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7-Chloro-1,5-dipropargyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione

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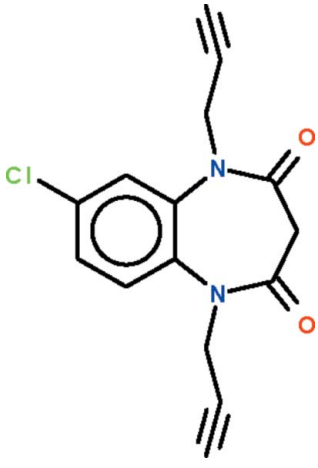
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.172; data-to-parameter ratio = 17.8.

The seven-membered ring of the title compound, $C_{15}H_{11}ClN_2O_2$, adopts a boat-shaped conformation (with the C atoms of the fused-ring as the stern and the methylene C atom as the prow). The N atoms exists in a trigonal–planar coordination; one of the acetylenic H atoms forms a C—H···O hydrogen bond to the O atom of an adjacent molecule, generating a linear chain along a body diagonal.

Related literature

For the crystal structure of 1,5-dimethyl-1,5-benzodiazepin-2,4-dione, see: Mondieig *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{11}ClN_2O_2$
M_r = 286.71
Monoclinic, $P2_1/n$
a = 10.7755 (3) Å
b = 7.6580 (2) Å
c = 16.7221 (5) Å
β = 103.621 (1)°
V = 1341.08 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 293 K
0.42 × 0.10 × 0.08 mm

Data collection

Bruker X8 APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.889, T_{max} = 0.977
17112 measured reflections
3359 independent reflections
2679 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.172
S = 1.07
3359 reflections
189 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O1^{i}	0.95 (3)	2.24 (3)	3.176 (3)	171 (3)

Symmetry code: (i) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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: NK2074).

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supporting information

Acta Cryst. (2010). E**66**, o3228 [https://doi.org/10.1107/S1600536810047008]

7-Chloro-1,5-dipropargyl-1*H*-1,5-benzodiazepine-2,4(3*H*,5*H*)-dione

Rachid Dardouri, Youssef Kandri Rodi, Natalie Saffon, El Mokhtar Essassi and Seik Weng Ng

S1. Comment

We have reported the alkylation of 1,5-benzodiazepine-2,4-dione by alkylating agents in the presence of tetra-*n*-butyl-ammonium bromide as catalyst (Mondieig *et al.*, 2005). In the present study, the amino H atoms are replaced by propargyl groups in the substituted 1,5-benzodiazepin-2,4-dione. The seven-membered ring of $C_{15}H_{11}ClN_2O_2$ (Scheme I, Fig. 1) adopts a boat-shaped conformation (with the C atoms of the fused-ring as the stern and the methylene C atom as the prow). The nitrogen atoms exists in a trigonal-planar coordination; one of the acetylenic H atoms forms a C–H···O hydrogen bond to the oxygen atom of an adjacent molecule to generate a linear chain (Fig. 2).

S2. Experimental

To a solution of the 7-chloro-1,5-benzodiazepine-2,4-dione (0.5 g, 2.38 mmol) in DMF (15 ml) was added potassium carbonate (0.98 g, 7.14 mmol), propargyl bromide (0.45 ml, 5.24 mmol) and tetra-*n*-butylammonium bromide (0.007 g, 0.25 mmol). Stirring was continued under reflux and the reaction was monitored by thin layer chromatography. On completion of the reactin, the mixture was filtered and the solvent removed under reduced pressure. The residue was separated by chromatography on a column of silica gel with ethyl acetate-hexane (1:1) as eluent. Yellow crystals were isolated when the solvent was allowed to evaporate.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C). The final difference Fourier map had a peak in the vicinity of H4, and is 1.51 Å from C4. Attempts to treat this peak as a disorder component of the chlorine atom were unsuccessful. Furthermore, lowering to 2θ limit to 50 ° lead to a peak that has only 1 e Å^{-3} only.

