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10,10′-Methylenebis[2,3-dihydro-1*H*-benzo[e]pyrrolo[1,2-*a*][1,4]diazepine-5,11(10*H*,11a*H*)-dione] dihydrate

Hanane Benzeid,[a] Nathalie Saffon,[b] Bernard Garrigues,[c] El Mokhtar Essassi[a] and Seik Weng Ng[d]*

[a]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, [b]Service Commun Rayons X, Université Paul Sabatier, Bâtiment 2R1, 118 route de Narbonne, 31062 Toulouse, France, [c]Hétérochimie Fondamentale et Appliquée, Université Paul Sabatier, UMR 5069, 118 Route de Narbonne, 31062 Toulouse, France, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

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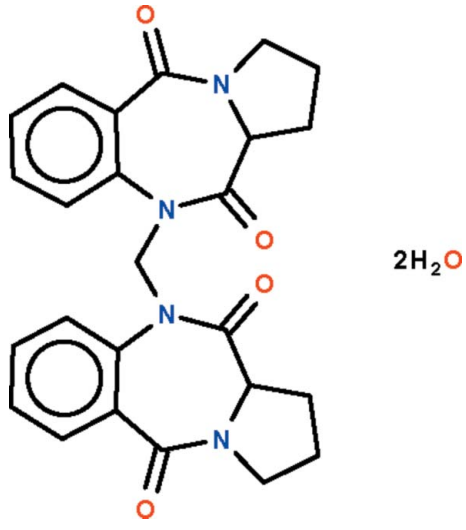
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.126; data-to-parameter ratio = 9.9.

The organic molecule and uncoordinated water molecule in the crystal of the title compound, $C_{25}H_{24}N_4O_4\cdot 2H_2O$, both lie on special positions of twofold symmetry. A twofold rotation axis passes through the methylene C atom connecting the two dihydrobenzopyrrolodiazepindionyl parts. The seven-membered C_5N_2 ring adopts a boat conformation.

Related literature

Pyrrolo[2,1-*c*][1,4]benzodiazepines are a group of potent chemicals produced by *Streptomyces* species. For their anti-cancer activity, see: Bose *et al.* (1992); Cargill *et al.* (1974); Gregson *et al.* (2004).



Experimental

Crystal data

$C_{25}H_{24}N_4O_4\cdot 2H_2O$
M_r = 480.51
Trigonal, $P3_121$
a = 11.9901 (2) Å
c = 13.6054 (2) Å
V = 1693.90 (4) Å^3
Z = 3
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 193 K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: none
23041 measured reflections
1572 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.18
1572 reflections
159 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2545).

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supporting information

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10,10′-Methylenebis[2,3-dihydro-1*H*-benzo[e]pyrrolo[1,2-*a*][1,4]diazepine-5,11(10*H*,11a*H*)-dione] dihydrate

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S1. Experimental

2,3-Dihydro-1*H*-benzo[*e*]pyrrolo[1,2-*a*][1,4]diazepine-5,11(10*H*,11a*H*)-dione (1.1 g, 6 mmol), dibromomethane (1.8 ml, 2.54 mmol), potassium carbonate (0.71 g, 6 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide were stirred in *N*,*N*-dimethylformamide (20 ml) for 24 h. The insoluble salts were filtered off and the solvent was removed under vacuum. The residue was separated by chromatography on silica gel with an *n*-hexane:ethyl acetate (8:2) solvent system. The compound was obtained as colorless crystals in 70% yield upon evaporation of the solvent.

S2. 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_{iso}(H) set to 1.2U_{eq}(C). The location of the water-bound H-atoms were ambiguous owing to disorder. The H1w1 atom was placed in a chemically sensible position on the basis of its hydrogen bonding to the O2 atom but was not refined. The water molecules are arranged along the c-axis with O1w···O1w separations of 2.741 (5) and 2.757 (5) Å, indicative of hydrogen bonding interactions. The assigned position for the second hydrogen atom (O-H = 0.84 Å), with full occupancy represents an intermediate position.

