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(1*R*,2*S*,3*R*,5*S*)-5-(1*H*-Benzimidazol-2-yl)-cyclohexane-1,2,3,5-tetraol monohydrate

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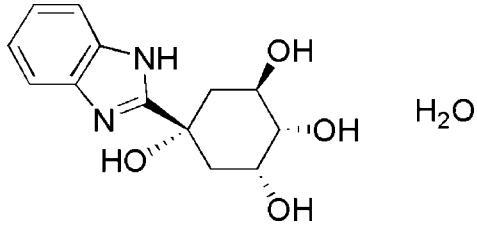
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 6.8.

In the crystal structure of the title compound, $C_{13}H_{16}N_2O_4\cdot H_2O$, intermolecular N—H···O, O—H···O and O—H···N hydrogen bonds form an extensive three-dimensional network, consolidating the crystal packing. The cyclohexane ring adopts a chair conformation.

Related literature

For the crystal structures of related compounds, see: Li *et al.* (1998); Gallagher *et al.* (2001); Howarth & Hanlon (2001); Huang *et al.* (2003); Kazak *et al.* (2006). For ring-puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{13}H_{16}N_2O_4\cdot H_2O$
M_r = 282.29
Orthorhombic, $P2_12_12_1$
a = 8.9684 (14) Å
b = 9.4809 (15) Å
c = 15.278 (4) Å
V = 1299.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.30 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.817, T_{max} = 0.906
12121 measured reflections
1716 independent reflections
1646 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.08
1716 reflections
253 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H6···O1^{i}	0.90 (3)	2.11 (3)	2.983 (2)	164 (2)
O3—H12···O5^{ii}	0.83 (3)	2.00 (3)	2.831 (2)	176 (3)
O4—H13···O2	0.89 (3)	1.87 (3)	2.660 (2)	148 (3)
O2—H14···O5^{iii}	0.84 (3)	1.93 (3)	2.743 (2)	163 (3)
O1—H15···O3^{iv}	0.86 (3)	2.21 (3)	3.066 (2)	172 (3)
O5—H17···O4^{v}	0.88 (3)	1.96 (4)	2.827 (2)	169 (3)
O5—H18···N1	0.93 (4)	1.81 (4)	2.740 (2)	176 (3)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (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: BX2237).

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supplementary materials

Acta Cryst. (2009). E**65**, o2547 [**doi:10.1107/S1600536809037957**]

(1*R*,2*S*,3*R*,5*S*)-5-(1*H*-Benzimidazol-2-yl)cyclohexane-1,2,3,5-tetraol monohydrate

Y. Cai

Comment

It has been generally accepted that benzimidazole systems continue to attract much attention due to applications in : chemical synthesis, structural science, applied biological and coordination chemistry (Gallagher *et al.*, 2001; Huang *et al.*, 2003; Kazak *et al.*, 2006). We report here the crystal structure of the title compound (Fig.1). The cyclohexane ring adopt a chair conformation as shown by the Cremer and Pople (1975) puckering parameters [Q_T= 0.573 (2) Å, θ =174.2 (2) ° and

φ=136 (2)°] and has the same configuration as the cyclohexane ring of (1*S*,3*R*,4*S*,5*R*)-1,3,4,5-tetrahydroxycyclohexane-carboxylic acid, used as started material . The crystal is stabilized by hydrogen bonds of N—H···O, O—H···O and hydrogen bond of O—H ···N, resulting in an extensive three-dimensional network (Fig. 2).

Experimental

(1*S*,3*R*,4*S*,5*R*)-1,3,4,5-tetrahydroxycyclohexanecarboxylic acid (0.02 mol, 3.84 g) and benzene-1,2-diamine (0.02 mol, 2.16 g) were dissolved in 5.5 N HCl (20 ml) at 110°C with stirring for 24 h at 110°C. After the solution was cooled to room temperature, the pH was adjusted to 8–9 with NaOH solution. The product formed was filtered, washed with ethanol and dried. Further purification was done by recrystallization from methanol. Single crystals suitable for X-ray analysis were obtained with about 50% yield.

Refinement

All H atoms were located in a difference Fourier map and refined isotropically; the C-H and O-H bond distances are in the ranges 0.95 (2)-1.04 (3) and 0.83 (3)-0.93 (4) Å; N-H = 0.90 (3) Å. Friedel pairs were merged.

