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3-Bromo-1-methyl-6,7-dihydropyrrolo-[2,3-*c*]azepine-4,8(1*H*,5*H*)-dione

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The title compound, $C_9H_9BrN_2O_2$, was synthesized by the cyclization of 3-(4-bromo-1-methylpyrrole-2-carboxamido)-propanoic acid in the presence of phosphorus oxychloride in 67.3% yield. In the crystal structure, intermolecular hydrogen-bond interactions link the molecules into two-dimensional sheets.

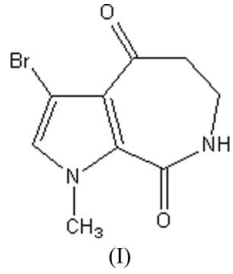
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.094
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Pyrrole derivatives are well known in many marine organisms (Faulkner, 2001) and some are bioactive substances (Tasdemir *et al.*, 2002). In our search for bioactive compounds, a series of 6,7-dihydropyrrolo[2,3-*c*]azepine-4,8(1*H*,5*H*)-diones, including the title compound, (I), has been synthesized by the cyclization of 3-(pyrrole-2-carboxamido)propanoic acids. Here, the crystal structure of (I) is reported.



(I)

The bond lengths and angles of (I) are unexceptional and are in good agreement with corresponding parameters in aldisin (Zeng *et al.*, 1991) and 1-methylaldisin (Zeng *et al.*,

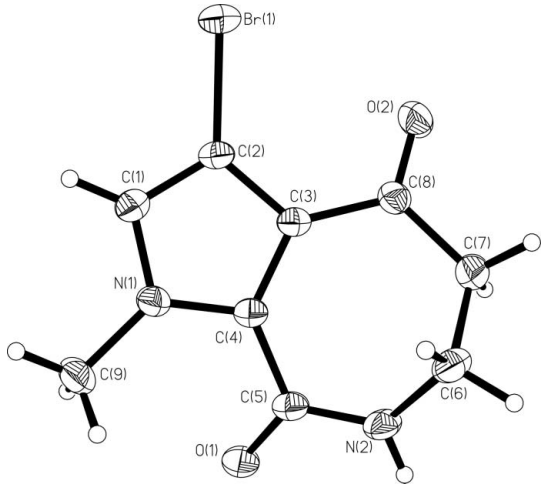


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

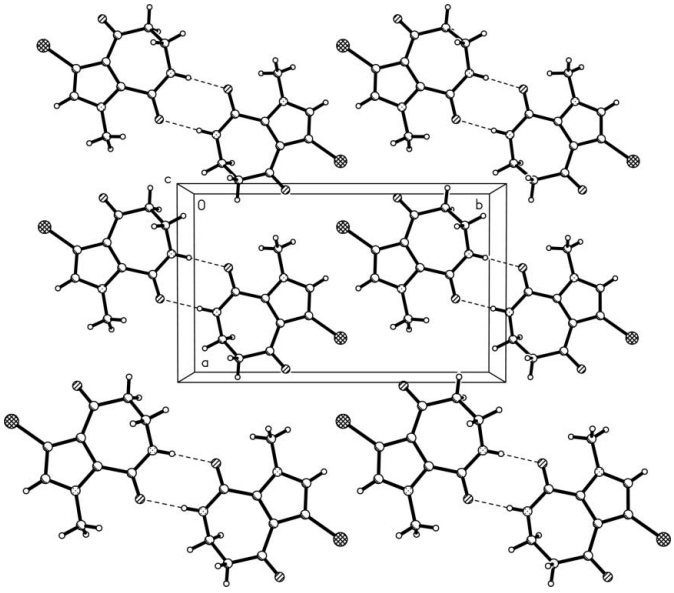


Figure 2
The centrosymmetric dimers formed by hydrogen bonds (dashed lines).

