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Crystal structure of (*Z*)-7,8-dichloro-4-(2-oxopropylidene)-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

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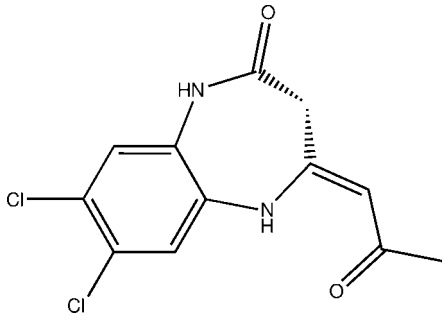
In the title compound, $C_{12}H_{10}Cl_2N_2O_2$, the seven-membered heterocycle displays a half-chair conformation. The mean plane through the oxopropylidene group makes a dihedral angle of 36.44 (9)° with the fused benzene ring. An intramolecular N—H···O hydrogen bond to close an *S*(6) loop is noted. An important feature of the molecular packing are N—H···O hydrogen bonds that lead to the formation of helical supramolecular chains along the *b* axis.

Keywords: crystal structure; 2-oxopropylidene; 1,5-benzodiazepinone; hydrogen bonding.

CCDC reference: 1441702

1. Related literature

For the pharmaceutical and biochemical properties of 1,5-benzodiazepine and their derivatives, see: El Azzaoui *et al.* (1999); Gringauz (1999); Swamy *et al.* (2008). For related structures, see: El Abbassi *et al.* (1997); Akkurt *et al.* (2006).



2. Experimental

2.1. Crystal data

$C_{12}H_{10}Cl_2N_2O_2$
M_r = 285.12
Monoclinic, $P2_1/n$
a = 7.6789 (4) Å
b = 13.2199 (6) Å
c = 12.4129 (7) Å
β = 102.561 (3)°
V = 1229.93 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 296 K
0.36 × 0.33 × 0.24 mm

2.2. Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.672, T_{max} = 0.746
25693 measured reflections
3299 independent reflections
2692 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.112
S = 1.02
3298 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.86	1.96	2.6410 (18)	135
N1—H1···O2^{i}	0.86	1.95	2.8010 (19)	173

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5415).

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supporting information

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Sanae Lahmidi, Abdelhanine Essaghouani, El Mokhtar Essassi, Mohamed Saadi and Lahcen El Ammari

S1. Comment

1,5-Benzodiazepines are used as starting materials in the synthesis of several heterocyclic compounds studied for potential biological activities (El Azzaoui *et al.* 1999). They are used for the purpose of hypnotic effects, owing to their less toxic and less severe withdrawal effects when compared with barbiturates (Gringauz, 1999). Some benzodiazepine derivatives have been widely used as anti-bacterial, anti-fungal, analgesic and anti-convulsant agents (Swamy *et al.*, 2008). In our laboratory we were interested in the synthesis of new 1,5-benzodiazepine derivatives (El Abbassi *et al.*, 1997; Akkurt *et al.*, 2006). The purpose of this work is to synthesize (Z)-7,8-dichloro-4,5-dihydro-4-(2-oxopropyl-idene)-1H-benzo[b][1,4] diazepin-2(3H)-one by condensation of 4,5-dichloro-o-phenylenediamine with 4-hydroxy-6-methyl-2H-pyran-2-one.

The molecule of the title compound, Fig. 1, is build up from two fused six- and seven-membered rings linked to a 2-oxopropylidene group. The seven-membered ring displays a half-chair conformation as indicated by the puckering amplitude QT = 0.811 (2) Å and spherical polar angle $\theta 2$ = 73.9 (2)°, $\varphi 2$ = 129.07 (12)° and $\varphi 3$ = -76.3 (4)°. Moreover, the dihedral angle between the mean plane through the oxopropylidene group and the dichlorobenzene ring is of 36.44 (9)°.

In the crystal, the molecules are linked by hydrogen bonds in the way to build an helical chain along the *b* axis as shown in Fig. 2 and Table 1. An intramolecular hydrogen bond N2—H2···O2 is also observed in this structure.

S2. Experimental

A mixture of 4,5-dichloro-o-phenylenediamine (1.13 g) and of 4-hydroxy-6-methyl-2H- pyran-2-one (0.25 g) in xylene (30 mL) was heated at reflux for 4 h with azeotropic distillation. The completion of the reaction was confirmed by TLC. The solid obtained upon cooling the mixture was recrystallized from ethanol to afford colourless crystals in 75% yield.

S3. Refinement

The H atoms were located in a difference map and treated as riding with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(C, N).