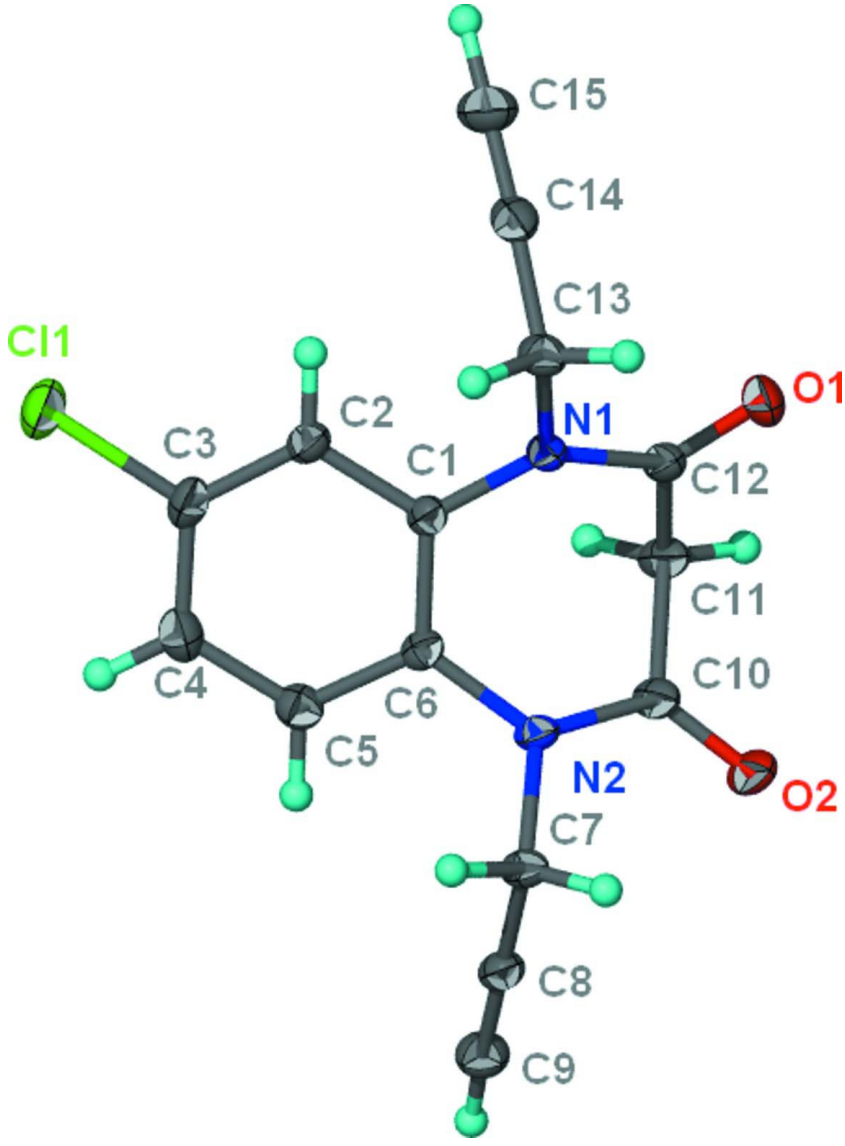

Figure 1

Displacement ellipsoid plot of $C_{15}H_{11}ClN_2O_2$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

