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2-Hydroxy-10-propargylpyrrolo[2,1-*c*]-[1,4]benzodiazepine-5,11-dione monohydrate

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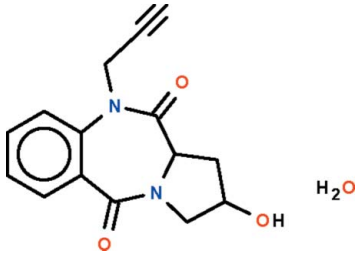
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.094; data-to-parameter ratio = 8.6.

The title compound, $C_{15}H_{14}N_2O_3\cdot H_2O$, consists of a benzodiazepinedione system fused to a pyrrole system. The seven-membered ring adopts a boat-shaped conformation (with the methine C atom as the prow); the five-membered ring adopts an enveloped-shaped conformation (with the hydroxy-bearing C atom as the flap). In the crystal, adjacent molecules are linked by O—H···O hydrogen bonds into sheets parallel to (102). In addition, $C_{acetylinic}$—H···O hydrogen bonds occur.

Related literature

Pyrrolo[2,1-*c*][1,4]benzodiazepines are potent antibiotics produced by *Streptomyces* species; see: Cargill *et al.* (1974). For the design of DNA inter-strand cross-linking and conjugate agents to enhance the sequence selectivity and selectivity for tumor cells, see: Gregson *et al.* (2004).



Experimental

Crystal data

$C_{15}H_{14}N_2O_3\cdot H_2O$
M_r = 288.30
Monoclinic, $P2_1$
a = 6.8977 (1) Å
b = 7.9761 (1) Å
c = 13.0680 (2) Å
β = 99.194 (1)°
V = 709.72 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.3 × 0.3 × 0.3 mm

Data collection

Bruker APEXII diffractometer
9524 measured reflections
1744 independent reflections
1680 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.094
S = 1.10
1744 reflections
202 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1w	0.84 (1)	1.85 (1)	2.686 (2)	177 (3)
O1w—H11···O2^{i}	0.84 (1)	1.92 (1)	2.7485 (19)	172 (4)
O1w—H12···O3^{ii}	0.84 (1)	1.92 (1)	2.767 (2)	177 (3)
C15—H15···O1^{iii}	0.93	2.29	3.166 (3)	157

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

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5199).

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supporting information

Acta Cryst. (2010). E**66**, o731 [doi:10.1107/S1600536810006896]

2-Hydroxy-10-propargylpyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione monohydrate

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S1. Experimental

2-Hydroxy-pyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione (0.5 g, 2.15 mmol), propargyl bromide (0.26 g, 2.15 mmol) and potassium carbonate (0.6 g, 4.3 mmol) along with a catalytic amount of tetra-*n*-butyl ammonium bromide were stirred in *N*,*N*-dimethylformamide (20 ml) for 72 h. The solid material was removed by filtration and the solvent evaporated under vacuum. The residue was separated by chromatography on silica gel with an *n*-hexane:ethyl acetate (1:9) solvent system. The compound was obtained as colorless crystals in 50% yield upon evaporation of the solvent.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93-0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The oxygen-bound H-atoms were located in a difference Fourier map, and were refined isotropically with a distance restraint of O–H 0.84±0.01 Å. Due to the absence of anomalous scatterers Friedel pairs were merged and the absolute configuration was arbitrarily set.

