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2,2-Dichloro-6-methoxy-3,4-dihydro-2H-naphthalene

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Key indicators

Single-crystal X-ray study

$T = 295\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

R factor = 0.042

wR factor = 0.125

Data-to-parameter ratio = 18.3

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

The structure of the title compound, $\text{C}_{11}\text{H}_{10}\text{Cl}_2\text{O}_2$, shows that the asymmetric unit contains two closely related independent molecules that pack in a head-to-tail arrangement.

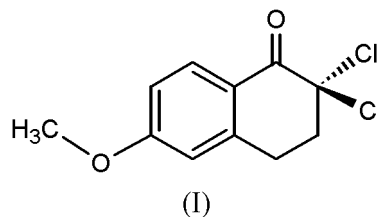
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Comment

The title compound, (I), was isolated as a by-product in the synthesis of the mono chloro compound, being synthesized as part of an ongoing program of discovery of biologically active compounds based on the α and β tetralone core. The asymmetric unit of (I) comprises two independent molecules, which are separated by normal van der Waals distances (Fig. 1). The two molecules lie approximately orthogonal to each other in a head-to-tail arrangement. The bond lengths and angles of both molecules (Table 1) are very similar and are in accord with conventional values (Allen *et al.*, 1987). The conformational structure of the two molecules is also similar. The methoxy group, the aromatic ring and the carbonyl group of the tetralone group are all approximately coplanar. The cyclohexanone rings adopt a half chair conformation, with C3 lying out of the plane of the molecule by approximately 0.5 Å.



Experimental

Sulfuryl chloride (9.18 g, 52 mmol) in anhydrous carbon tetrachloride (60 ml) was added dropwise to a stirred solution of 6-methoxy-3,4-dihydro-2H-naphthalene (7.01 g, 52 mmol) in anhydrous carbon tetrachloride (60 ml), over 1 h at 273 K under an inert atmosphere. The mixture was then stirred at room temperature for 24 h, followed by heating at 328–333 K for 6 h. After this time a further aliquot of sulfuryl chloride (9.18 g, 52 mmol) in carbon tetrachloride (120 ml) was added and heating maintained at 328–333 K for a further 12 h. Removal of the solvent and column chromatography of the resulting crude product (ether:hexane 1:1 *v/v*, then dichloromethane:hexane 1:1 *v/v*) yielded (I) as a white solid (9.18 g, 71.8%), m.p. 358–360 K. ^1H NMR (200 MHz, CDCl_3); δ (p.p.m.) 2.90–3.30 (2H, *m*, CH_2), 3.18 (2H, *t*, CH_2), (2H, *t*, CH_2), 3.89 (3H, *s*, CH_3), 6.72 (1H, *s*, Ar–H), 6.95 (1H, *dd*, Ar–H), 8.15 (1H, *d*, Ar–H). Crystals suitable for X-ray diffraction studies were grown by slow evaporation of the solvent from a saturated solution of (I) in diethyl ether.

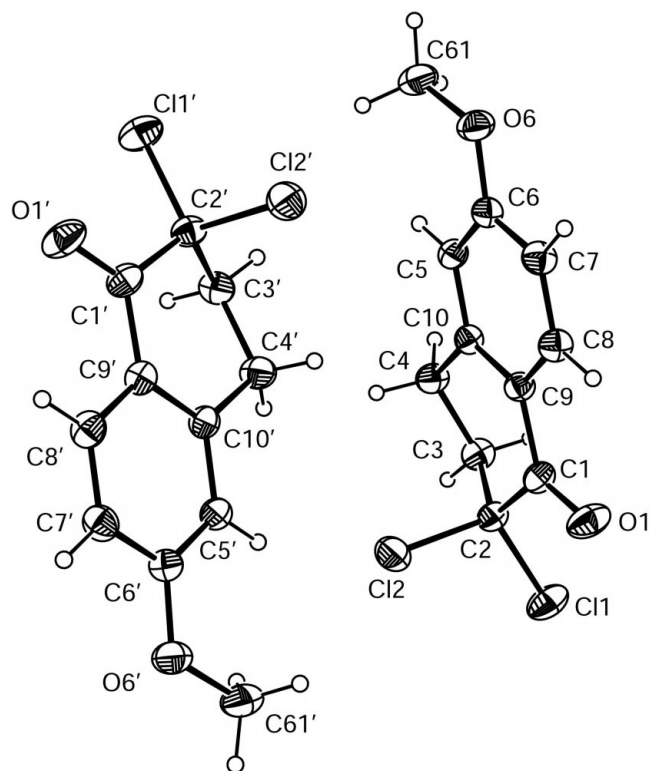


Figure 1
ORTEP-3 (Farrugia, 1997) plot, showing the atomic numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level for non-H atoms.

Crystal data

$C_{11}H_{10}Cl_2O_2$
 $M_r = 245.09$
 Monoclinic, $P2_1/c$
 $a = 17.569$ (5) Å
 $b = 10.745$ (3) Å
 $c = 12.239$ (4) Å
 $\beta = 109.83$ (2)°
 $V = 2173.5$ (12) Å³
 $Z = 8$

Data collection

Rigaku AFC-7R diffractometer
 ω -2 θ scans
 Absorption correction: none
 6026 measured reflections
 4967 independent reflections
 2849 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.040$

$D_x = 1.498$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 12.6$ – 17.1 °
 $\mu = 0.57$ mm⁻¹
 $T = 295$ K
 Prismatic, colorless
 $0.30 \times 0.30 \times 0.14$ mm

$\theta_{max} = 27.5$ °
 $h = -10 \rightarrow 22$
 $k = -13 \rightarrow 6$
 $l = -15 \rightarrow 14$
 3 standard reflections every 150 reflections
 intensity decay: 4.8%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.125$
 $S = 0.99$
 4967 reflections
 271 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.7378P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.24$ e Å⁻³
 $\Delta\rho_{min} = -0.30$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C11–C2	1.762 (2)	O6–C6	1.362 (3)
C12–C2	1.805 (3)	O6–C61	1.435 (4)
C11'–C2'	1.770 (2)	O1'–C1'	1.215 (3)
C12'–C2'	1.808 (3)	O6'–C6'	1.359 (3)
O1–C1	1.212 (3)	O6'–C61'	1.435 (4)
C6–O6–C61	118.2 (2)	O6–C6–C7	115.4 (2)
C6'–O6'–C61'	117.5 (2)	O1'–C1'–C2'	120.4 (2)
O1–C1–C9	123.7 (3)	O1'–C1'–C9'	123.4 (2)
O1–C1–C2	120.2 (2)	C11'–C2'–C12'	108.26 (15)
C11–C2–C12	108.24 (14)	C11'–C2'–C1'	111.38 (17)
C11–C2–C3	109.61 (16)	C11'–C2'–C3'	109.64 (18)
C12–C2–C1	104.85 (16)	C12'–C2'–C1'	104.99 (18)
C11–C2–C1	111.31 (18)	C12'–C2'–C3'	109.94 (19)
C12–C2–C3	110.33 (19)	O6'–C6'–C5'	124.6 (2)
O6–C6–C5	124.0 (2)	O6'–C6'–C7'	115.2 (2)

H atoms were located at calculated positions with C–H set to 0.95 Å. U_{iso} values for the H atoms were set at $1.2U_{eq}$ of the parent atoms.

Data collection: *MSC/AFC-7 Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC-7 Diffractometer Control Software*; data reduction: *teXsan* (Molecular Structure Corporation, 1997–2001); program(s) used to solve structure: *teXsan*; program(s) used to refine structure: *teXsan*; *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *teXsan*; *PLATON* (Spek, 2001).

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