Figures

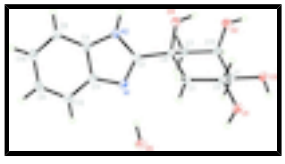

Fig. 1. A view of compound (1) with the atomic numbering scheme. Displacement ellipsoids were drawn at the 30% probability level.

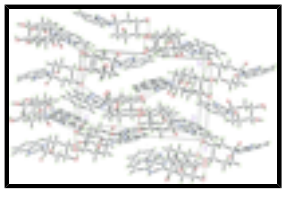

Fig. 2. The crystal packing of the title compound viewed along the *a* axis and all water molecules were omitted for clarity.

(1*R*,2*S*,3*R*,5*S*)-5-(1*H*-Benzimidazol-2- yl)cyclohexane-1,2,3,5-tetraol monohydrate

Crystal data

$C_{13}H_{16}N_2O_4 \cdot H_2O$	$F_{000} = 600$
$M_r = 282.29$	$D_x = 1.443$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 3450 reflections
$a = 8.9684$ (14) Å	$\theta = 2.6$–27.4º
$b = 9.4809$ (15) Å	$\mu = 0.11$ mm^{-1}
$c = 15.278$ (4) Å	$T = 293$ K
$V = 1299.0$ (4) Å^3	Prism, pink
$Z = 4$	0.30 × 0.30 × 0.30 mm

Data collection

Rigaku Mercury CCD diffractometer	1716 independent reflections
Radiation source: fine-focus sealed tube	1646 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
Detector resolution: 13.6612 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 293$ K	$\theta_{min} = 2.6$º
ω scans	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$k = -12 \rightarrow 12$
$T_{min} = 0.817$, $T_{max} = 0.906$	$l = -18 \rightarrow 19$
12121 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2 + 0.2437P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.079$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.08$	$\Delta\rho_{max} = 0.14$ e Å^{-3}
1716 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
253 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008)
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0476 (15)
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 $F^2 > \sigma(F^2)$ 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}
O5	0.72771 (16)	0.24370 (18)	0.64625 (9)	0.0354 (3)
C11	0.6615 (2)	0.3526 (2)	0.86815 (13)	0.0301 (4)
O3	0.07767 (15)	0.25080 (16)	0.51676 (10)	0.0348 (3)
O1	0.31588 (16)	0.45480 (17)	0.50248 (9)	0.0356 (3)
N1	0.57765 (19)	0.33374 (19)	0.79210 (11)	0.0348 (4)
N2	0.43215 (18)	0.42648 (18)	0.89530 (10)	0.0292 (3)
O2	0.01088 (17)	0.3204 (2)	0.69454 (10)	0.0435 (4)
C1	0.1920 (2)	0.2640 (2)	0.58171 (12)	0.0276 (4)
C2	0.4434 (2)	0.3787 (2)	0.81175 (12)	0.0286 (4)
C3	0.3703 (2)	0.4282 (2)	0.65804 (12)	0.0280 (4)
C4	0.5722 (2)	0.41153 (19)	0.93338 (12)	0.0264 (4)
C5	0.2540 (2)	0.4136 (2)	0.58515 (11)	0.0258 (4)
C6	0.3142 (2)	0.3786 (2)	0.74770 (12)	0.0266 (4)
C7	0.2462 (3)	0.2298 (2)	0.74082 (13)	0.0347 (4)
C8	0.6284 (2)	0.4454 (2)	1.01561 (13)	0.0332 (4)
C9	0.8684 (2)	0.3580 (2)	0.96474 (15)	0.0368 (5)
C10	0.7779 (2)	0.4184 (2)	1.02923 (14)	0.0351 (4)
C12	0.8121 (2)	0.3239 (2)	0.88366 (15)	0.0371 (5)
C13	0.1264 (2)	0.2240 (2)	0.66954 (13)	0.0340 (4)
O4	0.20679 (15)	0.47505 (16)	0.78330 (10)	0.0363 (3)
H1	0.272 (2)	0.196 (2)	0.5665 (14)	0.029 (5)*
H2	0.328 (3)	0.161 (3)	0.7259 (17)	0.045 (7)*
H3	0.456 (3)	0.376 (2)	0.6402 (15)	0.033 (6)*
H4	0.976 (3)	0.341 (2)	0.9775 (15)	0.038 (6)*
H5	0.173 (3)	0.477 (2)	0.5973 (13)	0.026 (5)*
H6	0.347 (3)	0.459 (3)	0.9186 (16)	0.042 (7)*
H7	0.824 (3)	0.440 (3)	1.0846 (16)	0.041 (6)*
H8	0.875 (3)	0.290 (3)	0.8364 (19)	0.061 (8)*
H9	0.198 (3)	0.206 (3)	0.8007 (16)	0.042 (6)*
H10	0.401 (3)	0.527 (3)	0.6663 (15)	0.043 (7)*
H11	0.567 (3)	0.488 (3)	1.0588 (18)	0.045 (7)*
H12	0.126 (3)	0.252 (3)	0.4704 (19)	0.058 (9)*
H13	0.119 (3)	0.445 (3)	0.7643 (18)	0.054 (8)*

