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(1*S*,5*R*)-1-(3,4-Dichlorophenyl)-3-oxabicyclo[3.1.0]hexan-2-one

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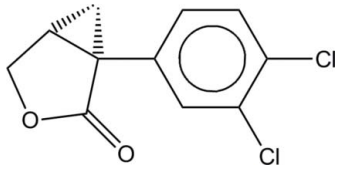
Key indicators: single-crystal X-ray study; T = 102 K; mean σ(C–C) = 0.002 Å; R factor = 0.024; wR factor = 0.063; data-to-parameter ratio = 14.6.

The absolute structure has been determined by X-ray analysis for the title compound, $C_{11}H_8Cl_2O_2$. The five-membered ring of the molecule is best described as a flattened envelope conformation with the methylene C atom located 0.208 (2) Å below the plane formed by the other four atoms. A weak intermolecular C—H···O hydrogen bond is present in the crystal structure

Related literature

The title compound was prepared as an intermediate in the search for potential triple neurotransmittor reuptake inhibitors, see: Milewska *et al.* (1996); Lin & Charette (2005); Tsuji *et al.* (1999); Džolić *et al.* (2003).



Experimental

Crystal data

$C_{11}H_8Cl_2O_2$
M_r = 243.07
Orthorhombic, $P2_12_12_1$
a = 7.0597 (4) Å
b = 11.1343 (7) Å
c = 12.6756 (8) Å
V = 996.36 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 102 K
0.58 × 0.36 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.680, T_{max} = 0.894
8562 measured reflections
2341 independent reflections
2278 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.063
S = 1.10
2341 reflections
160 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), 952 Friedel pairs
Flack parameter: 0.04 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H71···O2^{i}	0.880 (18)	2.366 (18)	3.2443 (16)	175.6 (16)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2480).

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supporting information

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(1*S*,5*R*)-1-(3,4-Dichlorophenyl)-3-oxabicyclo[3.1.0]hexan-2-one

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S1. Comment

The title compound was prepared as an intermediate in the search for potential triple neurotransmittor reuptake inhibitors (Milewska *et al.*, 1996; Lin & Charette, 2005; Tsuji *et al.*, 1999; Džolić *et al.*, 2003); details will be published elsewhere.

The molecular structure is shown in Fig. 1. The five-membered ring of the molecule is best described as a flat envelope conformation with C1 located 0.208 (2) Å below the plane constituted by C2, C4, C5 and O1, on the opposite side of C3. In the crystal structure the weak C—H···O hydrogen bonding presents between benzene ring and the carbonyl O atom of the neighboring molecule (Table 1).

S2. Experimental

Block-shaped single crystals were obtained from an acetonitrile solution by slow evaporation at room temperature.

S3. Refinement

Positional parameters were refined for all H atoms, U_{iso}(H) values were set to 1.2U_{eq}(C).

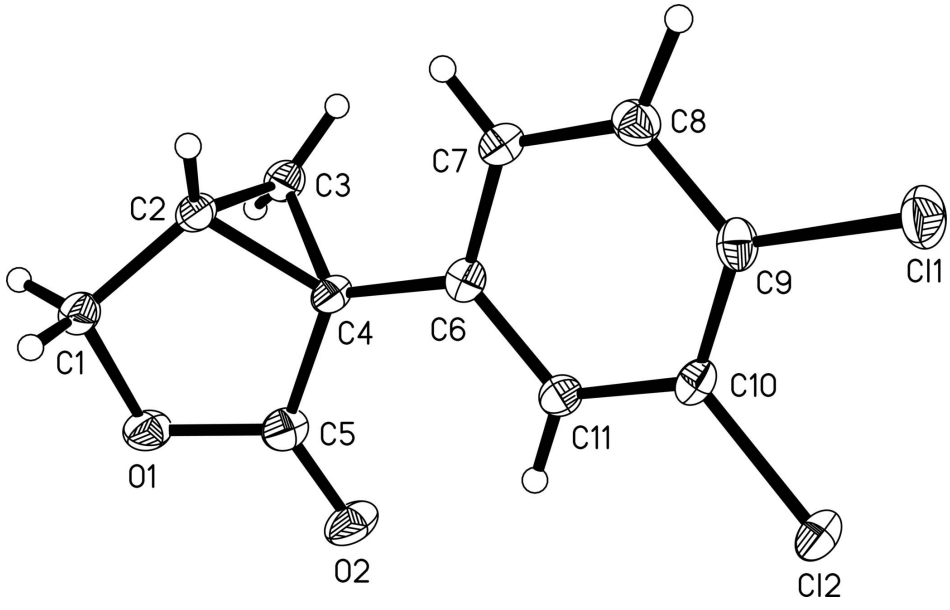

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary size.