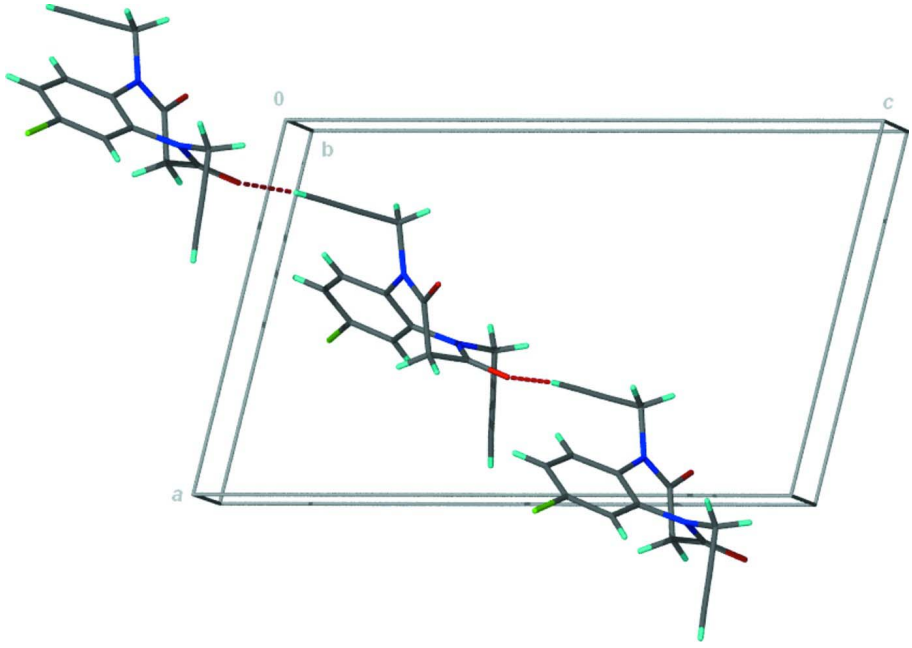

Figure 2

The hydrogen-bonded chain structure.

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Crystal data

$C_{15}H_{11}ClN_2O_2$
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$b = 7.6580$ (2) Å
$c = 16.7221$ (5) Å
$\beta = 103.621$ (1)°
$V = 1341.08$ (7) Å^3
$Z = 4$
$F(000) = 592$
$D_x = 1.420$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4883 reflections
$\theta = 2.5$–28.0°
$\mu = 0.29$ mm^{-1}
$T = 293$ K
Prism, yellow
0.42 × 0.10 × 0.08 mm

Data collection

Bruker X8 APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.889$, $T_{max} = 0.977$
17112 measured reflections
3359 independent reflections
2679 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 28.6°$, $\theta_{min} = 2.1°$
$h = -14\rightarrow14$
$k = -10\rightarrow10$
$l = -22\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.172$
$S = 1.07$
3359 reflections
189 parameters
2 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2 + 1.9878P]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} = 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 1.36$ e Å^{-3}
	$\Delta\rho_{min} = -0.55$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.57023 (7)	0.66801 (10)	0.13231 (5)	0.0407 (2)
O1	0.6840 (2)	0.0626 (3)	0.47422 (12)	0.0409 (5)
O2	0.44544 (18)	−0.2487 (2)	0.33490 (13)	0.0369 (5)
N1	0.59018 (19)	0.2396 (3)	0.36901 (12)	0.0224 (4)
N2	0.41244 (18)	0.0024 (3)	0.26154 (12)	0.0222 (4)
C1	0.5433 (2)	0.2716 (3)	0.28363 (14)	0.0206 (5)
C2	0.5776 (2)	0.4280 (3)	0.25108 (15)	0.0245 (5)
H2	0.6347	0.5038	0.2845	0.029*
C3	0.5270 (2)	0.4702 (3)	0.16968 (16)	0.0275 (5)
C4	0.4440 (3)	0.3598 (4)	0.11757 (16)	0.0309 (6)
H4	0.4107	0.3895	0.0628	0.037*
C5	0.4119 (2)	0.2030 (3)	0.14941 (16)	0.0270 (5)
H5	0.3575	0.1262	0.1148	0.032*
C6	0.4589 (2)	0.1576 (3)	0.23202 (14)	0.0210 (5)
C7	0.2750 (2)	−0.0392 (3)	0.23355 (15)	0.0232 (5)
H7A	0.2456	−0.0894	0.2790	0.028*
H7B	0.2279	0.0682	0.2176	0.028*
C8	0.2469 (2)	−0.1612 (3)	0.16395 (16)	0.0252 (5)
C9	0.2219 (3)	−0.2596 (4)	0.10749 (17)	0.0323 (6)
C10	0.4874 (2)	−0.1152 (3)	0.31164 (16)	0.0263 (5)
C11	0.6273 (2)	−0.0655 (3)	0.33946 (18)	0.0304 (6)
H11A	0.6770	−0.1651	0.3648	0.037*
H11B	0.6595	−0.0288	0.2926	0.037*
C12	0.6388 (2)	0.0817 (3)	0.40064 (16)	0.0273 (5)
C13	0.6048 (2)	0.3883 (3)	0.42683 (16)	0.0272 (5)
H13A	0.5387	0.4737	0.4057	0.033*
H13B	0.5928	0.3471	0.4793	0.033*
C14	0.7301 (3)	0.4731 (3)	0.43968 (16)	0.0298 (5)
C15	0.8290 (3)	0.5466 (4)	0.4487 (2)	0.0416 (7)
H9	0.204 (3)	−0.342 (4)	0.0641 (16)	0.050 (10)*
H15	0.906 (2)	0.611 (4)	0.457 (2)	0.057 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0390 (4)	0.0351 (4)	0.0491 (4)	−0.0006 (3)	0.0124 (3)	0.0178 (3)
O1	0.0477 (12)	0.0307 (10)	0.0343 (10)	−0.0038 (9)	−0.0102 (9)	0.0106 (8)
O2	0.0341 (10)	0.0184 (9)	0.0518 (12)	−0.0044 (7)	−0.0027 (9)	0.0070 (8)
N1	0.0227 (9)	0.0176 (9)	0.0237 (10)	−0.0010 (7)	−0.0011 (7)	0.0009 (7)