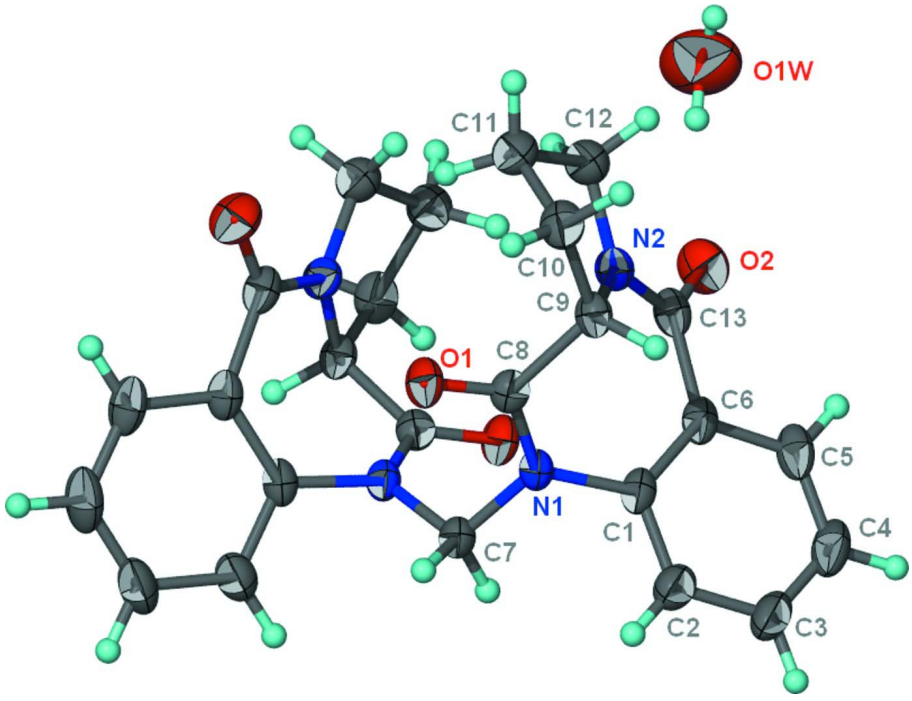

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{25}H_{24}N_4O_4{\cdot}H_2O$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. Unlabelled atoms are related by a twofold axis.

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Crystal data

$C_{25}H_{24}N_4O_4{\cdot}2H_2O$
M_r = 480.51
Trigonal, $P3_121$
Hall symbol: P 31 2"
a = 11.9901 (2) Å
c = 13.6054 (2) Å
V = 1693.90 (4) Å^3
Z = 3
F(000) = 762
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9284 reflections
θ = 5.0–28.3°
μ = 0.10 mm^{-1}
T = 193 K
Block, colorless
0.20 × 0.20 × 0.10 mm

Data collection

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
23041 measured reflections
1572 independent reflections
1472 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°, θ_{min} = 5.2°
h = −13→0
k = 0→15
l = 0→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.126$
$S = 1.18$
1572 reflections
159 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.3837P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}
Absolute structure: nd

