411 (8)	0.0330 (7)	0.0028 (6)	0.0029 (6)	−0.0026 (6)
C12	0.0362 (8)	0.0372 (8)	0.0316 (7)	0.0019 (6)	0.0033 (6)	−0.0009 (6)
N13	0.0440 (7)	0.0350 (7)	0.0362 (6)	0.0021 (5)	0.0120 (5)	−0.0008 (5)
O14	0.0749 (9)	0.0543 (8)	0.0530 (7)	−0.0113 (6)	0.0232 (6)	0.0087 (6)
C15	0.1093 (18)	0.0520 (12)	0.0610 (12)	−0.0196 (11)	0.0274 (12)	0.0019 (9)
C1'	0.0378 (8)	0.0352 (8)	0.0367 (8)	−0.0002 (6)	0.0081 (6)	0.0004 (6)
C2'	0.0472 (9)	0.0451 (9)	0.0389 (8)	0.0062 (7)	0.0038 (7)	−0.0023 (7)
C3'	0.0501 (9)	0.0387 (8)	0.0365 (8)	−0.0028 (6)	0.0061 (7)	−0.0041 (6)
O4'	0.1062 (11)	0.0451 (7)	0.0477 (7)	−0.0034 (7)	0.0302 (7)	−0.0001 (5)
O5'	0.0916 (10)	0.0433 (7)	0.0528 (8)	0.0155 (6)	0.0297 (7)	0.0040 (5)
OW	0.1082 (13)	0.0425 (7)	0.0594 (9)	0.0149 (7)	0.0382 (8)	0.0023 (6)

Geometric parameters (Å, °)

C1—N2	1.3447 (19)	C10—C11	1.431 (2)
C1—C12	1.385 (2)	C11—C12	1.421 (2)
C1—C1'	1.4955 (19)	C12—N13	1.3683 (19)
N2—C3	1.355 (2)	N13—H13A	0.8600
C3—C4	1.367 (2)	O14—C15	1.426 (2)
C3—H3A	0.9300	C15—H15A	0.9600
C4—C11	1.399 (2)	C15—H15B	0.9600
C4—H4A	0.9300	C15—H15C	0.9600
C5—C6	1.367 (2)	C1'—C2'	1.535 (2)
C5—C10	1.401 (2)	C1'—H1'A	0.9700
C5—H5A	0.9300	C1'—H1'B	0.9700
C6—C7	1.412 (2)	C2'—C3'	1.518 (2)
C6—H6A	0.9300	C2'—H2'A	0.9700
C7—O14	1.364 (2)	C2'—H2'B	0.9700
C7—C8	1.378 (2)	C3'—O4'	1.228 (2)
C8—C9	1.392 (2)	C3'—O5'	1.261 (2)
C8—H8A	0.9300	O5'—H5'A	0.8200

C9—N13	1.3728 (19)	OW—HWA	0.85 (3)
C9—C10	1.414 (2)	OW—HWB	0.87 (3)
N2—C1—C12	117.01 (13)	N13—C12—C1	128.81 (13)
N2—C1—C1'	119.18 (13)	N13—C12—C11	109.50 (13)
C12—C1—C1'	123.80 (13)	C1—C12—C11	121.69 (14)
C1—N2—C3	123.20 (13)	C12—N13—C9	108.49 (12)
N2—C3—C4	121.64 (15)	C12—N13—H13A	125.8
N2—C3—H3A	119.2	C9—N13—H13A	125.8
C4—C3—H3A	119.2	C7—O14—C15	117.49 (14)
C3—C4—C11	118.33 (14)	O14—C15—H15A	109.5
C3—C4—H4A	120.8	O14—C15—H15B	109.5
C11—C4—H4A	120.8	H15A—C15—H15B	109.5
C6—C5—C10	119.07 (15)	O14—C15—H15C	109.5
C6—C5—H5A	120.5	H15A—C15—H15C	109.5
C10—C5—H5A	120.5	H15B—C15—H15C	109.5
C5—C6—C7	121.16 (15)	C1—C1'—C2'	112.56 (12)
C5—C6—H6A	119.4	C1—C1'—H1'A	109.1
C7—C6—H6A	119.4	C2'—C1'—H1'A	109.1
O14—C7—C8	124.26 (16)	C1—C1'—H1'B	109.1
O14—C7—C6	114.16 (15)	C2'—C1'—H1'B	109.1
C8—C7—C6	121.58 (15)	H1'A—C1'—H1'B	107.8
C7—C8—C9	116.74 (15)	C3'—C2'—C1'	110.62 (13)
C7—C8—H8A	121.6	C3'—C2'—H2'A	109.5
C9—C8—H8A	121.6	C1'—C2'—H2'A	109.5
N13—C9—C8	127.69 (14)	C3'—C2'—H2'B	109.5
N13—C9—C10	109.52 (13)	C1'—C2'—H2'B	109.5
C8—C9—C10	122.79 (14)	H2'A—C2'—H2'B	108.1
C5—C10—C9	118.65 (14)	O4'—C3'—O5'	123.37 (15)
C5—C10—C11	134.97 (15)	O4'—C3'—C2'	120.80 (15)
C9—C10—C11	106.38 (13)	O5'—C3'—C2'	115.82 (14)
C4—C11—C12	118.13 (14)	C3'—O5'—H5'A	109.5
C4—C11—C10	135.75 (14)	HWA—OW—HWB	104 (2)
C12—C11—C10	106.11 (13)		
C12—C1—N2—C3	−0.5 (2)	C5—C10—C11—C12	−179.98 (18)
C1'—C1—N2—C3	−179.45 (15)	C9—C10—C11—C12	0.48 (17)
C1—N2—C3—C4	−0.2 (3)	N2—C1—C12—N13	−178.44 (14)
N2—C3—C4—C11	0.6 (3)	C1'—C1—C12—N13	0.4 (2)
C10—C5—C6—C7	0.6 (3)	N2—C1—C12—C11	0.8 (2)
C5—C6—C7—O14	179.06 (15)	C1'—C1—C12—C11	179.69 (14)
C5—C6—C7—C8	−0.3 (3)	C4—C11—C12—N13	178.96 (13)
O14—C7—C8—C9	−179.74 (15)	C10—C11—C12—N13	−0.36 (17)
C6—C7—C8—C9	−0.5 (3)	C4—C11—C12—C1	−0.4 (2)
C7—C8—C9—N13	−179.63 (16)	C10—C11—C12—C1	−179.75 (14)
C7—C8—C9—C10	0.9 (2)	C1—C12—N13—C9	179.43 (15)
C6—C5—C10—C9	−0.3 (2)	C11—C12—N13—C9	0.09 (17)
C6—C5—C10—C11	−179.79 (17)	C8—C9—N13—C12	−179.34 (16)
N13—C9—C10—C5	179.93 (14)	C10—C9—N13—C12	0.22 (17)
C8—C9—C10—C5	−0.5 (2)	C8—C7—O14—C15	−0.7 (3)