N2	0.0183 (9)	0.0161 (9)	0.0303 (10)	−0.0016 (7)	0.0017 (8)	−0.0010 (8)
C1	0.0173 (10)	0.0184 (10)	0.0255 (11)	0.0020 (8)	0.0041 (8)	0.0019 (8)
C2	0.0220 (11)	0.0196 (11)	0.0312 (12)	−0.0015 (9)	0.0051 (9)	0.0020 (9)
C3	0.0253 (12)	0.0252 (12)	0.0346 (13)	0.0026 (9)	0.0121 (10)	0.0092 (10)
C4	0.0299 (13)	0.0353 (14)	0.0276 (12)	0.0053 (11)	0.0070 (10)	0.0063 (10)
C5	0.0267 (12)	0.0269 (12)	0.0269 (12)	0.0007 (10)	0.0054 (10)	−0.0025 (9)
C6	0.0197 (10)	0.0173 (10)	0.0267 (11)	0.0008 (8)	0.0068 (9)	−0.0005 (9)
C7	0.0183 (11)	0.0194 (11)	0.0310 (12)	0.0003 (8)	0.0039 (9)	−0.0027 (9)
C8	0.0212 (11)	0.0212 (11)	0.0321 (12)	−0.0026 (9)	0.0040 (9)	0.0006 (9)
C9	0.0345 (14)	0.0296 (14)	0.0319 (14)	−0.0037 (11)	0.0062 (11)	−0.0052 (11)
C10	0.0245 (11)	0.0154 (11)	0.0352 (13)	0.0015 (9)	−0.0007 (10)	−0.0015 (9)
C11	0.0223 (12)	0.0169 (11)	0.0466 (15)	0.0027 (9)	−0.0029 (10)	0.0005 (10)
C12	0.0225 (11)	0.0211 (12)	0.0337 (13)	−0.0020 (9)	−0.0025 (10)	0.0049 (10)
C13	0.0272 (12)	0.0247 (12)	0.0276 (12)	−0.0006 (10)	0.0022 (10)	−0.0039 (10)
C14	0.0353 (14)	0.0217 (12)	0.0287 (12)	0.0010 (10)	0.0001 (10)	−0.0026 (10)
C15	0.0342 (15)	0.0341 (15)	0.0510 (18)	−0.0059 (12)	−0.0007 (13)	−0.0018 (13)

Geometric parameters (Å, º)