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.57170 (18)	0.14709 (17)	0.22350 (11)	0.0274 (4)	
O2	0.7198 (2)	0.0623 (2)	0.57106 (13)	0.0362 (5)	
O1W	0.9339 (4)	0.0162 (4)	0.5795 (2)	0.0819 (10)	
H1W1	0.8758	0.0349	0.5692	0.123*	
H1W2	1.0069	0.0798	0.5645	0.123*	
N1	0.47771 (19)	0.1028 (2)	0.37359 (13)	0.0219 (4)	
N2	0.7341 (2)	0.16144 (19)	0.42630 (14)	0.0235 (4)	
C1	0.4814 (2)	0.1364 (2)	0.47532 (15)	0.0233 (5)	
C2	0.3891 (3)	0.1666 (3)	0.50682 (18)	0.0303 (5)	
H2	0.3310	0.1689	0.4606	0.036*	
C3	0.3812 (3)	0.1934 (3)	0.60537 (19)	0.0361 (6)	
H3	0.3195	0.2160	0.6261	0.043*	
C4	0.4645 (3)	0.1867 (3)	0.67300 (18)	0.0382 (7)	
H4	0.4581	0.2019	0.7407	0.046*	
C5	0.5571 (3)	0.1577 (2)	0.64133 (16)	0.0303 (6)	
H5	0.6133	0.1529	0.6882	0.036*	
C6	0.5699 (2)	0.1355 (2)	0.54191 (16)	0.0243 (5)	
C7	0.3561 (3)	0.0000	0.3333	0.0239 (6)	
H7A	0.2900	−0.0353	0.3858	0.029*	0.50
H7B	0.3253	0.0353	0.2809	0.029*	0.50
C8	0.5813 (2)	0.1646 (2)	0.31219 (16)	0.0224 (5)	
C9	0.7077 (2)	0.2487 (2)	0.36580 (15)	0.0225 (5)	
H9	0.7020	0.3141	0.4078	0.027*	
C10	0.8238 (2)	0.3128 (3)	0.29721 (17)	0.0296 (5)	
H10A	0.8938	0.3930	0.3265	0.036*	
H10B	0.7997	0.3332	0.2329	0.036*	
C11	0.8645 (3)	0.2109 (3)	0.28641 (18)	0.0320 (6)	
H11A	0.9549	0.2499	0.2641	0.038*	
H11B	0.8080	0.1425	0.2397	0.038*	
C12	0.8482 (3)	0.1579 (3)	0.39173 (18)	0.0317 (6)	
H12A	0.8334	0.0690	0.3918	0.038*	
H12B	0.9245	0.2133	0.4327	0.038*	
C13	0.6801 (2)	0.1157 (2)	0.51428 (16)	0.0251 (5)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0330 (9)	0.0290 (9)	0.0170 (7)	0.0132 (8)	−0.0008 (6)	0.0000 (6)
O2	0.0427 (11)	0.0404 (11)	0.0284 (9)	0.0229 (9)	−0.0039 (8)	0.0068 (8)
O1W	0.082 (2)	0.126 (3)	0.0681 (18)	0.075 (2)	−0.0051 (16)	0.0110 (19)
N1	0.0235 (9)	0.0246 (9)	0.0173 (8)	0.0117 (8)	−0.0014 (7)	−0.0033 (7)
N2	0.0260 (10)	0.0253 (10)	0.0196 (8)	0.0131 (8)	−0.0018 (7)	0.0005 (7)
C1	0.0273 (11)	0.0201 (10)	0.0184 (9)	0.0088 (9)	0.0018 (9)	−0.0020 (8)
C2	0.0304 (13)	0.0304 (12)	0.0296 (12)	0.0148 (11)	0.0025 (9)	−0.0054 (10)
C3	0.0323 (13)	0.0361 (14)	0.0334 (13)	0.0122 (12)	0.0061 (11)	−0.0124 (11)
C4	0.0365 (15)	0.0363 (14)	0.0242 (11)	0.0050 (12)	0.0091 (11)	−0.0074 (10)
C5	0.0330 (13)	0.0259 (12)	0.0178 (10)	0.0042 (10)	0.0009 (9)	0.0005 (9)
C6	0.0267 (11)	0.0209 (10)	0.0181 (10)	0.0064 (9)	0.0014 (8)	−0.0009 (8)
C7	0.0248 (11)	0.0276 (16)	0.0203 (13)	0.0138 (8)	−0.0021 (6)	−0.0041 (12)
C8	0.0271 (11)	0.0212 (10)	0.0206 (10)	0.0134 (9)	−0.0005 (8)	0.0010 (8)
C9	0.0271 (11)	0.0212 (10)	0.0173 (9)	0.0106 (9)	−0.0013 (8)	0.0014 (8)
C10	0.0287 (12)	0.0278 (12)	0.0247 (10)	0.0084 (10)	0.0034 (9)	0.0017 (9)
C11	0.0294 (12)	0.0387 (14)	0.0277 (12)	0.0168 (11)	0.0022 (10)	−0.0014 (11)
C12	0.0299 (13)	0.0382 (14)	0.0287 (12)	0.0183 (11)	−0.0029 (10)	−0.0031 (10)
C13	0.0293 (12)	0.0227 (11)	0.0193 (9)	0.0099 (9)	−0.0037 (9)	0.0002 (8)

Geometric parameters (Å, º)

O1—C8	1.220 (3)	C5—C6	1.402 (3)
O2—C13	1.240 (3)	C5—H5	0.9500
O1W—H1W1	0.8429	C6—C13	1.502 (4)
O1W—H1W2	0.8500	C7—N1^{i}	1.466 (3)
N1—C8	1.367 (3)	C7—H7A	0.9900
N1—C1	1.436 (3)	C7—H7B	0.9900
N1—C7	1.466 (3)	C8—C9	1.522 (3)
N2—C13	1.341 (3)	C9—C10	1.526 (3)
N2—C12	1.466 (3)	C9—H9	1.0000
N2—C9	1.485 (3)	C10—C11	1.533 (4)
C1—C2	1.393 (3)	C10—H10A	0.9900
C1—C6	1.399 (3)	C10—H10B	0.9900
C2—C3	1.393 (3)	C11—C12	1.540 (3)
C2—H2	0.9500	C11—H11A	0.9900
C3—C4	1.389 (4)	C11—H11B	0.9900
C3—H3	0.9500	C12—H12A	0.9900
C4—C5	1.387 (4)	C12—H12B	0.9900
C4—H4	0.9500		
H1W1—O1W—H1W2	109.8	O1—C8—N1	121.9 (2)
C8—N1—C1	122.97 (19)	O1—C8—C9	124.4 (2)
C8—N1—C7	118.68 (17)	N1—C8—C9	113.59 (18)
C1—N1—C7	118.32 (17)	N2—C9—C8	106.93 (18)
C13—N2—C12	122.8 (2)	N2—C9—C10	103.41 (19)