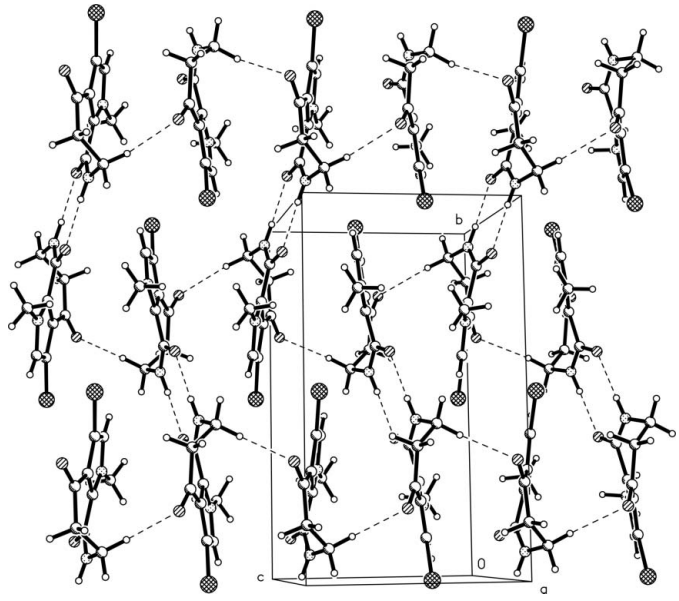


Figure 3
The crystal packing of the title compound, showing the two-dimensional sheets formed by hydrogen bonds (dashed lines).

2005). The conformation of the seven-membered ring can be described as follows: atoms C3, C4, C5, C8 are coplanar, while atoms C6, C7 and N2 deviate from the plane by 1.365 (1), 0.510 (2) and 0.562 (2) Å, respectively.

In the crystal structure, there are two types of intermolecular hydrogen bonds (Table 1). Molecules are linked through N—H···O hydrogen bonds to form centrosymmetric dimers (Fig. 2) of graph-set motif $R_2^2(8)$ (Bernstein *et al.*, 1995), not the one-dimensional chains observed in 1-methylaldisin (Zeng *et al.*, 2005). The dimers are connected by weak C—H···O hydrogen-bond interactions, generating two-dimensional sheets (Fig. 3).

Experimental

3-(4-Bromo-1-methylpyrrole-2-carboxamido)propanoic acid (2.75 g, 10 mmol) was added to phosphorus oxychloride (20 ml) at about 373 K. The mixture was reacted at gentle reflux for 2 h, and was then poured into ice–water and neutralized with NaOH solution. After filtration, the aqueous solution was extracted with ethyl acetate (4 × 15 ml). The organic phase was dried with sodium sulfate overnight. The solvent was removed by distillation under reduced pressure, and the yellow solid residue was collected. The crude product was dissolved in MeOH at room temperature and normal pressure. Colourless crystals of (I) suitable for X-ray analysis (yield 67.3%; m.p. 481 K) grew over a period of one week when the solution was exposed to air. Elemental analysis, calculated for $C_9H_9BrN_2O_2$: C 42.05, H 3.53, N 10.90%; found: C 42.32, H 3.63, N 11.08%.

Crystal data

$C_9H_9BrN_2O_2$
M_r = 257.09
Monoclinic, $P2_1/c$
a = 8.108 (2) Å
b = 14.020 (4) Å
c = 8.482 (2) Å
β = 92.137 (5)°
V = 963.5 (4) Å^3
Z = 4
D_x = 1.772 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 4.24 mm^{-1}
T = 293 (2) K
Prism, colourless
0.49 × 0.26 × 0.25 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.277, T_{max} = 0.346
5474 measured reflections
1887 independent reflections
1536 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.094
S = 1.08
1887 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.5851P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6B···O2^{i}	0.97	2.57	3.385 (4)	142
N2—H2···O1^{ii}	0.86	1.98	2.841 (3)	177

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

H atoms were positioned geometrically, with C—H = 0.96 Å for CH_3, 0.97 Å for CH_2 and 0.93 Å for aromatic CH, and with N—H = 0.86 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N), or 1.5U_{eq}(C) for the methyl group.

Data collection: *SMART* (Bruker,1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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