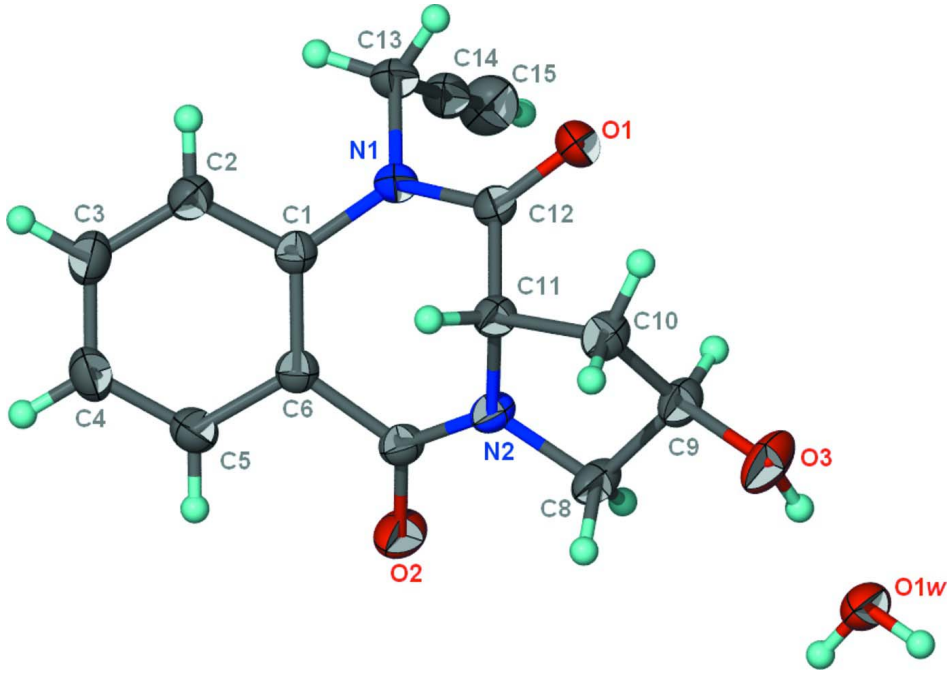

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{15}H_{14}N_2O_3\cdot H_2O$ at the 50% probability level; hydrogen atoms are drawn as spheres of an arbitrary radius.

2-Hydroxy-10-propargylpyrrolo[2,1-*c*][1,4]benzodiazepine-5,11-dione monohydrate

Crystal data

$C_{15}H_{14}N_2O_3 \cdot H_2O$
$M_r = 288.30$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.8977$ (1) Å
$b = 7.9761$ (1) Å
$c = 13.0680$ (2) Å
$\beta = 99.194$ (1)°
$V = 709.72$ (2) Å^3
$Z = 2$
$F(000) = 304$
$D_x = 1.349$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6455 reflections
$\theta = 2.5$–$34.3°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colorless
0.3 × 0.3 × 0.3 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
9524 measured reflections
1744 independent reflections
1680 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.094$
$S = 1.10$
1744 reflections
202 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.0541P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2007 (2)	0.5000 (2)	0.37937 (11)	0.0464 (4)
O2	0.0918 (2)	1.01924 (19)	0.13444 (13)	0.0503 (4)
O3	0.4060 (2)	0.4426 (2)	0.06723 (14)	0.0561 (4)
O1W	0.6986 (2)	0.6295 (2)	0.01457 (11)	0.0465 (4)
N1	−0.0173 (2)	0.7109 (2)	0.37874 (10)	0.0320 (3)
N2	0.1101 (2)	0.74500 (19)	0.17437 (11)	0.0317 (3)
C1	−0.1606 (2)	0.8238 (2)	0.32657 (12)	0.0301 (3)
C2	−0.3329 (2)	0.8493 (3)	0.36900 (14)	0.0377 (4)
H2	−0.3559	0.7850	0.4252	0.045*
C3	−0.4683 (3)	0.9684 (3)	0.32844 (16)	0.0456 (5)
H3A	−0.5816	0.9835	0.3576	0.055*
C4	−0.4380 (3)	1.0654 (3)	0.24530 (15)	0.0466 (5)
H4	−0.5281	1.1478	0.2194	0.056*