H14	−0.069 (3)	0.299 (3)	0.6691 (18)	0.055 (8)*
H15	0.393 (3)	0.403 (3)	0.4937 (18)	0.055 (8)*
H16	0.088 (3)	0.123 (2)	0.6650 (14)	0.029 (6)*
H17	0.745 (4)	0.156 (4)	0.661 (2)	0.074 (10)*
H18	0.673 (4)	0.272 (4)	0.695 (2)	0.077 (10)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0314 (7)	0.0459 (9)	0.0290 (7)	0.0014 (7)	0.0037 (6)	−0.0055 (6)
C11	0.0283 (9)	0.0381 (10)	0.0239 (9)	0.0032 (8)	−0.0024 (7)	−0.0007 (7)
O3	0.0279 (7)	0.0504 (8)	0.0260 (7)	−0.0044 (7)	−0.0035 (6)	−0.0059 (7)
O1	0.0302 (7)	0.0533 (9)	0.0235 (7)	−0.0023 (7)	−0.0003 (6)	0.0087 (6)
N1	0.0289 (8)	0.0517 (10)	0.0238 (8)	0.0084 (8)	−0.0033 (7)	−0.0078 (7)
N2	0.0233 (7)	0.0420 (9)	0.0224 (8)	0.0030 (7)	−0.0015 (6)	−0.0036 (7)
O2	0.0236 (7)	0.0740 (11)	0.0330 (8)	−0.0094 (7)	0.0042 (6)	−0.0140 (8)
C1	0.0251 (8)	0.0350 (9)	0.0226 (8)	0.0022 (8)	−0.0019 (7)	−0.0036 (7)
C2	0.0266 (9)	0.0364 (9)	0.0228 (9)	0.0013 (8)	0.0000 (7)	−0.0028 (7)
C3	0.0236 (8)	0.0362 (10)	0.0240 (9)	−0.0015 (8)	−0.0020 (7)	−0.0011 (7)
C4	0.0247 (8)	0.0320 (8)	0.0224 (9)	−0.0004 (7)	−0.0013 (7)	0.0006 (7)
C5	0.0223 (8)	0.0359 (9)	0.0192 (8)	0.0004 (8)	0.0002 (7)	0.0010 (7)
C6	0.0239 (8)	0.0362 (9)	0.0197 (8)	0.0016 (7)	−0.0004 (7)	−0.0047 (7)
C7	0.0413 (11)	0.0391 (10)	0.0239 (9)	−0.0045 (10)	−0.0038 (8)	0.0038 (8)
C8	0.0332 (9)	0.0429 (10)	0.0235 (9)	0.0001 (9)	−0.0019 (8)	−0.0028 (8)
C9	0.0264 (9)	0.0447 (11)	0.0394 (11)	0.0008 (9)	−0.0063 (8)	0.0066 (9)
C10	0.0341 (10)	0.0412 (10)	0.0300 (10)	−0.0045 (9)	−0.0109 (8)	0.0034 (8)
C12	0.0273 (9)	0.0486 (12)	0.0355 (11)	0.0068 (9)	0.0003 (8)	−0.0015 (9)
C13	0.0353 (10)	0.0387 (11)	0.0281 (10)	−0.0111 (9)	−0.0022 (8)	−0.0012 (8)
O4	0.0248 (7)	0.0507 (8)	0.0335 (8)	0.0058 (7)	−0.0027 (6)	−0.0141 (6)

Geometric parameters (Å, °)