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Crystal data

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$b = 11.1343$ (7) Å
$c = 12.6756$ (8) Å
$V = 996.36$ (11) Å^3
$Z = 4$
$F(000) = 496$
$D_x = 1.620$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6286 reflections
$\theta = 2.4$–$27.9°$
$\mu = 0.62$ mm^{-1}
$T = 102$ K
Block, colourless
0.58 × 0.36 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm^{-1}
Sets of exposures each taken over 0.5° ω rotation scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.680$, $T_{max} = 0.894$
8562 measured reflections
2341 independent reflections
2278 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.4°$
$h = -9 \rightarrow 8$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.063$
$S = 1.10$
2341 reflections
160 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
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 0.112P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 952 Friedel pairs
Absolute structure parameter: 0.04 (5)

Special details

Experimental. Crystallized from acetonitrile solution

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

Refinement. Data were collected by measuring three sets of exposures with the detector set at $2\theta = 29°$, crystal-to-detector distance 6.00 cm. Refinement of F^2 against ALL reflections.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.76710 (6)	1.02347 (3)	0.53880 (3)	0.02383 (10)
Cl2	0.76768 (6)	0.86335 (3)	0.33094 (3)	0.02003 (10)
O1	0.89020 (18)	0.30003 (10)	0.51020 (9)	0.0219 (2)
O2	0.7597 (2)	0.41878 (9)	0.38886 (8)	0.0258 (3)

C1	0.9599 (2)	0.30985 (14)	0.61831 (12)	0.0195 (3)
H11	1.095 (3)	0.3243 (17)	0.6145 (14)	0.023*
H12	0.928 (3)	0.2377 (17)	0.6515 (15)	0.023*
C2	0.8628 (2)	0.41790 (14)	0.66439 (13)	0.0162 (3)
H21	0.921 (2)	0.4618 (19)	0.7169 (15)	0.019*
C3	0.6531 (2)	0.41869 (15)	0.65659 (14)	0.0183 (3)
H31	0.586 (3)	0.469 (2)	0.6993 (15)	0.022*
H32	0.590 (3)	0.3471 (17)	0.6365 (14)	0.022*
C4	0.7718 (2)	0.48380 (12)	0.57217 (10)	0.0153 (3)
C5	0.8034 (2)	0.40360 (13)	0.47933 (12)	0.0191 (3)
C6	0.7654 (2)	0.61725 (12)	0.56086 (11)	0.0154 (3)
C7	0.7598 (2)	0.68907 (12)	0.65134 (10)	0.0174 (3)
H71	0.756 (3)	0.6558 (15)	0.7143 (14)	0.021*
C8	0.7586 (2)	0.81285 (12)	0.64365 (11)	0.0183 (3)
H81	0.746 (3)	0.8709 (14)	0.7047 (13)	0.022*
C9	0.7631 (2)	0.86805 (11)	0.54561 (11)	0.0167 (3)
C10	0.7664 (2)	0.79786 (12)	0.45517 (10)	0.0155 (3)
C11	0.7674 (2)	0.67269 (12)	0.46254 (11)	0.0156 (3)
H111	0.776 (3)	0.6262 (14)	0.4035 (14)	0.019*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0310 (2)	0.01352 (15)	0.02696 (19)	0.00112 (17)	−0.00448 (17)	0.00012 (12)
Cl2	0.02456 (18)	0.01897 (16)	0.01656 (16)	−0.00227 (16)	−0.00294 (15)	0.00519 (12)
O1	0.0341 (6)	0.0156 (5)	0.0160 (5)	−0.0008 (5)	0.0016 (5)	−0.0023 (4)
O2	0.0423 (7)	0.0211 (5)	0.0141 (5)	−0.0054 (6)	−0.0031 (6)	−0.0004 (4)
C1	0.0258 (8)	0.0161 (7)	0.0165 (7)	0.0021 (6)	0.0009 (6)	0.0023 (6)
C2	0.0203 (7)	0.0157 (7)	0.0126 (7)	0.0001 (6)	−0.0008 (6)	0.0010 (6)
C3	0.0216 (7)	0.0158 (7)	0.0175 (8)	−0.0008 (6)	0.0022 (6)	0.0030 (6)
C4	0.0186 (7)	0.0153 (6)	0.0120 (6)	−0.0015 (6)	−0.0015 (5)	0.0013 (5)
C5	0.0255 (8)	0.0142 (6)	0.0176 (7)	−0.0050 (6)	0.0003 (6)	−0.0004 (5)
C6	0.0142 (6)	0.0156 (6)	0.0165 (6)	−0.0012 (6)	−0.0012 (6)	0.0015 (5)
C7	0.0194 (7)	0.0192 (6)	0.0137 (6)	−0.0003 (7)	−0.0009 (6)	0.0018 (5)
C8	0.0179 (7)	0.0194 (6)	0.0176 (6)	0.0010 (7)	−0.0016 (6)	−0.0029 (5)
C9	0.0157 (6)	0.0129 (6)	0.0215 (7)	−0.0006 (6)	−0.0034 (6)	0.0003 (5)
C10	0.0140 (7)	0.0171 (6)	0.0153 (6)	−0.0010 (6)	−0.0019 (6)	0.0045 (5)
C11	0.0154 (7)	0.0168 (6)	0.0144 (6)	−0.0012 (6)	−0.0010 (6)	0.0003 (5)

Geometric parameters (Å, º)