C13—N2—C9	123.6 (2)	C8—C9—C10	113.32 (18)
C12—N2—C9	111.90 (18)	N2—C9—H9	111.0
C2—C1—C6	120.4 (2)	C8—C9—H9	111.0
C2—C1—N1	116.9 (2)	C10—C9—H9	111.0
C6—C1—N1	122.6 (2)	C9—C10—C11	103.3 (2)
C1—C2—C3	120.7 (2)	C9—C10—H10A	111.1
C1—C2—H2	119.6	C11—C10—H10A	111.1
C3—C2—H2	119.6	C9—C10—H10B	111.1
C4—C3—C2	119.4 (3)	C11—C10—H10B	111.1
C4—C3—H3	120.3	H10A—C10—H10B	109.1
C2—C3—H3	120.3	C10—C11—C12	102.4 (2)
C5—C4—C3	119.8 (2)	C10—C11—H11A	111.3
C5—C4—H4	120.1	C12—C11—H11A	111.3
C3—C4—H4	120.1	C10—C11—H11B	111.3
C4—C5—C6	121.6 (2)	C12—C11—H11B	111.3
C4—C5—H5	119.2	H11A—C11—H11B	109.2
C6—C5—H5	119.2	N2—C12—C11	102.4 (2)
C1—C6—C5	117.9 (2)	N2—C12—H12A	111.3
C1—C6—C13	124.7 (2)	C11—C12—H12A	111.3
C5—C6—C13	117.4 (2)	N2—C12—H12B	111.3
N1^{i}—C7—N1	109.8 (3)	C11—C12—H12B	111.3
N1^{i}—C7—H7A	109.7	H12A—C12—H12B	109.2
N1—C7—H7A	109.7	O2—C13—N2	122.4 (2)
N1^{i}—C7—H7B	109.7	O2—C13—C6	121.4 (2)
N1—C7—H7B	109.7	N2—C13—C6	116.2 (2)
H7A—C7—H7B	108.2		

C8—N1—C1—C2	124.6 (2)	C12—N2—C9—C8	113.4 (2)
C7—N1—C1—C2	−53.5 (3)	C13—N2—C9—C10	158.9 (2)
C8—N1—C1—C6	−57.4 (3)	C12—N2—C9—C10	−6.5 (2)
C7—N1—C1—C6	124.5 (2)	O1—C8—C9—N2	−112.1 (2)
C6—C1—C2—C3	−1.6 (4)	N1—C8—C9—N2	64.2 (2)
N1—C1—C2—C3	176.4 (2)	O1—C8—C9—C10	1.1 (3)
C1—C2—C3—C4	−1.6 (4)	N1—C8—C9—C10	177.5 (2)
C2—C3—C4—C5	2.1 (4)	N2—C9—C10—C11	29.1 (2)
C3—C4—C5—C6	0.4 (4)	C8—C9—C10—C11	−86.3 (2)
C2—C1—C6—C5	4.0 (3)	C9—C10—C11—C12	−40.6 (2)
N1—C1—C6—C5	−173.9 (2)	C13—N2—C12—C11	175.9 (2)
C2—C1—C6—C13	−174.4 (2)	C9—N2—C12—C11	−18.6 (3)
N1—C1—C6—C13	7.7 (4)	C10—C11—C12—N2	35.9 (2)
C4—C5—C6—C1	−3.4 (4)	C12—N2—C13—O2	−3.0 (4)
C4—C5—C6—C13	175.1 (2)	C9—N2—C13—O2	−166.9 (2)
C8—N1—C7—N1^{i}	59.77 (17)	C12—N2—C13—C6	175.5 (2)
C1—N1—C7—N1^{i}	−122.0 (2)	C9—N2—C13—C6	11.7 (3)
C1—N1—C8—O1	−169.7 (2)	C1—C6—C13—O2	−148.6 (2)
C7—N1—C8—O1	8.4 (3)	C5—C6—C13—O2	33.0 (3)
C1—N1—C8—C9	13.9 (3)	C1—C6—C13—N2	32.9 (3)

C7—N1—C8—C9	−168.02 (19)	C5—C6—C13—N2	−145.5 (2)
C13—N2—C9—C8	−81.2 (3)		

Symmetry code: (i) *x*−*y*, −*y*, −*z*+2/3.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1w—H1w1···O2	0.84	2.06	2.886 (4)	168