C5	−0.2715 (3)	1.0388 (3)	0.20077 (14)	0.0410 (4)
H5	−0.2512	1.1033	0.1442	0.049*
C6	−0.1333 (2)	0.9169 (2)	0.23914 (12)	0.0311 (3)
C7	0.0340 (3)	0.8983 (2)	0.17963 (13)	0.0330 (3)
C8	0.2643 (3)	0.7095 (2)	0.11252 (15)	0.0392 (4)
H8A	0.3734	0.7867	0.1290	0.047*
H8B	0.2141	0.7161	0.0389	0.047*
C9	0.3253 (3)	0.5312 (2)	0.14444 (15)	0.0380 (4)
H9	0.4195	0.5329	0.2091	0.046*
C10	0.1339 (3)	0.4509 (2)	0.16327 (16)	0.0387 (4)
H10A	0.1590	0.3553	0.2094	0.046*
H10B	0.0562	0.4146	0.0986	0.046*
C11	0.0298 (2)	0.5904 (2)	0.21314 (12)	0.0308 (3)
H11A	−0.1126	0.5841	0.1909	0.037*
C12	0.0799 (2)	0.5931 (2)	0.33103 (12)	0.0319 (3)
C13	0.0327 (3)	0.7196 (3)	0.49276 (13)	0.0362 (4)
H13A	−0.0758	0.7703	0.5204	0.043*
H13B	0.0502	0.6067	0.5204	0.043*
C14	0.2105 (3)	0.8159 (3)	0.52714 (13)	0.0402 (4)
C15	0.3548 (3)	0.8909 (4)	0.55819 (18)	0.0568 (6)
H15	0.4689	0.9501	0.5827	0.068*
H3	0.498 (3)	0.499 (4)	0.049 (2)	0.065 (8)*
H11	0.760 (4)	0.587 (4)	−0.0291 (19)	0.065 (8)*
H12	0.663 (4)	0.725 (2)	−0.009 (2)	0.058 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0455 (7)	0.0516 (9)	0.0432 (7)	0.0169 (7)	0.0107 (6)	0.0127 (6)
O2	0.0618 (9)	0.0320 (7)	0.0644 (9)	0.0021 (7)	0.0322 (7)	0.0110 (7)
O3	0.0670 (10)	0.0393 (8)	0.0728 (11)	−0.0023 (8)	0.0442 (9)	−0.0149 (8)
O1W	0.0522 (8)	0.0481 (9)	0.0430 (7)	0.0059 (7)	0.0196 (6)	0.0032 (6)
N1	0.0346 (7)	0.0360 (8)	0.0258 (6)	0.0029 (6)	0.0058 (5)	0.0028 (6)
N2	0.0376 (7)	0.0276 (7)	0.0324 (7)	−0.0025 (6)	0.0138 (6)	−0.0006 (5)
C1	0.0315 (7)	0.0298 (7)	0.0288 (7)	0.0000 (6)	0.0046 (6)	−0.0032 (6)
C2	0.0358 (8)	0.0443 (10)	0.0344 (8)	0.0016 (8)	0.0098 (6)	0.0005 (7)
C3	0.0353 (9)	0.0591 (13)	0.0434 (9)	0.0101 (9)	0.0097 (7)	−0.0037 (9)
C4	0.0435 (9)	0.0510 (12)	0.0439 (9)	0.0178 (9)	0.0024 (7)	0.0002 (9)
C5	0.0481 (10)	0.0391 (10)	0.0360 (8)	0.0083 (8)	0.0072 (7)	0.0042 (8)
C6	0.0341 (7)	0.0290 (8)	0.0305 (7)	0.0003 (6)	0.0060 (6)	−0.0019 (6)
C7	0.0377 (8)	0.0304 (8)	0.0324 (7)	−0.0006 (7)	0.0106 (6)	0.0011 (6)
C8	0.0488 (10)	0.0324 (9)	0.0416 (9)	−0.0030 (8)	0.0233 (8)	−0.0029 (7)
C9	0.0418 (9)	0.0337 (9)	0.0419 (9)	0.0012 (7)	0.0169 (7)	−0.0049 (7)
C10	0.0466 (9)	0.0272 (8)	0.0455 (10)	−0.0029 (7)	0.0169 (8)	−0.0051 (7)
C11	0.0339 (7)	0.0262 (7)	0.0334 (7)	−0.0019 (6)	0.0089 (6)	−0.0001 (6)
C12	0.0307 (7)	0.0324 (8)	0.0338 (7)	−0.0001 (7)	0.0086 (6)	0.0043 (7)
C13	0.0412 (9)	0.0408 (9)	0.0265 (7)	0.0014 (8)	0.0054 (6)	0.0031 (7)
C14	0.0420 (9)	0.0468 (11)	0.0313 (7)	0.0063 (8)	0.0040 (7)	0.0016 (8)

C15	0.0472 (10)	0.0728 (16)	0.0483 (11)	−0.0071 (12)	0.0014 (8)	−0.0078 (12)

Geometric parameters (Å, º)

O1—C12	1.215 (2)	C4—H4	0.9300
O2—C7	1.230 (2)	C5—C6	1.398 (2)
O3—C9	1.416 (2)	C5—H5	0.9300
O3—H3	0.838 (10)	C6—C7	1.498 (2)
O1W—H11	0.835 (10)	C8—C9	1.523 (3)
O1W—H12	0.843 (10)	C8—H8A	0.9700
N1—C12	1.362 (2)	C8—H8B	0.9700
N1—C1	1.428 (2)	C9—C10	1.522 (3)
N1—C13	1.476 (2)	C9—H9	0.9800
N2—C7	1.336 (2)	C10—C11	1.526 (2)
N2—C8	1.463 (2)	C10—H10A	0.9700
N2—C11	1.474 (2)	C10—H10B	0.9700
C1—C6	1.400 (2)	C11—C12	1.524 (2)
C1—C2	1.404 (2)	C11—H11A	0.9800
C2—C3	1.377 (3)	C13—C14	1.456 (3)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.377 (3)	C13—H13B	0.9700
C3—H3A	0.9300	C14—C15	1.176 (3)
C4—C5	1.384 (3)	C15—H15	0.9300
C9—O3—H3	109 (2)	N2—C8—H8B	111.2
H11—O1W—H12	106 (3)	C9—C8—H8B	111.2
C12—N1—C1	124.80 (13)	H8A—C8—H8B	109.1
C12—N1—C13	116.23 (14)	O3—C9—C10	110.79 (16)
C1—N1—C13	118.96 (14)	O3—C9—C8	113.19 (16)
C7—N2—C8	122.10 (15)	C10—C9—C8	103.21 (15)
C7—N2—C11	125.15 (13)	O3—C9—H9	109.8
C8—N2—C11	111.97 (14)	C10—C9—H9	109.8
C6—C1—C2	118.58 (15)	C8—C9—H9	109.8
C6—C1—N1	123.42 (14)	C9—C10—C11	103.99 (14)
C2—C1—N1	117.90 (14)	C9—C10—H10A	111.0
C3—C2—C1	120.83 (17)	C11—C10—H10A	111.0
C3—C2—H2	119.6	C9—C10—H10B	111.0
C1—C2—H2	119.6	C11—C10—H10B	111.0
C4—C3—C2	120.81 (17)	H10A—C10—H10B	109.0
C4—C3—H3A	119.6	N2—C11—C12	107.39 (14)
C2—C3—H3A	119.6	N2—C11—C10	103.60 (12)
C3—C4—C5	119.09 (19)	C12—C11—C10	113.32 (15)
C3—C4—H4	120.5	N2—C11—H11A	110.8
C5—C4—H4	120.5	C12—C11—H11A	110.8
C4—C5—C6	121.35 (17)	C10—C11—H11A	110.8
C4—C5—H5	119.3	O1—C12—N1	122.04 (15)
C6—C5—H5	119.3	O1—C12—C11	122.86 (16)
C5—C6—C1	119.23 (15)	N1—C12—C11	115.08 (14)