Cl1—C3	1.743 (3)	C5—C6	1.399 (3)
O1—C12	1.221 (3)	C5—H5	0.9300
O2—C10	1.219 (3)	C7—C8	1.467 (3)
N1—C12	1.373 (3)	C7—H7A	0.9700
N1—C1	1.419 (3)	C7—H7B	0.9700
N1—C13	1.478 (3)	C8—C9	1.188 (4)
N2—C10	1.359 (3)	C9—H9	0.95 (3)
N2—C6	1.422 (3)	C10—C11	1.517 (3)
N2—C7	1.479 (3)	C11—C12	1.508 (4)
C1—C2	1.401 (3)	C11—H11A	0.9700
C1—C6	1.402 (3)	C11—H11B	0.9700
C2—C3	1.380 (3)	C13—C14	1.468 (4)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.381 (4)	C13—H13B	0.9700
C4—C5	1.390 (4)	C14—C15	1.183 (4)
C4—H4	0.9300	C15—H15	0.95 (3)
C12—N1—C1	123.5 (2)	N2—C7—H7A	109.0
C12—N1—C13	117.0 (2)	C8—C7—H7B	109.0
C1—N1—C13	118.91 (19)	N2—C7—H7B	109.0
C10—N2—C6	124.11 (19)	H7A—C7—H7B	107.8
C10—N2—C7	117.3 (2)	C9—C8—C7	178.9 (3)
C6—N2—C7	118.57 (19)	C8—C9—H9	177 (2)
C2—C1—C6	119.1 (2)	O2—C10—N2	122.8 (2)
C2—C1—N1	118.3 (2)	O2—C10—C11	121.9 (2)
C6—C1—N1	122.5 (2)	N2—C10—C11	115.2 (2)
C3—C2—C1	120.3 (2)	C12—C11—C10	108.2 (2)
C3—C2—H2	119.9	C12—C11—H11A	110.1
C1—C2—H2	119.9	C10—C11—H11A	110.1

C2—C3—C4	121.7 (2)	C12—C11—H11B	110.1
C2—C3—Cl1	118.7 (2)	C10—C11—H11B	110.1
C4—C3—Cl1	119.6 (2)	H11A—C11—H11B	108.4
C3—C4—C5	118.0 (2)	O1—C12—N1	121.3 (2)
C3—C4—H4	121.0	O1—C12—C11	122.9 (2)
C5—C4—H4	121.0	N1—C12—C11	115.7 (2)
C4—C5—C6	121.9 (2)	C14—C13—N1	112.9 (2)
C4—C5—H5	119.1	C14—C13—H13A	109.0
C6—C5—H5	119.1	N1—C13—H13A	109.0
C5—C6—C1	119.0 (2)	C14—C13—H13B	109.0
C5—C6—N2	118.4 (2)	N1—C13—H13B	109.0
C1—C6—N2	122.5 (2)	H13A—C13—H13B	107.8
C8—C7—N2	113.11 (19)	C15—C14—C13	177.7 (3)
C8—C7—H7A	109.0	C14—C15—H15	177 (2)

C12—N1—C1—C2	−136.1 (2)	C10—N2—C6—C1	−47.3 (3)
C13—N1—C1—C2	35.0 (3)	C7—N2—C6—C1	135.5 (2)
C12—N1—C1—C6	47.5 (3)	C10—N2—C7—C8	−80.8 (3)
C13—N1—C1—C6	−141.4 (2)	C6—N2—C7—C8	96.6 (2)
C6—C1—C2—C3	1.1 (3)	C6—N2—C10—O2	−178.7 (2)
N1—C1—C2—C3	−175.4 (2)	C7—N2—C10—O2	−1.5 (4)
C1—C2—C3—C4	−1.4 (4)	C6—N2—C10—C11	3.5 (3)
C1—C2—C3—Cl1	178.89 (18)	C7—N2—C10—C11	−179.3 (2)
C2—C3—C4—C5	0.1 (4)	O2—C10—C11—C12	−106.1 (3)
Cl1—C3—C4—C5	179.86 (19)	N2—C10—C11—C12	71.8 (3)
C3—C4—C5—C6	1.4 (4)	C1—N1—C12—O1	176.3 (2)
C4—C5—C6—C1	−1.6 (4)	C13—N1—C12—O1	4.9 (4)
C4—C5—C6—N2	174.4 (2)	C1—N1—C12—C11	−6.2 (3)
C2—C1—C6—C5	0.3 (3)	C13—N1—C12—C11	−177.5 (2)
N1—C1—C6—C5	176.7 (2)	C10—C11—C12—O1	107.6 (3)
C2—C1—C6—N2	−175.5 (2)	C10—C11—C12—N1	−69.9 (3)
N1—C1—C6—N2	0.9 (3)	C12—N1—C13—C14	83.2 (3)
C10—N2—C6—C5	136.9 (2)	C1—N1—C13—C14	−88.6 (3)
C7—N2—C6—C5	−40.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O1^{i}	0.95 (3)	2.24 (3)	3.176 (3)	171 (3)

Symmetry code: (i) *x*−1/2, −*y*−1/2, *z*−1/2.