Cl1—C9	1.7329 (13)	C3—H32	0.949 (19)
Cl2—C10	1.7353 (13)	C4—C6	1.4934 (18)
O1—C5	1.3631 (19)	C4—C5	1.4940 (19)
O1—C1	1.4602 (19)	C6—C11	1.3909 (18)
O2—C5	1.1996 (18)	C6—C7	1.3988 (19)
C1—C2	1.503 (2)	C7—C8	1.3817 (19)
C1—H11	0.97 (2)	C7—H71	0.880 (17)

C1—H12	0.93 (2)	C8—C9	1.3868 (19)
C2—C3	1.484 (2)	C8—H81	1.012 (17)
C2—C4	1.522 (2)	C9—C10	1.3877 (19)
C2—H21	0.92 (2)	C10—C11	1.3968 (19)
C3—C4	1.541 (2)	C11—H111	0.912 (17)
C3—H31	0.91 (2)		

C5—O1—C1	110.94 (12)	C5—C4—C3	110.31 (12)
O1—C1—C2	105.73 (13)	C2—C4—C3	57.96 (9)
O1—C1—H11	107.3 (11)	O2—C5—O1	120.62 (14)
C2—C1—H11	109.6 (12)	O2—C5—C4	129.07 (14)
O1—C1—H12	106.0 (12)	O1—C5—C4	110.28 (13)
C2—C1—H12	113.7 (11)	C11—C6—C7	118.77 (12)
H11—C1—H12	113.8 (16)	C11—C6—C4	121.82 (12)
C3—C2—C1	115.72 (15)	C7—C6—C4	119.40 (12)
C3—C2—C4	61.65 (11)	C8—C7—C6	120.84 (13)
C1—C2—C4	106.25 (13)	C8—C7—H71	118.9 (11)
C3—C2—H21	119.1 (11)	C6—C7—H71	120.3 (11)
C1—C2—H21	120.2 (12)	C7—C8—C9	120.34 (12)
C4—C2—H21	119.1 (13)	C7—C8—H81	125.7 (9)
C2—C3—C4	60.40 (11)	C9—C8—H81	113.8 (9)
C2—C3—H31	118.9 (12)	C8—C9—C10	119.42 (12)
C4—C3—H31	113.9 (13)	C8—C9—Cl1	119.18 (10)
C2—C3—H32	118.9 (11)	C10—C9—Cl1	121.40 (10)
C4—C3—H32	117.8 (11)	C9—C10—C11	120.45 (12)
H31—C3—H32	115.4 (18)	C9—C10—Cl2	120.87 (10)
C6—C4—C5	121.57 (12)	C11—C10—Cl2	118.68 (10)
C6—C4—C2	124.48 (12)	C6—C11—C10	120.18 (13)
C5—C4—C2	104.70 (11)	C6—C11—H111	118.9 (10)
C6—C4—C3	121.22 (12)	C10—C11—H111	120.8 (10)

C1—C2—C4—C5	5.85 (15)	C3—C4—C5—O1	−57.45 (16)
O1—C1—C2—C4	−12.38 (16)	C2—C4—C6—C11	148.81 (15)
C2—C4—C5—O1	3.36 (16)	C3—C4—C6—C11	−140.85 (15)
C5—C4—C6—C11	7.2 (2)	C5—C4—C6—C7	−171.84 (15)
C5—O1—C1—C2	15.19 (17)	C2—C4—C6—C7	−30.3 (2)
O1—C1—C2—C3	53.50 (19)	C3—C4—C6—C7	40.1 (2)
C1—C2—C3—C4	−95.34 (15)	C11—C6—C7—C8	−0.7 (3)
C3—C2—C4—C6	108.17 (17)	C4—C6—C7—C8	178.38 (15)
C1—C2—C4—C6	−140.96 (15)	C6—C7—C8—C9	−0.1 (3)
C3—C2—C4—C5	−105.02 (14)	C7—C8—C9—C10	0.8 (3)
C1—C2—C4—C5	5.85 (15)	C7—C8—C9—Cl1	−178.46 (14)
C1—C2—C4—C3	110.87 (16)	C8—C9—C10—C11	−0.7 (2)
C2—C3—C4—C6	−113.68 (15)	Cl1—C9—C10—C11	178.55 (12)
C2—C3—C4—C5	95.02 (14)	C8—C9—C10—Cl2	178.79 (13)
C1—O1—C5—O2	169.94 (16)	Cl1—C9—C10—Cl2	−1.94 (19)
C1—O1—C5—C4	−11.76 (17)	C7—C6—C11—C10	0.8 (2)
C6—C4—C5—O2	−30.5 (3)	C4—C6—C11—C10	−178.25 (14)

C2—C4—C5—O2	−178.54 (17)	C9—C10—C11—C6	−0.1 (2)
C3—C4—C5—O2	120.66 (19)	Cl2—C10—C11—C6	−179.64 (12)
C6—C4—C5—O1	151.37 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H71···O2^{i}	0.880 (18)	2.366 (18)	3.2443 (16)	175.6 (16)

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