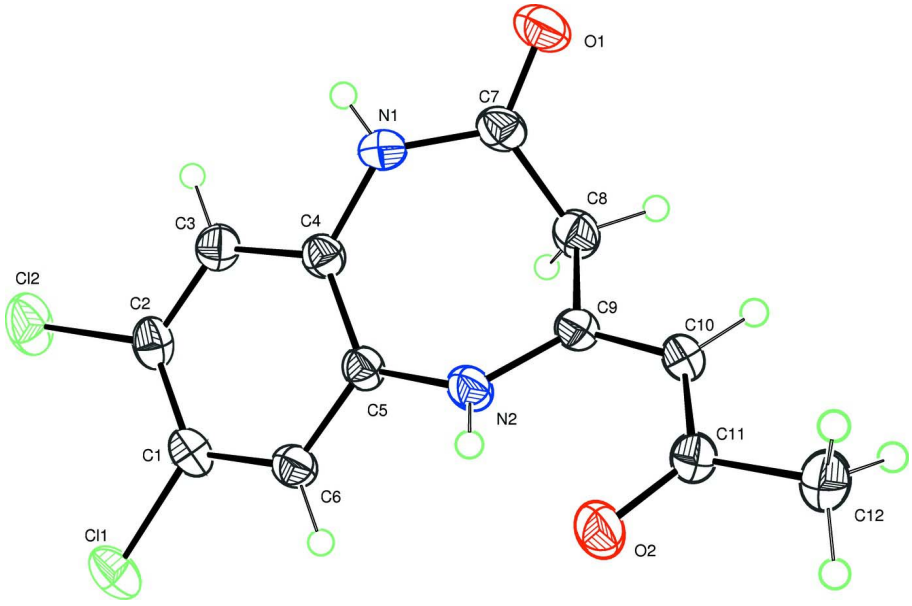

Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

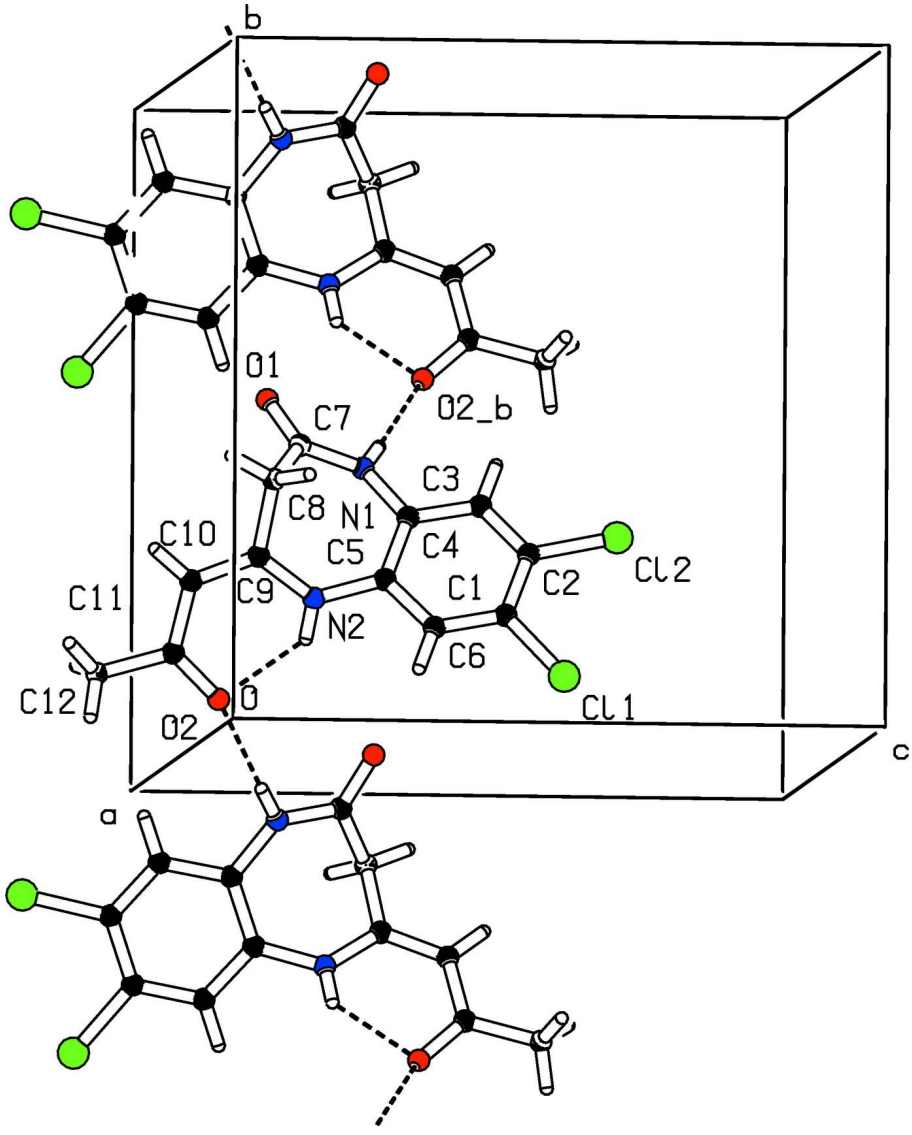

Figure 2

Structure of the title compound, showing molecules linked through N1—H1···O1 hydrogen bonds and the intramolecular hydrogen bond N2—H2···O2 (dashed lines).