N13—C9—C10—C11	−0.44 (17)	C6—C7—O14—C15	−179.95 (18)
C8—C9—C10—C11	179.14 (15)	N2—C1—C1'—C2'	95.18 (16)
C3—C4—C11—C12	−0.3 (2)	C12—C1—C1'—C2'	−83.68 (18)
C3—C4—C11—C10	178.79 (18)	C1—C1'—C2'—C3'	173.08 (13)
C5—C10—C11—C4	0.9 (3)	C1'—C2'—C3'—O4'	108.25 (18)
C9—C10—C11—C4	−178.66 (18)	C1'—C2'—C3'—O5'	−70.68 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N13—H13A···OW^{i}	0.86	1.86	2.7224 (18)	180
O5'—H5'A···N2^{ii}	0.82	1.79	2.5965 (18)	169
OW—HWA···O5'^{iii}	0.85 (3)	1.92 (3)	2.7514 (19)	169 (2)
OW—HWB···O4'^{iv}	0.87 (3)	1.89 (3)	2.764 (2)	175 (2)

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

Fig. 1

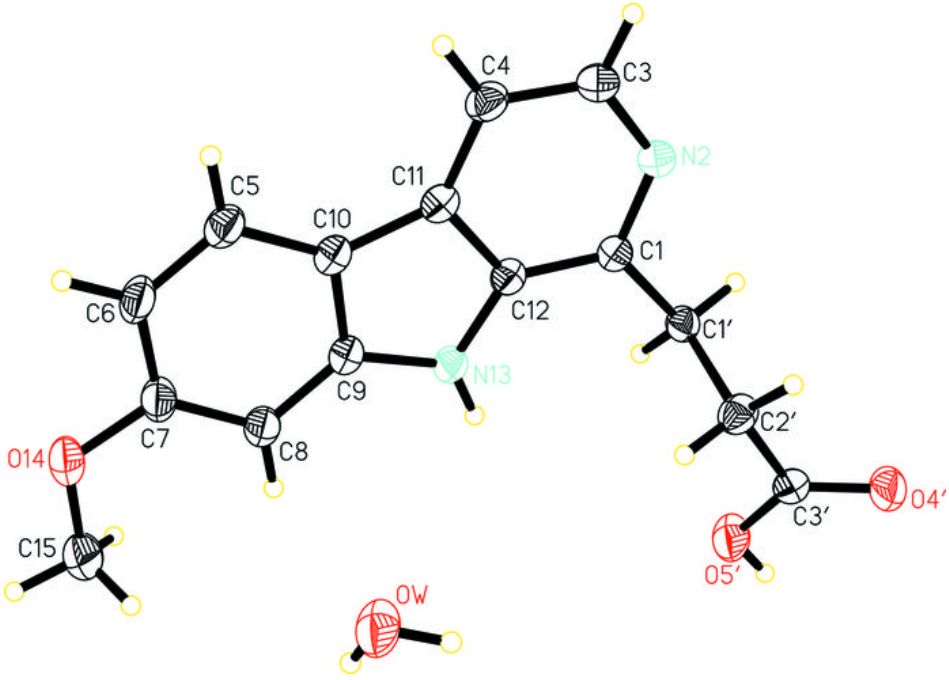

Fig. 2