O5—H17	0.88 (3)	C3—C6	1.533 (3)
O5—H18	0.93 (4)	C3—H3	0.95 (2)
C11—C4	1.395 (3)	C3—H10	0.98 (3)
C11—N1	1.395 (2)	C4—C8	1.391 (3)
C11—C12	1.398 (3)	C5—H5	0.96 (2)
O3—C1	1.432 (2)	C6—O4	1.435 (2)
O3—H12	0.83 (3)	C6—C7	1.540 (3)
O1—C5	1.434 (2)	C7—C13	1.531 (3)
O1—H15	0.86 (3)	C7—H2	1.01 (3)
N1—C2	1.312 (2)	C7—H9	1.04 (2)
N2—C2	1.358 (2)	C8—C10	1.381 (3)
N2—C4	1.392 (2)	C8—H11	0.95 (3)
N2—H6	0.90 (3)	C9—C12	1.376 (3)
O2—C13	1.434 (3)	C9—C10	1.399 (3)
O2—H14	0.84 (3)	C9—H4	1.00 (2)
C1—C13	1.514 (3)	C10—H7	0.96 (2)
C1—C5	1.524 (3)	C12—H8	0.97 (3)

C1—H1	0.99 (2)	C13—H16	1.02 (2)
C2—C6	1.517 (3)	O4—H13	0.89 (3)
C3—C5	1.532 (2)		
H17—O5—H18	99 (3)	C1—C5—H5	108.0 (13)
C4—C11—N1	109.71 (16)	C3—C5—H5	108.7 (12)
C4—C11—C12	120.72 (19)	O4—C6—C2	105.52 (14)
N1—C11—C12	129.55 (19)	O4—C6—C3	111.29 (16)
C1—O3—H12	102.6 (19)	C2—C6—C3	109.00 (15)
C5—O1—H15	107.0 (19)	O4—C6—C7	110.09 (16)
C2—N1—C11	105.19 (16)	C2—C6—C7	110.33 (15)
C2—N2—C4	106.96 (16)	C3—C6—C7	110.49 (15)
C2—N2—H6	123.3 (16)	C13—C7—C6	111.08 (16)
C4—N2—H6	129.7 (16)	C13—C7—H2	109.1 (15)
C13—O2—H14	110 (2)	C6—C7—H2	108.6 (15)
O3—C1—C13	108.30 (15)	C13—C7—H9	109.1 (15)
O3—C1—C5	111.52 (15)	C6—C7—H9	107.7 (13)
C13—C1—C5	110.17 (15)	H2—C7—H9	111 (2)
O3—C1—H1	107.5 (12)	C10—C8—C4	116.39 (19)
C13—C1—H1	109.1 (12)	C10—C8—H11	122.5 (16)
C5—C1—H1	110.2 (12)	C4—C8—H11	121.1 (16)
N1—C2—N2	113.06 (17)	C12—C9—C10	121.11 (19)
N1—C2—C6	123.58 (17)	C12—C9—H4	119.5 (14)
N2—C2—C6	123.37 (16)	C10—C9—H4	119.4 (14)
C5—C3—C6	113.48 (15)	C8—C10—C9	122.24 (19)
C5—C3—H3	107.0 (14)	C8—C10—H7	120.5 (16)
C6—C3—H3	111.0 (14)	C9—C10—H7	117.3 (16)
C5—C3—H10	111.7 (14)	C9—C12—C11	117.5 (2)
C6—C3—H10	105.6 (14)	C9—C12—H8	122.3 (17)
H3—C3—H10	107.9 (19)	C11—C12—H8	120.0 (17)
C8—C4—N2	132.89 (18)	O2—C13—C1	110.91 (17)
C8—C4—C11	122.02 (17)	O2—C13—C7	107.13 (16)
N2—C4—C11	105.07 (16)	C1—C13—C7	110.40 (16)
O1—C5—C1	111.36 (15)	O2—C13—H16	111.8 (13)
O1—C5—C3	110.63 (14)	C1—C13—H16	107.9 (13)
C1—C5—C3	110.96 (15)	C7—C13—H16	108.7 (13)
O1—C5—H5	107.1 (12)	C6—O4—H13	105.7 (18)
C4—C11—N1—C2	−0.5 (2)	N1—C2—C6—C7	79.5 (2)
C12—C11—N1—C2	−179.1 (2)	N2—C2—C6—C7	−101.0 (2)
C11—N1—C2—N2	0.2 (2)	C5—C3—C6—O4	−71.7 (2)
C11—N1—C2—C6	179.75 (17)	C5—C3—C6—C2	172.34 (15)
C4—N2—C2—N1	0.2 (2)	C5—C3—C6—C7	50.9 (2)
C4—N2—C2—C6	−179.36 (17)	O4—C6—C7—C13	69.8 (2)
C2—N2—C4—C8	178.1 (2)	C2—C6—C7—C13	−174.09 (16)
C2—N2—C4—C11	−0.5 (2)	C3—C6—C7—C13	−53.5 (2)
N1—C11—C4—C8	−178.15 (18)	N2—C4—C8—C10	−178.0 (2)
C12—C11—C4—C8	0.6 (3)	C11—C4—C8—C10	0.4 (3)
N1—C11—C4—N2	0.6 (2)	C4—C8—C10—C9	−1.0 (3)
C12—C11—C4—N2	179.4 (2)	C12—C9—C10—C8	0.7 (3)