(*Z*)-7,8-Dichloro-4-(2-oxopropylidene)-4,5-dihydro-1*H*-1,5-benzodiazepin-2(3*H*)-one

Crystal data

$C_{12}H_{10}Cl_2N_2O_2$
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$c = 12.4129\ (7)$ Å
$\beta = 102.561\ (3)°$
$V = 1229.93\ (11)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.540$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3299 reflections
$\theta = 2.3–29.1°$
$\mu = 0.52$ mm^{-1}
$T = 296$ K
Block, colourless
0.36 × 0.33 × 0.24 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.672, T_{max} = 0.746
25693 measured reflections
3299 independent reflections
2692 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 29.1°, θ_{min} = 2.3°
h = −10→10
k = −18→18
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.112
S = 1.02
3298 reflections
163 parameters
0 restraints
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 0.6111P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.5232 (2)	0.21313 (13)	0.49879 (13)	0.0338 (3)
C2	0.5740 (2)	0.30850 (13)	0.54157 (13)	0.0355 (3)
C3	0.6157 (2)	0.38307 (12)	0.47350 (13)	0.0346 (3)
H3	0.6479	0.4470	0.5024	0.042*
C4	0.6104 (2)	0.36438 (11)	0.36221 (13)	0.0303 (3)
C5	0.5633 (2)	0.26785 (11)	0.31938 (12)	0.0291 (3)
C6	0.5189 (2)	0.19345 (12)	0.38896 (13)	0.0330 (3)
H6	0.4858	0.1294	0.3607	0.040*
C7	0.6022 (2)	0.46740 (11)	0.19419 (14)	0.0349 (3)
C8	0.4552 (2)	0.40089 (12)	0.13165 (15)	0.0375 (4)
H8A	0.3586	0.3984	0.1707	0.045*
H8B	0.4087	0.4298	0.0593	0.045*
C9	0.5204 (2)	0.29534 (11)	0.11872 (13)	0.0310 (3)
C10	0.5298 (2)	0.25845 (12)	0.01690 (13)	0.0333 (3)
H10	0.4958	0.3007	−0.0440	0.040*
C11	0.5889 (2)	0.15901 (12)	−0.00056 (13)	0.0330 (3)
C12	0.6037 (3)	0.12898 (15)	−0.11491 (14)	0.0424 (4)
H12A	0.5680	0.1847	−0.1645	0.064*
H12B	0.7249	0.1111	−0.1145	0.064*
H12C	0.5275	0.0720	−0.1388	0.064*
N1	0.6678 (2)	0.44232 (10)	0.30134 (12)	0.0352 (3)

H1	0.7364	0.4859	0.3410	0.042*
N2	0.5688 (2)	0.23921 (10)	0.21168 (11)	0.0339 (3)
H2	0.6012	0.1781	0.2028	0.041*
Cl1	0.46370 (7)	0.11838 (4)	0.57991 (4)	0.04762 (15)
Cl2	0.58703 (8)	0.33611 (4)	0.67921 (4)	0.05696 (17)
O1	0.6604 (2)	0.54018 (9)	0.15280 (11)	0.0496 (3)
O2	0.6297 (2)	0.09619 (9)	0.07595 (10)	0.0451 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0353 (8)	0.0360 (8)	0.0318 (7)	0.0068 (6)	0.0109 (6)	0.0083 (6)
C2	0.0383 (9)	0.0408 (8)	0.0278 (7)	0.0116 (7)	0.0080 (6)	0.0008 (6)
C3	0.0383 (9)	0.0313 (7)	0.0330 (8)	0.0052 (6)	0.0053 (6)	−0.0031 (6)
C4	0.0316 (8)	0.0283 (7)	0.0309 (7)	0.0032 (6)	0.0066 (6)	0.0017 (6)
C5	0.0317 (8)	0.0281 (7)	0.0278 (7)	0.0031 (6)	0.0069 (6)	0.0018 (5)
C6	0.0389 (9)	0.0276 (7)	0.0330 (8)	0.0009 (6)	0.0089 (6)	0.0022 (6)
C7	0.0438 (9)	0.0245 (7)	0.0383 (8)	0.0055 (6)	0.0136 (7)	0.0015 (6)
C8	0.0410 (9)	0.0325 (8)	0.0370 (8)	0.0075 (7)	0.0043 (7)	0.0044 (6)
C9	0.0328 (8)	0.0278 (7)	0.0309 (7)	−0.0020 (6)	0.0039 (6)	0.0031 (6)
C10	0.0407 (9)	0.0315 (7)	0.0258 (7)	−0.0023 (6)	0.0029 (6)	0.0055 (6)
C11	0.0353 (8)	0.0346 (8)	0.0273 (7)	−0.0037 (6)	0.0028 (6)	0.0009 (6)
C12	0.0495 (10)	0.0468 (10)	0.0306 (8)	−0.0034 (8)	0.0083 (7)	−0.0031 (7)
N1	0.0416 (8)	0.0275 (6)	0.0359 (7)	−0.0056 (5)	0.0073 (6)	−0.0001 (5)
N2	0.0487 (8)	0.0253 (6)	0.0287 (6)	0.0033 (5)	0.0104 (6)	0.0020 (5)
Cl1	0.0591 (3)	0.0464 (3)	0.0420 (2)	0.0054 (2)	0.0213 (2)	0.01498 (18)
Cl2	0.0855 (4)	0.0567 (3)	0.0300 (2)	0.0141 (3)	0.0155 (2)	−0.00200 (18)
O1	0.0731 (10)	0.0323 (6)	0.0465 (7)	−0.0059 (6)	0.0199 (7)	0.0064 (5)
O2	0.0680 (9)	0.0334 (6)	0.0318 (6)	0.0095 (6)	0.0061 (6)	0.0038 (5)