C5—C6—C7	114.86 (15)	C14—C13—N1	112.68 (14)
C1—C6—C7	125.91 (15)	C14—C13—H13A	109.1
O2—C7—N2	122.21 (15)	N1—C13—H13A	109.1
O2—C7—C6	120.45 (16)	C14—C13—H13B	109.1
N2—C7—C6	117.27 (15)	N1—C13—H13B	109.1
N2—C8—C9	102.88 (14)	H13A—C13—H13B	107.8
N2—C8—H8A	111.2	C15—C14—C13	177.6 (2)
C9—C8—H8A	111.2	C14—C15—H15	180.0
C12—N1—C1—C6	−47.5 (2)	C7—N2—C8—C9	171.01 (16)
C13—N1—C1—C6	132.32 (17)	C11—N2—C8—C9	−18.65 (19)
C12—N1—C1—C2	136.14 (18)	N2—C8—C9—O3	154.01 (16)
C13—N1—C1—C2	−44.0 (2)	N2—C8—C9—C10	34.20 (19)
C6—C1—C2—C3	−2.8 (3)	O3—C9—C10—C11	−159.11 (16)
N1—C1—C2—C3	173.70 (18)	C8—C9—C10—C11	−37.66 (19)
C1—C2—C3—C4	0.0 (3)	C7—N2—C11—C12	−74.4 (2)
C2—C3—C4—C5	1.8 (3)	C8—N2—C11—C12	115.58 (16)
C3—C4—C5—C6	−0.6 (3)	C7—N2—C11—C10	165.40 (18)
C4—C5—C6—C1	−2.2 (3)	C8—N2—C11—C10	−4.59 (18)
C4—C5—C6—C7	177.42 (19)	C9—C10—C11—N2	26.07 (18)
C2—C1—C6—C5	3.9 (2)	C9—C10—C11—C12	−89.97 (17)
N1—C1—C6—C5	−172.45 (16)	C1—N1—C12—O1	−179.99 (17)
C2—C1—C6—C7	−175.72 (16)	C13—N1—C12—O1	0.2 (2)
N1—C1—C6—C7	8.0 (3)	C1—N1—C12—C11	1.6 (2)
C8—N2—C7—O2	−1.4 (3)	C13—N1—C12—C11	−178.26 (14)
C11—N2—C7—O2	−170.47 (17)	N2—C11—C12—O1	−109.22 (19)
C8—N2—C7—C6	175.64 (16)	C10—C11—C12—O1	4.6 (2)
C11—N2—C7—C6	6.6 (3)	N2—C11—C12—N1	69.19 (18)
C5—C6—C7—O2	30.1 (3)	C10—C11—C12—N1	−177.03 (14)
C1—C6—C7—O2	−150.25 (19)	C12—N1—C13—C14	81.9 (2)
C5—C6—C7—N2	−146.98 (17)	C1—N1—C13—C14	−97.99 (19)
C1—C6—C7—N2	32.6 (3)	N1—C13—C14—C15	−158 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1w	0.84 (1)	1.85 (1)	2.686 (2)	177 (3)
O1w—H11···O2^{i}	0.84 (1)	1.92 (1)	2.7485 (19)	172 (4)
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Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*; (ii) −*x*+1, *y*+1/2, −*z*; (iii) −*x*+1, *y*+1/2, −*z*+1.