O3—C1—C5—O1	−59.24 (19)	C10—C9—C12—C11	0.3 (3)
C13—C1—C5—O1	−179.53 (15)	C4—C11—C12—C9	−0.9 (3)
O3—C1—C5—C3	177.05 (15)	N1—C11—C12—C9	177.6 (2)
C13—C1—C5—C3	56.76 (19)	O3—C1—C13—O2	−64.01 (19)
C6—C3—C5—O1	−177.00 (16)	C5—C1—C13—O2	58.20 (19)
C6—C3—C5—C1	−52.9 (2)	O3—C1—C13—C7	177.41 (16)
N1—C2—C6—O4	−161.58 (19)	C5—C1—C13—C7	−60.4 (2)
N2—C2—C6—O4	17.9 (2)	C6—C7—C13—O2	−61.7 (2)
N1—C2—C6—C3	−42.0 (3)	C6—C7—C13—C1	59.1 (2)
N2—C2—C6—C3	137.55 (19)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H6···O1^{i}	0.90 (3)	2.11 (3)	2.983 (2)	164 (2)
O3—H12···O5^{ii}	0.83 (3)	2.00 (3)	2.831 (2)	176 (3)
O4—H13···O2	0.89 (3)	1.87 (3)	2.660 (2)	148 (3)
O2—H14···O5^{iii}	0.84 (3)	1.93 (3)	2.743 (2)	163 (3)
O1—H15···O3^{iv}	0.86 (3)	2.21 (3)	3.066 (2)	172 (3)
O5—H17···O4^{v}	0.88 (3)	1.96 (4)	2.827 (2)	169 (3)
O5—H18···N1	0.93 (4)	1.81 (4)	2.740 (2)	176 (3)

Symmetry codes: (i) −*x*+1/2, −*y*+1, *z*+1/2; (ii) *x*−1/2, −*y*+1/2, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) *x*+1/2, −*y*+1/2, −*z*+1; (v) −*x*+1, *y*−1/2, −*z*+3/2.

Fig. 1

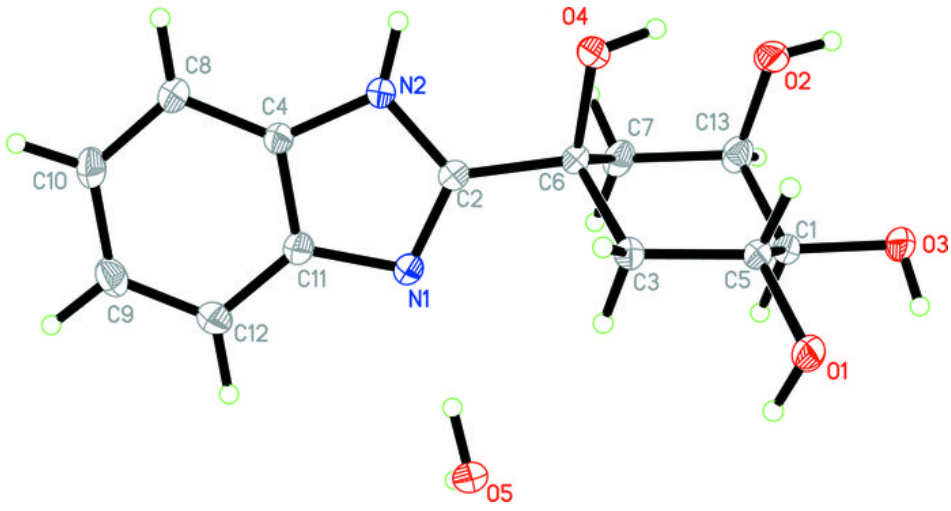

Fig. 2