Geometric parameters (Å, º)

C1—C6	1.381 (2)	C8—C9	1.503 (2)
C1—C2	1.391 (2)	C8—H8A	0.9700
C1—Cl1	1.7297 (16)	C8—H8B	0.9700
C2—C3	1.380 (2)	C9—N2	1.3541 (19)
C2—Cl2	1.7286 (17)	C9—C10	1.371 (2)
C3—C4	1.395 (2)	C10—C11	1.422 (2)
C3—H3	0.9300	C10—H10	0.9300
C4—C5	1.399 (2)	C11—O2	1.2491 (19)
C4—N1	1.404 (2)	C11—C12	1.502 (2)
C5—N2	1.3989 (19)	C12—H12A	0.9600
C5—C6	1.399 (2)	C12—H12B	0.9600
C6—H6	0.9300	C12—H12C	0.9600
C7—O1	1.220 (2)	N1—H1	0.8599
C7—N1	1.357 (2)	N2—H2	0.8600
C7—C8	1.506 (2)		

C6—C1—C2	119.49 (15)	C9—C8—H8B	109.3
C6—C1—Cl1	119.04 (13)	C7—C8—H8B	109.3
C2—C1—Cl1	121.46 (13)	H8A—C8—H8B	108.0
C3—C2—C1	119.79 (15)	N2—C9—C10	122.05 (14)
C3—C2—Cl2	118.88 (13)	N2—C9—C8	116.99 (14)
C1—C2—Cl2	121.34 (13)	C10—C9—C8	120.95 (14)
C2—C3—C4	121.26 (15)	C9—C10—C11	123.48 (14)
C2—C3—H3	119.4	C9—C10—H10	118.3
C4—C3—H3	119.4	C11—C10—H10	118.3
C3—C4—C5	119.15 (14)	O2—C11—C10	122.29 (15)
C3—C4—N1	117.25 (14)	O2—C11—C12	119.00 (15)
C5—C4—N1	123.37 (14)	C10—C11—C12	118.71 (15)
N2—C5—C4	123.42 (14)	C11—C12—H12A	109.5
N2—C5—C6	117.50 (14)	C11—C12—H12B	109.5
C4—C5—C6	118.95 (14)	H12A—C12—H12B	109.5
C1—C6—C5	121.34 (15)	C11—C12—H12C	109.5
C1—C6—H6	119.3	H12A—C12—H12C	109.5
C5—C6—H6	119.3	H12B—C12—H12C	109.5
O1—C7—N1	120.91 (17)	C7—N1—C4	127.87 (14)
O1—C7—C8	123.03 (16)	C7—N1—H1	116.6
N1—C7—C8	116.06 (14)	C4—N1—H1	114.1
C9—C8—C7	111.53 (14)	C9—N2—C5	127.32 (13)
C9—C8—H8A	109.3	C9—N2—H2	116.1
C7—C8—H8A	109.3	C5—N2—H2	116.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.86	1.96	2.6410 (18)	135
N1—H1···O2^{i}	0.86	1.95	2.8010 (19)	173

Symmetry code: (i) −*x*+3/2, *y*+1/2, −*z*+1/2.